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ADSORPTION OF CADMIUM (Cd) METAL IN SOLUTION USING POFA-BASED ZEOLITE (*PALM OIL FLY ASH*)

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Abstract: Cadmium is one of the heavy metals with high toxicity that can harm human health and the environment. A commonly used method to remove heavy metals is adsorption. One of the widely used adsorbents is zeolite, which is composed primarily of silica and alumina. Palm oil fly ash (POFA) is a waste from palm oil mills that is rich in silica and alumina so that it can be used as a basic material for zeolite. This research aims to study the factors that affect the adsorption process of Cd metal using zeolite synthesized from POFA and determine the kinetics and isotherms of adsorption. The factors observed were pH (3, 4, 5, 6; 7), adsorbent dosage (1, 1.5, 2, 2.5, 3 g/l), contact time (5, 10, 15, 30, 60, 90, 120, 150, 180 min), and initial concentration of Cd metal (2, 4, 6, 8, 10, 15 mg/L). Adsorbent characterization was performed by XRF and XRD analysis. The results showed that pofa-based zeolite can remove Cd metal with an efficiency above 95%. The adsorption process can follow well the pseudo-second-order kinetics model and both Langmuir and Freundlich isotherm models with a maximum adsorption capacity of 64.58 mg/g. The zeolite synthesized from POFA can remove Cd metal well and has the potential to be developed into an adsorbent.

Keywords: adsorption; cadmium; isoterm; kinetic; POFA; zeolite

Abstrak: Logam kadmium merupakan salah satu logam berat yang mempunyai toksisitas tinggi yang dapat membahayakan kesehatan manusia dan lingkungan. Metode yang umum digunakan untuk menyisihkan logam berat adalah adsorpsi. Salah satu adsorben yang banyak digunakan adalah zeolit yang terdiri dari unsur utama silika dan alumina. Palm oil fly ash (POFA) merupakan limbah dari pabrik kelapa sawit yang kaya dengan silika dan alumina sehingga bisa digunakan sebagai bahan dasar pembuatan zeolite. Penelitian ini bertujuan untuk mempelajari faktor-faktor yang mempengaruhi proses adsorpsi logam Cd menggunakan zeolite yang disintesis dari POFA dan menentukan kinetika serta isotherm adsorpsinya. Faktor-faktor yang diamati adalah pH (3, 4, 5, 6; 7), dosis adsorben (1, 1.5, 2, 2.5, 3 g/l), waktu kontak (5, 10, 15, 30, 60, 90, 120, 150, 180 menit), dan konsentrasi awal logam Cd (2, 4, 6, 8, 10, 15 mg/L). Karakterisasi adsorben dilakukan dengan analisis XRF dan XRD. Hasil penelitian menunjukkan zeolite berbahan dasar pofa dapat menyisihkan logam Cd dengan efisiensi di atas 95%. Proses adsorpsi dapat mengikuti dengan baik model kinetika pseudo-second order dan kedua model isoterm Langmuir dan Freundlich dengan kapasitas adsorpsi maksimum 64,58 mg/g. Zeolit yang disintesis dari POFA dapat menyisihkan logam Cd dengan baik dan berpotensi untuk dikembangkan menjadi adsorben.

Keywords: adsorpsi; kadmium; isoterm; kinetika; POFA; zeolite

INTRODUCTION

Industrial development is currently experiencing rapid growth, leading to the generation of wastes, such as heavy metal, which pose a significant threat to the environment. These wastes have the potential to cause human health risks and ecological disturbances. In addition, the primary concern with heavy metal lies in the non-degradability by living organisms and accumulation in the environment. Several studies have reported the presence at the bottom of waterbodies, forming complex compounds with organic and inorganic materials (Adhani and Husaini, 2017). Various sectors have been recognized to contribute to the release of heavy metal ions, including the batik industry (Deliza et al., 2021), electroplating (Kusuma et al., 2015), textiles (Komarawidjaja, 2017), mining (Sutrisno and Kuntyasti, 2017), and agriculture (Rofida et al., 2018).

According to previous reports, cadmium (Cd) is a commonly produced heavy metal with high toxicity. Cd poses severe risks to various physiological systems, including respiration, blood circulation, heart, reproduction, nerves, bone fragility, kidney health, and reduced Pulmo function (Sasongko et al., 2017). Based on the Regulation of the Indonesia Environment Minister Number 5 of 2014 concerning Wastewater Quality Standards, Cd content allowed in several businesses or industrial activities is 0.05mg/L to 0.1mg/L. To address the risks posed by the metal, various methods, such as adsorption, have been developed to facilitate the removal.

Adsorption has been widely reported to be suitable for wastewater treatment, offering several advantages, including simplicity, effectiveness, and economic nature. In addition, it often occurs when molecules adhere to the surface of adsorbent by exhibiting sorption capacity on the surface (Silvia, 2018). The selection of the appropriate material plays an essential role in achieving high processing efficiency (Mayangsari and Astuti, 2021). In this context, a commonly used adsorbent is zeolite, a porous crystalline mineral alumina silica tetrahydrate. Several studies have shown that it has a three-dimensional skeleton structure, formed by interconnected tetrahedral $[SiO_4]^{4-}$ and

$[AlO_4]^{5-}$ linked by oxygen atoms. This unique structure enables zeolite to absorb numerous smaller molecules or particles based on the size of the cavities. In the industrial field, it is often used as an ion exchanger, catalyst, molecular sieve, and adsorbent (Renni et al., 2018).

In line with these results, zeolite can be synthesized from various silica sources, such as Palm Oil Fly Ash (POFA). The material is typically derived from palm oil solid waste, including coir, shells, and empty bunches, which are burned at a temperature of approximately 800°-1,000°C in steam power plants (Arhamny, 2020). Despite the abundance, the use of POFA remains minimal and poorly managed, leading to a continuous increase in volume and necessitating extensive land disposal. The consistent increase in volume poses challenges to environmental sustainability (Satriawan et al., 2021), indicating the need to develop various strategies to reduce the amount produced. Despite being a waste, POFA is high in silica and alumina, so it still has the potential to be utilized. Materials containing silica and alumina can be used as basic materials to synthesize zeolite. This research is important to utilize POFA as well as an alternative solution to overcome the environmental problems it causes. Therefore, this study purposes to investigate the factors affecting adsorption process of Cd metal, isotherms, and kinetics using POFA-based zeolite as adsorbent.

METHOD

Materials

The tools used in this study included an 80 mesh sieve, magnetic stirrer, acidity level (pH) meter, closed polypropylene bottle, oven, shaker, and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Meanwhile, the materials comprised HCl (Merck) 2M, NaOH (Merck) 1M, KOH (Merck), Al_2O_3, and POFA obtained from PT Perkebunan Nusantara V (PTPN V) Sei. Galuh, Kampar Regency, Riau, and $ZnSO_4.7H_2O$ (Merck).

POFA Preparation

POFA waste that passed the 80 mesh sieve was weighed to 60 g, and 200 mL of 2M HCl was added, followed by stirring for 2 hours. Subsequently, POFA was filtered and rinsed using distilled water until pH of the filtrate was neutral. The solids were then dried in an oven at 105°C for 8 hours.

Zeolite Synthesis

Ingredients, such as KOH (9 g), SiO_2 from POFA (19 g), and Al_2O_3 (13

g) were each dissolved in 45 mL of H_2O (Zahro, 2014). The solution was then mixed, stirred for 30 minutes, and allowed to stand at room temperature for 1 hour. In addition, it was transferred into a sealed polypropylene bottle and crystallized in an oven at 80°C for 24 hours. The formed zeolite crystals were filtered, and the solids obtained were washed with distilled water until pH of the filtrate was neutral. Subsequently, zeolite solids were dried in an oven at 120°C for 12 hours.

Adsorption Process

Determination of Optimum Adsorption pH

In the pH determination procedure, 2 g/L zeolite was added to 100mL of Cd metal solution with a concentration of 10 mg/L. The pH of the solution was adjusted to values of 3, 4, 5, 6, and 7 using 1M HCl solution and 1M NaOH solution. The mixtures were stirred using a shaker at 120 rpm for 60 minutes. After stirring, the solution was filtered to obtain the filtrate, which was then analyzed using ICP-OES device, and all treatments were performed in duplicate.

Determination of Optimum Adsorption Dosage

Zeolite was weighed at 1, 1.5, 2, 2.5, and 3 g/L, and each was added to 100 mL of Cd metal solution with a concentration of 10 mg/L. The mixtures were stirred at 120 rpm for 60 minutes at the optimum pH. The solutions were then filtered to collect the filtrate, which was subsequently analyzed using ICP-OES, and all treatments were performed in duplicate.

Determination of Optimum Adsorption Contact Time

The optimum zeolite adsorbent dosage, determined from a previous study, was added to 100 mL Cd metal solution with a concentration of 10 mg/L. The mixture was stirred using a shaker at a speed of 120 rpm and the optimum pH. Contact time variations of 5, 10, 15, 30, 60, 90, 120, 150, and 180 minutes were utilized. Subsequently, the solution was filtered, and the filtrate was measured using ICP-OES, with all treatments being conducted in duplicate.

Determination of Initial Concentration of Adsorption Solution

The optimum zeolite adsorbent dosage obtained was added to 100 mL Cd metal solution with concentration variations of 2, 4, 6, 8, 10, and 15 mg/L. The mixture was stirred at 120 rpm and optimum pH during the contact time. The solution was then filtered, and the filtrate was measured using ICP-OES, with all treatments being performed in duplicate.

The removal efficiency of Cd metal was calculated using the formula below:

$$Efficiency\,(\%) = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

Where C_i is the initial Cd^{2+} concentration (mg/L) and C_e is the final Cd^{2+} concentration (mg/L). Adsorption capacity could be calculated using the f equation below:

$$q_t = \frac{(C_0 - C_e)}{m} \times V \quad (2)$$

Where q_t is adsorption capacity at time t (mg/g), C_o is the initial concentration of the solution (mg/l), C_e is the final concentration of a solution (mg/l), V is sample volume (L), and m is adsorbent mass (g).

Adsorption Isotherm

Langmuir Isotherm

$$q_e = \frac{q_m K_L C_e}{(1 + K_L C_e)} \pi r^2 \quad (3)$$

Freundlich Isotherm

$$q_e = K_F C_e^{1/n} \quad (4)$$

Where q_e, C_e, q_m, K_L, K_F, and n is adsorption capacity at equilibrium (mg/g), adsorbate concentration at equilibrium (mg/L), maximum adsorption capacity (mg/g), Langmuir constant, Freundlich constant (mg/g (L/mg)1/n), and adsorption intensity respectively.

Kinetics Model

Pseudo-first order kinetic model

$$q_t = q_e(1 - e^{k_1.t}) \quad (5)$$

Pseudo-second order kinetic model

$$q_t = \frac{q_e^2.k_2.t}{q_e.k_2.t + 1} \quad (6)$$

Where q_e is adsorption capacity at equilibrium state (mg/g), q_t is adsorption capacity at time t (minutes), k_1 is adsorption rate constant for pseudo-first-order kinetics model (min^{-1}), and k_2 is adsorption rate constant for pseudo-second-order kinetics model (g/mg min).

RESULT AND DISCUSSION

Zeolite Characterization Using XRF and XRD

The synthesized zeolite was characterized using XRF to determine the elemental composition. The composition of POFA and zeolite is presented in Table 1.

Table 1. Data hasil XRF POFA dan Zeolit

Component	(%)	
	POFA	zeolit
Al_2O_3	0,50	48,90
SiO_2	39,95	25,72
P_2O_5	8,70	4,88
K_2O	10,44	8,06
CaO	31,41	7,45
Fe_2O_3	3,22	3,77

Based on the results of XRF analysis, alumina and silica compounds were identified as the primary constituents of the composite. Zeolite also contained impurity metals, such as Ca and Fe but at lower levels compared to those in POFA. These metals were dissolved during the activation process of POFA, leading to lower concentrations in the final product. Zeolite was further characterized using X-ray diffraction (XRD) techniques. XRD analysis aimed to identify the crystal phase and determine the types of minerals present in the sample, as shown in Figure 1.

Based on the results of diffractogram in Figure 1, peak points occurred at 2θ= 20.81°, 26.58°, 42.40°, and 50.16°. The diffractogram was compared with JCPDS (Joint Committee Powder on Diffraction Standards) database, indicating the formation of a mixed zeolite, which was characterized by the presence of both zeolite X and Y peaks. In general, the synthesis from natural materials tended to produce mixed zeolite due to the effect of impurity metal contained in the silica source (Bahri, 2015).

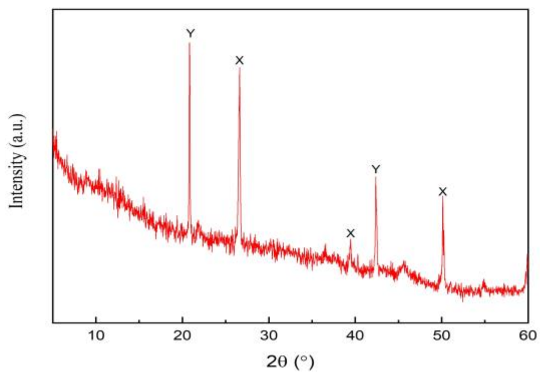

Figure 1. XRD spectra of zeolite

Effect of pH on Adsorption Efficiency

pH was a crucial factor influencing adsorption process of Cd metal. Variations in the solution's acidity could alter the types of ions present and the charge on adsorbent surface. The effect of pH on Cd adsorption efficiency is presented in Figure 2.

The optimum Cd metal removal efficiency occurred at pH 6, reaching 99.23%, as shown in Figure 2. The absorption efficiency increased significantly from pH 3 to 6 and decreased at pH 7. At a low level (pH 3), proton competition with metal ions occurred. Adsorbent surface was surrounded by H^+ ions, causing repulsion between adsorbent surface and metal ions, leading to a low adsorption efficiency (Pratomo et al., 2017). At pH 6, the removal of Cd metal occurred very high due to the number of H^+ ions beginning to decrease, causing competition with H^+, which was also reduced.

Figure 2. Effect of pH on Cd adsorption (dose 2 g/l, contact time 60 min, initial concentration 10 mg/l)

Meanwhile, at a level of 7, the removal of Cd metal decreased, where the higher pH of the solution caused more OH^- ions. The results showed that OH^- ions in the solution tended to bind with Cd^{2+} to form $Cd(OH)_2$ precipitate. The metal ions were absorbed less and were reduced for the ionic form. Consequently, it was more difficult to bind to the alumina group on zeolite. In acidic conditions, metal ions existed as free cations but in neutral to alkaline conditions, cations hydrolyzed to form their hydroxide, with the majority being insoluble (Lestari et al., 2020). The reaction of Cd metal precipitate formation is presented below:

$$Cd + 2OH^- \rightarrow Cd(OH)_2$$

Effect of Dosage on Adsorption Efficiency

Adsorbent dosage was another variable that significantly affected adsorption process, as it could determine adsorption capacity at the initial concentration of adsorbate.

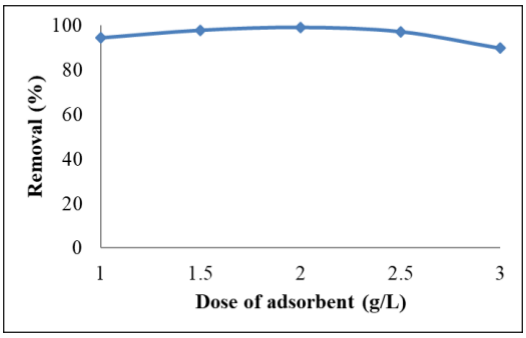

Figure 3. Effect of dose of adsorbent on Cd adsorption (pH 6, contact time 60 min, initial concentration 10 mg/l)

The effect of adsorbent dosage on removal efficiency is presented in Figure 3. Based on Figure 3, the optimum dosage for cadmium metal removal efficiency was 2 g/L, achieving an adsorption capacity of 5.13 mg/g. The high percentage of adsorption with increasing adsorbent dosage could be attributed to the increased surface area and availability of active sites for adsorbate binding during the process (Siringo-Ringo, 2019). However, at dosages of 2.5 and 3 g/L, there was a decrease in removal efficiency due to particulate interactions, leading to the formation of clumps, which could reduce the surface area. This reduction decreased the ability to adsorb, leading to decreased adsorption efficiency (Khodaie et al., 2013).

Effect of Contact Time on Adsorption Efficiency

Contact time referred to the duration required for adsorbent to adsorb adsorbate optimally, determining the kinetics of adsorption process. The effect of contact time on adsorption efficiency is presented in Figure 4. As shown in Figure 4, the percentage of metal removal increased with increasing contact time until it reached a constant level. The amount of Cd adsorbed reached the optimum point at 60 minutes, achieving 99.09% removal. According to Lestari et al. (2020), extending the contact time did not significantly affect the removal of Cd^{2+} by adsorbent. The optimum time was achieved when adsorbed Cd^{2+} levels reached their maximum, hence, the additional contact time did not significantly affect reduction from the solution.

Effect of Initial Concentration on Adsorption Efficiency

The results of determining the effect of initial concentration on adsorption efficiency are presented in Figure 5. As shown in Figure 5, at a low initial concentration of 2 mg/L, the highest efficiency was 99.06% with a removal capacity of 0.70mg/g. However, this efficiency decreased as Cd concentration increased up to 15mg/L. This was consistent with a previous study by Lestari et al. (2020), that with increasing concentrations of metal ions, the removal efficiency decreased because adsorbent capacity to remove metal ions was maximum. The decrease in removal efficiency occurred because the number of metal ions in the solution was not proportional to the number of adsorbent particles available, leading adsorbent to reach the saturation point and reducing the removal efficiency.

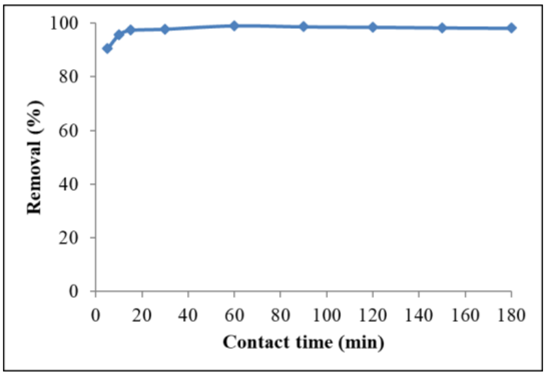

Figure 4. Effect contact time on Cd adsorption (pH 6, dose 2 g/l, initial concentration 10 mg/l)

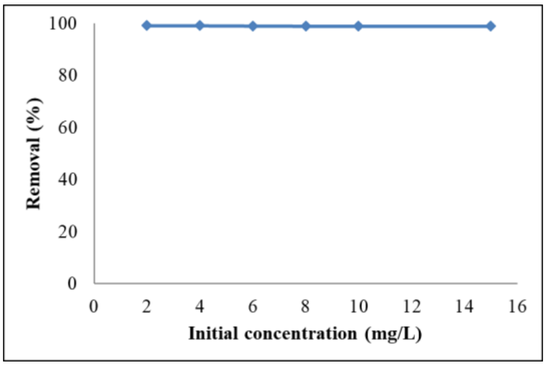

Figure 5. Effect initial concentration on Cd adsorption (pH 6, dose 2 g/l, contact time 120 min)

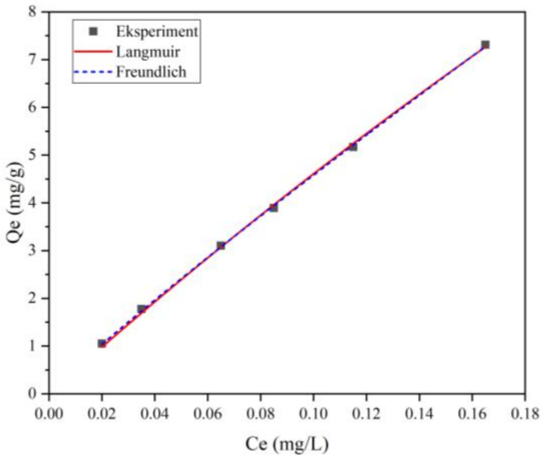

Figure 6. Adsorption isotherm of Cd

Table 2. Values of isotherm constant for Cd

Isotherm	Parameters	
Langmuir	Q_m(mg/g)	64,58
	K_L(L/mg)	0,7682
	R^2	0,99901
Freundlich	n	1,11
	K_F(mg/g (L/mg)$^{1/n}$)	38,46
	R^2	0,99968

Adsorption Isotherms

Adsorption isotherm type was determined by plotting the equation curve of Langmuir and Freundlich isotherm, as shown in Figure 6. The constants of each model were derived from the above graphs and were listed in Table 2.

The results showed that the correlation coefficient for both Langmuir and Freundlich isotherms was 0.99. This indicated that both isotherms effectively described adsorption of Cd, suggesting the presence of both physisorption and chemisorption in the absorption process. This occurred because this type of adsorption could bind adsorbed ions with chemical bonds, but the bonds were easily released to facilitate ion exchange (Masruhin et al., 2018).

According to Amran and Kartika (2021), RL (adsorption dimension) value indicated the suitability of Langmuir isotherm pattern for adsorption process. The study revealed that RL value at different initial concentrations of Cd^{2+} was in the range of 0 < RL < 1, namely between 0.08 - 0.38. This result demonstrated that POFA-based zeolite was effective for Cd^{2+} adsorption under the experimental conditions analyzed.

Adsorption Kinetics

Adsorption kinetics model helped to determine the rate at which fluid was adsorbed by adsorbent over a specified period, as shown in the curve in Figure 7. In addition, the constant values for each model are presented in Table 3.

Based on the calculation of adsorption kinetics parameters in Table 3, the pseudo-second-order equation best described adsorption kinetics of Cd metal. Adsorption process that followed the pseudo-second-order kinetics model indicated that absorption rate of zeolite adsorbent to cadmium metal per unit time was directly proportional to adsorbent capacity (Hasan et al., 2021).

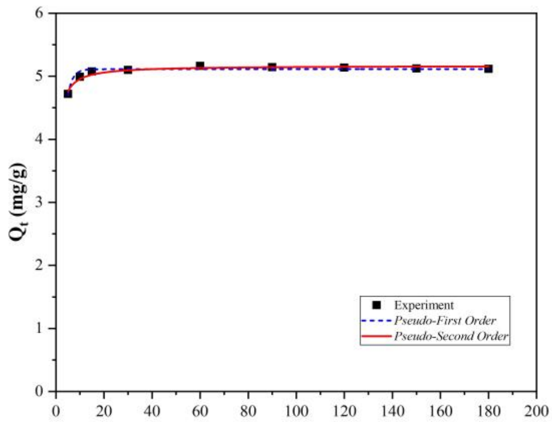

Figure 7. Kinetic Equation Fitting Curve

Table 3. Parameters for kinetic models for Cd

Kinetics model	Parameter	
	Q_{eks} (mg/g)	5,14
Pseudo-first order	Q_e (mg/g)	5,11
	K_1 (min^{-1})	0,51
	R^2	0,91
Pseudo-second order	Q_e (mg/g)	5,16
	K_2 (g/mg.min)	0,45
	R^2	0,94

CONCLUSION

The optimum conditions of adsorption were obtained at pH 6, 2 g/L adsorbent dosage, and 120 minutes of contact time with an efficiency above 95%. The adsorption isotherm model followed well Langmuir and Freundlich isotherms with a Qm value of 64.58 mg/g and a KL value of 0.77 L/mg. Meanwhile, in Freundlich isotherm, KF value was 38.46 (mg/g (L/mg)$^{1/1.11}$). The adsorption kinetics model in this study followed well the pseudo-second order with Q_e = 5.167 mg/g and a K_2 value of 0.449 (g/mg.min). The results showed that POFA-based zeolite has the potential to be developed as an adsorbent.

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