C.4b.6-Intercalation ZnCr Layered Double Hydroxides with K3 as Adsorbent of Rhodamine-B.pdf

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Research Paper

Intercalation Zn/Cr Layered Double Hydroxides with $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ as Adsorbent of Rhodamine-B

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Abstract

Synthesis of Zn-Cr layered double hydroxide intercalated with polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ was conducted successfully. The characterization of layered double hydroxide intercalated was performed using XRD and FT-IR analyses. After that layered double hydroxide intercalated used as an adsorbent of rhodamine B (RhB) dye. The results show Rhodamine-B (RhB) more effective adsorbed using the Zn-Cr adsorbent $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ than using an intercalated adsorbent $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ because the distance between the layers is smaller. Study of kinetics showed that the adsorption process is more fits with PSO than PFO based on coefficients correlation. The adsorption process described by Langmuir isotherm models for all adsorbent. The value of enthalpy and entropy shows that the adsorption process was endothermic and negative free energy value was indicated that the process was spontaneous.

Keywords

layered double hydroxides, intercalation, Keggin ion, rhodamine-B

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1. INTRODUCTION

The rapid economic growth has caused environmental dis-order with huge pollution problems. The demand for water has increased tremendously in agricultural, industrial and domestic sectors. This has resulted in the generation of large amounts of wastewater containing a high number of contaminants. Many industries, such as textiles, paper, and plastic, use dyestuffs in order to color their products (Aljeboree et al., 2017). Dyes that are often used in the textile industry rhodamine B (RhB). Dyes are resistant to direct biodegradation and photolysis and many N-containing dyes can experience degradation of undergoing natural reductive anaerobic degradation to yield potentially carcinogenic aromatic amines (Laipan et al., 2017). If rhodamine B (RhB) dye accumulates in the human body it can cause some health problems in humans (Han et al., 2016).

Many methods have been developed to remove dyes from wastewater including membrane filtration, precipitation, coagulation, flocculation, chemical oxidation, solvent extraction, biodegradation and adsorption (Taher et al., 2018). The adsorption method is an efficient technique to deal with the problems of domestic and industrial wastewater contamination because it can eliminate odors and reduce the level of the dye from the solution perfectly without turning it into a more dangerous compound (Gaini et al., 2009; Palapa et al., 2019b). One process for removing dyes through the adsorption process uses porous and layered materials, for example, layered double hydroxide. Layered double hydroxide compounds are compounds which in structure occur partial substitution of trivalent metal with divalent metal. This substitution creates a positive charge in the hydroxide layers (Palapa et al., 2019a; Tichit et al., 2019).

The advantages of this double layer hydroxide have great anion exchange properties and can be exchanged for various other anions (Antonyraj et al., 2010; Guo et al., 2013). The general formula for double layer hydroxide is $[M^{2+}{}_{(1-x)}M^{3+}x(OH)_2](An^-)x/n.nH_2O$ which in that positive charges are balanced by the interlayer anions such as Cl^-, NO_3^- and CO_3 (Antonyraj et al., 2010; Palapa et al., 2018). However, The use of these layered materials still has small surface area constraints and narrow layer spacing due to the small exchange ions which are generally alkali and alkaline earth metal ions (Han et al., 2016). Layered double hydroxide still needs to be modified to increase it's surface area and majority modifications made to this research are through intercalation of layered materials with atoms, molecules and complex compounds using ion exchange methods.

In this research, synthesis, and characterization of layered double hydroxide, polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ and layered double hydroxide interca-

lated polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]$ nH_2O has been done. Characterization has been conducted using XRD and FT-IR. Zn-Cr layered double hydroxide intercalated polyoxometalate compound $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]$ nH_2O can be used as rhodamine B (RhB) dye adsorbent. The factors that influence the adsorption process to be studied in this study are the effects, adsorption time, concentration and temperature. The investigation of kinetic, thermodynamic and isotherm adsorption was discussed.

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentation

Layered double hydroxide intercalated with $K_3[\alpha\text{-}PW_{12}O_{40}]$ nH_2O and $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ was performed by ion-exchange method. Characterization of the synthesized compounds was performed by different techniques such as FTIR and X-Ray Diffractometer (XRD) FTIR spectrophotometer was conducted using Shimadzu FTIR Prestige21 using KBr disk and scanning was performed at wavenumber 400-4000 cm^{-1}. XRD was conducted using Rigaku Miniflex-600 and scanned at 0.1 deg min^{-1}. Analysis of rhodamine B (RhB) was used UV-Vis spectrophotometer double beam Biobase BK-UV 1800.

2.2 Preparation of Zn-Cr LDH

Zn-Cr layered double hydroxides were synthesized by coprecipitation method at constant pH 10. $Zn(NO_3)_2 \cdot H_2O$ (100mL, 0.5 M) and $Cr(NO_3)_3 \cdot H_2O$ (100 mL, 0.25 M) with the molar ratio 2:1 were stirring for an hour. Then, 100 mL of sodium carbonate solution 2.5 M at 60 °C was added slowly. The solution was added with 25 mL of sodium hydroxides 3 M and the mixtures were slowly stirring at 60 °C for 24 hours. The solution kept at pH 10 to form gray solid bulky material. After that, the gray material was washed with water and kept overnight at 60 °C to obtain Zn-Cr layered double hydroxides. Characterization of Zn-Cr layered double hydroxides was performed using XRD powder analysis and IR spectroscopy (Oktriyanti et al., 2019).

2.3 Preparation of Polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]nH_2O$

125 g of sodium tungstate and 20 g of sodium phosphate were mixed with 187.5 ml of boiling water in 500 mL of a glass beaker. 100 ml of hydrochloric acid is added dropwise to the mixture and stirred using a magnetic stirrer. The stirring process is continued until all the solids dissolve. The phosphotungstate acid will begin to separate when half of the hydrochloric acid is added then the resulting solution becomes clear and cooled. A 75 mL of diethyl ether cold solution was added and then extracted. After the extraction process obtained three layers, the solution separated and taken from the bottom layer. The lowest layer was evaporate using a rotary evaporator to obtain a white solid that is $K_3[\alpha\text{-}PW_{12}O_{40}$. Characterization of $K_3[\alpha\text{-}PW_{12}O_{40}$ was performed using XRD and FT-IR spectrophotometer analysis.

2.4 Preparation of Polyoxometalate $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$

11 g of sodium metasilicate in 100 mL of water which is used as a solution. 182 g of sodium tungstate is dissolved in 300 mL of hot water and this solution is made as a solution B. 165 mL HCl 4 M was added dropwise for 5 minutes into solution B with strong stirring to dissolve the precipitate from tungstic acid. Then, solution A is added quickly to solution B followed by adding 50 mL of 4M HCl. The solution was maintained for 1 hour at 100 °C at a pH value of 5 to 6. 50 mL sodium tungstate concentrations of 1 M and 80 mL of 4M HCl were added to the solution quickly. This solution is filtered after being cooled at room temperature. The solution is used to obtain salt or α-$SiW_{12}O_{40}$. The potassium salt is obtained by adjusting the solution at a pH value of 5 using 50 g of KCl quickly to obtain white deposits from potassium salts to form $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$. Characterization was performed using XRD and FT-IR spectrophotometer analysis.

2.5 Intercalation of LDH-POM

Intercalation process of layered double hydroxide by polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]$ nH_2O or $K_4[\alpha\text{-}SiW_{12}O_{40}]nH_2O$ by ion exchange method was carried out by preparing 1 g of Polyoxometalate (solution A) mixed with 50 mL of distilled water, and 2 g of double layer hydroxide was added with 25 mL NaOH 1 M (solution B). Solution A and solution B are then mixed rapidly under conditions given N_2 gas for 24 hours. Then the suspension is cooled and the product is washed with water and dried at room temperature. Structural analysis, the thermal stability of the inserted product is carried out using XRD and FT-IR spectrophotometer analysis.

2.6 Adsorption Experiment

Preparing rhodamine B (RhB) solution by 1 g of solid rhodamine B(RhB) powder with 1000 mL of water to obtain 1000 mg/L as dye stock solution. The standard solution prepared by diluting dye stock solution. To obtain a standard curve of the dyes solution, dyes were measured of each standard solution using spectrophotometer UV-Vis at the maximum wavelength, respectively. The adsorption of dyes was conducted in a batch system. An amount of adsorbent was added into 50 mL of dyes solution then mixture using horizontal shaker at 250 rpm at a constant temperature for any predeterminate time.

The solution was filtered and measuring using UV-Vis spectrophotometer. In order to study the effect of time adsorption, the experiment set up as follow. 0.05 g of adsorbent was added to 50 mL of dyes solution then shake at a varying time with temperature 303 °K and the concentration of dyes solution was mg/L. The thermodynamic parameters investigated based on two kinetic parameter models, pseudo-first-order and pseudo-second-order. The thermodynamics studied by varying initial concentration, the experiment set up as follow. 0.05 g of adsorbent added to 50 mL of varying concentration dyes solution. Then shake in varying temperature 303-343 °K.

3. RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns of the products. The pattern in Fig. 1a clearly indicates the layer structure of the Zn-Cr-$K_4[\alpha$-$SiW_{12}O_{40}]$ nH_2O LDHs with characteristic peaks (assigned as) at 9.35°, 34.16° and 60.02° with each basal spacing at 9.45 Å, 4.14

Å, 2.64 Å and 1.53 Å was observed with low intensity, and Fig. 1b shows the interfraction pattern of layered double hydroxide material intercalated polyoxometalate $K_3\alpha$-$PW_{12}O_{40}$ there is the highest diffraction peak that is in the area at 9.36°, 23.34°, 27.90°, 32.98° and 60,17° with each basal spacing of 9.38 Å, 3.81 Å, 3.19 Å, 2.71 Å and 1,54 Å. Estimated the best formation of Zn–Cr–$K_4\alpha$-$SiW_{12}O_{40}$ and Zn-Cr-$K_3\alpha$-$PW_{12}O_{40}$ LDHs (Lafi et al., 2016).

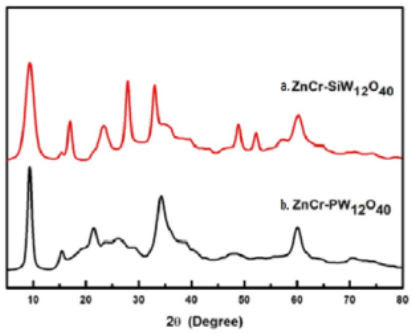

Figure 1. XRD pattern of layered double hydroxides

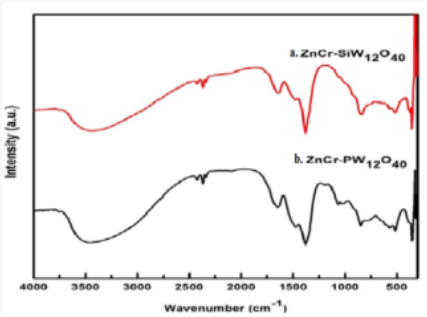

Figure 2. FT-IR spectra of layered double hydroxides

Fig. 2 shows the FT-IR spectra of the synthesized layered double hydroxide material intercalated polyoxometalate $K_3[\alpha$-$PW_{12}O_{40}]$ nH_2O and $K_4[\alpha$-$SiW_{12}O_{40}]nH_2O$. Fig. 2a-2b The intensity at wavenumber 3448 cm^{-1} is assigned to the OH stretching vibration [15]. Wavenumber at 1635 cm^{-1} is assigned as OH bending in the interlayer water molecule. Wavenumber at 354.9 cm^{-1} and 848.68 cm^{-1} is assigned as Zn-O and Cr-O vibration. Wavenumber 840.96 cm^{-1} shows the presence of vibrations from Si-O. Wavenumber 1064,71 cm^{-1} is assigned as P-O vibration. Wavenumber 1026,13 cm^{-1} is assigned as W=O vibration. Wavenumber 516.92 cm^{-1} is assigned as W-Oc-W group vibration (Palapa et al., 2019b; Oktriyanti et al., 2019). Wavenumber of 1381.03 cm^{-1} the width of the peak means that some nitrate anions have been replaced by anions $[\alpha SiW_{12}O_{40}]^{4-}$ and $[\alpha PW_{12}O_{40}]^{4-}$.

Table 1. Kinetics Parameters Adsorption of Dye Rhodamine B (RhB) by LDHs

Kinetics Models	Parameters	Adsorbent Zn-Cr-PW	Zn-Cr-SiW
PFO	Qe Exp (mg/g)	26.826	25.146
	Qe Calc (mg/g)	25.852	31.462
	k_1 (min^{-1})	0,031	0.036
	R^2	0,913	0.868
PSO	Qe Exp (mg/g)	26.826	25.146
	Qe Calc (mg/g)	29.94	27.1
	k_2 (min^{-1})	0.002	0.002
	R^2	0.985	0.97

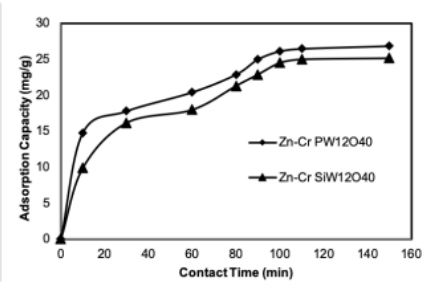

Figure 3. Time adsorption of dye rhodamine B (RhB) by LDHs

3.1 Effect of Contact Time and Kinetic Parameters

Effect of adsorption time of dye rhodamine B (RhB) by Layered double hydroxide intercalated shown in Fig. 3. The adsorption of rhodamine B (RhB) on Zn-Cr-$K_3[\alpha$-$PW_{12}O_{40}]$ nH_2O LDHs showed the adsorption faster than Zn-Cr-$K_4[\alpha$-$SiW_{12}O_{40}]nH_2O$. The maximum of adsorption time on rhodamine B (RhB) by Zn-Cr-$K_3[\alpha$-$PW_{12}O_{40}]$ LDHs at 100 min with maximum uptake 26,099 mg/g whereas the maximum adsorption on rhodamine B (RhB) by Zn-Cr-$K_4[\alpha$-$SiW_{12}O_{40}]nH_2O$ at 100 min with maximum uptake 24,501 mg/g. According to this research. adsorption use Zn-Cr-$K_3[\alpha$-$PW_{12}O_{40}]$ LDHs basal spacing greater, because it is influenced by the distance between layers. the greater the distance between layers, the more adsorbates are adsorbed by the adsorbent because of the more active sites on the adsorbent bind to rhodamine B (RhB) dyes. LDHs is better to remove the anionic dyes (Aguiar et al., 2013; Palapa et al., 2018).

In order to identify the kinetics parameter adsorption process, two kinetic models i.e, Pseudo-first-order and Pseudo-second-order are applied. The kinetic parameters are calculated using the pseudo-first-order and pseudo-second-order equations as follows:

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

$$t/Qt = (1/k_2Qe^2) + (1/Qe)t \quad (2)$$

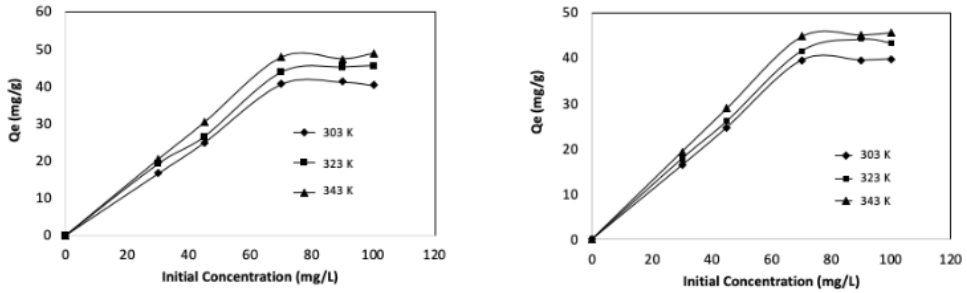

Figure 4. Adsorption isotherm of rhodamine B (RhB) by Zn-Cr- PW LDHs (a) Zn-Cr- SiW LDHs (b) at several temperatures

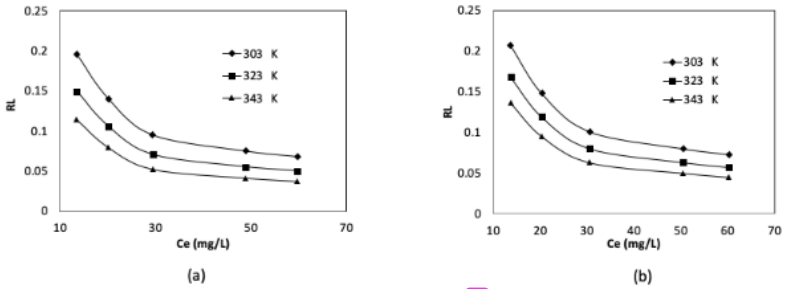

Figure 5. RL versus Ce Langmuir Isotherm Models by Zn-Cr- PW LDHs (a) Zn-Cr- SiW LDHs (b)

Where qe and qt are capacities of adsorbed (mg/g) at equilibrium and t is contact time (min), k_1 is the rate constant of Pseudo-First-Orde. Then, k_2 is the rate constant of pseudo-second-order. He calculated kinetics parameters for rhodamine B (RhB) by Zn-Cr-PW and Zn-Cr-SiW LDHs are given in Table 1. It shows that of the two models kinetic equations, the PSO model is more fit than PFO based on the correction coefficient R^2.

3.2 Adsorption Isotherm and Thermodynamic Study

Fig. 4 showed initial concentration vs concentration adsorbed with varying temperature of rhodamine B (RhB) adsorption by Zn–Cr– SiW and Zn-Cr-PW LDHs. In Fig. 4. Dyes adsorbed are higher at high temperature (343 K) its means the interaction of adsorbate-adsorbent is much stronger than solvent-adsorbent at adsorption sites. Fig. 4 when adding rhodamine B (RhB) concentration of 70, 90 and 100 (mg / L) LDHs has reached its saturation point. This is indicated dyes adsorption capacity of the double layer hydroxy material not experiencing a significant increase in capacity.

The adsorption isotherms were studied, as this provides information about the surface properties of adsorbent, the adsorption behavior and the design of adsorption systems. the process of rhodamine B (RhB) further investigated. The equilibrium isotherm adsorption explains how the adsorbent interacts with the adsorbate and Langmuir models. The Langmuir adsorption model is established on the following hypotheses, uniformly energetic adsorption sites, monolayer coverage, and no lateral interaction between adsorbed molecules. Therefore, at equilibrium, a saturation point is reached where no further adsorption can occur. The basic assumption is that sorption takes place at specific homogeneous sites within the adsorbent. Once a dye molecule occupies a site, no further adsorption can take place at that site (Abdellaoui et al., 2017; Aguiar et al., 2013; Laipan et al., 2017). Its used equation as follows:

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

where Ce is the concentration of adsorbate at equilibrium (mg/L); Qe is the equilibrium adsorption capacity (mg/g); qm is the maximum dye uptake for a complete monolayer (mg/g); and KL is the Langmuir constant (L/mg). The basic features of the Langmuir equation can be expressed in terms of a separation factors RL which is a dimensionless constant defined as:

$$RL = 1/(1 + (K_L Ci)) \quad (4)$$

where Ci is the initial concentration of rhodamine B (RhB) (mg/L) and RL indicates the adsorption nature. When RL >1 the adsorption is unfavorable, while is linear if RL=1, favorable if

Table 2. Langmuir Isotherm Models

Parameter	T= 303 K		T= 323 K		T= 343 K	
Langmuir	Zn-Cr-PW	Zn-Cr-SiW	Zn-Cr-PW	Zn-Cr-SiW	Zn-Cr-PW	Zn-Cr-SiW
Qmax (mg/g)	65.835	64.688	70.146	71,658	67.031	64.737
KL (L/mg)	0.136	0.127	0.189	0.164	0.259	0.211
RL	0.196-0.068	0.207-0.073	0.149-0.050	0.168-0.057	0.114-0.037	0.136-0.045
R2	0.839	0.855	0.903	0.877	0.93	0.926

Table 3. Values of Thermodynamic Parameters for The Adsorption of rhodamine B (RhB)

T (K)	Concentration (mg/L)	Zn-Cr-PW			Zn-Cr-SiW		
		ΔS (J/mol)	ΔH (J/mol)	ΔG (kJ/mol)	ΔS (J/mol)	ΔH (J/mol)	ΔG (kJ/mol)
303	100	21.404	7.455	-6.478	13.802	5.212	-4.176
323				-6.906			-4.452
343				-7.334			-4.728

0<RL<1, or irreversible if RL=0 [19]. As shown in Fig 5. the RL obtained in all the cases are between 0 and 1, thus indicating that the adsorption of Rhodamine B (RhB) is favorable in LDHs. Table 2. The Langmuir isotherm assumes that the adsorbent has a monolayer layer and also the surface of the adsorbent is homogeneous and there is no adsorption between the adsorbate molecules (Aguiar et al., 2013).

The thermodynamic parameters calculated from the values of the slopes and intercepts are reported in Table 3. For the rhodamine (RhB) dye the experimental results indicated that the amount adsorbed increased with decreasing temperature. The positive value of enthalpy shows that the adsorption process is endothermic. Process endothermic the adsorbate species replaces more than one water molecule to be dispersed. The low values of enthalpy indicated that the dyes are adsorbed via physical interactions. The positive values entropy indicates increasing randomness at the solid-solution interface during the adsorption process. Negative free energy value was indicated that the process is spontaneous and the decrease of free energy value with the increases of temperature indicated that the adsorption more favorable at low temperature.

4. CONCLUSIONS

In this study, adsorption experiments for the removal of rhodamine B (RhB) from aqueous solutions have been carried out using Zn-Cr layered double hydroxides (LDH) and intercalated with polyoxometalate $K_3[\alpha\text{-}PW_{12}O_{40}]\,nH_2O$ and $K_4[\alpha\text{-}SiW_{12}O_{40}]\,nH_2O$. The prepared LDH was characterized by XRD and FT-IR. The obtained results show that the materials were found to be excellent adsorbents for the used dyes. Kinetics study showed that the adsorption process more fits with PSO than PFO based correlation coefficients. The adsorption isotherms could be well fitted by the Langmuir equation. 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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