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Research Paper

Hydrochar and Humic Acid as Template of ZnAl Layered Double Hydroxide for Adsorption of Phenol

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Abstract
The adsorbents potential ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid were prepared using the coprecipitation method. The adsorbents were characterization by XRD, FTIR, and BET analysis. XRD peaks of ZnAl-LDH at 10.29°, 20.07°, 29.59°, 32.12°, 34.02°, 48.06°, and 60.16°. The FTIR absorption peak was observed at 3400-3500 cm^{-1}, 1600-1700 cm^{-1}, 1381 cm^{-1}, 1000 cm^{-1}, 500-700 cm^{-1}. All adsorbents exhibited N_2 adsorption-desorption isotherms type IV classified as a mesoporous structure (pore size= 2-50 nm). The surface areas of composites were higher than LDH and following order: ZnAl-Hydrochar > ZnAl-Humic acid > ZnAl-LDH. The kinetic parameter showed the pseudo-second-order kinetics model. The maximum adsorption capacity of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid were 48.077 mg/g, 90.090 mg/g, 94.340 mg/g, respectively; with Freundlich isotherm model. Reusability after 5 times of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid in the range 49.81-0.890%, 95.92-9.84%, and 70.02-5.72%, respectively. The adsorbent can be used up to 3 times.

Keywords
Hydrochar, Humic Acid, LDH, Adsorption, Phenol, Regeneration

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1. INTRODUCTION

Water is a basic human need to carry out activities. Drinking water used must meet physical, chemical, and biological requirements. Recently, rivers as water sources have been polluted by various kinds of waste, ranging from household and industrial to domestic waste (Selvanathan et al., 2017). The waste is not handled correctly, so the water becomes polluted (Chaari et al., 2020). One of the most dangerous wastes for the aquatic environment is phenol waste. Several industries that have the potential to produce phenol waste include the petroleum refining, textile, gas, pharmaceutical, and petrochemical industries, coal processing, pharmaceuticals, polymer resins, pesticides, and household industries that produce liquid phenol waste (Desmiarti et al., 2019; Zhang et al., 2021).

Phenol is a dangerous organic compound with a high level of toxicity, which can cause harm to humans and biota (De la Luz-Asunción et al., 2015; Dehbi et al., 2020; Dehmani et al., 2021; Girish and Ramachandra Murty, 2014) and accumulates in the environment. The limit of phenol concentration is 1.0 mg/L in water (Xie et al., 2020). Thus, developing an effective treatment to removal of phenol is necessary before being discharged to environment. Various methods have been proposed for phenol wastewater treatment, including oxidation, membrane separation, biodegradation, ion exchange, and adsorption (Tshemese et al., 2021). Adsorption is one technique for removal of phenol because it is fast, cost-effective, easy handling, regeneration, high selectivity, and high efficiency (Badhai et al., 2020; Ho and Adnan, 2021).

One of the materials used for water treatment in the adsorption process is layered double hydroxide (LDH). This material has become a material that has been widely developed because of its uniqueness and good absorption. Its application in water treatment as an adsorbent has excellent potential due to its low cost, exchangeable anionic features, and large surface area (Bouteraa et al., 2020; Zubair et al., 2021). According to Vithanage et al. (2020), LDH is adsorbent in water treatment applications to remove organic, inorganic species, dyestuffs, and toxic metal contaminants. Rathee et al. (2019) reported that LDH, which was applied as an adsorbent, had limitations in the regeneration process. Therefore, the double-layer hydroxyl needs to be modified with a carbon-based support material to form a composite.

Layered double hydroxide NiAl, ZnAl, and MgAl composited with carbon-based materials such as chitosan were effectively to remove congo red, and the ability of the composite to be used repeatedly was stable until the seventh cycle in the regeneration process (Siregar et al., 2021). Based on the literature, it can be concluded that carbon-based supporting materials to form composites, such as hydrochar and humic acid, are suitable for use in layered double hydroxide. According to Zubair et al. (2021), layered double hydroxide modified with carbon-based support material showed significant results, namely an increase in physicochemical characteristics such as surface area, structural stability, functional groups, and the resulting adsorption characteristics.

In this study, the synthesis of ZnAl-LDH using the coprecipitation method and the preparation of ZnAl composites with hydrochar and humic acid were carried out. The synthesized and prepared materials were characterized using X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Brunauer Emmet Teller (BET). Furthermore, the material will be applied as an adsorbent to adsorb phenol by studying various parameters including pH, contact time, temperatures, initial concentration, and regeneration.

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentation

The chemicals were used, including zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$), aluminum nitrate nonahydrate ($Al(NO_3)_3.9H_2O$), sodium carbonate (Na_2CO_3), humic acid, hydrochar, hydrogen chloride (HCl), distilled water (H_2O), sodium hydroxide (NaOH), phenol (C_6H_5OH), 4-aminoantipyrine ($C_{11}H_{13}N_3O$), potassium hexacyanoferrate(III) ($K_3[Fe(CN)_6]$), and acetate buffer solution (CH_3COONa) pH 10. Instrumentation was used in this study, including X-Ray Diffraction (XRD), Brunauer Emmet Teller (BET), and Fourier Transform Infra-Red (FTIR).

2.2 Synthesis of ZnAl-LDH

ZnAl-LDH synthesis was carried out with 100 mL $Zn(NO_3)_2.6H_2O$ 0.75 M mixed with 100 mL $Al(NO_3)_3.9H_2O$ 0.25 M, then dripped into 50 mL NaOH 2 M solution (Mohadi et al., 2022). The mixture was adjusted to pH 10, then stirred for 20 h at 353 K. After stirring, the precipitate was filtered and rinsed using distilled water to remove impurities. The precipitate was dried using an oven.

2.3 Preparation of ZnAl-Hidrochar and ZnAl-Humic Acid

ZnAl composites were prepared using the coprecipitation method with constant pH. A total of 15 mL of 0.75 M Zn solution and 15 mL of 0.25 M Al solution were mixed, and the pH was adjusted to pH 10. The mixture was stirred for 1 h until homogeneous, and a gel was formed, then 3 g of humic acid/hydrochar was added. The solution was kept at 353 K for 3 days. The precipitate from the preparation was filtered and dried using an oven.

2.4 Adsorption of Phenol

Phenol is a colorless solution, so it must be complexed before being measured using a UV-Vis spectrophotometer (Xie et al., 2020). 1 mL of phenol solution was put in a beaker, then 0.1 mL of 4-aminoantipyrine 2% was added, 0.1 mL of potassium hexacyanoferrate (III) 8%, 1 mL of pH 10 buffer solution, and 3 mL of distilled water were added. Then homogenized and allowed to stand for 5 min. The maximum wavelength of phenol after complexing is 510 nm.

2.5 Effect of pH

Adsorbent (0.05 g) was put into a 100 mL beaker filled with 50 mL of phenol solution, each at a concentration of 20 mg/L with a variation of pH 2-11 with stirring for 2 h.

2.6 Effect of Contact Time

Adsorbent (0.05 g) was added to 50 mL of 20 mg/L phenol solution, then stirred with variations contact time (0-180 min). The adsorbent was separated from the phenol solution.

2.7 Effect of Initial Concentration and Temperatures

Adsorbent (0.05 g) was added 50 mL of phenol solution with various initial concentrations (10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L and 50 mg/L and various temperatures (303 K, 313 K, 323 K, and 333 K). The solution was stirred for 2 h.

2.8 Reusability of Adsorbents

The adsorption process for phenol is carried out before being used repeatedly as an adsorbent. After that, the desorption of phenol solution using an ultrasonic device. The regeneration process is carried out by adding 50 mg/L of 50 mL phenol solution to adsorbent that has gone through the desorption process.

3. RESULT AND DISCUSSSION

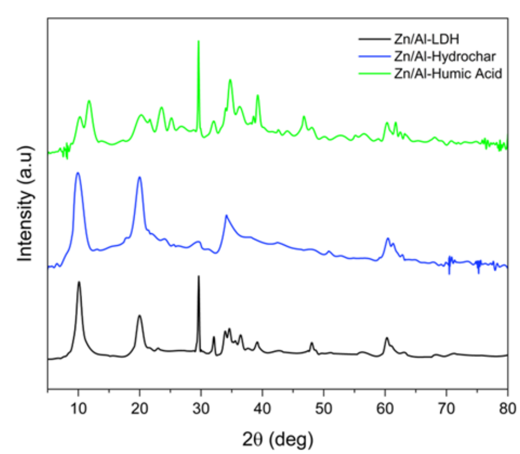

Figure 1. XRD Diffractogram of Adsorbents

XRD patterns of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid is displayed in Figure 1. ZnAl-LDH peaks at 10.29°, 20.07°, 29.59°, 32.12°, 34.02°, 48.06°, and 60.16° were indexed to (003), (006), (101), (012), (015), (107), and (110) corresponding to JCPDS No 05-0669 (Elhalil et al., 2017). The peaks at 10.29° and 60.16° indicated the anion in the interlayer of layered double hydroxide. After ZnAl-LDH was composited with hydrochar and humic acid, the peak at 20.3° indicated the humic acid, while the diffraction peaks of hydrochar at peaks 18.0°. The characteristic peaks of the constituents ZnAl-LDH, hydrochar, and humic acid indicated that the preparation of the composite has been successful.

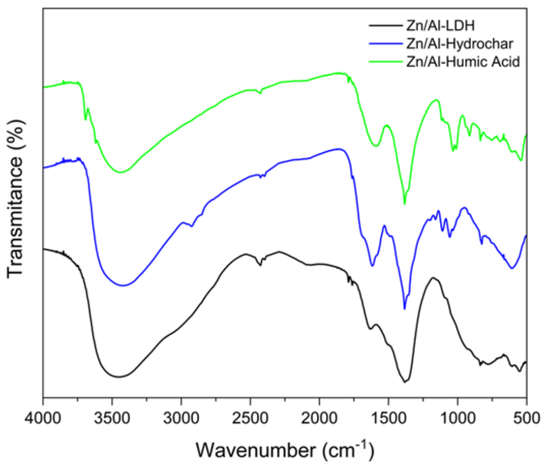

Figure 2. FTIR Spectra of Adsorbents

Figure 2 shows the FTIR spectra of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid. The absorption peak was observed at 3400-3500 cm^{-1} assigned to O-H vibration from water in LDH (Li et al., 2020). The peak at 1600-1700 cm^{-1} indicated vibration of -OH and carbonyl (COO) (Rashid et al., 2017). The peak at 1381 cm^{-1} corresponds to N-O from nitrate (Palapa et al., 2021). The new peak around 1000 cm^{-1} at ZnAl-Hydrochar and ZnAl-Humic acid indicated C-O stretching from hydrochar and humic acid (Lu et al., 2019; Shao et al., 2022). The peak at 500-700 cm^{-1} assigns to metal-oxide in LDH (Ahmad et al., 2022b).

Textural properties of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid give in Figure 3. All adsorbents exhibited N_2 adsorption-desorption isotherms type IV classified as a mesoporous structure (pore size = 2-50 nm) (Cao et al., 2022). In line with the data in Table 1, the pore size of adsorbent is 4-27 nm. The surface areas of composites were higher than LDH and were in the following order: ZnAl-Hydrochar > ZnAl-Humic acid > ZnAl-LDH. The pore volume of ZnAl-Hydrochar was higher than ZnAl-Humic acid and ZnAl-LDH. Thus, the surface area is directly proportional to the pore volume.

The effect of pH in adsorption phenol was displayed in Figure 4. pH is one of the key parameters in adsorption. pH

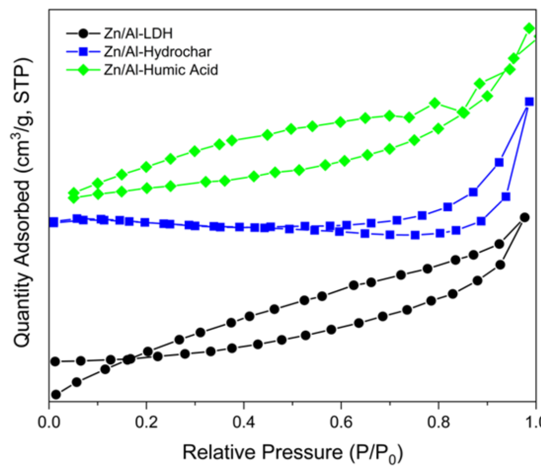

Figure 3. N_2 Adsorption-desorption Isotherms

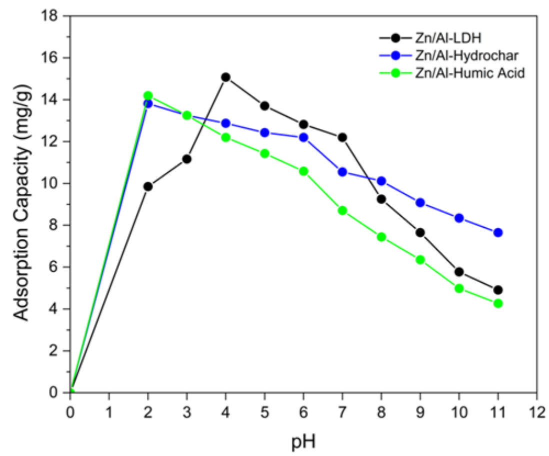

Figure 4. Effect of pH

optimum of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid was at pH 4, 2, and 2, respectively. Under acidic conditions, phenol adsorption is better because the phenol molecules do not dissociate, thereby reducing electrostatic repulsion and hydrogen bonding being the primary interaction (Asnaoui et al., 2022).

Kinetic parameters adsorption of phenol is shown in Figure 5. The pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were determined through the highest linear regression value. Based on Table 2, ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid followed the PSO kinetics model (R^2 closer to 1). PSO model assumes that the active site of the adsorbent is available more than the possible bond between the adsorbent and the adsorbate that occurs (de Farias et al., 2022).

Determination isotherm model seen the linear regression value (R^2) which is closer to 1. ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid tend to follow the Freundlich isotherm model because the R^2 value is closer to 1 compared to the

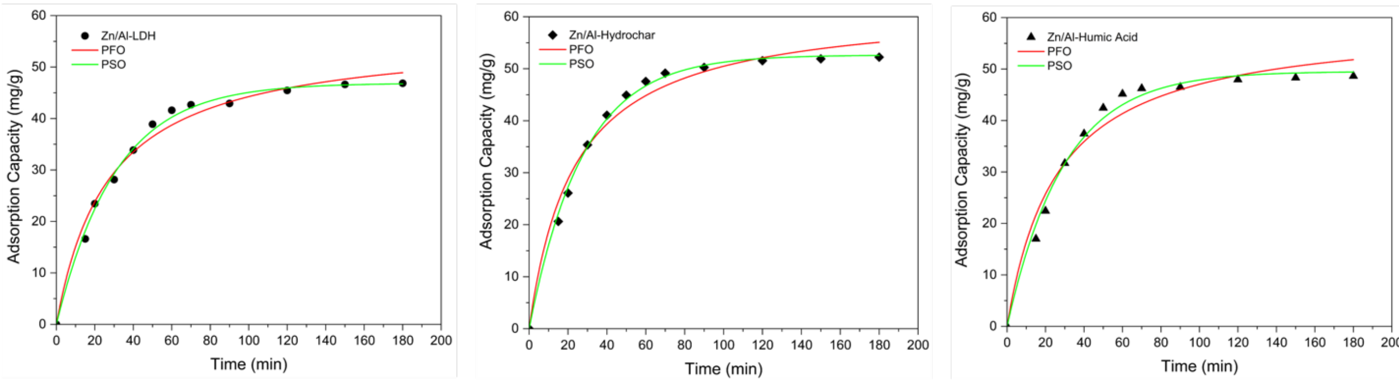

Figure 5. Effect of Contact Time

Table 1. BET Analysis of Adsorbents

Adsorbents	Surface Area (m^2/g)	Pore Size (nm), BJH	Pore Volume (cm^3/g), BJH
ZnAl-LDH	1.968	27.687	0.006
ZnAl-Hydrochar	29.874	24.420	0.042
ZnAl-Humic Acid	16.425	4.811	0.039

Table 2. Kinetics Parameters the Adsorption Process

Kinetic Parameter	Parameters	Adsorbents		
		ZnAl-LDH	ZnAl-Hydrochar	ZnAl-Humic Acid
PFO	Qe_{exp} (mg/g)	46.845	52.228	48.627
	Qe_{calc} (mg/g)	48.228	44.627	41.295
	k_1 (min^{-1})	0.033	0.034	0.034
	R^2	0.965	0.989	0.969
PSO	Qe_{exp} (mg/g)	46.845	52.228	48.627
	Qe_{calc} (mg/g)	56.644	59.524	56.497
	k_2 (min^{-1})	0.001	0.001	0.001
	R^2	0.993	0.993	0.986

Table 3. Isotherm Parameters the Adsorption Process

Adsorbents	T (K)	Langmuir			Freundlich		
		Qmax	kL	R^2	n	kF	R^2
ZnAl-LDH	303	20.492	0.167	0.974	5.149	15.765	0.994
	313	48.077	0.063	0.844	1.866	5.338	0.847
	323	43.487	0.140	0.930	2.482	9.643	0.822
	333	42.553	0.332	0.984	3.221	15.191	0.855
ZnAl-Hydrochar	303	27.027	0.019	0.961	1.989	6.703	0.947
	313	85.470	0.012	0.956	1.164	2.078	0.957
	323	75.188	0.038	0.7984	1.473	4.248	0.954
	333	90.090	0.071	0.8516	1.900	7.579	0.943
ZnAl-Humic Acid	303	26.882	0.078	0.969	3.837	12.552	0.989
	313	44.444	0.053	0.944	1.678	4.281	0.975
	323	64.935	0.049	0.900	1.616	5.018	0.962
	333	94.340	0.077	0.911	1.987	8.468	0.968

Table 4. Several Adsorbents to Adsorption of Phenol

Adsorbent	Qmax (mg/g)	References
Lignite	6.216	(Liu et al., 2021)
Tea waste biomass	7.62	(Gupta and Balomajumder, 2015)
MAG-CTAB-KH550	56.13	(Ge et al., 2018)
Bentonite	23.64	(Ahmadi and Igwegbe, 2018)
Fe-Biochar	39.23	(Dong et al., 2021)
ZnCl2-BFAC	17.02	(Sathya Priya and Sureshkumar, 2020)
Clarified sludge from basic oxygen furnace	1.052	(Mandal and Das, 2019)
α-Fe_2O_3	21.93	(Dehmani and Abouarnadasse, 2020)
Activated carbon	75.81	(da Silva et al., 2022)
ZnAl-LDH	48.077	This study
ZnAl-Hydrochar	90.090	This study
ZnAl-Humic Acid	94.340	This study

Table 5. Adsorption Thermodynamic Parameter

Adsorbents	ΔH (kJ/mol)	ΔS (J/K.mol)	ΔG (kJ/mol)				R^2
			303 K	313 K	323 K	333 K	
ZnAl-LDH	51.180	0.171	-0.586	-2.294	-4.003	-5.711	0.985
ZnAl-Hydrochar	33.152	0.110	-0.224	-1.326	-2.427	-3.529	0.993
ZnAl-Humic Acid	33.821	0.133	-0.488	-1.620	-2.752	-3.885	0.988

Langmuir isotherm model (See Table 3). Freundlich isotherm assumes that the adsorption process is physisorption and occurs in multilayers (Jain et al., 2022). The maximum adsorption capacity of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid were 48.077 mg/g, 90.090 mg/g, 94.340 mg/g, respectively. The comparison of adsorption capacity with other adsorbents can be seen in Table 4.

The thermodynamic parameters determined in the adsorption include enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG) as presented in Table 5. The enthalpy (ΔH) is positive in the range of 31-51 kJ/mol, this indicates that the reaction that occurs is endothermic (Dehmani et al., 2020). The entropy (ΔS) is positive in the range of 0.110 - 0.171 J/mol.K, this indicates that the degree of disorder is small during the adsorption process (Zhang et al., 2022). The Gibbs free energy (ΔG) is negative, indicating that adsorption of phenol is spontaneous (Ahmad et al., 2022a).

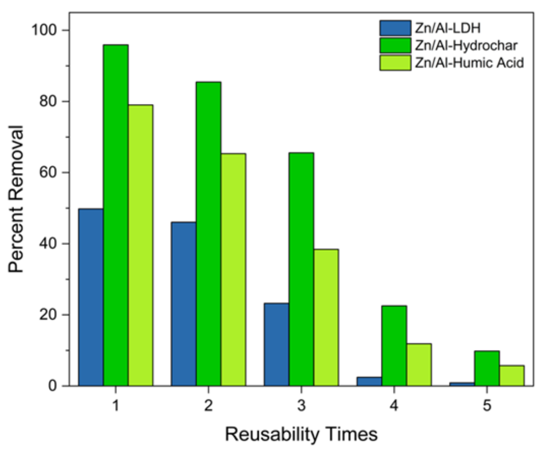

Figure 6. Reusability of Adsorbents

Reusability of adsorbent is the repeated use of the adsorbent by removing the adsorbate from the adsorbent (Qu et al., 2022). Based on Figure 6, reusability after 5 times of ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid in the range 49.81-0.890%, 95.92-9.84%, and 70.02-5.72%, respectively. The percentage of reusability decreases with the frequent adsorption process. The adsorbent can be used up to 3 times.

4. CONCLUSION

In summary, ZnAl-LDH, ZnAl-Hydrochar, and ZnAl-Humic acid were successfully prepared. The adsorbents were characterization by XRD, FTIR, and BET analysis. The maximum adsorption capacity is 94.340 mg/g on ZnAl-Humic acid. Parameters such as pH, contact time, concentration, temperature, and regeneration affected to adsorption process. Kinetic and isotherm data were fitted to the PSO kinetic model and Freundlich isotherm model, respectively. The adsorbent can be used up to 3 times in the adsorption of phenol.

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