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Kinetic Aspect of Direct Violet Adsorption on M^{2+}/M^{3+} (M^{2+}: Zn; M^{3+}: Al, Fe, Cr) Layered Double Hydroxides

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Abstract. As we know, synthetic dye such as direct violet is harmful to the environment. Synthetic dyes were adsorption using layered double hydroxides has been studied. The layered double hydroxides used were Zn/Al, Zn/Fe, and Zn/Cr, which were successfully synthesized using the co-precipitation method by adjusting at pH 10. Three obtained layered double hydroxide samples were characterized using X-ray Diffraction, Fourier Transform Infrared Spectroscopy, and surface area analysis by BET methods. The adsorption of direct violet onto Zn/Al, Zn/Fe and Zn/Cr layered double hydroxides. Based on the kinetics study, the result was tested using the *pseudo-first-order* and *pseudo-second-order*. The best models fit obtained with *pseudo-second-order* kinetics models.

Keywords: Layered double hydroxides, dyes removal, adsorption, kinetic study

INTRODUCTION

In recent years, industrial development harms environment such as dye wastewater is a very crucial problem and difficult to overcome. One of the most contributors to synthetic dyes sewer is textile industries. Synthetic dyestuffs wastewater in the form of stains which are anionic dyes or toxic metal cations [1,2]. Synthetic dyes are usually complex aromatic structures so that these dyes are hard to biodegradable [3]. The existing technologies for the elimination of heavy metals from wastewater include chemical precipitation, phytoextraction, ion exchange, membrane filtration and adsorption. However, all of these methods are expensive and hard to use this method. One of them has the most effective is adsorption method [4,5]. Adsorption has benefited such as easy to applied, high efficiency, and low costs. Adsorption is one of the most important industrial separation processes for the treatment of waste [6]. It is a mass transfer process through which the solid substance (adsorbent) can selectively remove dissolved constituents from an aqueous solution by attracting the dissolved solute toward its surface [7]. The accumulation of concentrated matter at the surface or the interphase is involved in this process. The adsorbent can exist in liquid, solid, gas, or dissolved solute phase [8]. Adsorption can be classified as chemical or physical [9]; the former procedure is due to the exchange of electrons, adsorbate is chemically bonded to the surface [10].

Layered double hydroxide have been considered to the effective adsorbent, as Palapa *et al.* (2018) reported that layered double hydroxides Ni-Al can effectively remove direct dyes in aqueous solution [2]. Layered double hydroxides (LDHs), as a type of anion exchange lamellar materials, have been well studied and widely used in various fields due to its excellent performance on anion exchange and easy preparation such as co-precipitation method. General formula of layered double hydroxides is $[M^{2+}(1-x)M^{3+}x(OH)2](A^{n-})x/nH_2O$, where M^{2+} and M^{3+} are Mg^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Ca^{2+} and Al^{3+}, Cr^{3+}, Mn^{3+}, or Fe^{3+} are divalent and trivalent metal cations, respectively then A^{n-} is anion in interlayer space and (x) is stoichiometry calculation [11]. Layered double hydroxides has positively charged and anion as a balancing counter in interlayer space [2,11]. Layered double hydroxides has a

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unique characteristic such as easy to synthesize, flexible interlayer so LDHs can change according to anion species interlayer.

In this study, the synthesis of Zn-M (M=Al,Fe,Cr) layered double hydroxides were using co-precipitation method. Then, Zn-M (M=Al,Fe,Cr) were using to removal direct violet in aqueous solution. The adsorption was conducted by varying times to get the kinetics aspect. The structure of Zn-M (M=Al,Fe,Cr) layered double hydroxides was analyzed by Fourier transform infrared (FTIR) spectra and X-ray diffraction (XRD).

MATERIAL AND METHODS

Materials

Zinc nitrate $Zn(NO_3)_2 \cdot 6H_2O$ were purchase by Merck, aluminum nitrate $Al(NO_3)_3 \cdot 9H_2O$ by Merck, chromium nitrate $Cr(NO_3)_3 \cdot 9H_2O$ by Merck, iron nitrate $Fe(NO_3)_3 \cdot 9H_2O$ by Merck, sodium hydroxide (NaOH) were obtain by Sigma Aldrich and sample direct violet dye were obtain from textile home industry wastewater Jumputan in Palembang. The solution was prepared by deionized purity water. Then characterized by XRD Diffractometer Rigaku Miniflex-600 and Spectrophotometer FT-IR Shimadzu FT-IR Prestige-21.

Methods

Synthetic methods

Layered double hydroxides has been prepared by co-precipitation method with molar ratio 1:3 (M^{2+}:M^{3+}) at constant pH. Metal solution zinc nitrate (100 mL, 3M) and M=Al,Cr,Fe (100 mL, 1M) was called A solution were added simultaneously with NaOH into boiled deionized water (10 mL) flowed nitrogen. Kept pH ten while 24 hours at 70 ^oC vigorous stirring. Then, layered double hydroxides has been obtain and kept dry at room temperature.

Adsorption experiment

The adsorption of direct violet on Zn-M (M=Al,Cr,Fe) layered double hydroxides was examined using a batch system, and kinetic parameters was evaluate. The stock solution (1000 mg/L) was prepared by 1 g of direct violet powdery dye the diluting with deionized water till 100 mL. After that, the sample solution (100 mg/L) has been prepared by gradually diluted. 0.05 g of Zn-M (M=Al,Cr,Fe) added to the sample solution. Then, placed to horizontal shaker at 250 rpm and shaken varying times (0, 5, 10, 25, 40, 50, 60, 70, 90, 120 and 150 minutes) until the equilibrium are reach. In kinetic study, the experiment can calculated by the eq:

$$q_t = (C_i - C_t) \text{ x } V/M \qquad (1)$$

where, C_i and C_t are direct violet initial concentration and concentration of time (mg/L), M is the mass (g) of Zn-M (M=Al,Cr,Fe) layered double hydroxides and V is volume (L) of solution. The schema of the adsorption process was shown in Fig. 1. The dye concentrations are measured by UV–vis spectrophotometer at 548 nm. The calibration curve was run range 5-25 mg/L (five points) has linear R^2= 0.998 before analyses. The linear Lambert-Beer relationships were used in the concentration analysis.

RESULT AND DISCUSSION

Characteristics of materials

The Zn-M (M=Al,Cr,Fe) layered double hydroxides has successfully synthesized and characterized using XRD and FTIR. The XRD pattern was shown in Fig. 2. The XRD pattern of Zn-M shows the sharp and symmetric peaks at lower 2θ values, which represent the typically characteristic reflections of layered double hydroxides [12]. The lower peaks at 2θ (about 9 to 11^o) reflections (003) were indicating interlayer distances of layered double hydroxides [2,4,10]. The interlayer distance was a difference because of the radii effect of metal cations. The higher value of

interlayer is Zn/Cr (7.68 Å) > Zn/Al (7.57 Å) > Zn/Fe (5.80 Å). the effect of ionic radii affects the distance between layers (interlayer) due to the difference in ionic radii where metal cation trivalent Cr^{3+} (0.64 Å), Al^{3+} (0.53 Å) and Fe^{3+} (0.61 Å) according to Forano *et al.* (2013) the influence of the biggest metal ionic radii will be transformed interlayer higher than interlayer of smaller metal ionic radii [13]. The FTIR spectra of Zn-M (M=Al,Cr,Fe) layered double hydroxides are showed in Fig. 3. All compounds had identical spectra where a broadband vibration from -OH stretching amount wavenumber 3400-3600 cm^{-1} while the weak vibration from water molecules in interlayer at wavenumber 1635 cm^{-1}. Then, the sharp vibration at wavenumber 1380 cm^{-1} denotes the anion of nitrate as balancing in the interlayer. The M-O bonding vibration was identify at wavenumber under 1000 cm^{-1}.

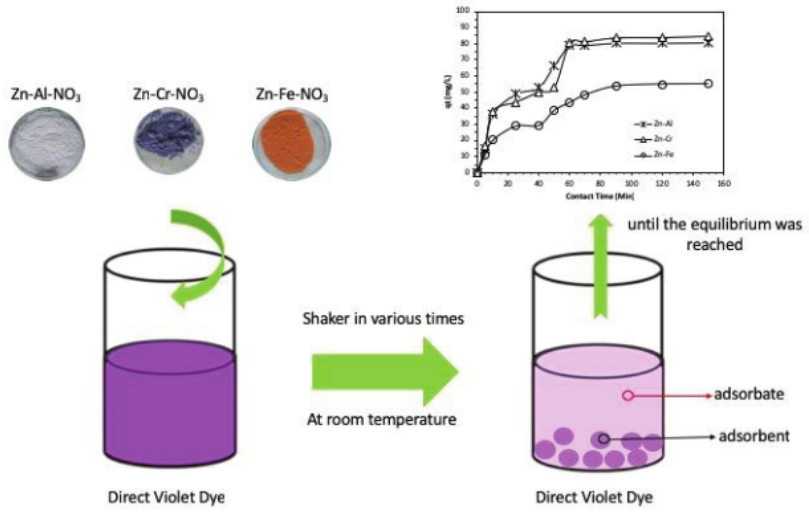

FIGURE 1. The schematic of the adsorption process direct violet into Zn-M (M=Al,Cr,Fe) layered double hydroxides in aqueous solution.

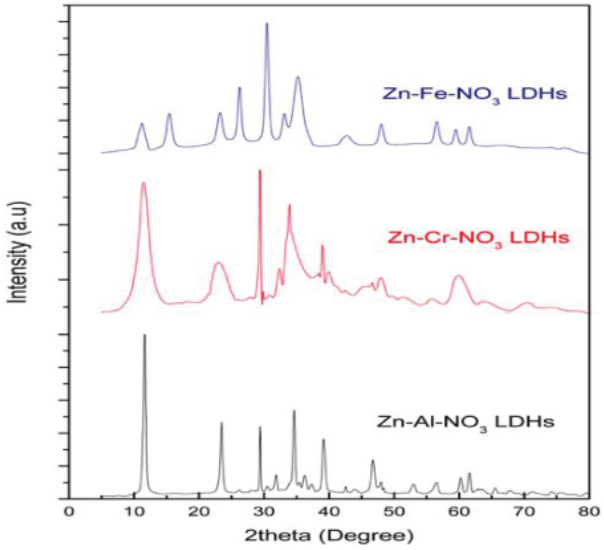

FIGURE 2. The XRD pattern of Zn-M (M=Al,Cr,Fe) layered double hydoxides.

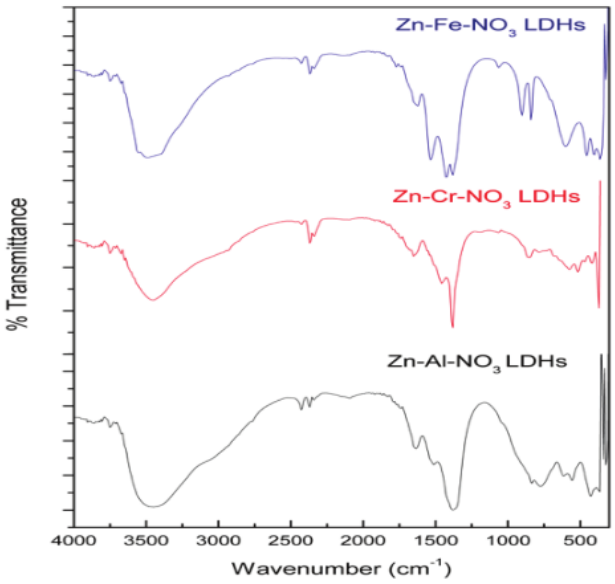

FIGURE 3. Spectra FT-IR of Zn-M (M=Al,Cr,Fe) layered double hydoxides.

Adsorption kinetic modeling

The adsorption kinetics is a significant factor for the tricky adsorption process and is essential for choosing the optimum operating conditions for adsorbent-adsorbate interaction [14,15]. To comprehend the behavior of the adsorbent and to investigate the controlling mechanism of the adsorption procedure, the pseudo-first-order, pseudo-second0order and intraparticle diffusion models are useful to check the kinetic information. According to Aguiar *et al*. (2013) were reported that layered double hydroxides has a greater coefficient correlation of pseudo-second-order than pseudo-first-order kinetic model [16]. Usually, the adsorption of dyes onto layered double hydroxides quite rapidly initially until reached equilibrium and the value is constant. In this case, were explained by the reason that a large number of vacant surface sites were available for adsorption during initial stage. In Fig. 4 shows the variation amount of absorbed (q_t) as a function of time. The result of the kinetics model fitted was shown in Table 1. The kinetic parameters are calculated using the pseudo-first-order:

$$\log (q_e - q_t) = \log q_e - k_1/(2.303)t \quad (2)$$

and pseudo-second-order equations as follows:

$$t/q_t = (1/k_2 q_e^2) + (1/q_e)t \quad (3)$$

where q_t is the amount of adsorbate adsorbed at time t (mg g^{-1}), q_e is the adsorption capacity in the equilibrium (mg g^{-1}), k_1 is the pseudo-first-order rate constant (min^{-1}), k_2 is the pseudo-second-order rate (g $mg^{-1}min^{-1}$) and t is the contact time (min).

Fig. 4. Were show the effect of contact time direct violet sorption onto Zn-Al-NO_3, Zn-Cr-NO_3 and Zn-Fe-NO_3 layered double hydroxides. The adsorption of direct violet onto layered double hydroxides quite rapidly increases in an hour and then slowly grows before reached equilibrium in 90 minutes to Zn-Al-NO_3, Zn-Cr-NO_3 and Zn-Fe-NO_3. In this case, these results were show that Zn-Cr-NO_3 and Zn-Al-NO_3 are better to adsorb direct violet than Zn-Fe-

NO_3 it because the interlayer space was work on adsorption process which is the most significant interlayer space would be higher to adsorb dyes.

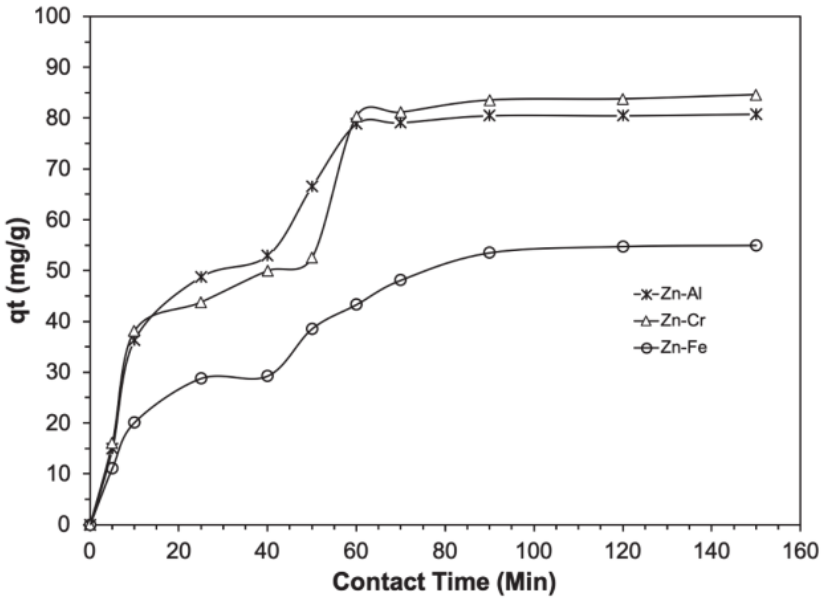

FIGURE 4. Effect of contact time on direct violet onto layered double hydroxides.

TABLE I. Pseudo-first-order and pseudo-second-order sorption rate constant kinetic models.

Kinetic models	Parameters	Layered double hydroxides		
		Zn-Al-NO_3	Zn-Cr-NO_3	Zn-Fe-NO_3
Pseudo-first-order	k_1	0.0597	0.0493	0.0339
	R^2	0.9127	0.873	0.891
Pseudo-second-order	k_2	0.0064	0.0050	0.0072
	R^2	0.9544	0.9077	0.9176
	q_e	80.791	84.565	54.965

The calculated parameters of the kinetic model were shown in Table 1. All the experimental data were shows pseudo-second-order has a good coefficient correlation with determination coefficient values higher than 0.9. It means, pseudo-second-order kinetic model is more fitted to the experimental data as compared to pseudo-first-order.

CONCLUSION

This research has been successfully prepared by the co-precipitation method and characterized by XRD and FTIR analyses. Zn-Al-NO_3, Zn-Cr-NO_3 and Zn-Fe-NO_3 layered double hydroxides was applied to adsorb direct violet in aqueous solution. The kinetic model are fitted by pseudo-second-order and pseudo-first-order. Based on the experiment, the results were shown that Zn-Al-NO_3, Zn-Cr-NO_3 and Zn-Fe-NO_3 layered double hydroxides more fit using pseudo-second-order model and after an hour, the adsorption reaches equilibrium.

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