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Research Paper

Layered Double Hydroxide Zn/M^{3+} (M^{3+}= Al and Cr) as Highly Efficient Adsorben of Heavy Metal Pb(II)

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Abstract
Synthesis of Zn-Al and Zn-Cr LDHs in this research was successfully carried out. The sucsessfully results can be seen from the XRD characterization which displays diffraction angles at 10 and 60 which indicates the presence of layered structures and anions. FT-IR data displays the presence of nitrate groups at wavenumber 1381 cm^{-1}. BET data shows surface area of Zn-Al and Zn-Cr at 1.965, 31.638 m^2/g, corresponding. pHpzc determination of Zn-Al material gets a pH below pHpzc which is 6.2 while Zn-Cr is at pHpzc which is 7.6. Able of Zn (Al/Cr) as adsorbent to adsorp Pb(II) was investigated through several parameters such as kinetics, isotherms, thermodynamics. Kinetic Adsorption for Zn-Al inclined follow PSO, while Zn-Cr follow PFO. Isotherm parameter for Zn-Al and Zn-Cr is Freundlich with a maximum adsorption capacity 74.127 mg/L, and 27.027 mg/L. Thermodynamic process shows that Zn-Al and Zn-Cr LDHs take place in endothermic and spontaneously.

Keywords
Layered Double Hydroxide, Pb(II), Adsorption

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1. INTRODUCTION

Untreated waste from industry causes environmentral pollution, especially in waters. Waste produced by industry includes both organic and inorganic waste which is difficult to degrade (Chazanah and Nandiyanto, 2022). Lead (Pb) is found in organic and inorganic materials which have a large bulk and atomic mass (Zhu et al., 2021). Organic lead can kill plants, while inorganic lead causes health risk in human such as kidneys, cancer, and blood circulation (Nag and Cummins, 2022). Pb is a metal that consists of 0.002% of the earth's crust which is categorized as the second toxic metal, nonbiodegradable and can be found Pb in the automobile industry, batteries, etc (Raj and Das, 2023).

Various techniques used in waste removal lead include coagulation, filtration (Qasem et al., 2021), electrcoagulation (Moussa et al., 2017), precipitation (Qasem et al., 2021), photodegradation (Wang et al., 2020), electrodialysis (Min et al., 2021), reverse osmosis (Samaei et al., 2020), ion exchange (Kaur and Jindal, 2018), and adsorption. Adsorption was selected as a lead effluent treatment method has the advantages of inexpensive cost and easy implementation (Alsawy et al., 2022). Adsorption occurs due to the interaction between the surface of the adsorbent (solid or liquid) and the adsorbate, which is the adsorption mechanism are classified into two: physical adsorption based on Van der Waals forces and dipole interaction, the other is chemical adsorption due to electrostatic interactions by covalent bonds (Joseph et al., 2019). The mechanism of Pb waste management involves the dispersion of metal ions through the micro sized pores of the adsorbent (El-Baz et al., 2020).

There are many types of adsorbents in the adsorption process i.e zeolite (Bai et al., 2022), activated carbon (Agarwal and Singh, 2017), and the other one is Layered Double Hydroxide (LDH). The strength of LDH include low cost, environmental friendliness, easy for modification such as can be anion exchange and can occur through intercalation (Gomes et al., 2016). Layered Double hydroxide (LDHs) is known as a brucite layer that has a positive charge and made from metal hydroxides with an anion in each layer. The chemical formula of LDH is $[M^{2+}{}_{1-x}M^{3+}{}_{x}(OH)_2]_{x+}(An-)_{x/n}]_nH_2O$; where, composting from bivalent (i.e., Ni^{2+}, Fe^{2+}, Zn^{2+}, Cu^{2+}, and Mg^{2+}), trivalent metal ions (Al^{3+}, Fe^{3+}, and Cr^{3+}), and anion in interlayer (Cl^-, $SO_4{}^{2-}$, NO^{3-}, and CO^{3-}) (Lesbani et al., 2020). Anion in LDH layer can be exchanged with other anions, because it has flexibility and reversible (Tang et al., 2022). LDH modified with organic materials or those with multiple functional groups can increase adsorption capacity to adsorb heavy metals (Liu et al., 2023).

There are a number of research projects that have used LDH as an adsorbent to remove organic or inorganic pollutants Mg-Al/citrate able to adsorp with capacity 298,5 mg/g (Chen et al., 2018). Refers to research conducated by Palapa et al. (2022) for Cu-Al the adsorption ability of Cd metal reached 13,947 mg/g used Langmuir equation. Pb^{2+} removal with Ca-Al LDH can be removed at a high concentration of 6 ppm from an initial concentration of 10 ppm (Rojas, 2014). The work conducted by Asiabi et al. (2017) with modified Ni-Cr/diphenylamine 4-sulfonate (DS) LDH to adsorp Pb with adsorpstion capacity reaching 309 mg/g at 293 K. Research by Zhang et al. (2016) for Cd(II) with Zn-Al/EDTA LDH, the maximum adsorption capacity was 16.1 $\mu g^{1-1/n} \cdot g^{-1} \cdot L^{-1}$ according to the Freundlich equation, indicating that adsorption occurs in a multilayer. Adsorption capacity of Fe(II) metal up to 12.618 mg/g using Zn/Cr LDH with Freundlich equation was observed by Oktriyanti et al. (2020). Qmax Zn-Al/POM for adsorption Pb^{2+} and Ni^{2+} 74.13, 55.56 consecutively research by Silaen et al. (2021).

Based on the above analysis, this study synthesized LDH M^{2+} (Zn) and M^{3+} (Al and Cr). LDH was prepared and characterized by XRD to determine the angle of LDH, FT-IR to determine the functional groups, and BET to determine the surface area of LDH adsorbent. Zn-Al and Zn-Cr LDHs conducts the effect of adsorption by the determining pHpzc to find out the optimum pH. Zn(Al/Cr) LDHs was used as an Pb waste removal adsorbent by regarding adsorption kinetic, isotherm, and thermodynamic.

2. EXPERIMENTAL SECTION

For this research, the materials used are $Zn(NO_3)_2.6H_2O$, $Al(NO_3)_3.9H_2O$, $Cr(NO_3)_3.9H_2O$, $PbCl_2$, NaOH, HCl, Na_2CO_3, aquadest. Laboratory equipment used are glassware, magnetic stirrer, hotplate, oven, also XRD device (Rigaku Mini Flex 600), FT-IR spectrophotometer (Shimadzu Prestige-21). BET adsorption-desorption apparatus (ASAP 2020) and UV-Vis spectrophotometer (Biobase BK-UV 1800 PC).

2.1 Zn-Al LDHs Synthesis

Zn-Al LDH was synthesised at pH 10 by the coprecipitation method (Juleanti et al., 2022). A 10 mL solution of 0.75 M $Zn(NO_3)_2.6H_2O$ and a 10 mL solution of 0.25 M $Al(NO_3)_3.9H_2O$ were slowly poured into 10 mL of 2 M NaOH and stirred at 60°C for 4 hours to produce a white crystalline solid. The resulting solid was then dried at 100°C for 24 hours. Zn/Al LDH was then characterised by XRD, FT-IR spectrophotometer and BET surface area analysis.

2.2 Zn-Cr LDHs Synthesis

Zn-Cr was synthesized using 3 M NaOH at pH 10 by the coprecipitation method with the subsequent addition of 2.5 M Na_2CO_3 (Hirata et al., 2015). Approximately 13.07 g of $Zn(NO_3)_2.6H_2O$ was reconstituted in a 100 mL volumetric flask. Then a total of 10.04 g of formula.$9H_2O$ was weighed and dissolved in a 100 mL volumetric flask. $Zn(NO_3)_2.6H_2O$ was added to $Cr(NO_3)_3.9H_2O$ solution which has the appropriate molarity ratio of 2:1, and reacted at 60°C for 24 hours to get grey crystalline solids. Solid was then dried at 100°C for 24 hours. After obtaining Zn/Al LDHs, they were characterised by XRD, FT-IR spectrophotometer and BET surface area analysis.

2.3 Adsorption Experiment

pHpzc determination using NaOH 0,1 M or HCl 0,1 M for set at pH range of 2-11 and added 0.01 grams of adsorbent and stirred for 24 hours, measure the initial pH and final pH. Time variation for kinetic 0, 5, 10, 20, 30, 50, 70, 90, 120, 150, and 180 minutes. Isotherms and thermodynamics involving variations in concentration (25, 30, 35, 40, 45) mg/L and temperature 30°C, 40°C, 50°C, 60°C, and 70°C. As much as 0.05 grams of Zn-Al and Zn-Cr LDHs was added 50 mL Pb(II) solutions with a concentration of 25 mg/L. At the end of the stirring process using optimum pH and contact time, LDHs adsorbent was separated from the remaining metal ions via filtration. The filtrate obtained was complexed with 1.10-phenanthroline, the absorbance value was measured using UV-Vis spectrophotometer.

3. RESULTS AND DISCUSSION

XRD characterization Zn-Al LDHs results show in Figure 1 (a) detected the presence of diffractograms at specific angle 10,290° with interval of interlayer 8,589 ∀, 60° with the reflecting field sequentially (003), (110) that had an anion in interlayer. Mohadi et al. (2022) refrence literature shows the XRD diffractogram of Zn-Al LDH at an angle of 10.150° (003), 29.616° (009), 60.002° (110) that represent the presence of layered structures, metal oxides (Zn-O, Al-O), and anions, also match with JCPDS 48-1023. Temporary Zn-Cr LDHs show peaks at 11,51°(003) with interval of interlayer 7,68 ∀. The other peaks of Zn-Cr LDHs (b) are 12,09°, 33°, and 60°. Typical peaks at 2θ values were observed by Hirata et al. (2015) at diffraction points of 12.09°, 33°, and 60° indicating the formation of a layer structure in the Zn-Cr LDH which is peaks of 33° and 60° are typical non-basal peaks of Zn-Cr LDH with JCPDS 38-0486.

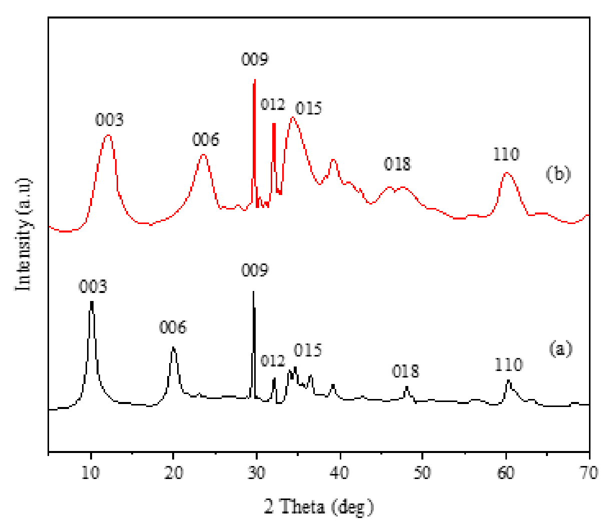

Figure 1. Diffractogram of Zn-Al (a), Zn-Cr (b)

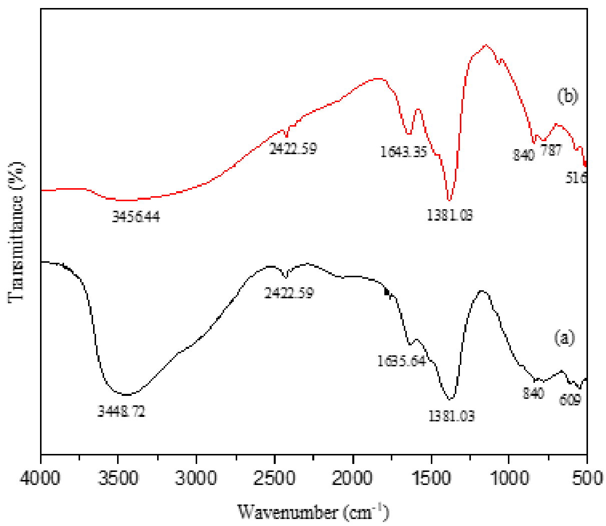

Figure 2. FT-IR Spectrum of Zn-Al (a), Zn-Cr (b)

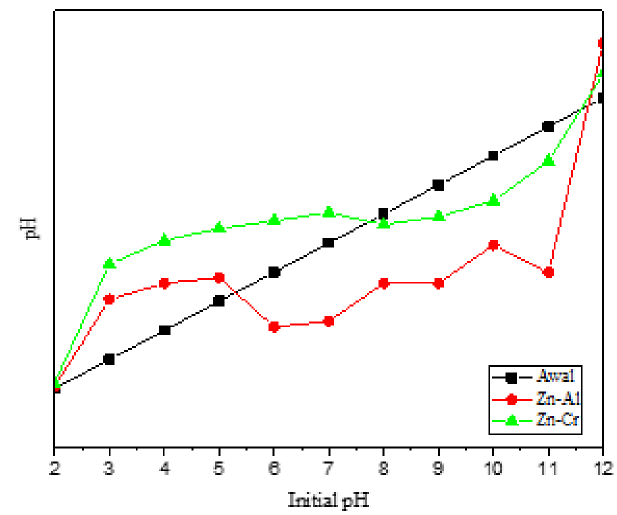

Figure 4. pH_{pzc} Analysis of Zn-Al and Zn-Cr LDHs

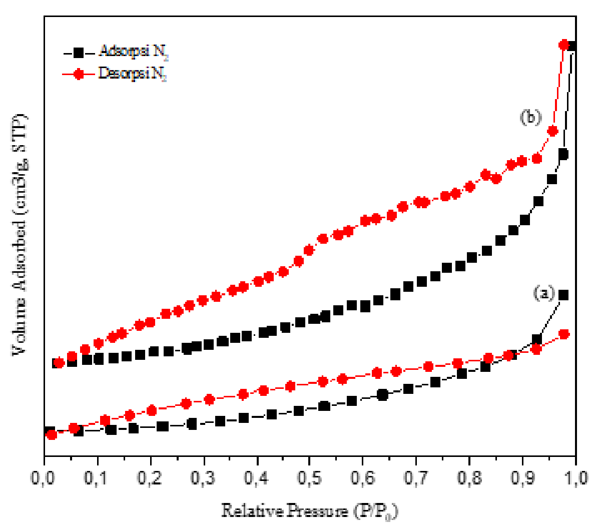

Figure 3. BET Isotherm Pattern of Zn-Al (a), Zn-Cr(b)

Zn-Al and Zn-Cr LDHs FT-IR spectrum presents in Figure 2 consecutively (a), (b) an intense peak in the wavenumber region of approximately 1381 cm^{-1} indicates nitrate anion, another extended vibration is found at a wavenumber around 3400 cm^{-1} which indicates the presence of -OH groups bound to the interlayer. Wavenumber 2422 cm^{-1} is a C=O as a result of stretching vibrations.group. Differences spectrum FT-IR of Zn-Al and Zn-Cr are located in the wavenumber for M-O. wave numbers of Zn-O, Al-O for Zn-Al are found at 840, 516 cm^{-1}. Meanwhile, Zn-O and Cr-O of Zn-Cr adsorbent are found at wavenumber 840, 609 cm^{-1}. Wavenumber for Zn-Al FT-IR spectrum results Dinari et al. (2016) explained at wavenumber 3100-3600 cm^{-1} is assigned to OH stretching vibrations, the bending vibration of interlayer OH is also shown in the broad band around 1620 cm^{-1}, nitrate group which shows LDHs is found at wavenumber 1385 cm^{-1}, and there's a low wavenumber of M-O group at 792 cm^{-1}, 552 cm^{-1}, and 431 cm^{-1}.

Table 1. BET Isotherm Measurement Data of Zn-Al and Zn-Cr LDHs

Materials	Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Diameter (nm)
Zn/Al LDH	1.965	0.006	27.687
Zn/Cr LDH	31.638	0.063	3.934

Table 2. Kinetic Adsorption Model for Removal Pb(II) use Zn-Al and Zn-Cr LDHs

Adsorbate	Kinetic Model	Parameter	Zn-Al	Zn-Cr
Pb(II)	Pseudo First Order (PFO)	qe eksperiment (mg/g)	15.239	12.233
		qe calculation (mg/g)	2.768	11.705
		k_1 (min^{-1})	0.067	0.038
		R^2	0.935	0.941
	Pseudo Second Order (PSO)	qe eksperiment (mg/g)	15.239	12.233
		qe calculation (mg/g)	15.527	13.280
		k_2 (g/mg.min)	0.052	0.002
		R^2	0.999	0.904

The general nitrogen adsorption-desorption isotherm is a fairly standard technique used to characterize the structure and pore network of a material. Figure 3 shows the surface adsorption-desorption profile of N_2 obtained from Zn-Al and

Table 3. Isotherm Model for Removal Pb(II) use Zn-Al and Zn-Cr LDHs

LDH	Adsorption Isotherm	Adsorption Constant	T (K)			
			30°C	40°C	50°C	60°C
Zn-Al	Langmuir	Qmax	74.129	64.516	52.632	48.544
		kL	0.032	0.051	0.097	0.158
		R^2	0.994	0.992	0.997	0.999
	Freundlich	n	2.796	1.609	1.983	2.320
		kF	7.698	5.155	7.982	10.852
		R^2	0.971	0.994	0.999	0.994
Zn-Cr	Langmuir	Qmax	27.027	12.987	66.225	68.966
		kL	0.041	0.121	0.041	0.053
		R^2	0.938	0.971	0.943	0.920
	Freundlich	n	1.073	1.183	1.566	1.552
		kF	1.244	2.017	4.438	5.351
		R^2	0.999	0.998	0.974	0.958

Zn-Cr LDHs. Zn-Al LDH shown in Figure 3 (a) with a type III isotherm adsorption-desorption. Type III is marked by molecules condensed at the same time on porous or ni-porous surfaces so the interaction between adsorbate and adsorbent is weak, thus resulting in weak adsorption energy due to the absence of monolayer coverage. Figure 3 (b) project result displayed a type IV isotherm for Zn-Cr LDH which is the overlap between the adsorption-desorption profile or as know as hysteresis, and mesoporous size (Kajama et al., 2015). Data pertaining to pore volume, surface area, and pore diameter are presented in Table 1. Based on the data shown in Table 1, the measurement results for BET isotherms surface area of Zn-Cr are higher than values of Zn-Al but pore diameter Zn-Al higher than Zn-Cr. This may be caused by the fact that Zn-Al occurs in a multi-layered.

pH_{pzc} was conducted to determine the exact condition of zero charge or no for Zn-Al and Zn-Cr LDHs retrieved on Figure 4. Determination on condition of the material being at pH_{pzc} is seen from the cross-over of the line between the pH of the material and the initial pH. Figure 4 (a) constitutes pHpzc Zn-Al an there is an intersection of lines at pH 6.2, while pHpzc Zn-Cr shown in Figure 4 (b) at pH 7.5. pH ≥ pHpzc (7.6) it is known that the cationic adsorption, meanwhile pH ≤ pHpzc is dominating to anionic adsorption. The pH_{pzc} obtained explains that Zn (Al and Cr) LDHs is neutrally charged and will be positively charged due to Pb(II) ions carrying a positive charge, so the amount of H^+ is more (Boukhalfa et al., 2017).

Increasing the adsorption time increases the adsorption capacity which can be seen in Figure 5. Analysis results of the adsorption pattern for pb(II) metal ions using Zn-Al LDH in Figure 5 (a) showed a significant increase at minute 10 and the adsorption tended to stabilise at minute 30. Figure 5 (b) shows the contact time for Zn-Cr LDH which increases at 30 minutes and tends to stabilise at 40 minutes. Kinetic adsorption dependent in contact time data shown at Figure 5 and used to calculate for rate using pseudo first order (PFO) and pseudo second order (PSO) equations. The pseudo first-order kinetic equation is treated

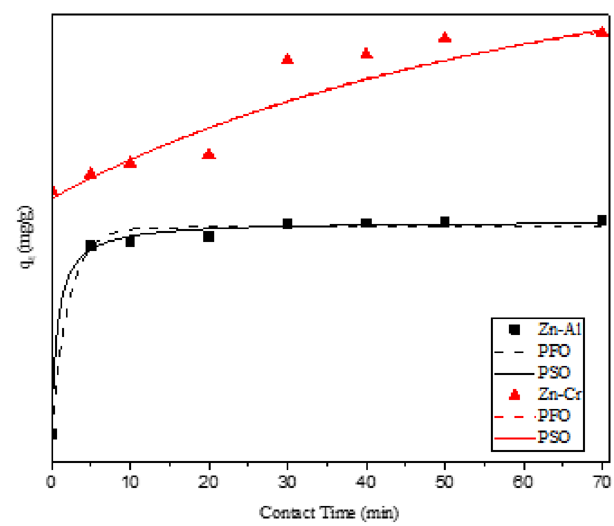

Figure 5. Effect of Adsorption Time of Pb(II) using Zn-Al and Zn-Cr

like first order in general by making one of the reagent components exaggerated, but actually the chemical reaction in this equation occurs at second order. Unlike the pseudo first-order kinetics, the pseudo second-order kinetics equation proceeds in a second-order manner. The value of the reaction rate constant can describe the speed of the Pb(II) ions adsorption process that occurs. Model constants of Pb(II) ions adsorption process kinetics against contact time can be seen in Table 2.

The kinetic adsorption process of Pb(II) ions shown in Table 2 with Zn-Al LDH adsorbent is inclined to follow a pseudo second-order equation that is directed towards chemisorption, but Zn-Cr LDH is more inclined to follow a pseudo first-order equation that is directed towards physisorption. This could be seen from the number of linear regression values which are getting closer to one, as well as from the calculated qe values which

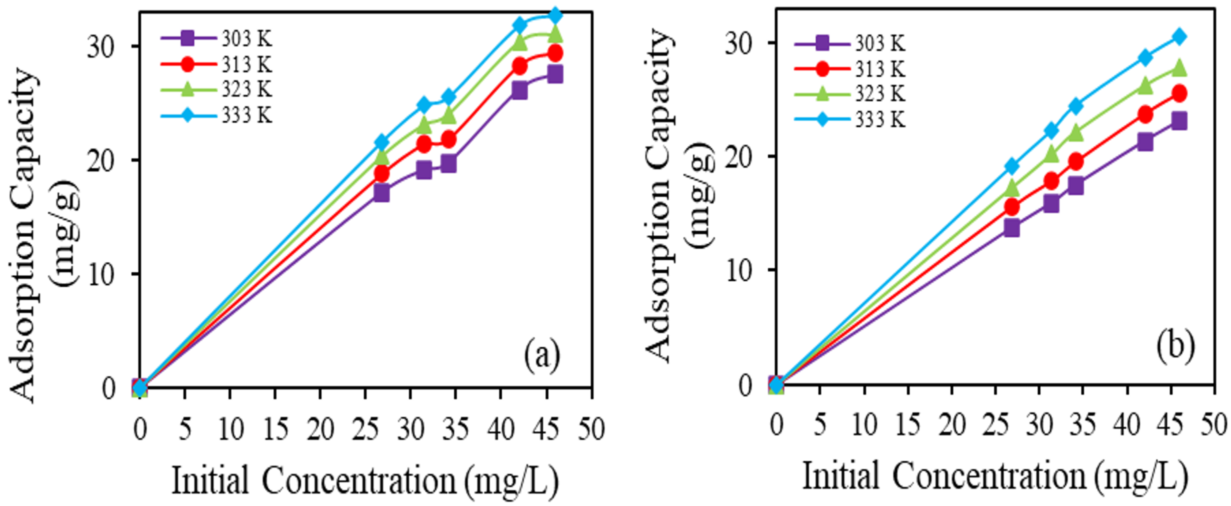

Figure 6. Effect of Initial Concentration and Temperature of Zn-Al (a), Zn-Cr (b)

Table 4. Thermodynamic Parameter for Removal Pb(II) use Zn-Al and Zn-Cr LDHs

Concentration (mg/L)	T (K)	Qe (mg/g)		ΔH (kJ/mol)		ΔS (kJ/mol)		ΔG (kJ/mol)	
		Zn-Al	Zn-Cr	Zn-Al	Zn-Cr	ZnAl	Zn-Cr	ZnAl	Zn-Cr
26.857	303	17.214	13.738	24.111	23.936	0.084	0.079	-1.431	-0.076
	313	18.857	15.571					-2.274	-0.868
	323	20.429	17.286					-3.117	-1.661
	333	21.707	19.143					-3.960	-2.453
31.429	303	19.143	15.929	24.539	24.358	0.085	0.080	-1.116	-0.004
	313	21.429	17.857					-1.963	-0.808
	323	23.143	20.236					-2.810	-1.612
	333	24.857	22.286					-3.656	-2.416
34.143	303	19.786	17.500	21.918	24.810	0.075	0.082	-0.794	-0.049
	313	21.857	19.546					-1.543	-0.870
	323	24.071	22.186					-2.293	-1.690
	333	25.571	24.479					-3.043	-2.511
42.043	303	26.257	21.329	18.026	20.550	0.064	0.068	-1.286	-0.038
	313	28.400	23.757					-1.923	-0.718
	323	30.480	26.257					-2.560	-1.397
	333	31.900	28.686					-3.198	-2.077
46.001	303	27.573	23.073	14.150	24.545	0.050	0.083	-1.010	-0.722
	313	29.501	25.573					-1.510	-1.556
	323	31.144	27.859					-2.010	-2.390
	333	32.821	30.501					-2.511	-3.224

are approaching the experimental qe. These data can also exhibit adsorption kinetics which can be seen from the k value which is increasingly close to zero. It suggests that the adsorption process is getting faster.

The data in Figure 6 indicate that the higher temperature, adsorption capacity of Pb(II) ions increases significantly process takes place using Zn-Al (a) and Zn-Cr (b) LDHs adsorbents. This situation is due to the fact that at high temperatures, adsorbent and adsorbate molecules are quite reactive, thus enabling interaction. Isotherm parameter data on Pb(II) adsorption process can be seen in Table 3.

Analysis of the data in Table 4 shows that the adsorption process of Pb(II) ions with Zn-Al and Zn-Cr LDHs follows the Freundlich isotherm equation model. This is evident from the correlation co-efficient value of the Freundlich isotherm equation which is closer to 1. Langmuir isotherm is applied when the adsorption process is monolayer, i.e. the adsorption-desorption surface rate is equal to zero under equilibrium conditions and has homogeneous sites. Freundlich isotherms are modelled when adsorption occurs in multilayers and heterogeneous sites are present (Kalam et al., 2021). Maximum capacity for Zn-Al is 74.129 mg/L at room temperature with kF value 7.698. Besides that Zn-Cr has maximum capacity 68.966 mg/L at temperature 60°C.

For each concentration when increasing temperature shown in Table 4, Gibss free energy (ΔG) values decreases gradually. It's indicates that the adsorption Pb(II) occurs depend on temperature. Gibbs free energy with a negative value indicates that the adsorption process occurs spontaneously. Value enthalpy energy (ΔH) and enthropy (ΔS) has a positive result and greater than 0, indicates that the adsorption process happens endothermally for Pb(II) ions. Endhotermic processes are the one that release energy from the system to the environment, while exothermic processes require energy from the environment which can be seen from negative enthalpy value. Enthalpy values produced are useful in determining the physical or chemical adsorption process. Physical adsorption has an enthalpy value of less than 40 kJ/mol, which means it does not require activation energy, because the physical adsorption process is existence of attractive forces between adsorbent and adsorbate. Chemical adsorption is characterized by having an enthalpy value of more than 40 kJ/mol because it requires high temperature and activation energy for chemical bonding to take place on the adsorbent surface, and chemical adsorption reactions are irreversible (Aljamali et al., 2021).

4. CONCLUSIONS

Zn-Al and Zn-Cr materials were successfully formed using the coprecipitation method. It can be seen from the XRD characterization results that show typical peaks of LDH Zn-Al and Zn-Cr, namely 10°, 30°, and 60°. Other supporting data are FT-IR and BET characterization data. pHpzc Zn-Al is 6.2 including below pHpzc (7.6), while Zn-Cr is 7.5 which means Pb(II) ions carries a positive charge on the neutrally charged LDH surface. Optimum time for Zn-Al occurs at time 30 minutes by following the PSO kinetics model with a k2 value of 0.052 g/mg.min that the adsorption process occurs chemically, for Zn-Cr the contact time stabilizes at minute 40 following the PFO kinetics model with a k_1 value of 0.038 min^{-1} which indicates the adsorption process occurs physically. Pb(II) ions adsorption with Zn-Al and Zn-Cr LDHs followed the Freundlich isotherm model with Qmax values of 74.129, 68.966 mg/L respectively. Enthalpy and entropy values obtained are positive, indicating that the process of adsorption occurs endotherm, releasing energy from the system to the environment, while the free energy value obtained is negative, meaning that it occurs spontaneously.

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