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Research Paper

Adsorption of Phenol from Aqueous Solution Using Zn/Al Layered Double Hydroxides-Cellulose Composite

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Abstract
The successful synthesis and preparation of Zn/Al layered double hydroxide, cellulose, and Zn/Al-cellulose materials were analyzed using XRD, FT-IR, and BET. The diffraction peaks of Zn/Al layered double hydroxide are at 10.3°; 20.3°; 34.8°; and 60.40°, while the diffraction peaks of cellulose are at 15.5°; 22.4°; and 34.5°. Diffraction peaks on Zn/Al layered double hydroxide and cellulose were observed at 3442 cm^{-1}, 1642 cm^{-1}, 1440-1620 cm^{-1}, 1351 cm^{-1}, 1153 cm^{-1}, and 400-800 cm^{-1}. The surface area of the material after the composite increased from 1.968 to 13.615 m^2/g. The optimum pH for Zn/Al LDH was pH 4, pH 10 for cellulose, and pH 2 for Zn/Al-cellulose. The isotherm data of Zn/Al LDH and cellulose followed the Freundlich model, while Zn/Al-cellulose followed the Langmuir model. The reuse of adsorbents in the adsorption process can be used up to 3 times.

Keywords
Cellulose, Phenol, Adsorption, Layered Double Hydroxide

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1. INTRODUCTION

Disruption to human health and ecosystems is of particular concern due to the presence of phenolic compounds produced from the petroleum and petrochemical industries, plastics, pesticides, paper and pharmaceuticals (Zhang et al., 2019). At low concentrations, this compound will have a negative impact on ecosystems and human health when ingested. Therefore, various world organizations such as World Health Organization (WHO), European Union (EU), and US Environmental Protection Agency (EPA) have set a threshold amount of phenolic compounds in drinking water and wastewater not more than 0.001 mg/L and 1 mg/L, respectively (Sas et al., 2019).

Various ways that can be done to remove phenolic compounds in wastewater include using photocatalyst, chemical and electrochemical oxidation, reverse osmosis, biological removal, photocatalyst, and adsorption methods (da Silva et al., 2022; Dehmani et al., 2023). Among the various methods above, the adsorption method is the most promising method for the removal of phenolic compounds in wastewater due to its high removal efficiency, simple application, flexibility, low cost (Haydari et al., 2023).

Recently, many other studies have been carried out regarding the types of adsorbents, both theoretically and practically, including layered double hydroxide (Alnasrawi et al., 2022). Layered double hydroxide (LDH) is known to have a unique two-dimensional flattened structure, high catalytic activity, and has a manageable composition, large surface area and good stability so it is very well used as an adsorbent (Kong et al., 2023; Zhang et al., 2023).

However, LDH has the limitation that it cannot be used for repeated use due to the ease of peeling off during application (Siregar et al., 2022). Therefore, it is necessary to improve the structure by compositing carbon-based materials. Many studies have conducted composites with carbon-based materials such as biochar (Luo et al., 2023), graphite (Hu et al., 2019), chitosan (Mallakpour et al., 2023), and hydrochar (Luo et al., 2020). According to Zubair et al. (2022), the addition of supporting materials such as carbon-based materials into the matrix in composites will be able to improve the properties of the material and can improve adsorption performance.

In this research, Zn/Al LDH composites will be made with other carbon-based materials, namely cellulose. The preparation of Zn/Al-cellulose composite material will then be carried out characterization tests using XRD, FT-IR, and BET. The effect of pH, time, temperature and temperature, as well as the reuse of adsorbent in adsorption will be studied in this research.

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentation

In this study using several chemicals including potassium hexacyanoferrate (III) ($K_3[Fe(CN)_6]$), pH 10 buffer solution, phenol solution, zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$), aluminum nitrate nonahydrate ($Al(NO_3)_3.9H_2O$), 4-aminoantipyrine ($C_{11}H_{13}N_3O$), sodium hydroxide (NaOH), hydrogen chloride (HCl), and cellulose carbon-based materials. Supporting tools in this research include Biobase BK-UV 1800 PC, X-Ray Diffraction (XRD) type Rigaku Miniflex-6000, and Surface Area & Pore Size Analyzer (BET) type NOVA 4200e.

2.2 Synthesis of Zn/Al LDH

Each 100 mL of 0.25 M $Al(NO_3)_3.9H_2O$ and 0.75 M $Zn(NO_3)_2.6H_2O$ solution was put into a glass beaker. After the solution mixture was added, sodium hydroxide with a concentration of 2 M was prepared as much as 50 mL to make the pH of the solution to pH 8. The solution mixture was stirred for 4 hours at 80°C until a white precipitate was obtained. Furthermore, the resulting precipitate was filtered, washed and dried.

2.3 Preparation of Zn/Al-Cellulose

Zn/Al LDH was first prepared using 30 mL each of 0.75 M $Zn(NO_3)_2.6H_2O$ and 0.25 M $Al(NO_3)_3.9H_2O$. Furthermore, 2 M NaOH was added to make the pH 8. The resulting solution was stirred for 1 hour before adding 3 grams of cellulose and stirred for 3 days at 80°C. The precipitate obtained was then filtered, washed using distilled water, and dried.

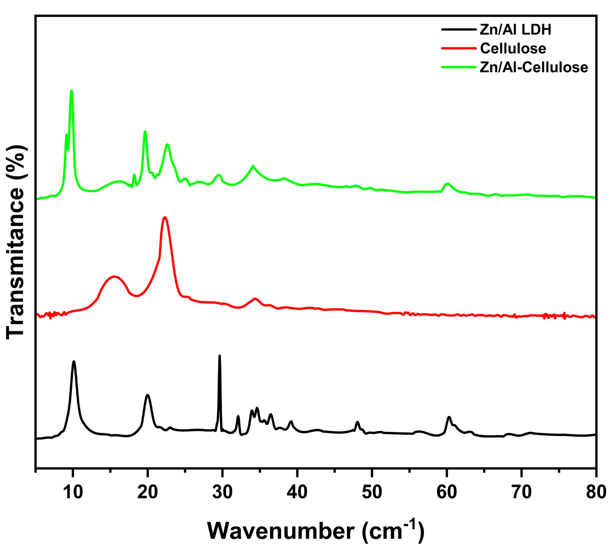

Figure 1. X-Ray Diffractogram of Adsorbents

2.4 Adsorption of Phenol

The adsorption process studied during the observation is the effect of pH, time, the effect of temperature and temperature, and the reuse of adsorbent. Observations were made by combining 30 mL of phenol solution and 0.03 grams of adsorbent material. The effect of pH was carried out with a pH range of 2-11, contact time was carried out at 0, 10, 20, 30, 40, 50, 60, 70, 90, 120, 150, and 180 minutes. The effect of temperature and temperature was carried out simultaneously using a temperature of 30-60°C at various concentrations of 20, 30, 40, 50, and 60 mg/L. The observation stage of adsorbent reuse in the adsorption process was carried out with 5 repetitions.

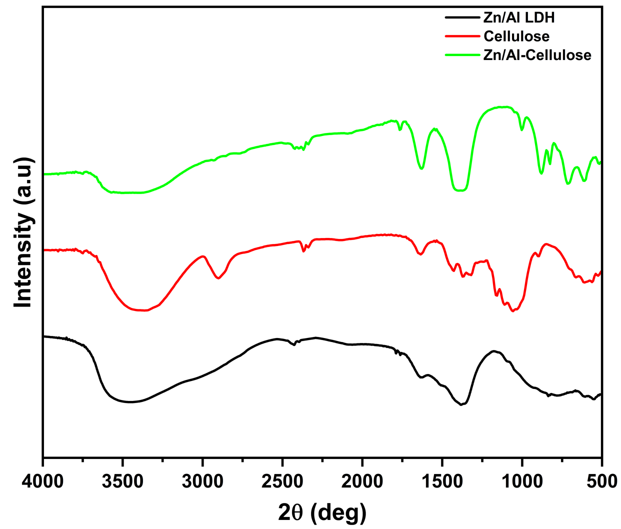

Figure 2. Fourier Transfer Infra-Red Spectrum of Adsorbents

Before measuring the concentration of penol solution on UV-VIS spectrophotometer, first the phenol solution was complexed first by combining 1 mL of each phenol solution and pH-10 buffer solution, then 0.1 mL of each solution was added 8% hexacyanoferrate (III) and 2% 4-aminoantipyrine reagent and 3 mL of distilled water. After that, it was allowed to stand for 15 minutes until homogeneous before measuring the absorbance.

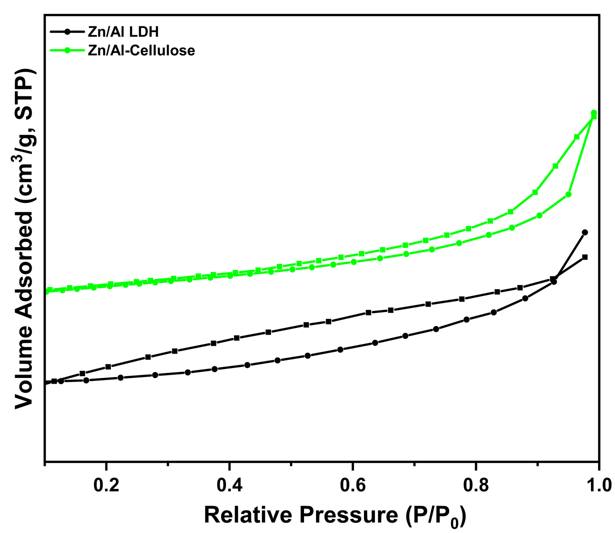

Figure 3. N_2 Adsorption-desorption Isotherms

3. RESULTS AND DISCUSSION

3.1 Characterization of the Adsorbent Materials

Figure 1 shows the appearance of the XRD patterns of Zn/Al LDH, cellulose, and Zn/Al-cellulose. The diffraction peaks at 10.3°, 20.3°, 34.8°, and 60.40° are indicated by the (003), (006), (012), (015), and (110) fields (Rezvani et al., 2023). The appearance of the peak indicates that the layered structure of the LDH has been successfully formed (Wu et al., 2022). Diffraction peaks in cellulose can be seen at peaks 15.5°, 22.4°, and 34.5° which are characteristic of cellulose according to fields (110), (200), and (040) (Jabli et al., 2022). The successful preparation of Zn/Al-cellulose can be seen in the diffraction peaks which show the characteristics of the diffraction peaks in Zn/Al LDH marked at 10.3°, 20.3°, 34.8°, and 60.40°. Whereas in cellulose it is characterized by diffraction peaks of 15.5°, 22.4°, and 34.5°.

Table 1. Brunauer Emmet Teller of Adsorbents

Material	Surface Area (m^2/g)	Pore Size (nm), BJH	Pore Volume (cm^3/g), BJH
Zn/Al LDH	1.968	27.687	0.006
Zn/Al-Cellulose	13.615	18.604	0.045

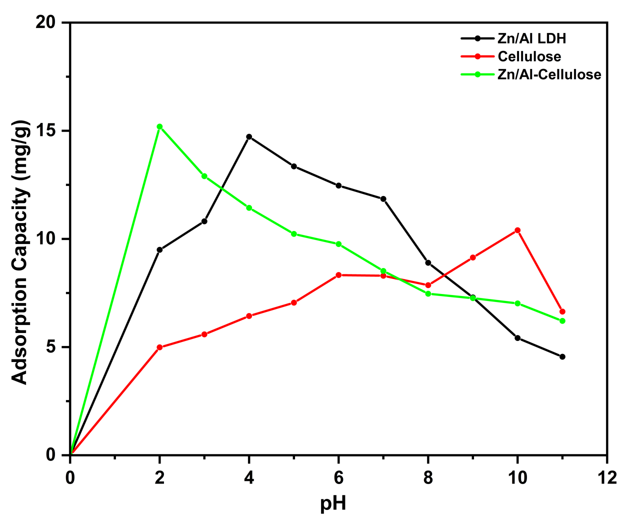

Figure 4. Effect of pH on Adsorption of Phenol

Figure 2 shows the FTIR spectra for Zn/Al LDH, cellulose, and Zn/Al-cellulose. In this material there is a band at 1351 cm^{-1} which indicates the presence of nitrate ions in the LDH interlayer (Çalışkan and Baran, 2022). The band originating from 3442 cm^{-1} indicates the presence of OH stretching vibrations of the hydroxyl, while the O-H bending vibrations in the water molecule interlayer are shown at the peak of 1642 cm^{-1} (Azad and Mohsennia, 2020; Çalışkan and Baran, 2022). The band at 400-800 cm^{-1} indicates the presence of M-O bonds in LDH (where M=Zn and Al) (Dinari et al., 2019). In addition, there are bands at 1440-1620 cm^{-1} indicating the presence of symmetrical and asymmetric stretching vibrations of the carboxylate groups. The peak at 1153 cm^{-1} indicates the presence of C-O-C groups associated with glucosidic cellulose biopolymers (Bessaies et al., 2021).

The textural characteristics of Zn/Al LDH and Zn/Al-cellulose can be observed in Figure 3. The results obtained show that the N_2 adsorption-desorption isotherms for all materials follow the IV hysteresis loop pattern. This indicates that all adsorbents have mesoporous characteristics (Zubair et al., 2022). In table 1 it can be seen that the Zn/Al-cellulose material has a larger surface area than the Zn/Al LDH material. The same thing with the size of the pore volume where Zn/Al-cellulose has a larger size than Zn/Al LDH. So it can be concluded that the surface area is directly proportional to the pore volume.

3.2 Effect of pH

Figure 4 illustrates the effect of pH on phenol adsorption using Zn/Al-LDH, cellulose and Zn/Al-cellulose materials. The results showed that Zn/Al-LDH, cellulose and Zn/Al-cellulose materials had optimum pH of 4, 10 and 2, respectively. The high adsorption capacity for acids was due to the large amount of protonation on the surface of the material thus creating an electrostatic attraction interaction between phenolic ion is negatively charged with more material (adsorbent) (Karamipour et al., 2020).

3.3 Effect of Time and Kinetics study

Figure 5 shows the effect of time on phenol adsorption. It can be seen that with increasing time the adsorption capacity increases. Determination of the adsorption kinetics model can be seen from its R^2 value (Wang et al., 2022). Based on the data in Table 2, it can be seen that all adsorbent materials follow the pseudo second order (PSO) kinetics model, where the R^2 value in PSO is closer to 1.

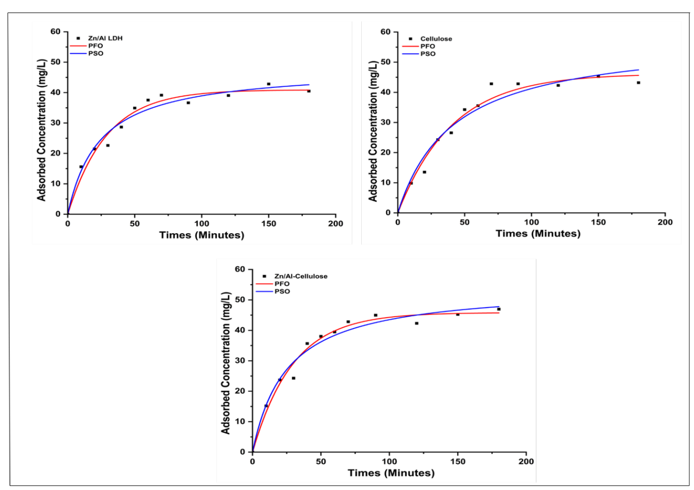

Figure 5. Effect of Contact Time

Table 2. Parameters of Adsorption Process Kinetics

Kinetic Model	Parameter	Adsorbents Zn/Al LDH	Cellulose	Zn/Al-Cellulose
pseudo first order	Qe_{exp} (mg/g)	40.481	43.209	46.952
	Qe_{calc} (mg/g)	26.965	54.150	32.576
	k_1 (min^{-1})	0.025	0.029	0.022
	R^2	0.831	0.767	0.843
pseudo second order	Qe_{exp} (mg/g)	40.481	43.209	46.952
	Qe_{calc} (mg/g)	46.948	57.143	53.191
	k_2 (min^{-1})	0.0010	0.0004	0.0008
	R^2	0.988	0.955	0.989

Table 3. Isoterm Parameters the Adsorption Process

Adsorbent	T (°C)	Langmuir			Freundlich		
		Q_{max}	kL	R^2	n	kF	R^2
Zn/Al LDH	30	20.492	0.167	0.974	5.149	15.765	0.994
	40	48.077	0.063	0.844	1.866	5.338	0.847
	50	43.478	0.140	0.930	2.482	9.643	0.822
	60	42.553	0.332	0.984	3.221	15.191	0.855
Cellulose	30	21.413	0.018	0.857	1.191	1.905	0.946
	40	20.450	0.014	0.829	1.405	3.187	0.959
	50	34.722	0.008	0.446	0.511	1.401	0.985
	60	35.336	0.006	0.689	2.002	7.084	0.920
Zn/Al-Cellulose	30	30.960	0.043	0.999	1.541	3.726	0.978
	40	20.450	0.235	0.999	2.016	6.750	0.989
	50	34.722	0.062	0.999	1.175	1.922	0.895
	60	35.336	0.076	0.999	2.755	12.897	0.989

3.4 Effect of Isotherms and Thermodynamic Studies

Adsorption isotherm data for the three adsorbent materials can be seen in Table 3. In addition to knowing the type of isotherm model that is suitable, adsorption isotherm is also used to observe the interaction between the adsorbent and the adsorbate molecule in order to achieve equilibrium. Based on Table 3, it can be seen that Zn/Al LDH and cellulose materials tend to be the Freundlich isotherm model, while Zn/Al-cellulose tends to the Langmuir isotherm model. This can be seen from the value of R^2 which is close to 1. The Langmuir isotherm model shows a uniform adsorbent surface and there are several absorption sites where the nearby adsorbed molecules do not interfere. Meanwhile, the Freundlich isotherm model shows that the absorption is layered and the surface is heterogeneous where the absorption distribution is uneven (Zhang et al., 2021). Comparison of this study with other studies is shown in Table 4.

Adsorption thermodynamic parameters such as ΔS (entropy), ΔG (Gibbs free energy), and ΔH (enthalpy) are determined in Table 5. A small ΔS value indicates a low entropy value when the adsorption process is running (Mahmoud, 2022). A positive ΔH value indicates that the adsorption process is endothermic. The spontaneity of the adsorption process can be seen from the negative ΔG value, where as the temperature rises it shows a negative ΔG value indicating that the adsorption process occurs spontaneously (Ali et al., 2022).

Table 4. Maximum Adsorption Capacity of Phenol and Comparison with Other Researches

Adsorbent	Q_{max} (mg/g)	Reference
DNNH	16.52	(Nakhjiri et al., 2021)
Graphene	28.26	(Li et al., 2012)
Exfoliated graphite	10.23	(Tshemese et al., 2021)
Fe_3O_4/chitosan/ZIF-8 nanocomposite	6.43	(Keshvardoostchokami et al., 2021)
Zinc oxide	0.28	(Safwat et al., 2019)
Zn/Al LDH	42.553	This work
Zn/Al-cellulose	35.336	This work

The use of material reuse against the adsorption process is

Table 5. Adsorption Thermodynamic Parameter

Adsorbent	Concentration (mg/L)	ΔH (kJ/mol)	ΔS (kJ/mol)	ΔG (kJ/mol)			
				303 K	313 K	323 K	333 K
Zn/Al LDH	60	17.798	0.057	0.460	-0.112	-0.684	-1.256
Cellulose	60	5.404	0.016	-10.344	-10.507	-10.670	-10.833
Zn/Al-Cellulose	100	13.350	0.042	0.487	0.063	-0.362	-0.786

shown in Figure 6. Based on the figure, it can be seen that all materials are carried out up to five cycles. The Zn/Al-cellulose material has a reduction in adsorption capacity from 39.07%-1.34%, Zn/Al LDH has a reduction in adsorption capacity from 49.81%-0.89%, and cellulose has a reduction from 23.25%-4.69%. The low adsorption capacity of Zn/Al-cellulose compared to Zn/Al LDH is possible because Zn/Al-cellulose material has a homogeneous surface and only has a few number of adsorption sites compared to Zn/Al LDH which has a heterogeneous surface so that it can adsorb in layers on the surface.

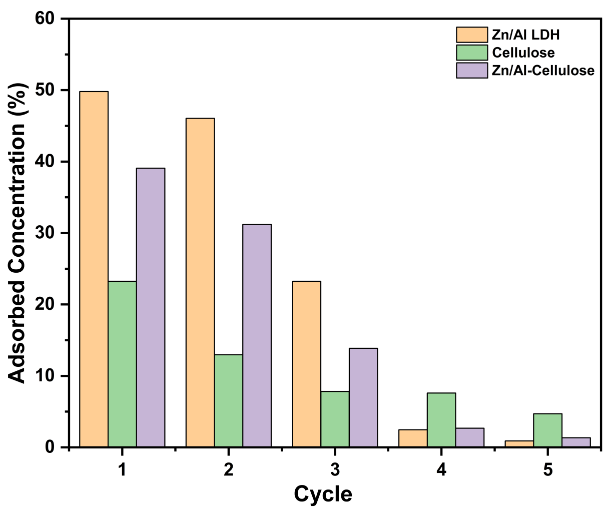

Figure 6. Reusability of Adsorbents

4. CONCLUSION

In summary, Zn/Al LDH, cellulose, and Zn/Al-cellulose materials were successfully synthesized. Adsorbent materials were characterized using XRD, FT-IR, and BET. The kinetic data of all adsorbents followed PSO. The isotherm data on Zn/Al LDH and cellulose followed the Freundlich model, while Zn/Al-cellulose followed the Langmuir model. The maximum adsorption capacity of Zn/Al-cellulose was 35,336 mg/g. Adsorbent reuse in the adsorption process can be used up to 3 times.

5. ACKNOWLEDGMENT

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