



Synthesis and Characterization of Zeolite-Geopolymer Composites for Water Treatment

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<https://doi.org/10.18280/acsm.470308>

ABSTRACT

Received: 4 April 2023

Accepted: 2 June 2023

Keywords:

geopolymerization, heavy metal adsorption, Cu²⁺ adsorption, Fe³⁺ adsorption, adsorption isotherm, adsorption kinetics, zeolite-geopolymer composite

This research has studied the synthesis of zeolite-based geopolymer materials with different Si/Al ratios using kaolin and zeolite A with simple, scalable, ambient condition and low-cost method. Along with that, the mechanical strength, density, and pellet formability of the materials were also investigated. Consequently, research introduced zeolite-based geopolymer material with high zeolite content but still kept acceptable strength in water. The metal adsorption capacity was conducted at various initial concentrations and between samples with different Si/Al ratios to provide a suitable kinetic and thermodynamic model for the adsorption process. This study demonstrated that optimized 1.15 Si/Al ratio zeolite-geopolymer composites, which contain 62.4% zeolite, can be a promising adsorbent for removing heavy metal ions from water with 23.15 mg/g for Cu²⁺ and 12.38 mg/g for Fe³⁺ maximum adsorption capacities.

1. INTRODUCTION

Environmental pollution, especially water pollution, is a serious problem today, especially for developing countries. The cause comes from domestic wastewater, agricultural activities, and industries. Copper and its compounds are commonly used in alloys, antirust paints, glasses, enamels, ceramics, and fungicides or pesticides for seeds and crops. Drinking water with large amounts of Cu can cause many health issues, for example, kidney, liver, or nervous system damage and mucosal irritation [1]. The allowable copper content in water should not exceed 2 mg/l [2]. Iron and its compounds are widely used in industry, such as computer memories, coatings for magnetic tapes, pigments, and polishes, or in the manufacture of fertilizers and pesticides. Iron is considered an aesthetic pollutant that affects the smell and color of tea, coffee, and food using contaminated water, along with the growth of bacteria and the blockage of pipes [3]. Currently, a number of biological, physical, and chemical techniques for wastewater treatment have been used to deal with this problem such as chemical precipitation, ion exchange, adsorption, or membrane filtration. Among them, adsorption is an effective treatment method for heavy metal removal from contaminated wastewater at a low cost. In recent years, the research on materials used for the adsorption process is constantly increasing, notably zeolite-based geopolymers [4].

Zeolite is a hydrated crystal of aluminosilicate and metal cations. Thanks to the crystal lattice connected by SiO₄ and AlO₄ tetrahedra with positive ions (Na⁺, K⁺, Ca²⁺, Mg²⁺, ...) [5], zeolite has created great applications such as industrial, domestic and drinking water wastewater treatment, industrial catalysts for many processes, sedimentation of sludge, control of waste liquid or exhaust gas and separation of gases. Geopolymer is an inorganic polymer with a disordered spatial

network structure formed from poly-sialate chains. Geopolymer has attracted much interest from scientists due to its inexhaustible source of raw materials with the possibility of reusing waste products of other industries. Thanks to their high mechanical strength, heat resistance, fire resistance, and acid resistance, geopolymers have many applications in life such as ceramics, construction materials, toxic and radioactive waste storage, and composite types.

The zeolite-based geopolymer material combines the superior properties of geopolymer and zeolite materials. While geopolymer is a "green" material that creates a strong and durable substrate for zeolite, zeolite contains highly applicable properties, typically a porous structure with a large specific surface area, suitable for adsorption as well as ion exchange in practical uses. With this hypothesis, Zeolite-geopolymer composites with optimized Si/Al ratios may introduce as a material with high adsorption capacities for heavy metal removal.

The aim of this study is to synthesize zeolite-geopolymer composites with different Si/Al ratios and evaluate their potential as adsorbents for removing Cu²⁺ and Fe³⁺ from water. By examining adsorption of ion Cu²⁺ and Fe³⁺, this study provides more comprehensive assessment by comparing the difference between two different valence metal behavior in kinetic and thermodynamic adsorption. In addition, mechanical strength, density, and pellet formability of the materials was also studied.

2. EXPERIMENTAL

In this study, commercial zeolite A from Chau Thinh Phat Co., kaolin from KL Mineral Co., and NaOH from Xilong Co. were the main raw materials used to synthesize zeolite-based

geopolymer materials. The synthetic process was carried out based on the published method [6] with some modifications. In detail, the powder zeolite A was mixed with distilled water to avoid changing the geopolymer reaction medium during the alkalization process. The amount of 2.4, 3.76, 5.2, 6.8 and 8.56 g of metakaolin was produced by heating the kaolin at 650°C for 2 hours [7]. Metakaolin was then alkalized with 6 M NaOH solution. Then 7.6, 6.24, 4.8, 3.2, 1.44 g of hydrated zeolite was mixed with the alkalized metakaolin. The mixture is then put into a mold with a diameter and height of 5 mm. The samples were then allowed to cure naturally for 24 hours at room temperature (28°C). After that, the materials were taken out and put in an 80°C oven for the next 24 hours. Then, the material samples were washed with 0.01 M H₂SO₄ solution and distilled water until the pH value is approximately 7. The samples were left to dry naturally for 15 minutes and dried at 80°C for 24 hours to obtain zeolite-based geopolymer pellets. The pellets with Si/Al ratio 1.10; 1.15; 1.20; 1.25; 1.30 was named GZA110, GZA115, GZA120, GZA125, and GZA130 with the mass percentage of zeolite in the sample being 76%, 62.4%, 48%, 32%, and 14.4%, respectively.

X-ray diffraction method (XRD) by Bruker D8 Advance diffractometer with $K_{\alpha} = 1.5418 \text{ \AA}$, 40kV and 25mA Cu anode, and scan rate of 0.0194°/s was used for determination of the crystal structure of the material. The binding present in the material was determined by FTIR on the Frontier FT-IR/NIR instrument. JSM-IT200 InTouchScope™ Scanning Electron Microscope was utilized for morphology and element analysis via the SEM-EDS method. The forming ability of the material samples was determined by the percentage of successfully formed pellets on 20 randomly selected ones. The successfully formed pellets are pellets that do not have visual crack or being crushed after curing. Besides, the unsuccessfully formed pellets were examined on size distribution by a 2 mm sieve. The density of the material samples was determined by measuring the mass and volume of the samples using a 4-digit analytical scale and a 150 mm caliper with a tolerance of 0.02 mm. The mechanical strength in the water of the material samples was determined through the percentage of weight remaining on the 2 mm sieve over 24 hours when 0.5 g of each material sample was added to 100 ml distilled water. The mechanical strength of the material will be investigated through the compressive strength by hydraulic compressors with rate of 0.01MPa/s.

To study the heavy metal removal of material over time, the solution containing metal ions was adsorbed at a concentration of 30 ppm, room temperature 30°C, a solid-liquid ratio of 500 ml/g, pH = 5 with Cu²⁺ and pH = 2 with Fe³⁺. pH of solution was adjusted by 0.01M NaOH and 0.01M H₂SO₄ solution and measured by pH meter. The solution containing metal ions was adsorbed under the above conditions in the concentration range of 20 - 80 ppm for Cu²⁺ and 20 - 70 ppm for Fe³⁺. Cu²⁺ solution with different concentration is prepared from 1000 ppm Cu²⁺ solution made by dissolving 3.93g CuSO₄.5H₂O into 1L distilled water. Fe³⁺ solution with different concentration is prepared from 1000 ppm Fe³⁺ solution made by dissolving 8.635 g NH₄ Fe(SO₄).12H₂O into 1L distilled water. The adsorption time was in 120 min with Cu²⁺ and 180 min with Fe³⁺. The sample was leaved on sieve in becher during adsorption and took out after the adsorption finish. The remaining metal concentration was vacuum filtered to remove any further residuals and determined by complexing PAN solution with Cu²⁺ and complexing NH₄SCN solution with Fe³⁺, then based on a calibration curve obtained by ultraviolet-

visible spectroscopy method (UV-Vis) measured by LABOMED Spectro 2000 RS to determine the concentration. Besides, the percentage of removal is determined by the formula:

$$\%R = (C_0 - C_t) * 100 / C_0 \quad (1)$$

The adsorption capacity is determined by the formula:

$$Q_t = (C_0 - C_t) * V / m \quad (2)$$

The first and second-order kinetic models are studied according to the following expressions, respectively:

$$1^{\text{st}} \text{ order: } \log(Q_e - Q_t) = \log(Q_e) - K_1 * t / 2.303 \quad (3)$$

$$2^{\text{nd}} \text{ order: } t / Q_t = (1 / K_2 Q_e^2) + (1 / Q_e) * t \quad (4)$$

The Freundlich model and the Langmuir model are studied according to the expression respectively:

$$\text{Freundlich: } \log(Q_e) = \log(K_F) + (1/n) * \lg(C_e) \quad (5)$$

$$\text{Langmuir: } C_e / Q_e = (1 / K_L Q_m) + (C_e / Q_m) \quad (6)$$

where, C₀ and C_t are concentration at the beginning and concentration at time t; Q_t is the adsorption capacity at time t; m and V are adsorbent mass and solution volume; Q_e is the adsorption capacity at the equilibrium time; K₁, K₂ are rate constants of the first- order kinetic models and second-order kinetic models, respectively; Q_m is the maximum adsorption capacity; K_L, K_F are Langmuir and Freundlich constants, where 1/n is the heterogeneity coefficient.

3. RESULTS AND DISCUSSION

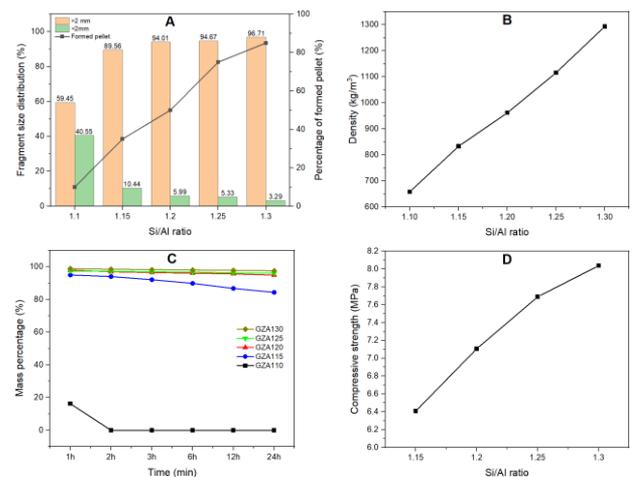


Figure 1. Percentage of formed pellets with the size distribution of the fragments (A), density (B), strength in water (C) and compressive strength (D) of the material

Mechanical properties of the geopolymers are reported in Graph (A) of Figure 1. It shows that when increasing the Si/Al ratio, the material has better pelletizing ability with the results gradually increasing from 10% to 85%. The fragment size distribution results show the same trend as the successfully formed pellets. The results in fragment size smaller than 2 mm at low Si/Al ratio 1.1 account for largely 40.55% of sample

weight. However, at higher Si/Al ratios from 1.2 to 1.3, the percentage of fragments less than 2 mm in size decreased significantly, the lowest reached 3.29%. Graph (B) of Figure 1 shows that the density of the material samples increased from 658 kg/m³ to 1293 kg/m³. Graph (C) of Figure 1 shows that at the Si/Al ratio 1.1, the material is almost unstable in water, after 1 hour, the weight percentage on the 2 mm sieve with pore size is only 16.27%. However, at the Si/Al ratio of 1.15, although the ratio difference is small, after 24 hours the weight percentage on the sieve still kept up to 84%. With higher Si/Al ratios than the sample, there was almost no decay over 24 hours. Graph (D) of Figure 1 shows that the higher the Si/Al ratio or the mass percentage of metakaolin, the higher the strength of the material. The highest strength of the material is 8.04 MPa corresponding to sample GZA130.

Table 1 compares the difference between zeolite-based geopolymer material with different Si/Al ratio, it can be seen that zeolite-based geopolymer material with 1.15 Si/Al ratio has the highest percentage amount of zeolite but still has the characteristic of geopolymer with stable formability, undecomposed in water and high compressive strength.

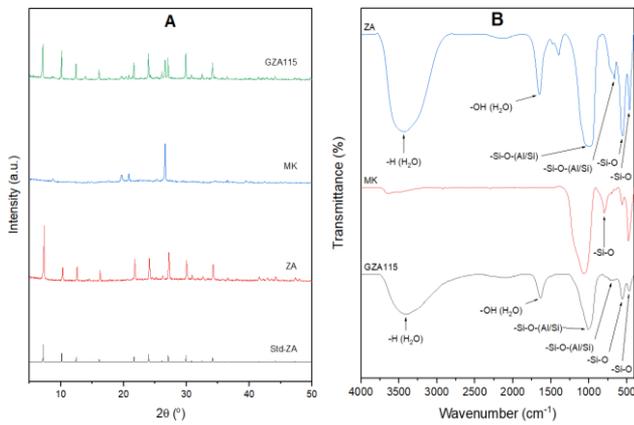


Figure 2. XRD patterns and FTIR spectra of GZA115, metakaolin-MK and zeolite A-ZA

From Graph (A) of Figure 2, The XRD results show that the geopolymerization process with alkaline agents has an influence on the structure but still retains the characteristics of zeolite A and there are almost no strange signals appearing. The decrease in XRD signal intensity in the material sample can be explained by the increase of the amorphous phase in the material structure formed from metakaolin during geopolymerization.

From Graph (B) of Figure 2, The results from FT-IR spectra show peaks that are typical for the bonding vibrations in zeolite A with wave numbers 3400 cm⁻¹, 1650 cm⁻¹, 1000 cm⁻¹, 662 cm⁻¹, 560 cm⁻¹, and 460 cm⁻¹ [8] appeared in sample GZA115. The results from FT-IR spectra also show the appearance of wavenumber 1000 cm⁻¹ and 460 cm⁻¹ characterizing metakaolin appearing in sample GZA115 [9]. The appearance of a 798 cm⁻¹ peak in the kaolin meta sample and the disappearance of this one in the zeolite-based geopolymer samples is typical for the Si-O valence vibration lost due to the change in the coordination number of Al from 6 to 4 after geopolymerization [10].

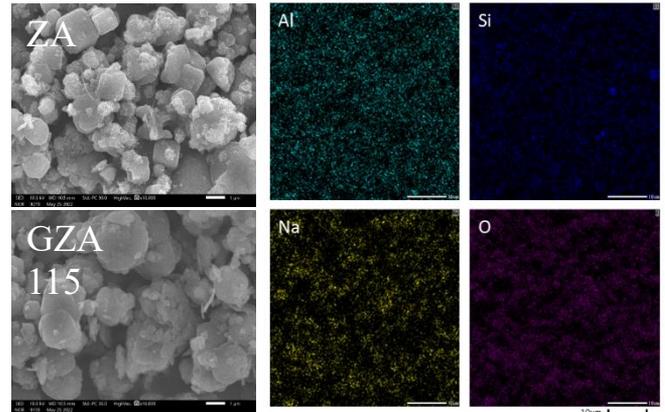


Figure 3. SEM image of zeolite A-ZA and GZA115 (left) and EDS mapping of GZA115 (right)

From SEM images of zeolite A and sample GZA115 in Figure 3, it can be seen that zeolite A crystals appear in the material structure. However, we see a deterioration in the structure of zeolite A crystals. This can be explained by the use of alkaline agents in the geopolymerization process, causing a partial collapse of the material structure. From the EDS results, it can be seen that Al, Si, O, and Na elements are evenly distributed in the material sample. The distribution of Na element can imply the even distribution of zeolite crystals in the material structure.

From Table 2, it can be calculated that the ratio between Si and Al elements is 1.145, approximately 1.15, which is approximately the theoretical calculation results. The result that the percentage of K element is very small, about 0.43 ± 0.1% also shows that most of the cations contained in zeolite are Na, which is consistent with the zeolite A formula used in the calculation.

Table 1. Characteristic comparison of different Si/Al ratio zeolite-based geopolymer material

Si/Al Ratio	1.1	1.15	1.2	1.25	1.3
Amount of Zeolite	76%	62.4%	48%	32%	14.4%
Formability	Formed, unstable	Formed, stable	Formed, stable	Formed, highly stable	Formed, highly stable
Density (kg/m ³)	658	833	962	1116	1293
Strength in Water	Highly decay	Stable	Highly stable	Highly stable	Highly stable
Comp. Strength (MPa)	-	6.41	7.11	7.69	8.04

Table 2. Table of elemental percentages of GZA115 obtained by EDS

Element	O	Na	Al	Si	K
Element Percentage (%)	64.62 ± 0.19	8.57 ± 0.07	12.30 ± 0.08	14.08 ± 0.10	0.43 ± 0.03

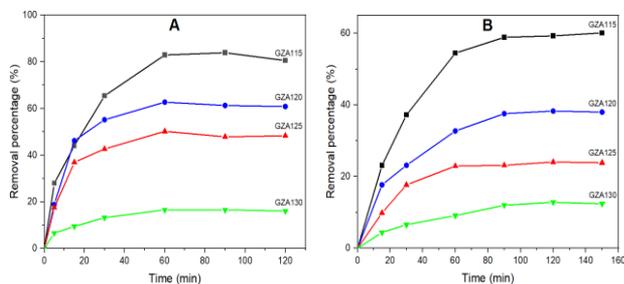


Figure 4. The plot of time versus removal percentage of Cu^{2+} (A) and Fe^{3+} (B)

For heavy metal removal, Figure 4 shows the adsorption with different Si/Al ratio zeolite-based geopolymer. It can be seen that the highest percentage of Cu^{2+} and Fe^{3+} removal belongs to GZA115 with 83.89% and 60.12%, respectively. Besides, the adsorption capacity is 12.6 mg/g and 6.03 mg/g toward Cu^{2+} and Fe^{3+} , respectively. However, when increasing the Si/Al ratio of zeolite-based geopolymer samples, the adsorption capacity of these samples tends to decrease. This can be explained that the low zeolite content in the samples with a high Si/Al ratio reducing the adsorption capacity of the material. Cu^{2+} removal results in samples GZA120, GZA125, GZA130 respectively reached 61.32%; 48.34%; 16.58% with Cu^{2+} and 38.31%; 24.08%; 12.83% with Fe^{3+} .

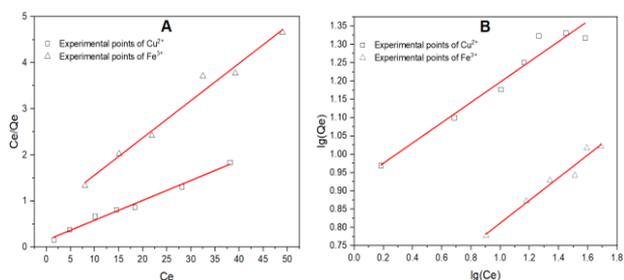


Figure 5. Thermodynamic models for Cu^{2+} and Fe^{3+} removal using GZA115: Freundlich model (A) and Langmuir model (B)

Table 3. Constants of the thermodynamic models for removal of Cu^{2+} and Fe^{3+} using GZA115

Metal ion		Cu^{2+}		
Model		K_L (L/mg)	Q_m (mg/g)	R^2
Langmuir		0.285	23.15	0.9888
Freundlich		K_F (mg/g)(L/mg) ^{1/n}	n	R^2
		8.319	3.61	0.9531
Metal ion		Fe^{3+}		
Model		K_L (L/mg)	Q_m (mg/g)	R^2
Langmuir		0.107	12.38	0.9806
Freundlich		K_F (mg/g)(L/mg) ^{1/n}	n	R^2
		3.180	3.22	0.9663

The data for C_e and Q_e in the Figure 5 has been obtained by experiment with different initial concentration. The maximum adsorption capacities for Cu^{2+} are 9.32 mg/g, 12.6 mg/g, 15.03

mg/g, 17.84 mg/g, 21.06 mg/g, 21.44 mg/g and 20.76 mg/g with 20-80 ppm initial Cu^{2+} concentration, respectively. The maximum adsorption capacities for Fe^{3+} are 5.99 mg/g, 7.45 mg/g, 8.26 mg/g, 9.76 mg/g, 10.40 mg/g, 10.51 mg/g for 20-70 ppm initial Fe^{3+} concentration, respectively.

Adsorption thermodynamics of heavy metal removal has been analyzed. From Table 3, it can be seen that Langmuir isotherm adsorption model is more suitable than Freundlich isotherm adsorption model with R^2 of 0.9888 with Cu^{2+} and 0.9806 with Fe^{3+} compared to 0.9531 and 0.9663, respectively. Thus, the Langmuir model is more appropriate for the $\text{Cu}^{2+}/\text{Fe}^{3+}$ adsorption on the geopolymer material. As seen in Table 2, the maximum adsorption capacity of the material is 23.15 mg/g with Cu^{2+} and 12.38 mg/g with Fe^{3+} .

Table 4. The kinetic model constants for Cu^{2+} and Fe^{3+} removal

Sample	Metal ion	Model	First-Order		
			K_1 (1/min)	Q_e (mg/g)	R^2
GZA115	Cu^{2+}		0.0732	15.08	0.9661
GZA120			0.0719	8.97	0.9798
GZA125			0.0633	6.67	0.9548
GZA130			0.0504	2.23	0.9746
GZA115	Fe^{3+}		0.0387	6.23	0.9797
GZA120			0.0417	4.39	0.9794
GZA125			0.0392	2.17	0.9676
GZA130			0.0279	1.38	0.9719
Sample	Metal ion	Model	Second-Order		
			K_2 (g/mg·min)	Q_e (mg/g)	R^2
GZA115	Cu^{2+}		0.0732	15.08	0.9661
GZA120			0.0719	8.97	0.9798
GZA125			0.0633	6.67	0.9548
GZA130			0.0504	2.23	0.9746
GZA115	Fe^{3+}		0.0387	6.23	0.9797
GZA120			0.0417	4.39	0.9794
GZA125			0.0392	2.17	0.9676
GZA130			0.0279	1.38	0.9719

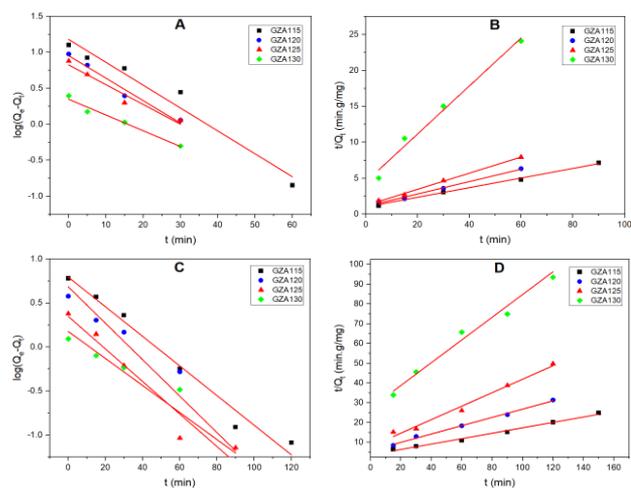


Figure 6. Kinetic analysis of Cu^{2+} removal (A- first order, B- second order), and Fe^{3+} removal (C- first order, D- second order) using the geopolymer materials

Figure 6 and Table 4 reports adsorption kinetics of metal

removal using the geopolymer materials. In both Cu^{2+} and Fe^{3+} cases, the pseudo-second-order kinetic model is more appropriate with the R^2 value being larger than the R^2 value of the pseudo-first-order kinetic model and greater than 0.98. The theory adsorption capacity at equilibrium Q_e calculated for GZA115, GZA120, GZA125, GZA130 samples, are 14.94 mg/g; 11.61 mg/g; 8.91 mg/g; 2.99 mg/g respectively with Cu^{2+} and 4.942 mg/g; 2.893 mg/g; 1.947 mg/g; 1.587 mg/g respectively with Fe^{3+} . The rate constants drawn from the model with samples GZA115, GZA120, GZA125, GZA130 are 0.0044 g/mg; 0.0068 g/mg.min; 0.0103 g/mg.min; 0.0249 g/mg.min respectively with Cu^{2+} and 0.0074 g/mg.min; 0.0119 g/mg.min; 0.0222 g/mg.min; 0.0223 g/mg.min respectively with Fe^{3+} .

4. CONCLUSION

The objective of this study was to synthesize zeolite-geopolymer composites for heavy metal removal from wastewater. Zeolite-geopolymer composites are promising adsorbents due to their high adsorption capacities, low costs and environmental friendliness. The research has demonstrated a path to prepare zeolite-based geopolymer with low-cost chemicals and simple synthetic conditions. The XRD, SEM, FTIR results showed that the zeolite structure was still kept in the material structure. The results of physical and mechanical properties revealed that when the Si/Al ratio was increased, the formability, density and strength of the materials all increased. However, the zeolite-geopolymer composite GZA115 with the highest adsorption capacity can be a potential adsorbent for the treatment of heavy metal-contaminated wastewater from various industries. This material also shows the ability to remove metal ions with the maximum adsorption capacity of 23.15 mg/g with Cu^{2+} and 12.38 mg/g with Fe^{3+} from data analysis by Langmuir isotherm model. The pseudo-second-order adsorption kinetic model can be the appropriate model for $\text{Cu}^{2+}/\text{Fe}^{3+}$ removal by the zeolite-based geopolymer. Nevertheless, further studies on the effects of other experimental conditions, material modifications and adsorption mechanisms are still needed to optimize the adsorbent performance.

ACKNOWLEDGMENTS

We acknowledge Ho Chi Minh City University of Technology (HCMUT), VNU-HCM for supporting this study.

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NOMENCLATURE

C_0, C_t	Concentration at the beginning and at time t, ppm
Q_t, Q_e, Q_m	Adsorption capacity at time t, at the equilibrium time and maximum adsorption capacity, mg/g
K_1	Rate constants of the first-order kinetic models, 1/min
K_2	Rate constants of the second-order kinetic models, (g/mg.min)
K_F	Freundlich constants, $(\text{mg/g})(\text{L/mg})^{1/n}$
K_L	Langmuir constants, (L/mg)