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Layered double hydroxides Mg/Fe intercalated $H_3 [\alpha - PW_{12}O_{40}] \cdot nH_2O$ as adsorbent of cadmium(II)

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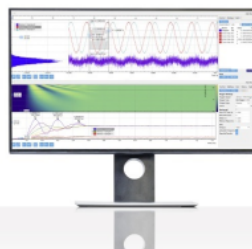
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Layered Double Hydroxides Mg/Fe Intercalated $H_3[\alpha\text{-PW}_{12}\text{O}_{40}]\cdot n\text{H}_2\text{O}$ as Adsorbent of Cadmium(II)

Aldes Lesbani^{1,2,a}, Della Risni Maretha¹, Tarmizi Taher², Miksusanti¹, Risfidian Mohadi^{1,2}, Roy Andreas³

¹Department of Chemistry, Faculty of Mathematic and Natural Sciences, Universitas Sriwijaya, Jl. Palembang Prabumulih Km.32 Ogan Ilir 30662, Indonesia

²Environmental Science Study, Graduate Program, Universitas Sriwijaya, Jl. Padang Selasa Bukit Besar Palembang 30139, Indonesia

³Department of Chemistry, Faculty of Mathematic and Natural Sciences, Universitas Jenderal Soedirman, Jl. Dr. Soeparno, Karangwangkal, Purwokerto Utara, Banyumas, 53123, Indonesia

^a) Corresponding Author: aldeslesbani@pps.unsri.ac.id

Abstract

Intercalation of Mg/Fe layered double hydroxides with $H_3[\alpha\text{-PW}_{12}\text{O}_{40}]\cdot n\text{H}_2\text{O}$ was successfully conducted. Characterization was carried out using FTIR and XRD analyses. Material was used as adsorbent of cadmium (II) in aqueous medium. Adsorption was studied through pH system, adsorption time, concentration of cadmium(II), and temperature. XRD analysis showed that Interlayer distance of Mg/Fe layered double hydroxides was increased after intercalation with $H_3[\alpha\text{-PW}_{12}\text{O}_{40}]\cdot n\text{H}_2\text{O}$ from 4.39 Å to 4.75 Å. Unique vibration of Mg/Fe before and after intercalation was found at wavenumber 1381 and 1635 cm^{-1} . Analysis of PZC showed that pH PZC of Mg/Fe layered double hydroxides and intercalated material was 9 and 8. Adsorption of cadmium(II) using Mg/Fe layered double hydroxides and intercalated material showed that intercalated material has better adsorption properties than Mg/Fe layered double hydroxides.

Keywords : layered double hydroxides, Mg/Fe, Keggin type polyoxometalate, $H_3[\alpha\text{-PW}_{12}\text{O}_{40}]\cdot n\text{H}_2\text{O}$, cadmium(II).

INTRODUCTION

The problem of heavy metal pollution has been recognized due to their toxicity and degradability. Heavy metals are produced mainly from industrial activity such as electroplating, steel industry, electronic recycling process, and so on. Various heavy metals are applied for these activities and will be released as pollutant in environment. The removal and decrease of metal ions from industrial activities is vital issues until this decade not only practical but also fundamental research [1,2]. Various methods have been applied to remove heavy metals from solution such as physical and chemical techniques and also combination with biological treatment [3,4]. Heavy metal containing unique properties such as redox and acid properties then removal using chemical method is appropriate following physical and biological methods. Chemical methods to remove heavy metal from waste water are applied such as coagulation [5], precipitation [6], ion exchange [7], membrane [8], and also adsorption [9,10]. Among these methods, adsorption is appropriate method due to simple way, easy to handle, fast process, and also can be applied for all heavy metals. The successfully process of adsorption is depending on ability adsorbent. Various adsorbents have been used to remove heavy metals from solution such as zeolites [11], clay minerals such as bentonite [12] and kaolin [13], metal oxides [14], and also layered double hydroxides [15,16].

Layered double hydroxides (LDH) is inorganic materials, namely hydrotalcite-like compounds. LDH with general formula $[M^{2+}_{1-x}M^{3+}_x(\text{OH})_2]^{x+}(\text{A}^{n-})_{x/n}\cdot n\text{H}_2\text{O}$ consist of M^{2+} and M^{3+} metal ions in positively charged metal-oxide layer and the layer surface charges is balanced by anions [17,18]. LDH has been used as adsorbent to remove various heavy metal ions from waste water. Due to anionic exchange capacity of LDH, the adsorption capacity of LDH can be increased by anionic exchange between interlayer using intercalation method. Small anions such as sulfate, nitrate, and chloride were used as intercalant for interlayer LDH [19]. On the other hand, large anion such as polyoxometalate has been applied to increase large interlayer distance between M^{2+} and M^{3+}

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of LDH. Keggin type polyoxometalate with general formula $[X_xM_mO_y]^{q-}$ (X : heteroatom; M : addenda atom) is common type of polyoxometalate which was used as intercalant of LDH [20,21].

Herein, Mg/Fe LDH was synthesized by co-precipitation method followed by calcination at 800 °C. These materials were intercalated with Keggin ion polyoxometalate $[\alpha\text{-PW}_{12}\text{O}_{40}]^{3-}$. All materials were characterized using XRD powder analysis and identification using FTIR spectroscopy. Mg/Fe LDH and intercalated Mg/Fe LDH were used as adsorbent of cadmium(II). Adsorption of cadmium(II) was conducted using batch system. Several factors that influencing adsorption process such as adsorption time, initial concentration of cadmium(II), and temperature were studied in this research. On the other hand, properties of materials were checked by pH pzc analysis and stability at various pH solution.

EXPERIMENTAL SECTION

Chemical and Instrumentation

Chemicals such as sodium phosphate, sodium tungstate, hydrochloric acid, potassium hydroxide, potassium chloride, diethyl ether, sodium hydroxide, sodium carbonate, iron (III) nitrate, and magnesium nitrate were supplied from Merck and Sigma Aldrich and directly used without further purification. Water was obtained from Integrated Research Laboratory Universitas Sriwijaya after cycling ion exchange process using Purite® water system technology at pH 7.

Analysis was conducted using Shimadzu FTIR Prestige-21 using KBr pellet and scanning was performed at wavenumber 400-4000 cm^{-1} . Analysis of XRD was conducted using X-Ray powder diffraction Rigaku Miniflex-600. XRD pattern was obtained after scanning at speed scan 0.1 deg min^{-1} . Cadmium(II) was determined using UV spectrophotometer EMC-61PC double beam spectrophotometer after complexation using 1,10-phenanthroline as ligand.

Synthesis of Mg/Fe Layered Double Hydroxides

Synthesis of Mg/Fe layered double hydroxides was carried out according to Elmoubarki et.al [22] with slightly modification. Magnesium nitrate (19.23 g) and iron(III) nitrate (10.10 g) was dissolved with water (solution 1). Sodium hydroxide (9 g) and sodium carbonate (1.33 g) was dissolved with water (solution 2). Solution 1 was added to solution 2 dropwise and solution was stirred slowly. pH solution was kept at 10 by addition of sodium hydroxide. Mixtures solution were kept at 110 °C for 10 hours to obtain brown solid material. Brown solid material was kept at 100 °C overnight. Solid material was washed with water and calcined at 800 °C for 3 hours to form Mg/Fe layered double hydroxides. Characterization of Mg/Fe layered double hydroxides was conducted using X-Ray powder analysis and FTIR spectrophotometer.

Synthesis of $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$

Synthesis of $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$ was conducted as follow: A sodium tungstate (125 g) and 20 g of disodium phosphate were dissolved in 187.5 mL of boiling water. A concentrated hydrochloric acid (100 mL) was added dropwise with constant stirring. Phosphotungstic acid begins to separate when about half of acid is added. When the solution has cooled, diethyl ether (75 mL) was added. After shaking, three layers remain and a little amount of water was added to dissolve the remaining of sodium chloride. The acid-ether complex in the lower layer was collected following the evaporation and recrystallization using water to produce $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$ [23]. Polyoxometalate $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$ was characterized using FT-IR spectroscopy and X-ray powder analysis.

Layered Double Hydroxides Intercalated Polyoxometalate

Intercalation of Mg/Fe LDH with $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$ was carried out using ion exchange method as follows. Polyoxometalate of $\text{H}_3[\alpha\text{-PW}_{12}\text{O}_{40}] \cdot n\text{H}_2\text{O}$ was dissolved with water. Mg/Fe layered double hydroxides was mixed with 1 M sodium hydroxide. Into this mixture was added solution of polyoxometalate with gentle stirring under nitrogen condition. Reaction was performed at 24 hours to form suspension. Suspension was filtered by vacuum and washed with water several times. Solid material was kept at 110 °C for 48 hours. Intercalated material was analyzed using XRD powder analysis and identification using FTIR spectroscopy.

pH PZC analysis

Analysis of pH point zero charge (pH PZC) was conducted using solution of sodium chloride 0.1 M. pH solution of sodium chloride 0.1 M was adjusted from pH 1 to 10 by addition of sodium hydroxide or hydrochloric acid 0.1 M. In the series of cadmium chloride solution with various pH, Mg/Fe LDH was added. The mixtures were shaken for 24 hours then pH of filtrate was determined by pH meter. pH PZC was obtained by comparison initial and final pH solution [24].

Adsorption Studies

Adsorption of cadmium(II) was studied by variation of pH, adsorption times, initial concentration of cadmium(II), and temperatures. pH of adsorption system was adjusted to 1, 3, 4, 5, 7, and 9 by addition of 0.1 M sodium hydroxides or 0.1 M hydrochloric acid. Adsorption times were studied to obtain kinetic parameter by variation of shaking time started i.e. 10, 20, 30, 40, 50, 60, and 90 minutes. Thermodynamic parameter was studied by variation of initial concentration of cadmium(II) and temperatures of adsorption system. The initial

concentration of cadmium(II) was 0.5, 1, 2, 3, 5, 7, and 10 mg/L. Temperature was controlled at 30, 40, 50, 60, and 70 °C. Adsorption was conducted at 60 minutes. Filtrate of adsorption was analyzed using UV after complexation with phenanthroline at λ 326 nm.

RESULTS AND DISCUSSION

XRD powder analysis of Mg/Fe LDH and intercalated materials is shown in Fig. 1. Figure 1a showed that diffraction of Mg/Fe LDH appeared at 11°, 22°, 35°, and 59° [25]. The existence of Mg/Fe layered material is identified at diffraction 59°. Calcination of Mg/Fe LDH at 800 °C resulted diffraction in Fig. 1b. Diffraction at 20°, 22°, 35°, and 59° was split due to loss of water of crystallization at high temperature. Figure 1c showed diffraction of polyoxometalate $H_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$. Several diffractions were found in polyoxometalate $H_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$ at lower diffraction angle. This is indicated high crystallinity of common polyoxometalate in Keggin type. Diffraction of intercalated Mg/Fe LDH was shown in Fig. 1d. Main diffraction of Mg/Fe LDH was identified in Figure 1d at 11°, and 59°. Diffraction at 35° was shifted to 40° and also 54°. Probably due to insertion of large anion to Mg/Fe LDH will shift diffraction of 22°. Interlayer distance or gallery of Mg/Fe LDH before intercalation was 4.39 Å and to be 4.75 Å after intercalation. Large anion such as polyoxometalate $[\alpha-PW_{12}O_{40}]^{3-}$ will increase gallery for large space. In this results the gallery is slightly increase due to intercalation occurred in diagonal position not in vertical position.

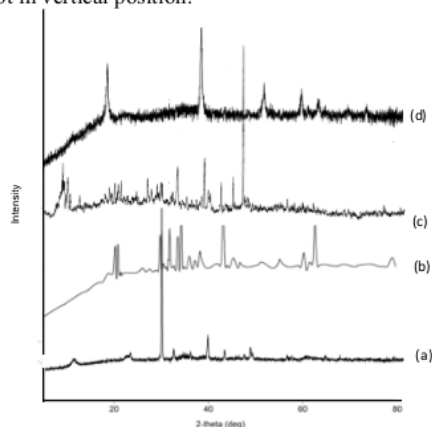


FIGURE 1. XRD patterns of (a) Mg/Fe LDH synthesized at r.t., (b) Mg/Fe LDH calcined at 800 °C, (c) polyoxometalate $H_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$, (d) intercalated Mg/Fe LDH.

FTIR spectrum of Mg/Fe LDH and intercalated Mg/Fe LDH is shown in Fig. 2. Mg/Fe LDH has strong vibration at 1381 cm^{-1} which was assigned as stretching vibration of nitrate anion. Another vibration was appeared at wavenumber 3300 cm^{-1} indicated vibration of OH from water. Vibration of metal-oxygen (M-O) was identified at 586 cm^{-1} and 378 cm^{-1} which were assigned as vibration Fe-O and Mg-O, respectively [26]. Figure 2b showed that vibration of nitrate was split after calcination at 800 °C probably due to unstable nitrate at high temperature. Figure 2c showed FTIR spectrum of polyoxometalate $H_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$ were appeared at wavenumber 1080 cm^{-1} (ν P-O), 987 cm^{-1} (ν W=O), 894 cm^{-1} (ν W-Oc-W), and 802 cm^{-1} (ν W-Oe-W) [27]. Intercalation Mg/Fe LDH with $[\alpha-PW_{12}O_{40}]^{3-}$ anion resulted FTIR spectra in Fig. 2d. Broad vibration at wavenumber 1380 cm^{-1} was indicated as nitrate anion was replaced with $[\alpha-PW_{12}O_{40}]^{3-}$ ion. Sharp vibration at 3695 cm^{-1} is assigned as water of crystallization of intercalated Mg/Fe LDH.

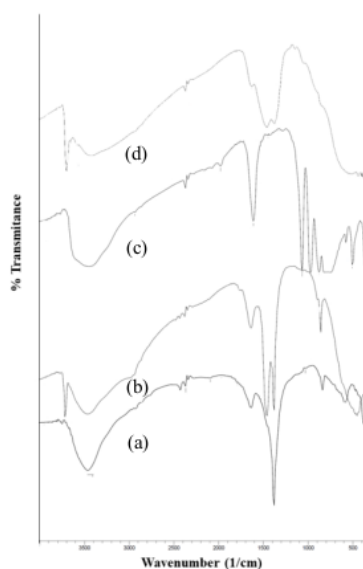


FIGURE 2. FTIR spectrum of (a) Mg/Fe LDH synthesized at r.t., (b) Mg/Fe LDH calcined at 800 °C, (c) polyoxometalate $H_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$, (d) intercalated Mg/Fe LDH.

Adsorbent of Mg/Fe LDH and intercalated Mg/Fe LDH was identified by pH pzc analysis before adsorption process. The aim of this experiment is to determine the type of adsorbent surface charge. The pH pzc graph for Mg/Fe and intercalated Mg/Fe LDH is shown in Fig. 3.

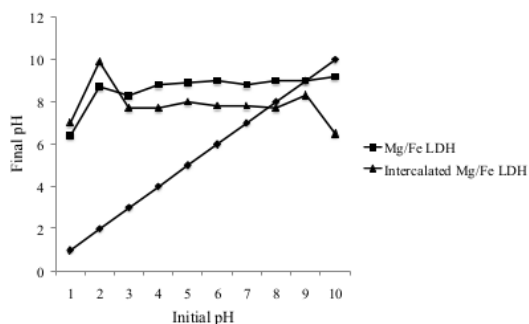


FIGURE 3. pH PZC graph.

Figure 3 showed that pH pzc for Mg/Fe LDH and intercalated Mg/Fe was obtained at pH 9 and pH 8, respectively. Mg/Fe LDH was synthesized at base condition resulted base material. Materials will be protonated at lower pH ($<$ pH pzc). Otherwise, the surface charge of material will negative at pH above pH pzc. This result is appropriate with results of determination of pH stability for adsorption of cadmium(II) using Mg/Fe LDH and intercalated Mg/Fe LDH as shown in Fig.4.

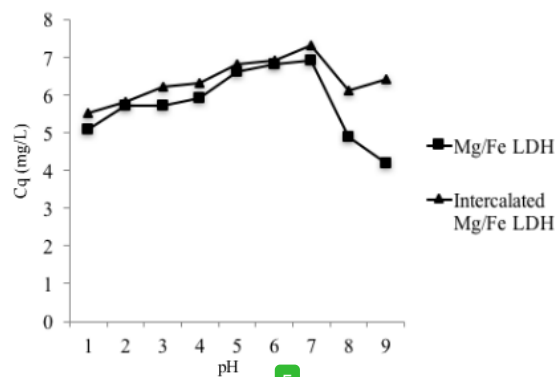


FIGURE 4. Effect of pH adsorption Cd(II) on Mg/Fe LDH and intercalated Mg/Fe LDH.

Effect of pH on adsorption of cadmium(II) using Mg/Fe LDH and intercalated Mg/Fe LDH was found pH at 7 for both adsorbents. Thus adsorption of cadmium(II) on LDH was performed at pH 9 for Mg/Fe LDH and pH 8 for intercalated Mg/Fe LDH. According to cadmium species in various pH as shown in Fig. 5, the results of Fig. 4 is meet with theoretical prediction in which cadmium ion stable in solution at pH below than 9.

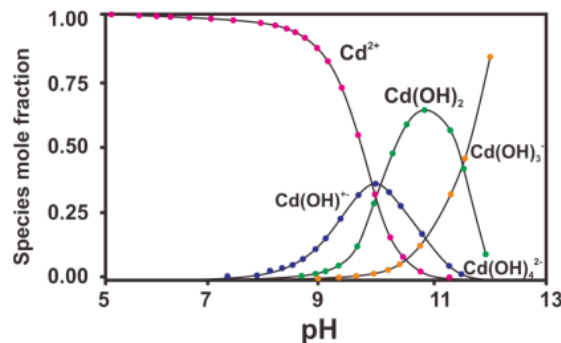


FIGURE 5. Species distribution of cadmium at various pH solution [28].

Adsorption of cadmium(II) using Mg/Fe LDH and intercalated Mg/Fe LDH was firstly investigated by effect of adsorption time as shown in Fig. 6. Adsorption cadmium(II) was occurred very fast below 20 minutes for Mg/Fe LDH and 50 minutes for intercalated Mg/Fe LDH. Adsorption after that times was stable and cadmium(II) was occupied on surface of LDH. Kinetic adsorption of cadmium(II) on Mg/Fe LDH and intercalated Mg/Fe LDH was calculated by applying Langmuir-Heinselwood [29] equation for data in Fig. 6.

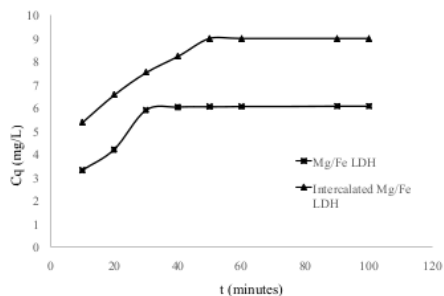


Figure 6. Effect of adsorption time on adsorption Cd(II).

Adsorption rate constant (k_1) for Mg/Fe LDH and intercalated Mg/Fe LDH was 0.052 min^{-1} and 0.082 min^{-1} , respectively. That results showed that intercalated Mg/Fe LDH is more faster than Mg/Fe LDH without intercalation of Keggin ion $[\alpha\text{-PW}_{12}\text{O}_{40}]^{3-}$.

Effect of initial concentration of cadmium(II) and various temperatures in the adsorption using Mg/Fe LDH and intercalated material were shown in Fig. 7.

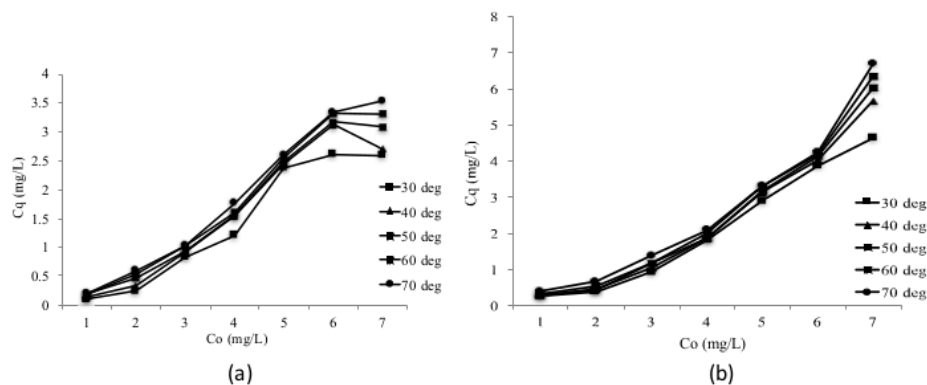


Figure 7. Effect of concentration and temperature on adsorption Cd(II). (a) Mg/Fe LDH, (b) intercalated Mg/Fe LDH

Data of initial concentration of cadmium(II) at various adsorption temperatures in Fig. 7 showed that by increasing temperature will increase adsorption of cadmium(II) from solution. Adsorption capacity of cadmium(II) on LDH can be obtained from Langmuir equation [30]. Adsorption capacity of cadmium(II) on Mg/Fe LDH is up to 0.13 mg.g^{-1} at $70 \text{ }^\circ\text{C}$ while adsorption capacity for intercalated Mg/Fe LDH is up to 0.16 mg.g^{-1} at $70 \text{ }^\circ\text{C}$. These results indicated that intercalated Mg/Fe LDH is suitable as adsorbent of cadmium(II) than Mg/Fe LDH without intercalation with ion $[\alpha\text{-PW}_{12}\text{O}_{40}]^{3-}$.

CONCLUSION

2 Intercalation of Mg/Fe LDH using anion $[\alpha\text{-PW}_{12}\text{O}_{40}]^{3-}$ was successfully conducted in which 2 layer of layer increased from 4.39 \AA to 4.75 \AA after intercalation process with diagonal position of anion. Adsorption of cadmium(II) using intercalated Mg/Fe LDH is faster and higher than Mg/Fe LDH without intercalation of large anion.

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