

ADSORPTION KINETICS AND ISOTHERMS OF Cu(II) AND Fe(II) IONS FROM AQUEOUS SOLUTIONS BY FLY ASH-BASED GEOPOLYMER

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Abstract. This paper describes the adsorption of Cu²⁺ and Fe²⁺ ions, common heavy metals found in industrial wastewater, by a fly ash-based geopolymer in batch adsorption experiments. Kinetics studies showed that the adsorption of each ion followed a pseudo-second order reaction. Moreover, adsorption isotherm of Cu²⁺ and Fe²⁺ ions followed the Langmuir model. Monolayer adsorption capacities were approximately 53.76 mg/g for Cu²⁺ ion and 52.63 mg/g for Fe²⁺ ion, respectively.¹

Keywords: adsorption, Cu(II) ion, Fe(II) ion, fly ash-based geopolymer, Langmuir isotherm, pseudo second order reaction.

1. Introduction

Geopolymers are mineral polymers with Si–O–Al bonds formed from aluminosilicate materials with alkaline activators at the temperature below 373 K. Aluminosilicate materials used as a geopolymer raw material can come from nature such as kaolin or metakaolin (calcined kaolin) and solid wastes such as fly ash and slag. Alkaline activators commonly used in geopolymer production are sodium hydroxide solution, potassium hydroxide solution, mixture of sodium hydroxide solution and sodium silicate solution, mixture of potassium hydroxide solution and potassium silicate solution. Geopolymers have been widely applied as a building material, coatings, adhesives, composites, and waste encapsulation.¹

Fly ash is a by-product from coal combustion at power plant. Fly ash which is solid waste can be utilized

as additives in cement industry, road construction, mine reclamation, *etc.* Fly ash containing silica and alumina can also be utilized as a geopolymer raw material.²⁻⁴ Fly ash-based geopolymer has been widely used as Portland cement substitute because it has a high mechanical strength, resistance to corrosive environment, and less CO₂ emissions.^{5,6}

Geopolymers with amorphous to semi-crystalline three dimensional structures have pores that potentially are to be used as an adsorbent, for example the heavy metal adsorbent. This is also supported by the presence of negative charge on aluminium in the geopolymer structure that will attract cation.^{7,8} Adsorption is one of the treatment methods of wastewater containing the heavy metal that is widely used due to relatively easy operation, high efficiency, low cost, and possibility of adsorbent regeneration.⁹⁻¹¹ Copper and iron ions are common heavy metals found in industrial wastewater.¹² Long-term exposure of copper can cause headache, stomachache, and irritation of nose, mouth, eyes; while excess amounts of iron can cause rapid pulse rates, blood vessels congestion, and hypertension.¹³

In this research, the fly ash-based geopolymer was used as the adsorbent for Cu²⁺ and Fe²⁺ ions. Adsorption kinetics and isotherms that can describe the adsorption process were studied. In addition, the fly ash-based geopolymer was also characterized before and after the adsorption process. The results of this study were expected to contribute to the solid waste utilization for wastewater treatment.

2. Experimental

2.1. Geopolymer Preparation

Fly ash from power plants in East Java, Indonesia, was sieved with 200 mesh standard sieve before being used as the geopolymer raw material. The oxides content

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in the fly ash based on X-ray fluorescence (XRF) analysis is shown in Table 1. The silica and alumina contents in the fly ash are high enough so that fly ash can be used as the raw material for the geopolymer.

Fly ash was mixed with an alkaline activator for 10 min to form the geopolymer. The ratio of fly ash to the alkaline activator was 2.5:1. Alkaline activator consisted of 10N NaOH solution and Na-silicate solution with the ratio of 1:1. Na-silicate solution had $\text{SiO}_2/\text{Na}_2\text{O}$

mole ratio of 3.2 and solid mass fraction of 0.35. The mixture was placed in a 5'5'5 cm mold and cured for 24 h. After removing from the mold, the geopolymer was cured in an oven at 333 K for 6 h and then at room temperature for 7 days. The geopolymer was crushed and sieved with 50 mesh standard sieve to obtain geopolymer powder used as the heavy metal adsorbent. Geopolymer powder had Brunauer-Emmett-Teller (BET) surface area of 27.205 m^2/g .

Table 1. Oxides content in fly ash

Oxide	wt %	Oxide	wt %	Oxide	wt %	Oxide	wt %
SiO_2	42.382	MgO	4.721	ZnO	0.011	Others	0.875
Al_2O_3	22.62	K_2O	1.118	TiO_2	0.957		
CaO	14.796	CuO	0.008	P_2O_5	0.604		
Fe_2O_3	9.447	MnO	0.092	SO_3	2.369		

2.2. Adsorption Experiment

Solutions of Cu^{2+} and Fe^{2+} were each prepared by dissolving the analytical grade $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ with distilled water. Batch adsorption experiments were carried out on 100 ml heavy metal solution at room temperature (301 K) with geopolymer dosage of 0.2 g, pH of 5, and stirring rate of 200 rpm. Kinetics studies were conducted with varying adsorption time (15–180 min) at the initial concentration of heavy metal solution of 100 ppm. Meanwhile, isotherm studies were conducted with a varying initial concentration of heavy metal solution (10–150 ppm) at adsorption time of 2 h. The concentrations of Cu^{2+} and Fe^{2+} solutions were each measured by atomic absorption spectroscopy (AAS) with a removal efficiency (%) as follows:

$$\text{Removal efficiency} = \frac{C_0 - C_e}{C_0} \cdot 100\% \quad (1)$$

where C_0 and C_e are the initial and equilibrium concentrations of heavy metal solution, respectively.

2.3. Characterization

Characterization of geopolymer before and after adsorption process comprised X-ray diffraction (XRD) analysis and Fourier transform infrared spectroscopy (FTIR) analysis. XRD analysis was conducted with a Shimadzu XRD-7000 instrument under following

conditions: 30 kV, 30 mA, $\text{CuK}\alpha$ radiation, and $2\theta = 5\text{--}90^\circ$. FTIR analysis was conducted with a Shimadzu IR Prestige-21 instrument with KBr pellet technique in the wavenumber range of 4000–400 cm^{-1} .

3. Results and Discussion

3.1. The Effect of Adsorption Time and Adsorption Kinetics

The effect of adsorption time on the removal efficiency of Cu^{2+} and Fe^{2+} ions is shown in Fig. 1. The increase in the adsorption time caused the increase in the removal efficiency until it did not change. This indicated that the adsorption process had reached equilibrium. The adsorption process of Cu^{2+} and Fe^{2+} ions by the fly ash-based geopolymer had reached equilibrium after 120 min.

Studies on the effect of adsorption time can provide information related to the mechanism of adsorption process that determined by a kinetic model. The expression for adsorption kinetics models are:¹⁴

- A pseudo-first order reaction:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

- A pseudo-second order reaction:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (3)$$

where k_1 and k_2 are the pseudo-first order and the pseudo-second order reaction constants, respectively; q_e

and q_t are the amounts of adsorbed metal ions or adsorption capacity at equilibrium and at time t , respectively.

Linear fittings of the pseudo-first order kinetics model, namely $\ln(q_e - q_t)$ vs. t , for adsorption of Cu^{2+} and Fe^{2+} ions by the fly ash-based geopolymer are shown in Fig. 2, while linear fittings of the pseudo-second order kinetics model, namely t/q_t vs. t , in Fig. 3. The obtained

adsorption kinetics parameters are listed in Table 2. Adsorption process followed the pseudo-second order kinetics model with a coefficient of correlation (R^2) of 0.992 and 0.997 for Cu^{2+} and Fe^{2+} , respectively. Thus the adsorption of Cu^{2+} and Fe^{2+} ions by the fly ash-based geopolymer was controlled by the chemical adsorption process.^{15,16} These result were similar to those obtained by other researches.^{14, 17-19}

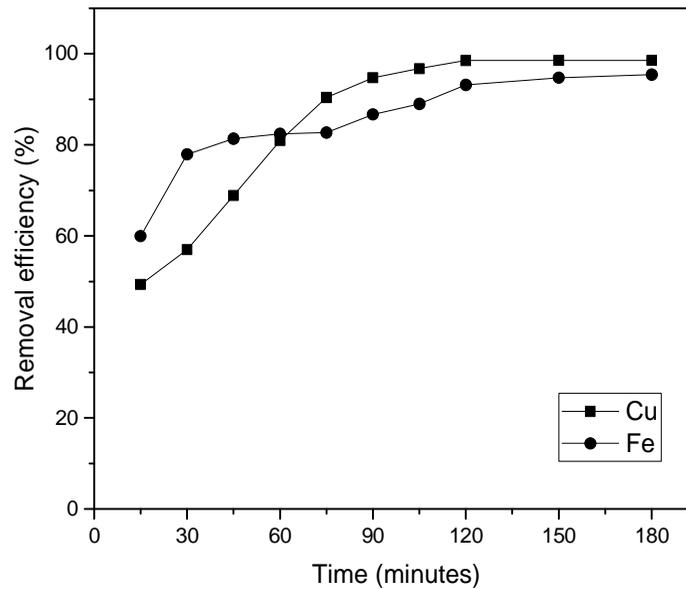


Fig. 1. Effect of adsorption time on Cu^{2+} and Fe^{2+} ions removal efficiency

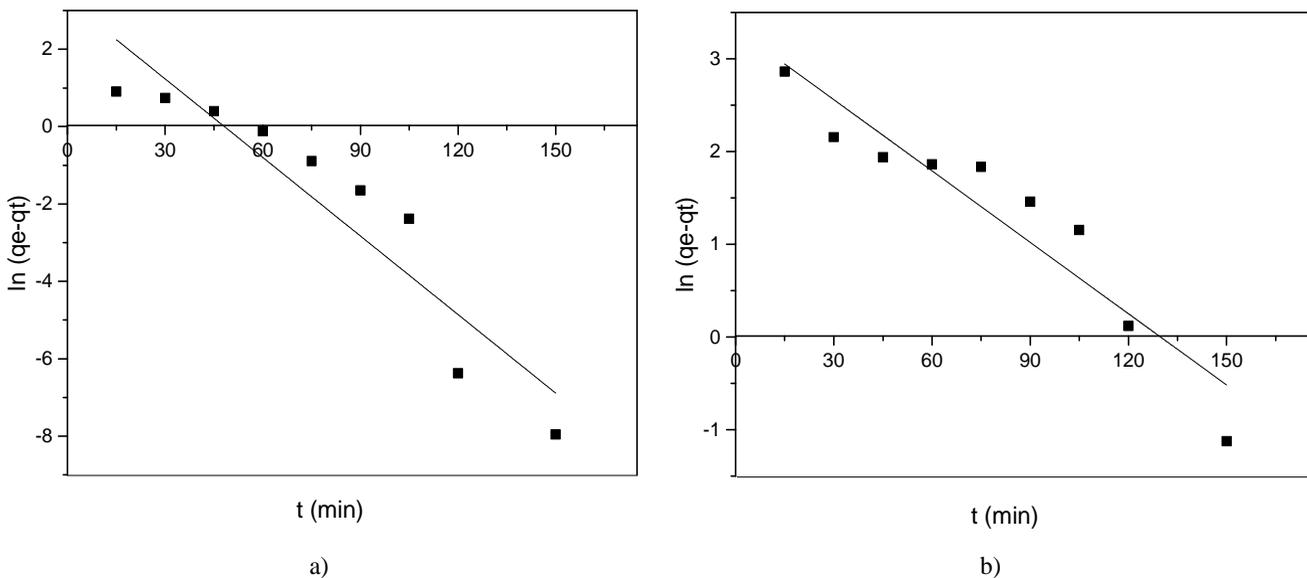


Fig. 2. Linear fittings of the pseudo-first order kinetics model for adsorption of Cu^{2+} (a) and Fe^{2+} (b) ions by the fly ash-based geopolymer

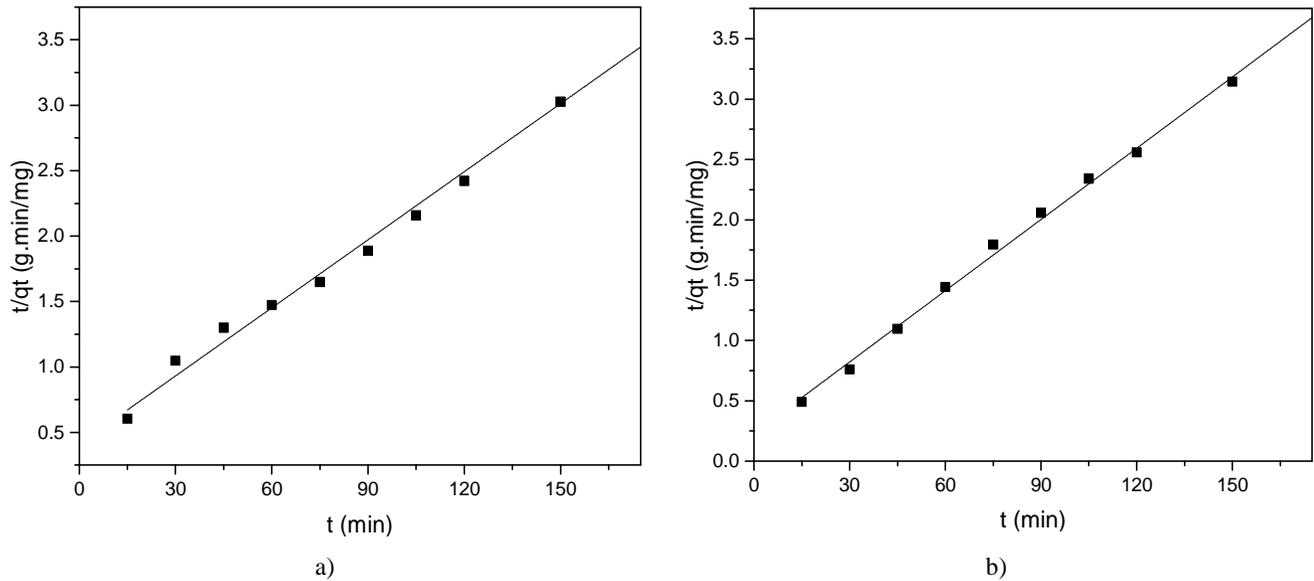


Fig. 3. Linear fitting of pseudo second order kinetics model for adsorption of Cu^{2+} (a) and Fe^{2+} (b) ions by the fly ash-based geopolymer

Table 2. Adsorption kinetics parameters for adsorption of Cu^{2+} and Fe^{2+} ions by the fly ash-based geopolymer

Metal ion	Pseudo-first order kinetics model			Pseudo-second order kinetics model		
	$q_e, mg \cdot g^{-1}$	k_1, min^{-1}	R^2	$q_e, mg \cdot g^{-1}$	$k_2, g \cdot mg^{-1} \cdot min^{-1}$	R^2
Cu^{2+}	26.102	0.067	0.872	52.804	0.000727	0.992
Fe^{2+}	27.910	0.025	0.889	51.020	0.001657	0.997

3.2. The Effect of Initial Concentration and Adsorption Isotherm

The effect of initial concentration on Cu^{2+} and Fe^{2+} ions removal efficiency is shown in Fig. 4. The increase of initial concentration would decrease the removal efficiency. At low concentration of adsorbate, the adsorbent had the sufficient surface to adsorb the adsorbate. However, if concentration of adsorbate was increased continuously then the surface of the adsorbent was no longer sufficient to adsorb the adsorbate.²⁰

The interaction between the adsorbent and the adsorbate can be studied from the adsorption isotherm equilibrium model. Adsorption isotherm equilibrium model can be determined with the varying initial concentration of adsorbate at constant adsorbent dosage. Common equilibrium models of adsorption isotherm are the Langmuir isotherm and the Freundlich isotherm.¹⁸

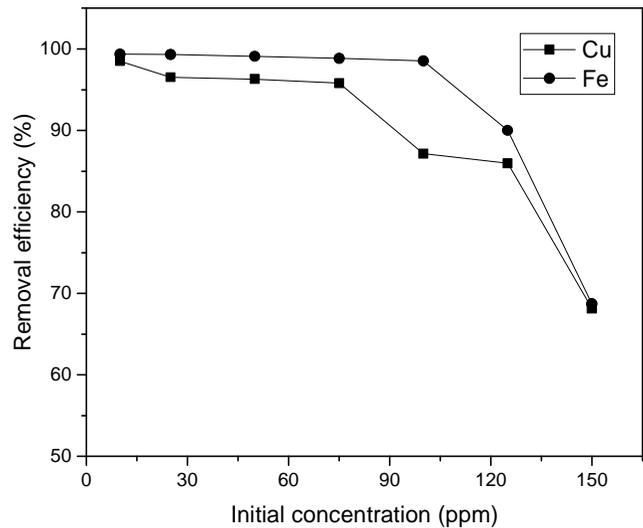


Fig. 4. Effect of initial concentration on Cu^{2+} and Fe^{2+} ions removal efficiency

Langmuir isotherm

In this model, the adsorbent is assumed to have a whole homogeneous surface and each adsorbent molecule

can only adsorb one adsorbate molecule so that there is only one layer of adsorbate on the surface of the adsorbent. The Langmuir isotherm equation is expressed as:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (4)$$

where C_e is the equilibrium concentration, q_m is the maximum adsorption capacity and K_L is the Langmuir constant.

Freundlich isotherm

The adsorbent is assumed to have a heterogeneous surface in this model so that each adsorbent molecule has different adsorption abilities. The equation for Freundlich isotherm is expressed as:

$$q_e = K_F C_e^{\frac{1}{n}} \quad (5)$$

where K_F is Freundlich constant which is related to the adsorption capacity, $1/n$ is related to the adsorption intensity.

Fig. 5 shows linear fittings of Langmuir isotherm (C_e/q_e vs. C_e) and Fig. 6 shows linear fittings of Freundlich isotherm ($\log q_e$ vs. $\log C_e$) for the adsorption process of Cu^{2+} and Fe^{2+} ions by the fly ash-based geopolymer with adsorption isotherm parameters listed in Table 3. Based on coefficient of correlation (R^2), the Langmuir isotherm model was more appropriate to explain the adsorption of Cu^{2+} and Fe^{2+} ions by the geopolymer. Therefore, the adsorption of Cu^{2+} and Fe^{2+} ions by the geopolymer was the monolayer adsorption. Similar results were also obtained by other researches.^{14, 17-19}

Characteristics of Langmuir isotherm can be expressed by a dimensionless separation factor (R_L) calculated with the following equation:

$$R_L = \frac{1}{1 + K_L C_0} \quad (6)$$

where K_L is Langmuir constant and C_0 is initial concentration.

The adsorption process can be categorized favorable when $0 < R_L < 1$; unfavorable when $R_L > 1$; linear when $R_L = 1$, and irreversible when $R_L = 0$.²¹ The adsorption of Cu^{2+} ion by geopolymer with initial concentration of 10–150 ppm had R_L values of 0.166–0.013 so it can be categorized as favorable adsorption. Likewise, the adsorption of Fe^{2+} ion by geopolymer with initial concentration of 10–150 ppm had R_L values of 0.021–0.001 and can also be categorized as favorable adsorption.

3.3. Characterizations

XRD pattern of geopolymer before and after the adsorption process of Cu^{2+} and Fe^{2+} ions are shown in Fig. 7. Broad diffraction hump in the region of 20–40° (2θ) indicated the presence of amorphous phases. Meanwhile, the sharp peak at around 26° (2θ) came from quartz.⁴ The intensity of the peaks in the geopolymer after the adsorption process did not seem to differ much compared to those in the geopolymer before the adsorption process. This indicated that the adsorption process of Cu^{2+} and Fe^{2+} ions had occurred on the surface of geopolymer.^{22,23}

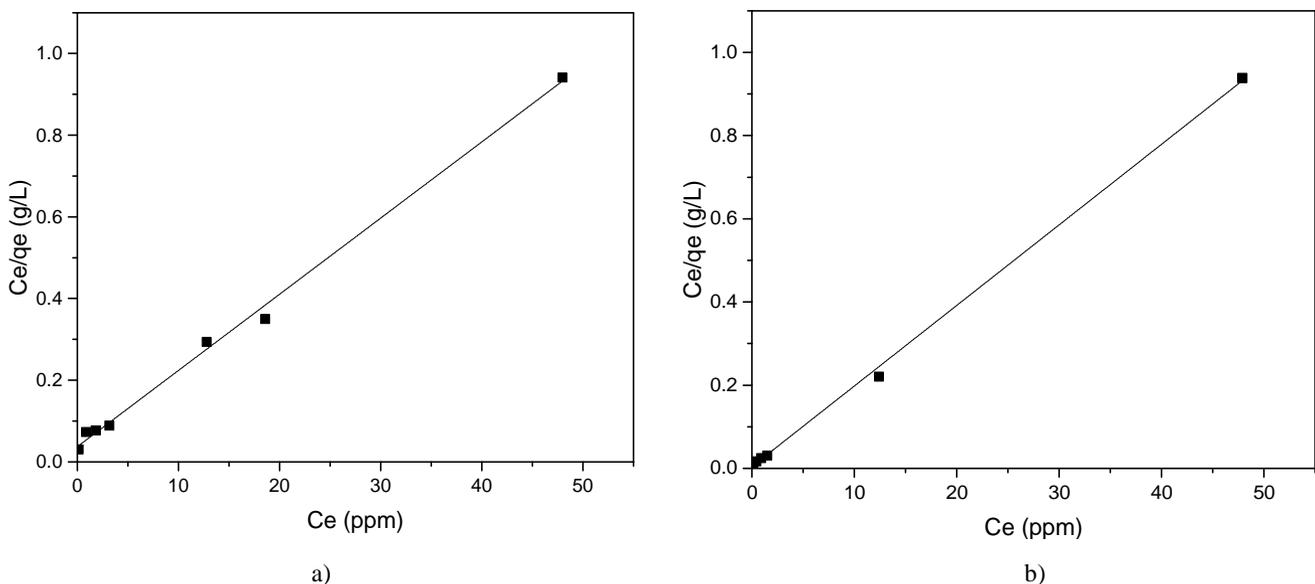


Fig. 5. Linear fitting of Langmuir isotherm for adsorption of Cu^{2+} (a) and Fe^{2+} (b) ions by the fly ash-based geopolymer

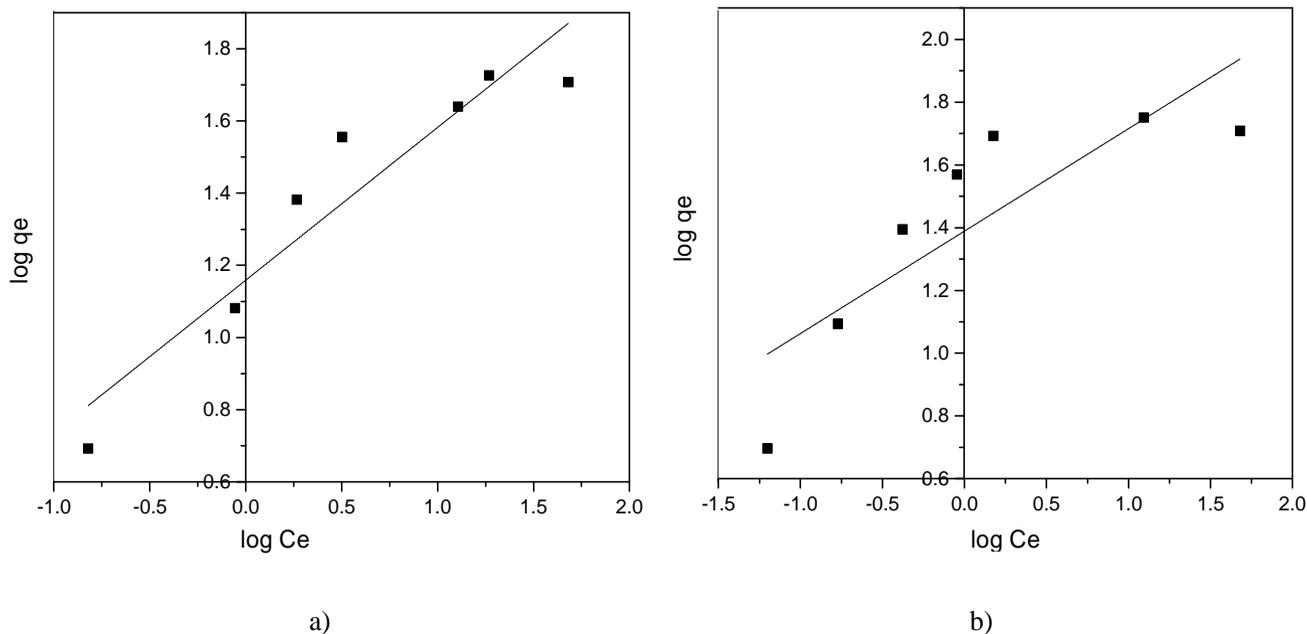


Fig. 6. Linear fitting of Freundlich isotherm for adsorption of Cu²⁺ (a) and Fe²⁺ (b) ions by the fly ash-based geopolymer

Table 3. Adsorption isotherm parameters for adsorption of Cu²⁺ and Fe²⁺ ions by the fly ash-based geopolymer

Metal ion	Langmuir model			Freundlich model		
	$q_m, \text{mg} \cdot \text{g}^{-1}$	$K_L, \text{L} \cdot \text{mg}^{-1}$	R^2	$1/n$	$K_F, \text{mg} \cdot \text{g}^{-1}(\text{L} \cdot \text{mg}^{-1})^{1/n}$	R^2
Cu ²⁺	53.763	0.503	0.996	0.423	14.388	0.897
Fe ²⁺	52.632	4.750	0.998	0.326	24.491	0.717

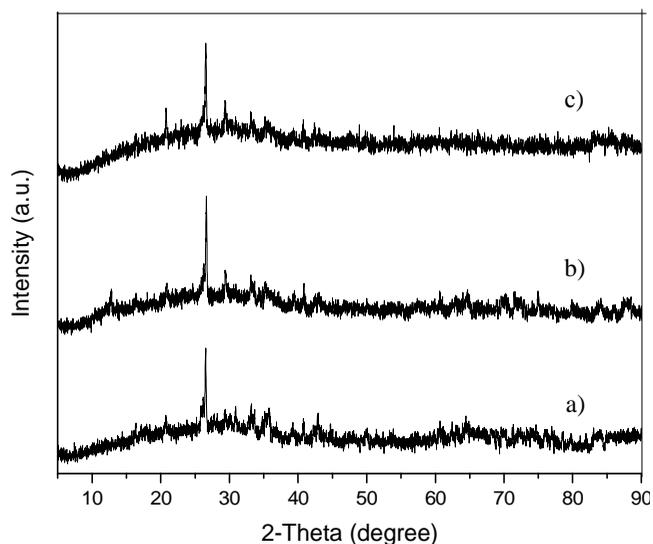


Fig. 7. XRD pattern of geopolymer before the adsorption process (a) and the geopolymer after the adsorption process of Cu²⁺ (b) and Fe²⁺ (c) ions

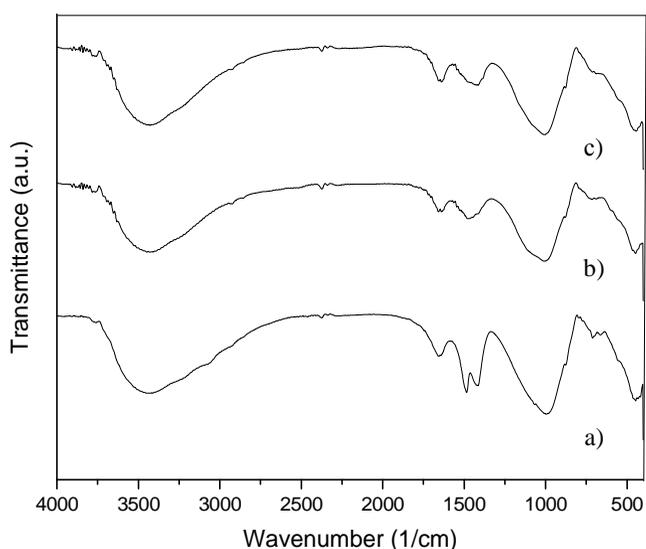


Fig. 8. FTIR spectra of geopolymer before the adsorption process (a) and the geopolymer after the adsorption process of Cu²⁺ (b) and Fe²⁺ (c) ions

Fig. 8 shows FTIR spectra of geopolymer before and after the adsorption process of Cu^{2+} and Fe^{2+} ions. Geopolymer before and after the adsorption process contained the same functional groups, namely Si–O–Si and O–Si–O (*bending vibration*) at the wavenumber of about 470 cm^{-1} , Si–O–Si (*symmetrical stretching vibration*) at about 795 cm^{-1} , Si(Al)–O (*asymmetrical vibration*) at about 1008 cm^{-1} , O–C–O (*stretching vibration*) at about 1410 cm^{-1} , HOH (*bending vibration*) at about 1650 cm^{-1} , –OH and HOH (*stretching vibration*) at about 3400 cm^{-1} .²⁴⁻²⁶ The presence of O–C–O group showed that the presence of carbonate that can be formed from the atmospheric CO_2 , while –OH and HOH groups showed the presence of adsorbed atmospheric water.²⁷

The peaks of functional group in the geopolymer after the adsorption process did not appear to change much compared to those in the geopolymer before the adsorption process. The apparent change in intensity came from O–C–O group in which the peak intensity in the geopolymer after adsorption process appeared to be smaller than that in the geopolymer before the adsorption process. This was due to the adsorption process that took place in the acidic solution (pH 5) that can dissolve the formed carbonate.²⁸

4. Conclusions

Fly ash-based geopolymer had been prepared and utilized as the adsorbent for Cu^{2+} and Fe^{2+} ions from the aqueous solutions. The kinetics studies showed that the adsorption of Cu^{2+} and Fe^{2+} ions followed the pseudo-second order reaction or controlled by the chemical adsorption process with the coefficient of correlation $R^2 > 0.99$. Furthermore, the adsorption isotherm of Cu^{2+} and Fe^{2+} ions followed Langmuir model or monolayer adsorption with each $R^2 > 0.99$ based on adsorption isotherm studies. Maximum adsorption capacity of fly ash-based geopolymer on Cu^{2+} ion was slightly higher than that on Fe^{2+} ion, namely 53.76 mg/g for Cu^{2+} ion and 52.63 mg/g for Fe^{2+} ion.

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КІНЕТИКА АДСОРБЦІЇ ТА ІЗОТЕРМИ ЙОНІВ Cu(II) І Fe(II) З ВОДНИХ РОЗЧИНІВ З ВИКОРИСТАННЯМ ГЕОПОЛІМЕРУ НА ОСНОВІ ЗОЛИ ВІНОСУ

Анотація. Експериментально досліджено адсорбцію йонів Cu^{2+} та Fe^{2+} , звичайних важких металів, що знаходяться в промислових стічних водах, геополімером на основі золи виносу. Встановлено, що адсорбція кожного йона відбувається за реакцією псевдодругого порядку. Доведено, що ізотерма адсорбції йонів Cu^{2+} та Fe^{2+} відповідає моделі Ленгмюра. Визначено, що моношарова адсорбційна здатність становила приблизно 53,76 мг/г та 52,63 мг/г для йонів Cu^{2+} та Fe^{2+} , відповідно.

Ключові слова: адсорбція, йон Cu(II), йон Fe(II), геополімер на основі золи виносу, ізотерма Ленгмюра, реакція псевдодругого порядку.