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Innovative Use of Palm Oil Fly Ash-Based Zeolite for Zinc (II) Removal from Wastewater

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ABSTRAK

Zinc (Zn) adalah logam berat yang sering ditemukan dalam limbah cair, konsentrasi ion Zn (II) di atas ambang batas dapat menyebabkan masalah kesehatan dan polusi lingkungan. Meskipun zinc penting, kelebihan zinc dapat menyebabkan keracunan. Oleh karena itu, ion Zn (II) harus disisihkan sebelum air dikembalikan ke lingkungan. Metode adsorpsi efektif dalam mengendalikan polusi logam berat, dengan zeolit berbahan dasar POFA (abu terbang minyak kelapa sawit) sebagai adsorben. Penelitian ini bertujuan untuk menganalisis pengaruh pH, dosis adsorben, waktu kontak, dan konsentrasi awal terhadap kemampuan zeolit berbasis POFA dalam menyisihkan ion Zn(II). Percobaan dilakukan dengan memvariasikan 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 ion Zn (II) (2, 4, 6, 8, 10, 15 mg/L). Hasil menunjukkan efisiensi penyisihan ion Zn(II) tertinggi pada pH 6, dosis adsorben 2 g/L, dan waktu kontak 60 menit, dengan tingkat penyisihan 98,77%. Proses adsorpsi ion Zn(II) sesuai dengan isoterm Langmuir dan Freundlich, dengan kapasitas adsorpsi maksimum 28,27 mg/g. Kinetika adsorpsi mengikuti model pseudo orde dua dengan qe 5,04 mg/g dan konstanta laju adsorpsi 0,197 g/mg.min.

Kata Kunci: Adsorpsi; Kinetika; POFA, Zeolit; Zinc

ABSTRACT

Zinc (Zn) is a heavy metal commonly found in wastewater, with Zn (II) ions levels above the threshold causing health problems and environmental pollution. Though zinc is essential, excess zinc can lead to poisoning. Thus, Zn (II) ions must be removed before releasing water into the environment. The adsorption method is effective for controlling heavy metal pollution, with zeolite based on POFA (palm oil fly ash) as a useful adsorbent. This research aims to analyze the influence of pH, adsorbent dose, contact time, and initial concentration on POFA-based zeolite's ability to remove Zn (II) ions. Experiments were conducted by varying 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 minutes), and initial Zn (II) ions concentration (2, 4, 6, 8, 10, 15 mg/L). Results showed the highest Zn removal efficiency at pH 6, an adsorbent dose of 2 g/L, and a contact time of 60 minutes, achieving 98.77% removal. The Zn (II) ions adsorption process fits the Langmuir and Freundlich isotherms, with a maximum adsorption capacity of 28.27 mg/g. The adsorption kinetics followed a pseudo-second-order model with qe 5.04 mg/g and an adsorption rate constant of 0.197 g/mg.min.

Keywords: Adsorption, Kinetics, POFA, Zeolite, Zinc

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1. INTRODUCTION

Industrial development not only has a positive impact on human life, but it also causes negative impacts in the form of waste. Waste that is not managed properly, especially those containing toxic substances such as heavy metals will pollute the environment and affect human health (Rizky, 2015). Waste containing heavy metals usually comes from the batik industry (Handayani et al., 2019), textiles (Panigrahi & Santhoskumar, 2020), electroplating (VANIA, 2016), mining (Ifa et al., 2018), ceramics (Priadi et al., 2014), batteries, wood, and paint (Suciandica et al., 2019).

Heavy metals are a type of pollutant that is very dangerous in the environmental system because they are non-biodegradable, toxic, and capable of bioaccumulation in the food chain. Heavy metal ions have atomic numbers 22-92 and are located in periods III and IV in the periodic system of chemical elements. One example of a heavy metal ion is Zn (II). Based on Regulation Number 5 of 2014 of the Minister of Environment of the Republic of Indonesia concerning Quality Standards for Industrial Wastewater, it is stated that the permitted Zn content in several industries is 1 mg/l.

Several techniques that can be used to overcome heavy metal pollution are precipitation, oxidation, reduction, solvent extraction, electrolysis extraction, evaporation, osmosis, ion exchange, and adsorption (Darmayanti et al., 2019). The adsorption process is more widely used because it is relatively simple, can work at low concentrations, the adsorbent can be recycled, and the costs required are relatively cheap (Siregar, 2021). Adsorption is the process of adsorbing a substance (adsorbate) on the surface of another substance (adsorbent) due to the molecular attraction that occurs between the adsorbate and the adsorbent (Ngapa & Gago, 2019). In the adsorption process, the selection of adsorbent is an important factor in producing high processing efficiency (Mayangsari & Astuti, 2021). One of the adsorbents that is widely used is zeolite. Zeolite is a multi-functional material widely used because it has dehydration properties, high cation exchangeability, is a good catalyst, and can adsorb other compounds (Pratomo et al., 2017).

Zeolite is a hydrated aluminosilicate mineral that has a unique structure, namely the presence of alumina group (AlO_4) and silica groups (SiO_4), which are interconnected by oxygen atoms, forming a three-dimensional framework (Fatha, 2021). Zeolite consists of two types, namely natural zeolite and synthetic zeolite. Synthetic zeolite is a zeolite that is engineered to resemble natural zeolite by containing alumina and silica. One alternative that can be used as a source of silica is fly ash palm oil (POFA). POFA is the result of burning palm oil waste at a temperature of around 800–1,000°C, which is burned in a steam power plant at a palm oil mill (Telaumbanua, 2017). As the palm oil industries expands, the amount of POFA will increase. POFA is not widely used or adequately managed, despite the fact that part of it has been used as fertilizer for palm oil plants. POFA that is not utilized correctly can harm the soil and contaminate the ecosystem. POFA has a high silica content, namely around 50-70% (Hamada et al., 2018), so it has the potential to be utilized. One use of silica content is as a basic material for making zeolites (Prihastuti et al., 2021). POFA zeolite is considered efficient because it reuses palm oil waste. Although POFA contains high levels of silica and has little potential to be synthesized into zeolite, research that examines it. Based on this, this research aims to utilize synthetic zeolite based on POFA as an adsorbent to reduce the Zn (II) ion content in wastewater by studying the factors that influence adsorption such as pH, dose, time, and initial concentration.

2. METHODOLOGY

2.1. Materials and Tools

POFA is used as raw materials taken from PT. Perkebunan Nusantara V (PTPN V) Sei. Galuh, Kampar Regency, Riau, $ZnSO_4.7H_2O$ (Merck), 2 M HCl, 1 M NaOH, KOH, and Al_2O_3. The tools used in this research were 80-mesh sieves, a magnetic stirrer, a pH meter, bottles of polypropylene, an oven, shakers, and inductively coupled plasma optical emission spectroscopy (ICP-OES Agilent 5100X).

2.2. Materials and Tools

POFA was sieved with an 80-mesh sieve. A total of 60 grams of POFA that passed through the sieve was added to 200 ml of 2M HCl and stirred for 2 hours, then filtered and rinsed with distilled water until the filtrate pH was neutral. The solid was dried in an oven at 105°C for 8 hours. The molar composition used to synthesize is $10Na_2O$: Al_2O_3: $15SiO_2$: $300H_2O$. Each ingredient (KOH, POFA, and Al_2O_3) dissolved in H_2O is then mixed. The solution was then stirred for 30 minutes until homogeneous. After that, the solution was left at room temperature for 1 hour. The solution is then transferred into a bottle of polypropylene and crystallized in an oven for 24 hours at a temperature of 80°C. The zeolite formed is filtered and then washed with distilled water until the filtrate pH is neutral. Next, the zeolite solid was dried in an oven at a temperature of 120°C for 12 hours.

2.3. Adsorption Experiments

Adsorption experiments were carried out using POFA-based zeolites at different conditions, such as pH (3, 4, 5, 6, and 7), adsorbent dosage (1, 1.5, 2, 2.5, and 3 g/L), contact time (5, 10, 15, 30, 60, 90, 120, 150, and 180 minutes), and initial concentrations (2, 4, 6, 8, 10, and 15 mg/l). A total of 0.2 grams of adsorbent was added to 100 ml of $ZnSO_4$ with a concentration of 10 mg/L with a different pH, then stirred with shakers at a speed of 120 rpm for 60 minutes, then filtered to separate the adsorbent from the liquid. The filtrate obtained was analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES). After obtaining the optimum pH, the same experiment was carried out for varying adsorbent dosages, contact times, and initial concentrations. To calculate adsorption efficiency, the following formula is used:

$$\% \text{ Removal} = \frac{C_0 - C_e}{C_o} \text{ X } 100\%$$

with Co and Ce indicating the initial and final concentrations of Zn (II) ion.

3. RESULTS AND DISCUSSION

3.1. Zeolite Characteristics Using XRF and XRD

The synthesized zeolite was characterized using X-ray fluorescence (XRF) to determine the composition of the elements in the zeolite. The composition of zeolite can be seen in Table 1.

Table 1. Zeolite XRF Result Data

Elements/Compounds	Elements Content (%)	
	POFA	Zeolite
Al_2O_3	0.50	48.74
SiO_2	39.95	25.61
P_2O_5	8.70	4.86
K_2O	10.44	8.06
CaO	31.41	7.40
Fe_2O_3	3.22	3.75
TiO_2	0.28	0.79

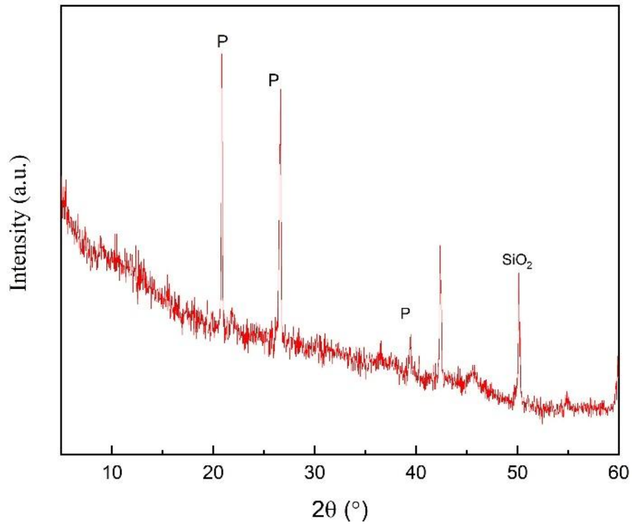

Figure 1. Zeolite XRD Result

Based on the results of the diffractogram in Figure 1, it can be seen that the peak point occurs at 2θ = 20.81°, 26.58°, 39.41°, 42.40°, and 50.16°. Then, the results of the diffractogram are adjusted to the JCPDS standards (Joint Committee Powder on Diffraction Standards), and obtained type P, Gismondine-Type, (JCPDS: 39-0219) zeolite mixed or contaminated with quartz (JCPDS 46-1045), which is characterized by the presence of zeolite peaks.

3.2. Zn Adsorption

3.2.1. Effect of pH on the removal of Zn (II)

The pH value is an important parameter in the absorption of heavy metals by zeolites because it influences the metal charge state and also the adsorbent surface. The effect of pH on the adsorption of Zn can be seen in Figure 2.

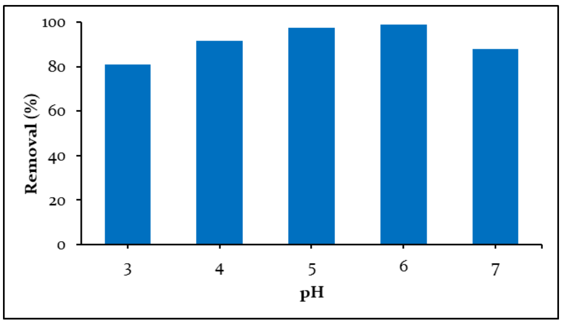

Figure 2. Effect of pH on Zn (II) Adsorption (dose 2 g/l, Contact Time 60 Minutes, Initial Concentration 10 mg/g)

According to Figure 2, the effectiveness of Zn (II) ions removal improved from pH 3 to 6 but dropped at pH 7. The maximum Zn (II) ion removal efficiency occurred at pH 6 of 98.77%, and the adsorption capacity was 4.81 mg/g. According to (Abd El-Azim & Mourad, 2018), in acidic conditions, namely pH 3-5, the absorption of Zn (II) ions is small because in acidic conditions adsorbent was protonated, thereby the surface became positively charged, so there is competition between H+ and Zn (II) ions to interact with the adsorbent surface, resulting in repulsion between Zn (II) ions and the adsorbent surface.

At pH 6, the removal of Zn (II) ion was very large; this was because the number of H^+ ions began to decrease, so that competition with H^+ also decreased, whereas at pH 7 the removal of Zn (II) ions decreased. This is because the higher the pH of the solution, the more OH^- ions there will be in the solution. OH^- ions in solution tend to bind with Zn (II) ions forming $Zn(OH)_2$ precipitate. As a result, Zn (II) ions is less adsorbed, or, in other words, Zn (II) ions is no longer in ionic form, so it becomes more difficult for them to bind to the alumina groups in the zeolite. In general, in acidic conditions, metal ions exist as free cations, but in neutral to basic conditions, the cations will hydrolyze to form their hydroxides, where most metal hydroxides are insoluble (Naat et al., 2020). The reaction for the formation of Zn (II) ion deposits is as follows:

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

3.2.2. Effect of Adsorbent Dosage on Zn (II) Removal

Adsorbent dosage is an important parameter in adsorption studies because it can influence removal efficiency and metal ion adsorption capacity, where the removal efficiency is inversely proportional to the adsorption capacity. The effect of adsorbent dose on the adsorption of Zn (II) ions can be seen in Figure 3.

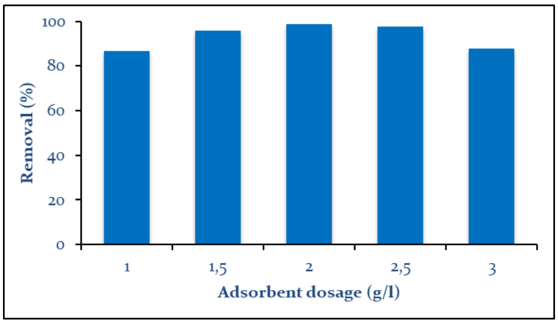

Figure 3. Effect of Adsorbent Dosage on Zn (II) Adsorption (pH, Contact Time 60 Minutes, Initial Concentration 10 mg/g)

Based on Figure 3, the removal efficiency rises with increasing adsorbent dosage. The maximum Zn (II) ions removal efficiency was 98.56% with the addition of an adsorbent dose of 2 g/L. The high percentage of adsorption with increasing adsorbent dosage can be attributed to the surface area and availability of active sites to bind adsorbate in the adsorption process (Siringo-Ringo, 2019). However, when the dose was increased above 2 g/L, the removal efficiency decreased. According to (Agusriyadin, 2020) and (Jasem, 2015), this is because adding large doses can cause particle buildup or aggregation on the adsorbent, which can result in a decrease in the total surface area of the adsorbent, resulting in many active sites being blocked and unable to interact freely with Zn (II) ions. This reduced surface area of the adsorbent causes the adsorbent's ability to adsorb the adsorbate to be reduced, resulting in a decrease in the adsorption capacity of the adsorbent (Khodaie et al., 2013).

3.2.3. Effect of Contact Time on Zn (II) Ion Removal

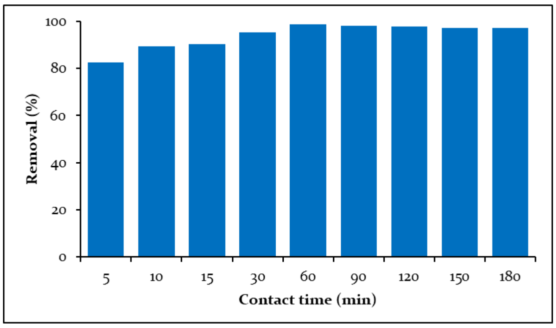

Figure 4. Effect of Contact Time on Zn (II) Adsorption (pH 6, Dose 2 g/l, Initial Concentration 10 mg/l)

According to Figure 4, the highest Zn (II) ions removal effectiveness occurred during a contact time of 60 minutes, or 98.68%. The efficacy of Zn (II) ions removal increased with contact duration from 5 to 60 minutes. In the adsorption process, the ability to remove Zn(II) ions increases because, at the beginning of the adsorption process, the availability of positive sites or active sites and the large space in the pores of the adsorbent cause the Zn(II) mass transfer process from the adsorbate to the adsorbent to run quickly, while For a contact time of 90 to 180 minutes, the ability of POFA-based zeolite to remove Zn (II) ions decreases until it reaches constant. According to (Fatha, 2021), there was a decrease in the removal of adsorbed Zn (II) ion metal because the positive site or active site on it was filled with Zn (II) ions, so the adsorbent saturation caused a desorption process or release of Zn (II) ions from the adsorbent back into the adsorbate.

3.2.4. Effect of Initial Concentration on Zn (II) Ion Removal

The absorption ability of an adsorbent is also influenced by the concentration of the metal ion solution. The effect of initial concentration on Zn (II) ions adsorption can be seen in Figure 5.

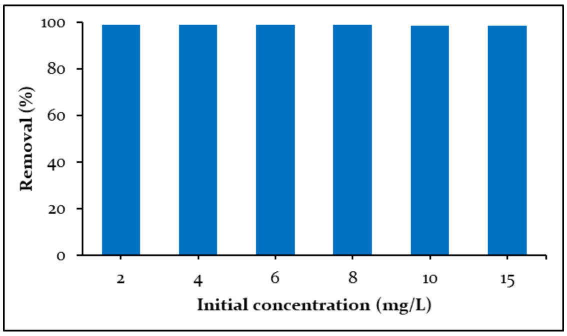

Figure 5. Effect of Initial Concentration on Zn (II) Ions Adsorption (pH 6, Dose 2 g/l, Contact Time 120 Minutes)

Based on Figure 5, the highest Zn removal efficiency was obtained at a concentration of 2 mg/L, namely reaching 98.71%, then experienced an insignificant decrease as the concentration of the Zn (II) ions solution increased. This is by research by (Nyamunda et al., 2019), which states that with increasing metal ion concentrations, the absorption efficiency will decrease. The high removal efficiency at a concentration of 2 mg/L is because, at a low metal ion concentration, most of the metal ions in the solution are adsorbed onto the empty active site of the adsorbent, resulting in a very high adsorption efficiency, whereas when the metal ion concentration increases, the active site becomes saturated, leaving most metal ions in solution. The concentration of metal ions is closely related to the number of active sites on the surface of the adsorbent. If the number of active sites is large enough compared to the number of metal ions, the removal capacity will be high.

3.3. Adsorption Isotherms

In general, the isotherms used to study adsorption mechanisms are the Freundlich and Langmuir isotherms. The purpose of determining the type of isotherm is to determine the adsorption capacity and mechanism (Mayangsari & Astuti, 2021). Determining the type of adsorption isotherm is carried out by creating a Freundlich isotherm and a Langmuir isotherm equation curve, as shown in Figure 6.

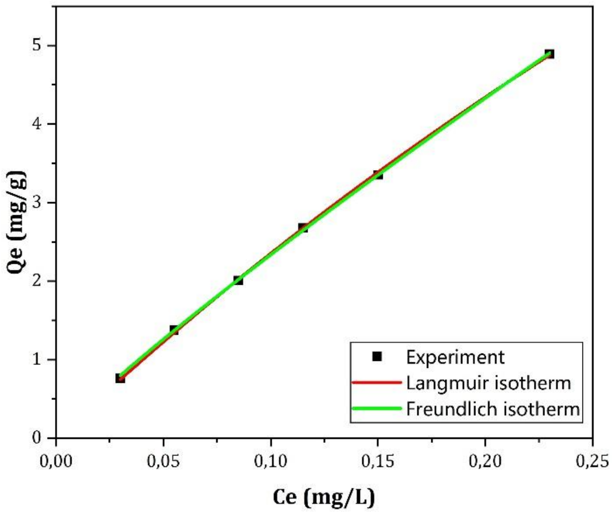

Figure 6. Adsorption isotherm of Zn (II) ions

Based on the graph plot of the Langmuir and Freundlich equations, the constant values for each model are obtained, which are presented in Table 2.

Table 2. Adsorption Isotherm Constants

Isotherm Equation	Constant	
Isotherm Freundlich	n	1,12
	K_F(mg/g) (L/mg)$^{1/n}$)	18,17
	R^2	0,99975
Isotherm Langmuir	Qm (mg/g)	28,27
	K_L(L/mg)	0,92
	R^2	0,99973
	R_L	

According to Table 2, the R^2 value for the Langmuir isotherm is 0.99973, while the Freundlich isotherm is 0.99975. The results of the data analysis above demonstrate that the adsorption of Zn (II) ions with POFA-based zeolite is suitable for utilizing the two isotherm models, indicating that the adsorption process happens in both multilayer and monolayer modes. This shows that the Zn adsorption process occurs via physisorption and chemisorption because adsorption is a complex process involving several mechanisms simultaneously, namely physisorption (weak van der Waals/electrostatic interactions) and chemisorption (through ion exchange or surface complex formation). Ion exchange, as a form of chemisorption with reversible chemical bonds, is often identified as the main mechanism causing the release/recovery of easily released ions (Kalam et al., 2021).

Another parameter that can be used to determine the adsorption process is the value R_L. R_L is Langmuir separation factor associated with adsorption favourability (dimensionless), calculate with equation:

$$R_L = \frac{1}{1 + K_L C_0}$$

If $R_L>1$ adsorption process is unfavorable, $R_L = 1$ linear isothermal adsorption process, $0<R_L<1$, namely between 0.069 and 0.322, which means the adsorption process is very profitable (favorable).

3.4. Adsorption Kinetic

Adsorption kinetics is used to determine the binding rate constant (k), which is a parameter of the speed of the Zn (II) ion adsorption process with wide variations in contact time. The adsorption kinetics model applied to this research data is pseudo-first-order and pseudo-second-order. The kinetic model graph can be seen in Figure 7.

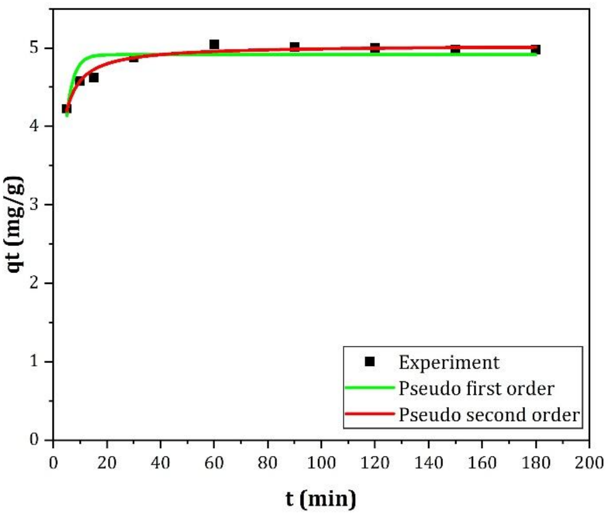

Figure 7. Kinetics of Zn (II) Adsorption

Table 3 shows the constant values produced from each model using the pseudo-first-order and pseudo-second-order equations.

Table 3. Kinetic Parameters for Zn (II) Ions Adsorption

Kinetic Model	Parameter	Mark
Pseudo First Order	Qe exp (mg/g)	4,98
	K1 (min-1)	0,37
	qe (mg/g)	4,92
	R^2	0,7240
Pseudo Second Order	K2 (g/mg.min)	0,20
	qe (mg/g)	5,04
	R^2	0,9640

The adsorption kinetics are determined by the pseudo-second-order equation, as indicated by the R^2 values in Table 3. This is because the 0.9640 second-order R^2 is rather near to one. The substantial R^2 value ensures that a second-order reaction will take place during the binding reaction of Zn (II) ions on the adsorbent. The theoretical values of adsorption equilibrium (qe) obtained from the pseudo-second-order reaction model are 5.04 mg/g. This number is more consistent with the experimental qe estimate of 4.98 mg/g. The second-order nature of the adsorption process confirms that chemical sorption of metal ions has occurred on the POFA-based zeolite (Nejadshafiee & Islami, 2019).

4. CONCLUSION

Zeolite-based materials were successfully synthesized. Based on the results of Zn (II) ions adsorption research using POFA-based zeolite, it was found that the highest Zn removal efficiency occurred at pH 6, an adsorbent dose of 2 g/L, a contact time of 60 minutes, and an initial Zn concentration of 2 mg/L amounting to 98.77%. The adsorption isotherm model that is suitable to describe the mechanism of Zn (II) ions adsorption by POFA-based zeolites follows the isotherm equation, Langmuir and Freundlich, with a maximum adsorption capacity of 28.27 mg/g and a K value 0.92 l/mg.A suitable kinetic model to describe the rate of Zn (II) ions adsorption by POFA-based zeolites follows Eq pseudo-second-order. The research results show that POFA-based zeolite can be used as an adsorbent.

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