Adsorption Study of Heavy Metals Cu and Cd by Ciapus Zeolite Activated Physically and Chemically with Acid

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Abstrak

Zeolit alami telah banyak dieksplorasi sebagai adsorben untuk mengurangi kontaminasi logam berat, khususnya tembaga (Cu) dan kadmium (Cd), di lingkungan. Studi ini bertujuan untuk mengevaluasi karakteristik, kapasitas adsorpsi, dan kinetika adsorpsi zeolit alami terhadap ion Cu dan Cd. Zeolit diaktifkan secara fisik pada suhu 400°C selama 3 jam dan secara kimia menggunakan HCl 0,5 M selama 30 menit untuk meningkatkan kinerja adsorpsinya. Karakterisasi dilakukan menggunakan analisis FTIR, BET, dan SEM. Hasil FTIR mengkonfirmasi keberadaan gugus fungsional Si–O–Si dan Si–O–Al pada 1007,49 cm^{-1}, yang menunjukkan struktur aluminosilikat khas zeolit. Analisis BET menunjukkan bahwa zeolit memiliki luas permukaan yang lebih besar dibandingkan dengan cangkang telur, sedangkan SEM mengungkapkan keberadaan struktur berpori setelah aktivasi. Percobaan adsorpsi menunjukkan bahwa kapasitas adsorpsi maksimum adalah 0,139 mg/g untuk Cu dan 0,169 mg/g untuk Cd. Waktu kontak optimum untuk kedua logam tersebut adalah 30 menit pada konsentrasi 10 mg/L untuk Cu dan 15 mg/L untuk Cd. Studi kinetik menunjukkan bahwa proses adsorpsi mengikuti model pseudo-orde kedua, dengan konstanta laju 0,0001 min^{-1} untuk Cu dan 0,0002 min^{-1} untuk Cd, yang menunjukkan kemisorpsi sebagai mekanisme dominan.

Kata kunci: aktivasi, adsorpsi, logam Cd, logam Cu, zeolit alam

Abstract

Natural zeolite has been widely explored as an adsorbent to reduce heavy metal contamination, particularly copper (Cu) and cadmium (Cd), in the environment. This study aimed to evaluate the characteristics, adsorption capacity, and adsorption kinetics of natural zeolite toward Cu and Cd ions. The zeolite was activated physically at 400°C for 3 hours and chemically using 0.5 M HCl for 30 minutes to enhance its adsorption performance. Characterization was conducted using FTIR, BET, and SEM analyses. FTIR results confirmed the presence of Si–O–Si and Si–O–Al functional groups at 1007.49 cm^{-1}, indicating the typical aluminosilicate structure of zeolite. BET analysis showed that zeolite has a larger surface area compared to eggshell, while SEM images revealed the presence of porous structures after activation. Adsorption experiments indicated that the maximum adsorption capacities were 0.139 mg/g for Cu and 0.169 mg/g for Cd. The optimum contact time for both metals was 30 minutes at concentrations of 10 mg/L for Cu and 15 mg/L for Cd. Kinetic studies demonstrated that the adsorption process followed a pseudo-second-order model, with rate constants of 0.0001 min^{-1} for Cu and 0.0002 min^{-1} for Cd, suggesting chemisorption as the dominant mechanism.

Keywords: activation, adsorption, cadmium metal, copper metal, natural zeolite

INTRODUCTION

Adsorption is one of the methods employed by several companies to reduce the levels of heavy metals in the environment (Estiaty, 2012). Heavy metals are highly toxic to water, soil, and air. In addition to their toxicity, heavy metal contamination can have various effects on living organisms, including damage to the nervous system, disruption of the human immune system, and when accumulated in the food chain, it can have adverse effects on the metabolism of living organisms (Khimantoro et al., 2014). Based on toxicology, heavy metals are divided into two types. The first type is essential heavy metals, such as Cu, and the second type is non-essential heavy metals, such as Cd (Said, 2010). Materials like zeolite can be used to reduce the levels of heavy metals. Generally, zeolite is used as an adsorbent. Silicon atoms in zeolite are replaced by aluminum atoms that are coordinated

with only three oxygen atoms. The presence of aluminum atoms causes zeolite to have a negative charge, allowing it to bind to cations. Zeolite optimization is carried out through physical and chemical activation. Physical activation is conducted at temperatures of 300°C-400°C with the aim of removing trapped water from the pores. Chemical activation is achieved using an acid solution, namely hydrochloric acid. The purpose of chemical activation is to clean the pore surfaces and remove impurities (Sugiarti and St. Zaenab, 2008).

Research on the adsorption of heavy metals Cu and Cd by natural zeolite has been conducted extensively. Suyanta and Cerry (2016) conducted a study on the effectiveness of natural zeolite in adsorbing Cu metal using natural zeolite from Gunung Kidul. The study utilized two particle sizes and obtained a maximum adsorption efficiency of 37.1698% for the 10-mesh size with an optimum time of 30 minutes and 35.9976% for the 5-mesh size with an optimum time of 120 minutes. The utilization of zeolite in reducing the concentration of Cd metal has also been studied by Raziah et al. (2017) using natural zeolite from Aceh, achieving an adsorption efficiency of 81.8560% with an optimum time of 40 minutes. Building on the research by Suyanta & Cerry (2016) and Raziah et al. (2017), this study is conducted using commercial natural zeolite sourced from the Ciapus area in Bogor, West Java, to investigate the rate of adsorption of Cu and Cd metal ions from a solution with specific concentrations by zeolite that has been physically activated at 400°C and chemically activated with HCl acid solution.

RESEARCH METHODS

Tools and materials

The equipment used includes Atomic Absorption Spectrometry-Flame XplorAA Dual (AAS), 50 mL macro burette, 20 mL volumetric pipette, 5 mL Mohr pipette, dropper pipette, 100 mL measuring flask, volumetric flasks (200 mL, 500 mL, and 1000 mL), beakers (100 mL, 250 mL, and 1000 mL), glass stirring rod, 100 mL glass funnel, stand, clamp, fritted glass, anodized aluminum spring clamp, vacuum pump, filter paper (0.45 μm), magnetic stirrer, oven, furnace, Sieve Stainless Mesh 10 (2.00 mm), analytical balance HWH, pH universal indicator, mortar and pestle, desiccator, porcelain crucible, porcelain filtering crucible, bulb, and aluminum tray.

The materials used include Cu metal solutions (5, 10, and 15 mg/L), Cd metal solutions (5, 10, and 15 mg/L), Ciapus zeolite, 0.5 M hydrochloric acid (HCl) solution, 0.05 M nitric acid (HNO_3) dilution solution, and distilled water.

0.5 M HCl Solution

The preparation of 0.5 M HCl solution was carried out using the dilution formula $M_1V_1 = M_2V_2$, where M_1 is the molarity of the concentrated HCl stock (37%, approximately 12.06 M), V_1 is the required volume to be calculated, M_2 is the target molarity (0.5 M), and V_2 is the final volume (1000 mL). Based on the calculation, 41.39 mL of 37% HCl solution was dispensed from a burette into a 1000 mL volumetric flask containing distilled water. The solution was then diluted with distilled water up to the mark and homogenized thoroughly.

0.05 M HNO_3 Dilution Solution

The preparation of 0.05 M HNO_3 dilution solution was performed using the dilution formula $M_1V_1 = M_2V_2$. Using concentrated HNO_3 (65%, approximately 13.5 M) as the stock, a volume of 3.7 mL was calculated. A 1000 mL volumetric flask was partially filled with distilled water, then 3.7 mL of 65% HNO_3 was carefully pipetted into the flask (acid added to water, never the reverse). The solution was then diluted with distilled water to the 1000 mL mark and homogenized thoroughly.

Cu Solutions at Concentrations (5, 10, dan 15) mg/L

The stock Cu solution of 1000 mg/L was pipetted (20 mL) into a 200 mL volumetric flask. Distilled water was added up to the 200 mL mark and homogenized, yielding a 100 mg/L Cu intermediate solution. To prepare the working standard series, volumes of 25 mL, 50 mL, and 75 mL of this 100 mg/L Cu solution were each dispensed into separate 500 mL volumetric flasks. Each flask was then diluted with 0.05 M HNO_3 dilution solution to the 500 mL mark and homogenized, resulting in Cu standard solutions of 5, 10, and 15 mg/L, respectively.

Cd Solutions at Concentrations (5, 10, dan 15) mg/L

The stock Cd solution of 1000 mg/L was pipetted (20 mL) into a 200 mL volumetric flask. Distilled water was added up to the 200 mL mark and homogenized, yielding a 100 mg/L Cd intermediate solution. To prepare the working standard series, volumes of 25 mL, 50 mL, and 75 mL of this 100 mg/L Cd solution were each dispensed into separate 500 mL volumetric flasks. Each flask was then diluted with 0.05 M HNO_3 dilution solution to the 500 mL mark

and homogenized, resulting in Cd standard solutions of 5, 10, and 15 mg/L, respectively.

Activation of Ciapus Zeolite

The zeolite rock was crushed using a mortar and pestle to obtain smaller-sized zeolite particles. The obtained zeolite was then sieved using a stainless mesh with a size of 10 and washed thoroughly with distilled water. For the physical activation, the crushed and sieved zeolite was subjected to activation. Activation was carried out by heating it in a furnace at a temperature of 400°C for 3 hours. Subsequently, chemical activation was performed by immersing the zeolite in a 0.5 M HCl solution for 30 minutes at a ratio of 1g/25mL in a beaker. The zeolite was then washed with distilled water until it reached a neutral pH of 7.0 ± 1.0 and dried in an oven at a temperature of 105°C for 90 minutes.

Volatile Matter Content

The unactivated zeolite rock was weighed, approximately ± 2 g, into a porcelain crucible with a known weight. The crucible and zeolite were heated up to 950°C. Once the temperature was reached, the crucible was transferred to a desiccator. This process was carried out in duplicate. The volatile matter content was calculated using the following formula:

$$\text{Volatile Matter (\%)} = [(W_2 - W_3) / (W_2 - W_1)] \times 100\% \quad \ldots(1)$$

where W_1 = weight of empty crucible (g), W_2 = weight of crucible + sample before heating (g), W_3 = weight of crucible + sample after heating at 950°C (g) (BSN, 1995).

Moisture Content

The unactivated zeolite rock was weighed, approximately ± 1 g, into a porcelain crucible with a known weight. The crucible and zeolite were placed in an oven at a temperature of 105°C for 3 hours and then cooled in a desiccator. This process was carried out in duplicate. The moisture content was calculated using the following formula:

$$\text{Moisture Content (\%)} = [(W_2 - W_3) / (W_2 - W_1)] \times 100\% \quad \ldots(2)$$

where W_1 = weight of empty crucible (g), W_2 = weight of crucible + sample before drying (g), W_3 = weight of crucible + sample after drying at 105°C (g) (BSN, 1995).

Ash Content

The unactivated zeolite rock was weighed, approximately ± 3 g, into a porcelain crucible with a known weight. The zeolite was slowly burned and then transferred to a furnace at a temperature of 900°C until it turned into ash. The crucible was cooled in a desiccator. This process was carried out in duplicate. The ash content was calculated using the following formula:

$$\text{Ash Content (\%)} = [(W_3 - W_1) / (W_2 - W_1)] \times 100\% \quad \ldots(3)$$

where W_1 = weight of empty crucible (g), W_2 = weight of crucible + sample before ashing (g), W_3 = weight of crucible + residue after ashing at 900°C (g) (BSN, 1995).

Fixed Carbon Content

The calculation involves subtracting 100% from the sum of volatile matter, moisture content, and ash content. This calculation is performed for each test. The fixed carbon content formula is as follows:

$$\text{Fixed Carbon (\%)} = 100\% - (\text{Volatile Matter} + \text{Moisture Content} + \text{Ash Content}) \quad \ldots(4)$$

All values expressed as percentages (%) (BSN, 1995).

Analysis of Cu and Cd Content by AAS

The activated zeolite rock, weighed approximately ± 1 g, was then placed into a beaker containing 100 mL of each Cu and Cd metal solution. The solutions were stirred using a magnetic stirrer at approximately 50 rpm at room temperature. The adsorption process was conducted with variations in contact time (0, 15, 30, 45, and 60) minutes and concentrations (5, 10, and 15) mg/L. The filtered adsorption samples were analyzed for residual Cu and Cd concentrations using Atomic Absorption Spectrometry-Flame XplorAA Dual (AAS).

Calculation of Adsorption Capacity and Efficiency

The adsorption capacity (q) of zeolite for Cu and Cd ions was calculated using the following formula (Renni et al., 2018):

$$q = [(C_0 - C_t) \times V] / m \quad \ldots(5)$$

where q = adsorption capacity (mg/g), C_0 = initial concentration (mg/L), C_t = concentration at time t (mg/L), V = volume of solution (L), m = mass of adsorbent (g). The adsorption efficiency was calculated as follows:

$$\text{Efficiency (\%)} = [(C_0 - C_t) / C_0] \times 100\% \quad \ldots(6)$$

Adsorption Kinetics Model

The adsorption kinetics model is used to describe the rate at which metal ions are removed from solution by the adsorbent over time. Three kinetic models were applied in this study: zero-order, first-order, and second-order models. The best-fitting model is determined based on the highest coefficient of determination (R^2) obtained from linear regression (Estiaty, 2012).

Zero-Order Kinetics

Zero-order kinetics assumes that the adsorption rate is independent of the concentration of the adsorbate. The linear form of the zero-order equation is obtained by plotting C_A against time (t) (Estiaty, 2012):

$$C_A = C_{A0} - k_0 t \quad \ldots(7)$$

where C_A = concentration at time t (mg/L), C_{A0} = initial concentration (mg/L), k_0 = zero-order rate constant (mg/L·min), t = contact time (minutes).

First-Order Kinetics

First-order kinetics assumes that the adsorption rate is proportional to the remaining concentration of the adsorbate in solution. The linear form is obtained by plotting $\ln(C_{A0}/C_A)$ against time (t) (Estiaty, 2012):

$$\ln(C_{A0}/C_A) = k_1 t \quad \ldots(8)$$

where k_1 = first-order rate constant (min^{-1}), C_{A0} = initial concentration (mg/L), C_A = concentration at time t (mg/L), t = contact time (minutes).

Second-Order Kinetics

Second-order kinetics assumes that the adsorption rate is proportional to the square of the adsorbate concentration. The linear form is obtained by plotting $1/C_A$ against time (t) (Estiaty, 2012):

$$1/C_A = 1/C_{A0} + k_2 t \quad \ldots(9)$$

where k_2 = second-order rate constant (L/mg·min), C_{A0} = initial concentration (mg/L), C_A = concentration at time t (mg/L), t = contact time (minutes).

RESULTS AND DISCUSSION

Characteristics of Ciapus Zeolite

According to the classification of zeolite in the Indonesian National Standard (SNI 13-3594-1994), Ciapus zeolite typically falls under types B and C, with a zeolite content ranging between 50-90%. Therefore, it can still be utilized as a commodity (Muta'alim, 2002). Due to the various properties and types of zeolite, it is necessary to conduct characteristic tests. The Ciapus zeolite used in this study originates from Ciapus, Bogor, West Java. Characteristic tests for Ciapus zeolite as an adsorbent follow the Technical Active Carbon Standard (SNI 06-3730-1995) and include analyses for volatile matter content, moisture content, ash content, and fixed carbon content. The results of the characteristic tests for Ciapus zeolite can be seen in Table 1.

Table 1. Characteristics of Ciapus Zeolite

Parameter	Results (%)	Quality Standards (%)
Volatile Matter Content	4,26	Max. 25
Moisture Content	3,03	Max. 15
Ash Content	8,63	Max. 10
Fixed Carbon Content	78,78	Max. 65

Source: SNI 06-3730-1995, Technical Active Carbon Standard (Badan Standarisasi Nasional, 1995)

Volatile Matter Content

The volatile matter content test is conducted to determine the presence of fly ash in zeolite. High volatile matter content can lower the quality of zeolite (Junary et al., 2015). The analysis results for the first and second crucibles showed volatile matter contents of 2.87% and 5.65%, respectively, with an average of 4.26%. These results were compared to the quality requirements for volatile matter content in SNI 06-3730-1995. The volatile matter content in both crucibles did not exceed the maximum limit of 25% according to SNI 06-3730-1995. According to Elvitriana et al. (2017), the low volatile matter content is due to the evaporation of non-carbon compounds that are volatile. The more silica content in a briquette, the higher its volatile matter content (Yuliah et al., 2017).

Moisture Content

The purpose of the moisture content test is to determine the hygroscopic properties of active zeolite (Nizar et al., 2018). The analysis results for the first

and second crucibles showed moisture contents of 3.29% and 2.78%, respectively, with an average of 3.03%. These results were compared to the quality requirements for moisture content in SNI 06-3730-1995. The moisture content in both crucibles did not exceed the maximum limit of 15% according to SNI 06-3730-1995. According to Nizar et al. (2018), a high moisture content can reduce the adsorption capacity of zeolite towards liquids. Activated zeolite easily absorbs and captures water vapor from the air due to its structure, which has large cavities and pores and a wide surface area. The lower the moisture content, the more empty pores in the zeolite, resulting in a larger surface area and improved zeolite performance as an adsorbent or catalyst.

Ash Content

Ash content is related to the mineral content in the material. The determination of ash content aims to determine the content of metal oxides present in active zeolite (Nizar et al., 2018). The analysis results for the first and second crucibles showed ash contents of 8.58% and 8.68%, respectively, with an average of 8.63%. These results were compared to the quality requirements for ash content in SNI 06-3730-1995. The ash content in both crucibles did not exceed the maximum limit of 10% according to SNI 06-3730-1995. The high ash content in zeolite is due to its high silica content. According to Elvitriana et al. (2017), the ash content is influenced by the amount of silica, and the higher the silica content, the higher the ash content produced.

Fixed Carbon Content

The fixed carbon content test is conducted to determine the level of pure or bound carbon contained in zeolite. The analysis results for the first and second crucibles showed fixed carbon contents of 79.94% and 77.63%, respectively, with an average of 78.78%. These results were compared to the quality requirements for fixed carbon content in SNI 06-3730-1995. The fixed carbon content in both crucibles met the minimum quality requirement of 65% according to SNI 06-3730-1995. The low fixed carbon content is due to the absence of carbonization in the zeolite. According to Rahman et al. (2018), the determination of bound carbon content aims to determine the carbon content after carbonization and activation processes. Therefore, the higher the bound carbon content, the better the activated carbon will be in adsorbing heavy metals.

Adsorption Capacity and Efficiency

Adsorption capacity measurements with variations in contact time were conducted to determine the influence of time on the adsorption of the adsorbent, while variations in concentration were carried out to determine the amount of adsorbate concentration adsorbed by the adsorbent (Renni et al., 2018). The measurement of adsorption efficiency with variations in contact time and concentration can be seen in Tablel 2.

Table 2. Adsorption Capacity of Cu Metal Ions and Cd Metal Ion

Metals Ion	Co (mg/L)	Ce (mg/L)	q (mg/g)	W (g) / V (L)	% Removal
Cd	5.060	3.848	0.1212	1 / 0,1	23.95 %
	10.434	8.913	0.1521	1 / 0,1	14.58 %
	14.510	12.593	0.1917	1 / 0,1	13.21 %
Cu	4.803	3.620	0.1183	1 / 0,1	24.63 %
	10.182	8.683	0.1499	1 / 0,1	14.72 %
	14.272	12.765	0.1507	1 / 0,1	10.56 %

The data in Table 2 were obtained at equilibrium (t = 60 minutes) from measurements of Cd and Cu concentrations using AAS, with a zeolite weight (W) of 1 g and an adsorbate solution volume (V) of 0.1 L. The adsorption capacity (q) was calculated using the equation $q = (Co - Ce) \times V / W$, while the percentage removal was calculated using the equation $(Co - Ce)/Co \times 100\%$.

The adsorption capacity (q) of zeolite for Cd increased with increasing initial concentration (Co), reaching a maximum value of 0.1917 mg/g at Co = 14.510 mg/L, while the adsorption capacity for Cu also increased, reaching a maximum value of 0.1507 mg/g at Co = 14.272 mg/L. This pattern indicates that within the tested concentration range, the zeolite had not yet reached full saturation, so an increase in the initial concentration was still able to drive more metal ions to bind to the adsorbent surface.

Conversely, the removal percentage for both metals actually tended to decrease as the initial

concentration increased, from around 23.95% to a lower value for Cd, and a similar pattern occurred for Cu. This occurs because the number of active sites on the zeolite is fixed (the zeolite mass is constant); thus, at higher initial concentrations, the proportion of metal ions that can be adsorbed relative to the total number of ions in the solution becomes smaller. In general, the adsorption capacities of the zeolite for Cd and Cu fall within a relatively close range, indicating that the zeolite's affinity for these two metals is not significantly different under the experimental conditions. The graphs depicting variations in contact time and concentration are shown in Figure 1.

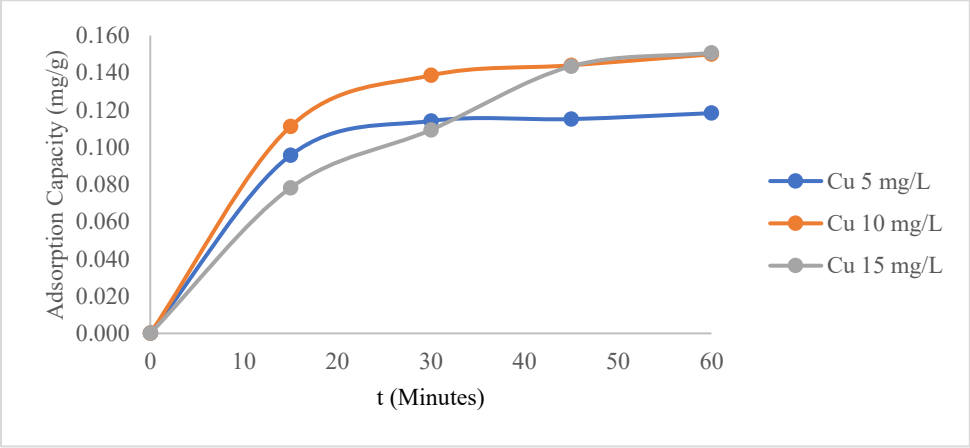

Figure 1. Adsorption Capacity Curve for Variation in Contact Time and Concentration of Cu Metal

The graph in Figure 1 shows that the adsorption of Cu metal ions by zeolite increases with increasing contact time. From 15 to 30 minutes of contact time, the Cu metal curve exhibits an increase because the metal ions have not been fully absorbed by the zeolite. However, from 30 to 60 minutes of contact time, the Cu metal curve no longer increases because the remaining surface area of the zeolite decreases with increasing contact time, and the zeolite is approaching its saturation point. The duration of contact time between the adsorbate and adsorbent affects the amount of adsorbate adsorbed (Sugiarti & St. Zaenab, 2008). This is because the adsorbent has not yet reached saturation, and there is still empty space on the surface of the adsorbent that has not been filled with adsorbate (Renni et al., 2018). Based on the adsorption process and variations in time, the optimum adsorption contact time for Cu metal ions is found to be 30 minutes. Several factors influence the adsorption process, including the type of adsorbent, the type of substance being absorbed, the surface area of the adsorbent, the concentration of the substance being adsorbed, and the temperature (Wijayanti et al., 2018). The increase in concentration affects the adsorption capacity of zeolite for metal ions. Based on the graph in Figure 1, the concentration of Cu metal ions at a concentration of 5 mg/L results in less adsorption by zeolite compared to a concentration of 10 mg/L. However, at a concentration of 15 mg/L, zeolite adsorbs fewer metal ions compared to a concentration of 5 mg/L. Based on the concentration variation process, the optimum concentration is found to be 10 mg/L. The adsorption capacity achieved under optimum conditions is 0.139 mg/g.

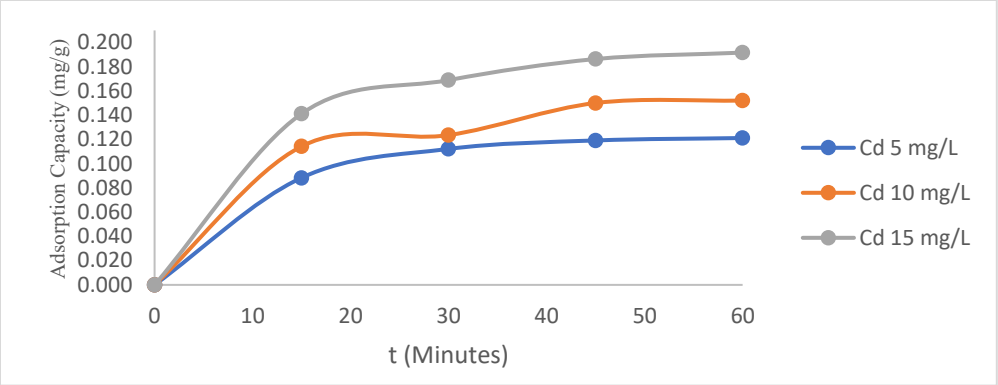

Figure 2. Adsorption Capacity Curve for Variation in Contact Time and Concentration of Cd Metal

The graph in Figure 2 shows that the adsorption of Cd metal ions by zeolite increases with increasing contact time. From 15 to 30 minutes of contact time, the Cd metal curve exhibits an increase, while from 30 to 60 minutes of contact time, the Cd metal curve no longer increases. Based on the adsorption process and variations in time, the optimum adsorption contact time for Cd metal ions is found to be 30 minutes. The graph in Figure 5 shows that the adsorption of Cd metal ions at concentrations of 5, 10, and 15 mg/L results in different levels of adsorption by zeolite. At a concentration of 5 mg/L, zeolite adsorbs fewer metal ions compared to a concentration of 10 mg/L.Similarly, adsorption at a concentration of 10 mg/L is lower than that at a concentration of 15 mg/L. This is due to differences in particle collisions between the two concentrations. According to Estiaty (2012), as the concentration increases, the amount of adsorbate adsorbed per gram of adsorbent also increases. Based on the concentration variation process, the optimum concentration is found to be 15 mg/L. The adsorption capacity achieved under optimum conditions is 0.169 mg/g.

The measurement of adsorption efficiency with variations in contact time and concentration was conducted to determine the reduction in the levels of Cu and Cd metal ions under optimum conditions. The results of the measurements for contact time and concentration variations can be seen in Table 3

Table 3. Adsorption Efficiency of Cu Metal Ions and Cd Metal Ions

Contact Time (minutes)	Efficiency of Copper Ion Adsorption			Efficiency of Cadmium Ion Adsorption		
	5 mg/L	10 mg/L	15 mg/L	5 mg/L	10 mg/L	15 mg/L
0	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
15	19,93%	10,92%	5,48%	17,47%	10,95%	9,74%
30	23,76%	13,61%	7,66%	22,17%	11,85%	11,65%
45	23,96%	14,14%	10,05%	23,56%	14,39%	12,85%
60	24,63%	14,72%	10,56%	23,95%	14,58%	13,21%

The percentage of adsorption efficiency represents the percentage of metal content removed by the adsorbent (Sylvia et al., 2021). Based on Table 3, the percentage of adsorption efficiency for Cu and Cd metal ions increases with contact time. In contrast, the percentage of adsorption efficiency for Cu and Cd metal ions decreases with increasing metal ion concentration. According to Asih et al. (2015), the quantity of metal ions in the solution may not be proportional to the available adsorbent particles, leading to the saturation of the adsorbent surface and a decrease in adsorption efficiency. The highest percentage of adsorption efficiency for Cu and Cd metal ions is observed at a contact time of 60 minutes and a concentration of 5 mg/L.

Adsorption Kinetics

Adsorption kinetics represents the rate of adsorption occurring on the surface of the adsorbent for a certain period of time. The adsorption capacity can be determined by measuring the changes in the concentration of the adsorbed substance, analyzing the rate constant (k) values, typically represented as the slope, and plotting them on a graph (Muslich et al., 2010). Adsorption kinetics are influenced by the adsorption rate, which is the amount of substance adsorbed per unit of time (Yao et al., 2009). The determination of adsorption kinetics can be carried out by observing zero-order, first-order, and second-order kinetic models (Estiaty, 2012). The general forms of these models are as follows. Zero-order kinetics: $C_A = C_{A0} - k_0 t$ …(7); first-order kinetics: $\ln(C_{A0}/C_A) = k_1 t$ …(8); second-order kinetics: $1/C_A = 1/C_{A0} + k_2 t$ …(9); where C_{A0} is the initial concentration (mg/L), C_A is the concentration at time t (mg/L), k is the rate constant, and t is the contact time (minutes) (Estiaty, 2012).

Zero-Order Kinetics Model

The determination of zero-order kinetics is carried out using the linear regression method. The graph is obtained by plotting the data of CA against time (t). The linear regression for zero-order kinetics can be seen in Figure 3.

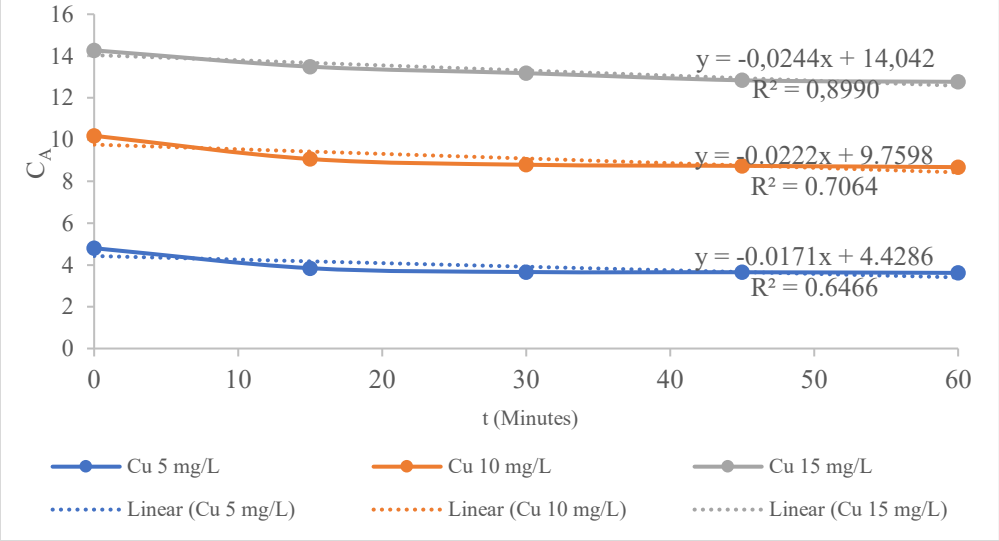

Figure 3. Zero-Order Kinetics Curve of Copper Ion

The graph in Figure 3 shows that the copper ion at a concentration of 15 mg/L is more suitable to follow the zero-order kinetics because it has the highest R^2 value, which is 0.8890. A higher R^2value indicates a better predictive model compared to the proposed research model (Ghozali, 2016). Based on the equation, the value of the rate constant (k) for copper ion at a concentration of 15 mg/L is -0,0244 minutes^{-1}.

The graph in Figure 4 indicates that the cadmium ion at a concentration of 10 mg/L is more suitable for following the zero-order kinetics because it has the highest R^2 value of 0.7380. The difference in R^2 values for cadmium (Cd) and copper (Cu) ions is due to the variance in metal adsorption. Based on the equation, the rate constant (k) for cadmium ion at a concentration of 10 mg/L is -0,0222 minutes^{-1}. This k value represents the rate of adsorption of copper and cadmium ions by zeolite for each unit of time (in minutes). The negative sign in the k value indicates a decrease in the reactant's concentration over time, and the curve is presented in an inverted manner.

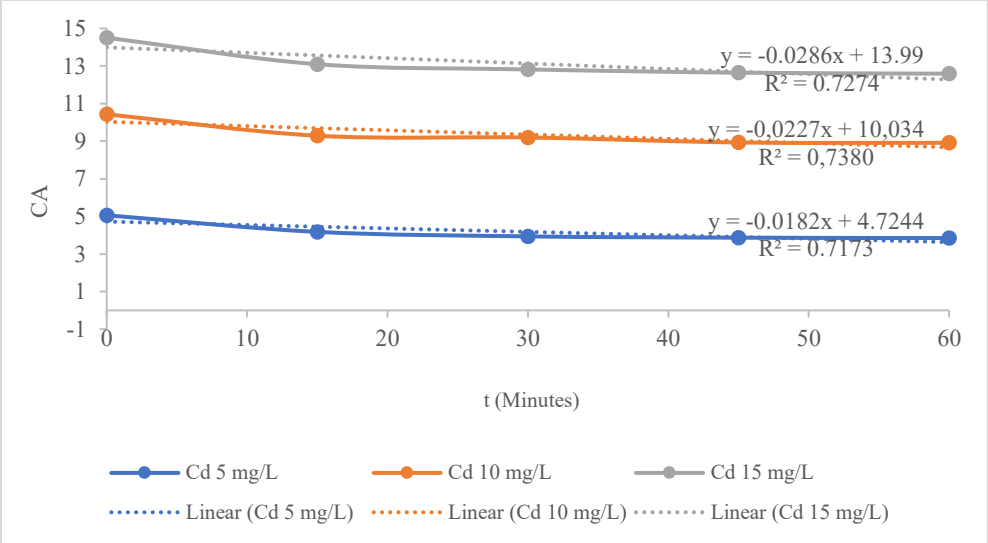

Figure 4. Zero-Order Kinetics Curve of Cadmium Ion

This result indicates that Cd ions at 10 mg/L most closely follow a concentration-independent (zero-order) rate law, even though the maximum adsorption capacity of Cd is instead achieved at 15 mg/L (Table 2). This is not contradictory, since capacity and kinetic order describe different aspects of the process: capacity is governed by the concentration gradient driving ion transport to the zeolite surface, while the best-fitting kinetic order reflects which step film diffusion, pore diffusion, or surface reaction controls the rate at that concentration, a mechanism known to shift with initial concentration, particle size, and ion-adsorbent affinity (Sahmoune et al., 2024). Moreover, the rate constants and R^2 values used to select the best kinetic model are themselves strongly dependent on the initial concentration (Bullen et al., 2021), so the concentration giving the best statistical fit need not coincide with the concentration giving the highest equilibrium uptake. The same reasoning explains the reversed pattern observed for Cu, where the best zero-order fit occurs at 15 mg/L while its maximum capacity is obtained at 10 mg/L.

First-Order Kinetics Model

The determination of first-order kinetics is performed using the linear regression method. The graph is obtained by plotting the data $\ln(C_{A0}/C_A)$ against time (t). The linear equation for first-order kinetics can be seen in Figure 5.

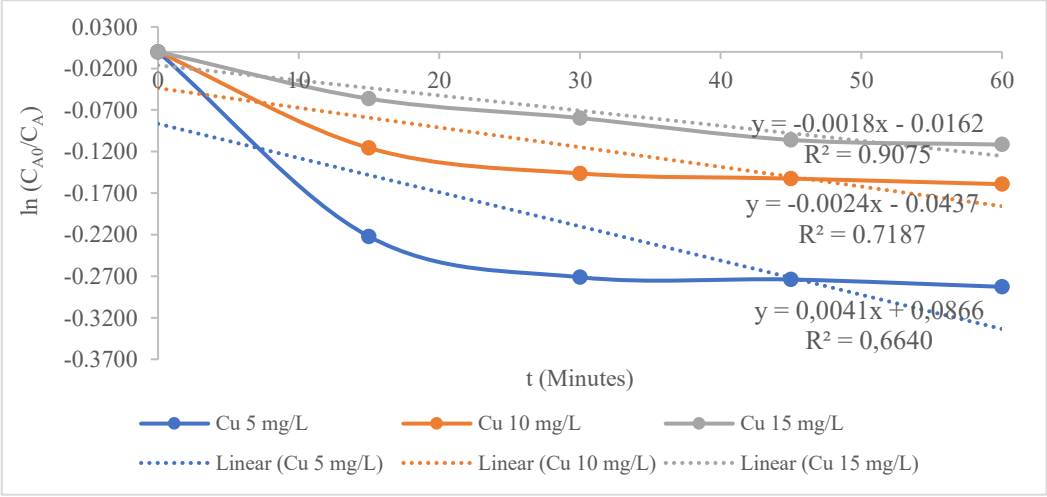

Figure 5. First-Order Kinetics Curve of Copper Ion (Cu)

The graph in Figure 5 shows that copper ions at a concentration of 15 mg/L are more in line with first-order kinetics. The equation has the highest R^2 value, which is 0.9075, with a rate constant (k) of -0.0021 minutes^{-1}. This result is consistent with the findings of Suyanta and Cerry (2016), who reported that the adsorption of Cu ions by natural zeolite from Gunung Kidul also showed a tendency to follow first-order kinetics at higher metal concentrations, reflecting a concentration-dependent adsorption rate. Similarly, Khimantoro et al. (2014) found that the kinetics of heavy metal adsorption using activated natural zeolite followed first-order behavior, indicating that the adsorption rate is proportional to the concentration of remaining metal ions in solution. The negative k value obtained in this study confirms that the concentration of Cu ions decreases over time as adsorption progresses, which is characteristic of first-order reaction kinetics (Estiaty, 2012).

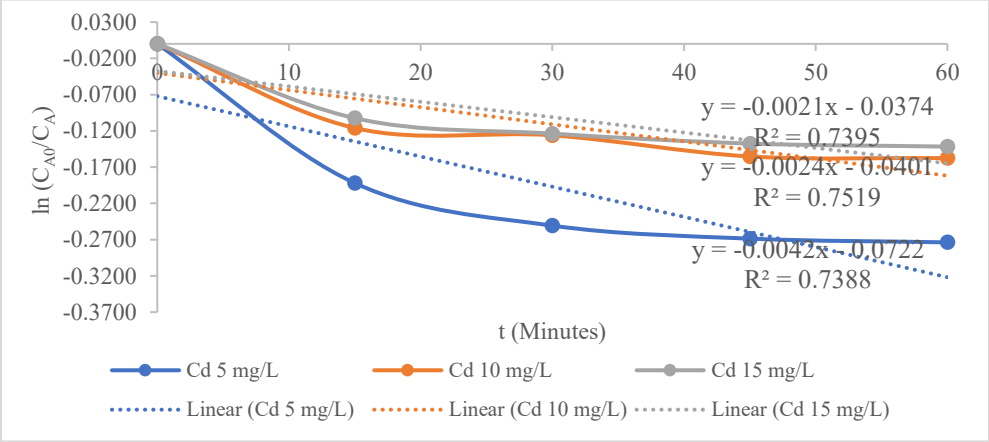

Figure 6. First-Order Kinetics Curve of Cadmium Ion (Cd)

The graph in Figure 6 shows that cadmium ions at a concentration of 10 mg/L are more in line with first-order kinetics. This is because at this concentration, it has the highest R^2 value, which is 0.7519. The rate constant (k) for cadmium ions at a concentration of 10 mg/L is -0.0024 minutes^{-1}. This result is in agreement with the study conducted by Raziah et al. (2017), who investigated the adsorption of Cd ions using natural zeolite from Aceh and observed that adsorption kinetics at moderate concentrations (around 10 mg/L) tended to follow first-order behavior, suggesting that the adsorption rate is dependent on the initial concentration of the adsorbate. Furthermore, Muslich et al. (2010) noted that first-order kinetic models are generally applicable when the adsorbent surface still has sufficient active sites available, so that the rate of adsorption remains proportional to the solute concentration. The relatively lower R^2 value of Cd compared to Cu may reflect greater variability in Cd adsorption behavior, as also noted by Estiaty (2012), who reported that differences in ionic radii and hydration energy between Cu and Cd ions can influence the regularity of the adsorption process on zeolite surfaces.

Second-Order Kinetics Model

The determination of first-order kinetics is carried out using linear regression method. The graph is obtained by plotting $1/C_A$ against time (t). The result of the linear equation for first-order kinetics can be seen in Figure 7.

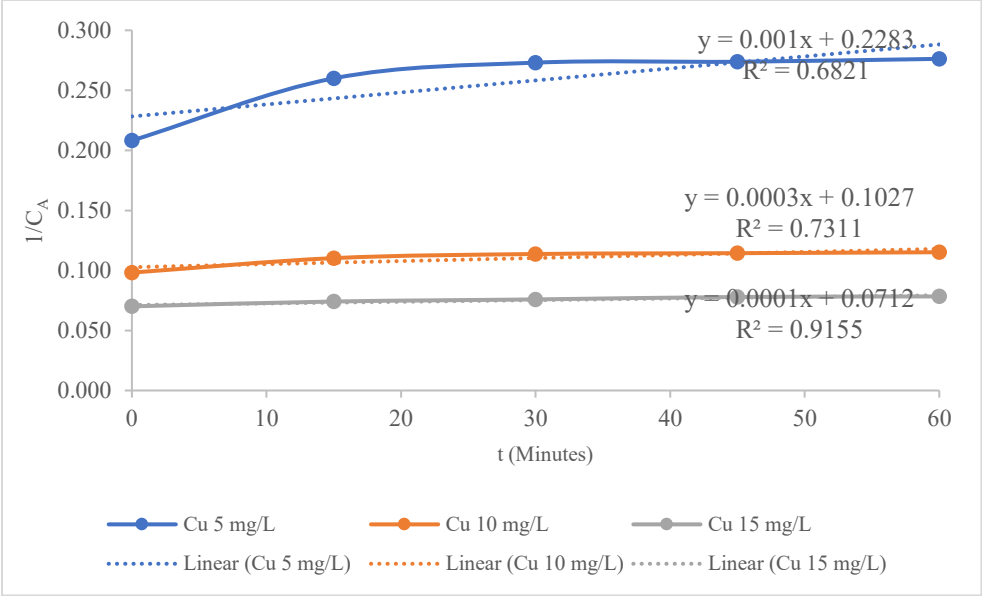

Figure 7. Second-Order Kinetics Curve of Copper Ion

The graph in Figure 7 shows that copper ion at a concentration of 15 mg/L follows second-order kinetics. The equation has the highest R^2 value, which is 0.9155, with a k value of 0,0001 $minutes^{-1}$. Meanwhile, for cadmium ion, a concentration of 10 mg/L follows second-order kinetics as well. This is because at this concentration, it has the highest R^2 value of 0.7659, with a k value of 0,0002 $minutes^{-1}$ (Figure 8).

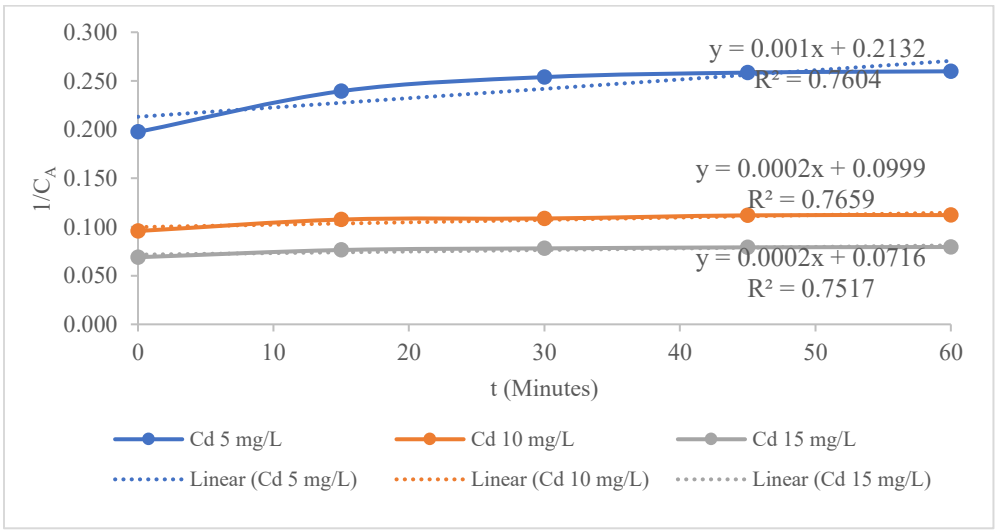

Figure 8. Second-Order Kinetics Curve of Cadmium Ion

4. CONCLUSIONS

The characterization of Ciapus zeolite confirmed that all quality parameters, including volatile matter, moisture, ash, and fixed carbon contents, comply with SNI 06-3730-1995 standards, establishing its suitability as an adsorbent for liquid waste treatment. Physical activation at 400°C and chemical activation with 0.5 M HCl effectively enhanced the adsorption performance of zeolite, achieving maximum adsorption capacities of 0.139

mg/g for Cu and 0.169 mg/g for Cd at optimum contact times of 30 minutes. Kinetic analysis demonstrated that the adsorption of both Cu and Cd ions best follows the pseudo-second-order model, indicating chemisorption as the dominant mechanism governing the interaction between metal ions and the zeolite surface.

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