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Research Paper

Removal of Anionic Direct Dye Using Zn/Al, Zn/Fe and Zn/Cr Layered Double Hydroxides Toward Interlayer Distance

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Abstract
In order to minimize harmful effect of synthetic dye (direct yellow and direct violet) the capacity of Zn/Al, Zn/Cr and Zn/Fe layered double hydroxides of these contaminant was studied in this work. Batch adsorption experiment was conduct to investigate the effect of various operating parameters such as contact time, initial dye concentration and adsorption temperature in order to provide optimal condition in removal synthetic dye. Based on result, the sorption of direct dye onto LDHs followed pseudo-second-order rate model. The equilibrium adsorption data for both direct dye was fitted Freundlich isotherm model.

Keywords
adsorption, synthetic dye, layered double hydroxide, kinetic, thermodynamic

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1. INTRODUCTION

Water is the most important for all living organism, development and also the environment (Gu et al., 2018). Unfortunately, now days water source has been containing many pollutants (Faust and Aly, 2018). Its caused by many industries doesn't care about waste, they throwing away their waste in water irrigation, a small river and also in pond retention (Faust and Aly, 2018). That's why water treatment become a serious problem environment and also many researchers are search the effective ways to get clean water (Guimares et al., 2019; Boubakri et al., 2018; Palapa et al., 2018). However, rapidly demand new and diverse products based on technology influences many industries more prepared to using synthetic chemicals (Kefif et al., 2019), which is increasing the pollutant and harmful to human and environment (Oladipo et al., 2019).

Nowadays, almost 78% of industries such as textile, paper, leather, paints, food and also cosmetics are using synthetic dyes (Faust and Aly, 2018; Marzbali et al., 2017). In the large scale, synthetic dye continuously causing environmental water gets worse (Taher et al., 2019b). Indeed the presence of these substances in the aquatic environment reduces the oxygen and disturbs the biological cycle of aquatic biota (Taher et al., 2019b; Kefif et al., 2019). Moreover, the presence of a pollutant in drinking water is more dangerous to human, its can caused reproductive system disorder, caused cancer liver, brain, mutagenic and carcinogenic (Palapa et al., 2018; Saria et al., 2018; Faust and Aly, 2018). Generally, many textile industries used direct dye, due to their excellent binding ability (Boubakri et al., 2018).

Removal the pollutant into wastewater has been studied taking the efficiency and cost, various methods such as biological treatment (Faust and Aly, 2018), membrane filtration (Xiao et al., 2019), electrochemical, photochemical (Zhu et al., 2018), coagulation (Huang et al., 2019), degradation and adsorption (Taher et al., 2019a). Most of them have high cost and not easy to operation, except, adsorption. The adsorption process has widely used, easy to operation, low cost and high effectiveness water treatment. In fact, the main weakness of the adsorption method is the high cost and impurity of the conventional sorbent. Hence, cheaper and effective sorbent is still required to solve this problem (Miandad et al., 2018). Now, researchers have been focused on the development of natural clays.

Layered double hydroxides are the one of synthetic clay mineral which has ability to removal various contaminant from aqueous solution. Layered double hydroxides (LDHs) has consist of brucite layers by metal cation divalent and trivalent substituted (Palapa et al., 2019). Generally, LDHs has formula $[M^{2+}(1-x)M^{3+}x(OH)_2](An-)x/nH_2O$, (denoted as M^{2+} and M^{3+} , where M^{2+} is a divalent metal cation, such as Mg^{2+}, Fe^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, Ca^{2+}, and M^{3+} is a trivalent metal cation, such as Al^{3+}, Cr^{3+}, Ga^{3+}, Mn^{3+} or Fe^{3+}. LDHs has unique structure properties favorable for adsorption of dye, which is they have specific features like no toxic, anion exchange properties, good thermal stability, has high flexibility, high surface area, and easy to han-

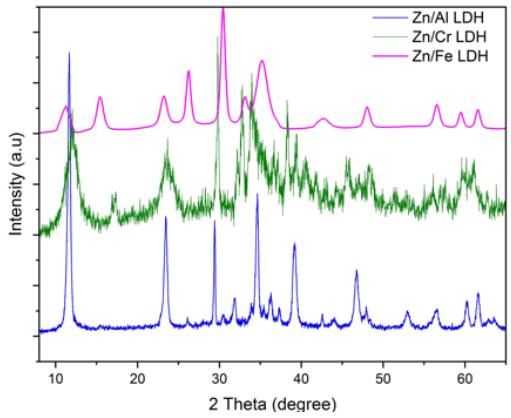

Figure 1. XRD Pattern of Zn/Al, Zn/Cr and Zn/Fe LDHs

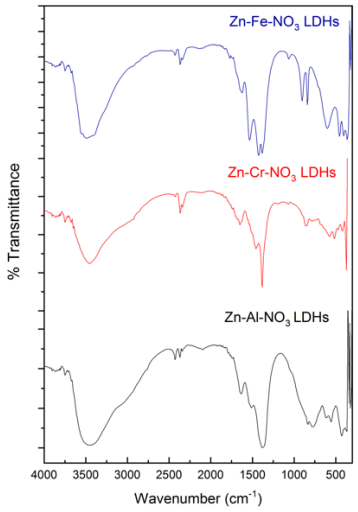

Figure 2. FTIR Spectra of Zn/Al, Zn/Cr and Zn/Fe LDHs

dling (Taher et al., 2019a; Palapa et al., 2019; Boubakri et al., 2018; Nidheesh et al., 2018; Tajuddin et al., 2018).

In this work, LDHs was made to study of removal ability direct dye onto LDHs from aqueous solution. LDHs has been prepared by co-precipitation method and characterized using XRD, FTIR and Surface Area Analyses by BET method. the adsorption factors performance such as time, initial dye concentration and temperature was investigated. Sorption capacity was studied using kinetic laws and isotherm models.

2. EXPERIMENTAL SECTION

2.1 Materials

The chemical used in this experiment are analytical grade, such as zinc nitrate, aluminum nitrate, iron nitrate, chromium nitrate and also sodium nitrate was purchased by Merck, dyes were obtained in local textile industry in Palembang and the water was used as solvent was obtained by purifying water de-ionized

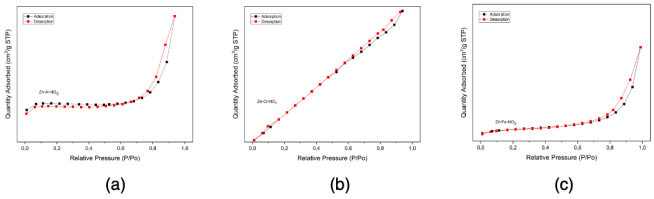

Figure 3. Isotherm Graphic adsorption-desorption of (a) Zn/Al, (b) Zn/Cr and (c) Zn/Fe LDHs

'Purite'. LDH was characterized using a Rigaku Miniflex 600, Shimadzu Prestige-21 and BET (ASAP Micromeritics 2020).

2.2 Methods

2.2.1 Synthesis of Layered Double Hydroxide

According to Palapa et al. (2019), the synthesis of layered double hydroxide was conducted to alkaline precipitation with divalent and trivalent metal cation ratio 3:1 at nitrogen atmosphere. Prepare the alkaline solution for zinc nitrate (0.75 M), aluminum nitrate (0.25 M), chromium nitrate (0.25 M) and also iron nitrate (0.25 M). divalent and trivalent metal cation are mixed as long as an hour and added to 50 mL of sodium hydroxide (2 M) with continuous stirring 240 rpm for 4 hours at 60 °C. then the solution was adjusted at pH 10 using sodium hydroxide and let the mixing solution at 60 °C overnight. After that, the solution kept in oven at 80 °C until we obtain gel, then LDH was filtered, washed for several times using de-ionized water and finally dried at 60 °C for 24 h. The obtained solid then keep in a sealed bottle and ready for further used as an adsorbent.

2.2.2 Adsorption Experiment

As much as 0.05 g of adsorbent was added to 50 mL of dyes solution in a certain concentration, in this work we used direct yellow and direct violet dye as the adsorbate. The dye solution was prepared by 1 g of each dye were diluted into 1000 mL (as stock solution) then dissolve gradually. Then 0.05 g adsorbent added into 50 mL adsorbate and shaken with specified time at 240 rpm. After that, the solution was filtered and the final concentration was calculated using UV-Vis spectrophotometer.

3. RESULTS AND DISCUSSION

3.1 Adsorbent Characterization

Zn/Al, Zn/Cr and Zn/Fe layered double hydroxides (LDHs) were successfully prepared and each LDHs were characterized by XRD, FT-IR and Surface Area Analyses. The result of X-ray Diffraction was shown in Fig. 1. The pattern was shown the unique diffraction of LDHs amount 10°, 22°, 30°, 35°, 49° and overlapping peaks at 60° are characterized by LDHs. For all LDHs, the sharp peaks at amount 10o were indicated interlayer of LDHs with higher distance is Zn/Al 7.57 Å, Zn/Cr 7.32 Å and Zn/Fe 5.80 Å. Then all of LDHs has been characterized using FT-IR and the spectra were shown in Fig 2. Zn/Al, Zn/Cr and Zn/Fe spectra have identically vibration were indicating nitrate band, OH band and M-O band. All identical spectra were shown in Fig

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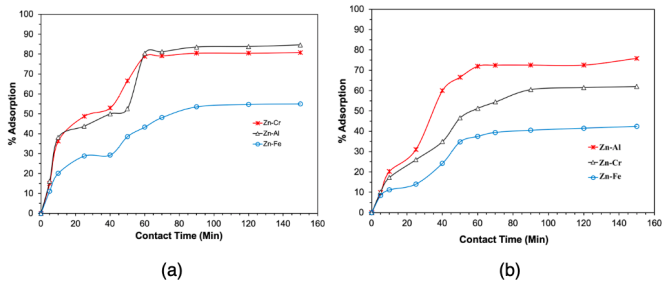

Figure 4. Effect of contact time adsorption (a) direct yellow and (b) direct violet onto layered double hydroxides (LDHs)

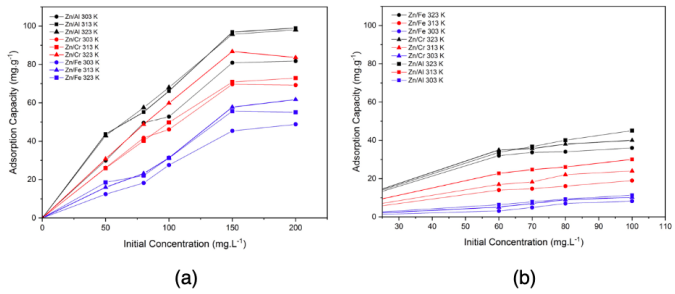

Figure 5. Effect of initial concentration of (a) direct yellow dye and (b) direct violet dye onto LDHs at various temperature

2. amount 3400-3600 cm^{-1} for OH band, a vibration of nitrate anions at 1380 cm^{-1} and vibration under 1000 cm^{-1} generally indicate M-O band.

According to Boubakri et al. (2018); Taher et al. (2019a), layered double hydroxides has much potential application its because layered double hydroxides have unique structure as layer materials which is interlayer easily to exchangeable. So, in this work, LDHs were characterized by surface area analyses by BET method. the result of each LDHs shows in Fig 3 and Table 1. Fig. 3 were shows the curve of adsorption and desorption of Zn/Al, Zn/Cr and Zn/Fe LDH, respectively. The typical nitrogen adsorption-desorption isotherms and the corresponding pore size distribution and the Fig. 3 indicating Zn/Al, Zn/Cr and Zn/Fe LDHs have mesoporous pore. Table 1. Listed the volume pore and pore size based on digital data of characterization. According to IUPAC, diameter pore of material such as micropore has pore <2 nm, mesopore between 2-50 nm and macropore >50 nm.

Table 1. Data of volume pore and pore size of layered double hydroxides

Adsorbent	SBET (m^2/g)	Volume Pore (BJH, cm^3 /g)	Pore Size (ϕ, nm)
Zn-Al-NO_3	9.4128	0.045351	19.2743
Zn-Fe-NO_3	2.8834	0.010334	14.6964
Zn-Cr-NO_3	11.865	0.112128	3.77051

3.2 Adsorption Study

3.2.1 Kinetic Adsorption Study

Effect of adsorption time of direct dyes by Zn/Al, Zn/Cr and Zn/Fe LDHs was shown in Fig. 4. The adsorption process of direct dye onto layered double hydroxides was setting up at room temperature and 100 mg/L adsorbate for each LDHs. The result of adsorption was calculated using UV-Vis Spectrophotometer is the final concentration at 403 nm and 549 nm. After that, we calculated the % adsorbed, then we identify the kinetic parameters adsorption process. The maximum adsorption time direct yellow onto Zn/Al, Zn/Cr and Zn/Fe LDHs at an hour with % adsorbed are amount 83%, 81% and 38%, respectively. However the adsorption direct violet onto Zn/Al, Zn/Cr and Zn/Fe LDHs have maximum adsorption time at an hour amount 71%, 49% and also 27%, respectively. Based on result, layered double hydroxides more effective to adsorb direct yellow dye than direct violet dye. In this work, we fitted two kinetic models. i.e. pseudo-first-order and pseudo-second-order. These kinetic models equation as follows:

Pseudo-first-order:

$$log(Qe - Qt) = logQe - k_1/(2.303)t \quad (1)$$

and pseudo-second-order:

$$t/Qt = (1/k_2 Qe^2) + (1/Qe)t \quad (2)$$

where qe and qt are capacities of adsorbed ($mg.g^{-1}$), t is time adsorption process, $k_1$is the rate constant of the first order and k_2 the rate constant of second-order. Table 2. Shows the data of kinetic parameters of direct dyes adsorption onto LDHs.

Based on coefficient correlation of direct dye adsorption onto layered double hydroxides was followed pseudo-second-order kinetic model. Generally, the adsorption of dye onto LDHs is quite rapidly initially until the equilibrium was reached and the value of adsorbed are constant. Its caused, active site of layered double hydroxides at equilibrium are full than before reached equilibrium which is the surface active site is more available.

3.2.2 Isotherm and Thermodynamic Study

The thermodynamic was studied by varying initial concentration and temperature. In Figure 5, dye adsorbed is higher at temperature 343 K so that the interaction of adsorbate and adsorbent are higher than solvent-adsorbent at active site adsorption. Therefore, the adsorption for both direct dye onto LDHs at varying temperature were shown in Fig. 5. Fig 5 were shows the effect of initial concentration at various temperature. The higher adsorption capacity of both dyes (direct yellow and direct violet) is Zn/Al layered double hydroxides at 323 K it's amount 98 mg/g and 36 mg/g, respectively.

The isotherms models Langmuir and Freundlich are used for this data. The Langmuir assumed that adsorbate was occupied into monolayer. Its used equation as follows:

$$Ce/Qe = 1/(K_L Qmax) + (1/Qmax)Ce \quad (3)$$

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Table 2. Pseudo-first-order and pseudo-second-order sorption rate constant kinetic models.

Kinetic models	Dye	Parameters	Layered double hydroxides		
			Zn-Al-NO_3	Zn-Cr-NO_3	Zn-Fe-NO_3
Pseudo-first-order	Direct Yellow	k_1	0.0597	0.0493	0.0339
		R^2	0.9127	0.873	0.891
Pseudo-second-order		k_2	0.0064	0.005	0.0072
		R^2	0.9544	0.9077	0.9176
		qe exp	80.791	84.565	54.965
Pseudo-first-order	Direct Violet	k_1	0.0414	0.0364	0.0339
		R^2	0.938	0.922	0.912
Pseudo-second-order		k_2	0.004	0.0045	0.0072
		R^2	0.9297	0.9883	0.948
		qe exp	75.791	62.012	44.056

Table 3. Langmuir and Freundlich Isotherm Model Fitted for adsorption direct yellow onto LDHs

Adsorbent	Isoterm	Parameters	T (K)		
			303	313	323
Zn/Al	Langmuir	qmax (mg.g-1)	100.706	118.7815	121.3768
		kL (Lmg^{-1})	0.009	0.0328	0.0515
		R^2	0.6023	0.7954	0.905
	Freundlich	kF ($mg.g^{-1}$)(Lmg^{-1})1/n	3.5136	13.506	19.5108
		n	1.3646	2.3814	2.6625
		R^2	0.9025	0.9045	0.9363
Zn/Cr	Langmuir	qmax (mg.g-1)	86.4756	91.1563	104.3262
		kL (Lmg^{-1})	0.0052	0.0041	0.0033
		R^2	0.6999	0.992	0.9855
	Freundlich	kF ($mg.g^{-1}$)(Lmg^{-1})1/n	1.8634	1.788	1.8525
		n	1.2733	1.183	1.133
		R^2	0.9734	0.998	0.9986
Zn/Fe	Langmuir	qmax ($mg.g^{-1}$)	39.654	39.2063	32.8447
		kL (Lmg^{-1})	0.034	0.0228	0.0443
		R^2	0.6075	0.89	0.9793
	Freundlich	kF ($mg.g^{-1}$)(Lmg^{-1})1/n	0.1918	1.7759	2.4524
		n	0.7738	1.5642	1.6492
		R^2	0.9699	0.9081	0.9848

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Table 4. Langmuir and Freundlich Isotherm Model Fitted for adsorption direct violet onto LDHs

Adsorbent	Isoterm	Parameters	T (K) 303	313	323
Zn/Al	Langmuir	qmax ($mg.g^{-1}$)	39.654	45.2025	66.093
		kL (Lmg^{-1})	0.1472	0.1246	0.0387
		R^2	0.9808	0.9473	0.998
	Freundlich	kF ($mg.g^{-1}$)(Lmg^{-1})1/n	20.5059	20.6027	9.1501
		n	7.4144	6.189	2.5095
		R^2	0.9902	0.9942	0.998
Zn/Cr	Langmuir	qmax ($mg.g^{-1}$)	37.6209	56.7362	47.5136
		kL (Lmg^{-1})	0.0122	0.0099	0.0239
		R^2	0.9202	0.7694	0.9839
	Freundlich	kF ($mg.g^{-1}$)(Lmg^{-1})1/n	1.6529	1.5044	4.6399
		n	1.8141	1.544	2.2851
		R^2	0.9621	0.902	0.987
Zn/Fe	Langmuir	qmax (mg.g-1)	23.709	28.332	31.095
		kL (Lmg^{-1})	0.0071	0.0046	0.0409
		R^2	0.847	0.713	0.829
	Freundlich	kF ($mg.g^{-1}$)($Lmgg^{-1}$)1/n	2.604	6.882	9.571
		n	1.303	1.265	2.507
		R^2	0.992	0.949	0.906

Table 5. Values of Thermodynamic Parameters for The Adsorption of Direct Yellow By LDHs

T (K)	C (mg/L)	Zn/Al LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)	Zn/Cr LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)	Zn/Fe LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)
303	60	-1.049	64.961	18.634	-2.975	39,00	9,819	-0,989	32,01	8,71
313		-2.348			-5.714			-1,282		
323		-3.647			-0.009			-1,575		
303	70	-0.199	45.216	13.501	-2.18	39,75	5,223	-0,990	26,47	4,50
313		-1.104			-4.351			-1,631		
323		-2.008			-0.227			-2,271		
303	80	-0.245	46.498	13.844	-2.975	35,59	9,650	-1,355	17,879	7,35
313		-1.175			-5.714			-1,742		
323		-2.105			-0.009			-2,129		
303	90	-0.378	30.336	8.814	-2.18	47,50	9,311	-0,838	11,06	1,96
313		-0.984			-4.351			-1,379		
323		-1.591			-0.227			-1,920		
303	100	-0.831	28.635	7.093	-0.009	33,402	6.991	-0.012	12.3543	3.889
313		-2.075			-2.18			-1.05		
323		-3.318			-4.351			-2.087		

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Table 6. Values of Thermodynamic Parameters for The Adsorption of Direct Violet By LDHs

T (K)	C (mg/L)	Zn/Al LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)	Zn/Cr LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)	Zn/Fe LDH ΔG (kJ/mol)	ΔS (J/mol.K)	ΔH (kJ/mol)
303	60	-1.049	64.961	18.634	-2.975	39,00	9,819	-0,989	32.01	8.71
313		-2.348			-5.714			-1,282		
323		-3.647			-0.009			-1,575		
303	70	-0.199	45.216	13.501	-2.18	39,75	5,223	-0,990	26.47	4.5
313		-1.104			-4.351			-1,631		
323		-2.008			-0.227			-2,271		
303	80	-0.245	46.498	13.844	-2.975	35,59	9,650	-1,355	18	7.35
313		-1.175			-5.714			-1,742		
323		-2.105			-0.009			-2,129		
303	90	-0.378	30.336	8.814	-2.18	47,50	9,311	-0,838	11.06	1.96
313		-0.984			-4.351			-1,379		
323		-1.591			-0.227			-1,920		
303	100	-0.831	28.635	7.093	-0.009	33,402	6.991	-0.012	12.3543	3.889
313		-2.075			-2.18			-1.05		
323		-3.318			-4.351			-2.087		

Where qe is the equilibrium adsorption, Ce is equilibrium concentration, qmax is the maximum adsorption and kL is the equilibrium adsorption constant. Then, the essential features of Langmuir isotherm namely RL (equilibrium parameters). Value RL has indicated the models of isotherm. If irreversible, the RL calculated zero (RL = 0), liniear when RL = 1, and favorable when 0> RL>1. The Freundlich isotherm model identified the heterogenous adsorbent surface. The equation is following:

$$Logqe = LogkF + nLogCe \quad (4)$$

Where k_F is adsorption capacity when equilibrium, the value of n gives information of favorability of adsorption process, if n=1 linear, n<1 is chemisorption and n>1 is favorable.

The isotherm data were shown in Table 3. Tabel 3 was shown the parameters of Freundlich and Langmuir. The result was shows Freundlich model is more fitted of the experimental data based on coefficient correlation. This result indicated the adsorbate interact physisorption each other on surface site active of layered double hydroxides. Thermodynamic study parameter was calculated and obtained enthalpy, entrophy and free Gibbs Energy. The value of enthalphy and entrophy shows that the adsorption process is endothermic. Negative free energy value was indicated that the process is spontaneous and the desreases of free energy value with the increases of temperature indicated that the adsorption more favorable at low temperature (room temperature).

4. CONCLUSIONS

In this work, Zn/Al, Zn/Cr and Zn/Fe has been used as adsorbent for removal dyes in aqueous solution. Based on result, Zn/Al LDHs has higher adsorption capacity than Zn/Cr and Zn/Fe LDHs. For removal direct dyes, LDHs more effective adsorb direct yellow dye than direct violet dye. Kinetics study showed that the adsorption process is more fits with PSO than PFO based correlation coefficients. The adsorption process is described by Freundlich isotherm models for all dyes. The value of enthalpy and entropy shows that the adsorption process is endothermic and negative free energy value was indicated that the process is spontaneous.

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