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Adsorptive Removal of Cd (II) Ion from Aqueous Solution by Polyoxometalate Intercalated MgAl LDH

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Abstract. Keggin type polyoxometalate (POM) α-$SiW_{12}O_{40}^{4-}$ has successfully intercalated into the interlayer space of MgAl LDH through facile ion exchange method. The pristine MgAl LDH was firstly synthesized by coprecipitation method. The structure of MgAl LDH and its intercalated form were characterized by X-ray diffraction and FTIR analysis. The obtained results indicated the formation of MgAl LDH with brucite-like layer structure. Moreover, the POM intercalated MgAl LDH sample exhibited an expansion of interlayer distance that indicates the success of the POM intercalation. Adsorptive removal of Cd(II) ion from aqueous solution by using pristine MgAl LDH and MgAl POM was conducted in batch method. The results showed that the MgAl POM has a higher adsorption capacity for Cd(II) removal than the pristine MgAl LDH.

Keywords: Adsorption, cadmium (II), Mg/Al LDH, polyoxometalate

INTRODUCTION

In the recent century, the growth of industrial activities has caused the increase of an industrial pollutant that affects the harmony of the ecosystem [1]. The pollutants that produced by industry such as dyes, heavy metal, and hazardous organic/inorganic compound has become a worldwide concern due to their noxious effect to the living organism including human life [2]. Among the detrimental industrial pollutant, heavy metal has considered as the most hazardous one due to its capability to be accumulated in the organs and tissue then caused various detrimental effect and even cancer [3].

Cadmium ion (Cd(II)) has considered as one of the most toxic heavy metal that unfortunately utilized widely by various industries such as metallurgy, electronic, mining, electroplating, etc [4]. Human exposure to the Cd(II) could cause serious organs damage such as kidney and lung and cancer [5]. Consequently, as stated by World Health Organization (WHO), the permissibility of the Cd(II) concentration in the drinking water is limited less than 3µg/L [6]. Regarding to the above consideration, it is mandatory to remove the contamination of Cd(II) ion from the industrial effluent in order maintain the environmental sustainability [7].

Recently, various methods based on the biological, chemical and physical technology such as bioremediation [8], flocculation [9], precipitation, ion exchange, and adsorption [10] have been extensively adapted for the removal of Cd(II) from the industrial effluent [11]. However, the established methods should be developed in order to increase their efficiency and economical cost. Adsorption has considered as one of the most effective and efficient methods for removal of heavy metal from wastewater due to its low cost, easy operational, safety, and the availability of numerous

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adsorbent materials. Although, activated carbon has been employed as the most common adsorbent, nowadays various alternative based adsorbents have been also developed [7].

Layered double hydroxides (LDHs) are one of the most promising material to be explored as low cost-based adsorbent [12]. They can be easily synthesized in laboratory and even industrial scale. Moreover, LDHs could be formed by numerous metal composition that produced various type and characteristic [13]. Due to its high anion exchange capability, LDHs are also commonly called as anion clay since they have similar properties with clay minerals. In order to expand its adsorption capability, LDH can be modified by intercalation technique by inserting a certain anion that has bigger size than their original anion [14]. In this work, MgAl LDH has been synthesized by facile coprecipitation method to be further developed by intercalating polyoxometalate (α-$SiW_{12}O_{40}^{4-}$) anion into its interlayer space. The obtained material was tested as adsorbent for removal of Cd(II) ion from aqueous solution.

EXPERIMENTAL

Materials

All the chemical used in this work including magnesium nitrate ($Mg(NO_3)_2{\cdot}6H_2O$), Aluminum nitrate ($Al(NO_3)_3{\cdot}9H_2O$), Sodium tungstate (Na_2WO_4), sodium metasilicate (Na_2SiO_3), sodium hydroxides (NaOH), and cadmium chloride ($CdCl_2{\cdot}H_2O$) were purchased from Merck and used as received without further purification.

Preparation of MgAl LDH

MgAl LDH was synthesized according to the previously reported paper by Palapa et al. using coprecipitation method [15]. In brief, 50 mL of 1 M $Mg(NO_3)_2{\cdot}6H_2O$ and 20 mL of 1 M $Al(NO_3)_3{\cdot}9H_2O$ were mixed with 250 mL of distilled water under vigorous stirring. In to the mixed metal solution, the solution of 2M NaOH and Na_2CO_3 was further introduced until the pH of the solution was constant at pH 10. The obtained mixed metal solution was then stirred for 3 h at 40 °C. After finished, the obtained solid was filtered and dried in an oven at 80 °C for overnight. The obtained dried white solid was then kept in a closed bottle and labelled as MgAl LDH.

Preparation of MgAl POM

The intercalated MgAl LDH was prepared by facile ion exchange procedure in which firstly the α-$SiW_{12}O_{40}^{4-}$ anion was prepared by following the previously reported paper[16]. Typically, the intercalated process was conducted by the following procedure. The α-$SiW_{12}O_{40}^{4-}$ solution was prepared by dissolving 1 g of $H_4[\alpha\text{-}SiW_{12}O_{40}^{4-}]{\cdot}nH_2O$ powder in 50 mL of distilled water. Into the prepared α-$SiW_{12}O_{40}^{4-}$ solution, 2 g of MgAl LDH was added then vigorously stirred under N_2 atmosphere for 12 h. After finished, the obtained solid was filtered and dried at 80 °C for 8 h and the product was labelled as MgAl POM.

Cd (II) Adsorption Study

The study of Cd(II) adsorption from aqueous solution was carried out in batch method using the pristine MgAl LDH and the intercalated MgAl POM as the adsorbent. The stock solution of Cd(II) ion was firstly prepared by dissolving 1.791 g of $CdCl_2$ solid in 1000 mL of distilled water then the standard and working solution of Cd(II) was prepared by diluting the prepared Cd(II) stock solution. The adsorption experiment was done by investigating the effect of the contact time to the adsorption capacity of the tested adsorbent. Typically, 0.1 g of adsorbent was added into a beaker flask containing 10 mL of 10 mg/L Cd(II) solution. The mixture was stirred for a predetermined time ranging from 0 to 90 min in a horizontal shaker at room temperature condition. After finished, the mixture was separated and the remaining concentration of Cd(II) solution was determined by UV-Vis spectrometry technique.

Material Characterization

The synthesized materials including MgAl LDH and MgAl POM was characterized by X-ray diffraction analysis and FTIR spectroscopy. The XRD analysis was carried out using Shimadzu lab X type 6000 XRD that scanned at 2θ 5° to 80°. The FT-IR analysis was conducted using Shimadzu Prestige-21 FTIR instrument using KBr disk method and measured at wavenumber of 4000 to 400 cm^{-1}.

RESULTS AND DISCUSSION

The synthesized materials including MgAl LDH and MgAl POM were characterized by X-ray diffraction analysis in order to investigate their structure and crystallinity. XRD pattern of the synthesized MgAl LDH and MgAl POM are shown in Fig. 1. The XRD pattern of the synthesized MgAl LDH showed the characteristic diffraction peaks that correspond to the LDH material as indexed by the JCPS card 038–0478. The typical diffraction peak of layered material was indicated by three symmetric diffraction peaks at low diffraction angle, i.e., 11.62°, 22.69°, and 34.48°. Another typical diffraction peak of LDH material was observed at 2θ around 60.56° and 61.78° that correspond to the (110) and (113) planes of layered double hydroxides structure, respectively. The typical characteristic peaks of (003) plane that correspond to the basal spacing of LDH was observed at 2θ of 11.62°. The interlayer distance of the MgAl LDH sample according to the (003) plane value was calculated as 7.59 A.

The synthesized MgAl LDH was further modified by intercalation method using polyoxometalate (α-$SiW_{12}O_{40}^{4-}$) anion. The synthesized MgAl POM was also characterized by XRD where the results were shown in Fig. 1. The XRD pattern of MgAl POM showed the typical diffraction pattern of LDH material as its pristine form (MgAl LDH). However, it can be observed that the characteristic peak of (003) plane was shifted to the lower angle. That finding indicated the expanding of the interlayer distance of the LDH structure. After intercalated by polyoxometalate (POM), the diffraction peak of (003) shifter from 11.62° to 11.48° that correspond to the increase of the interlayer distance from 7.59 A to 7.67 A. This finding showed that the POM anion was successfully inserted into the interlayer distance of the MgAl LDH.

The synthesized MgAl LDH and MgAl POM were also characterized by FT-IR in order to investigate their composition of the functional group. The FTIR spectra of the pristine MgAl LDH exhibited typical vibration band of LDH in which the characteristic of metal oxide (M—O) bonding was observed at wavenumber around 630 cm^{-1} [17]. The vibration band of the –OH functional group that belong to the interlayer water molecule and surface hydroxyl group were observed as broad band at wavenumber around 3464 cm^{-1}, and 1650 cm^{-1}. While the typical vibration band of the original interlayer anion (CO_3^{2-}/NO_3^-) was observed at strong and sharp band at wavenumber around 1381 cm^{-1} These observed vibration bands are the typical characteristic of LDH as also reported by Shafigh et al. [18]. The FTIR spectra of the intercalated MgAl LDH by α-$SiW_{12}O_{40}^{4-}$ anion, as can be shown in Fig. 2, indicate a major similarity as the pristine MgAl LDH. However, the prominent different can be observed at the typical vibration band of the interlayer anion at wavenumber around 1381 cm^{-1}. It can be identified that the band that

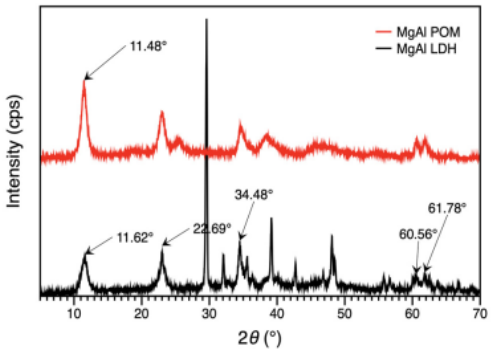

FIGURE 1. XRD pattern of pristine MgAl LDH and MgAl POM.

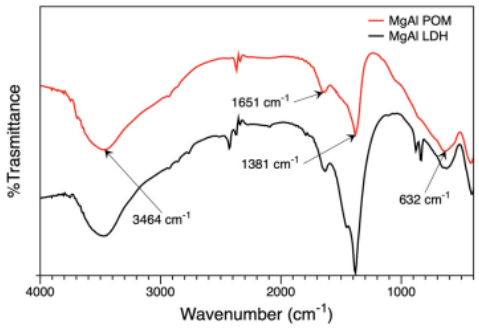

FIGURE 2. FTIR spectra of the synthesized MgAl LDH and MgAl POM.

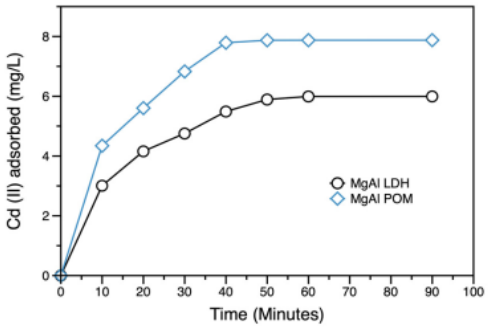

FIGURE 3. Effect of contact time vs Cd (II) adsorption capacity.

correspond to the presence of interlayer anion significantly decreased after intercalation process. Although it wasn't totally disappeared, the finding indicated that the original interlayer anion (CO_3^{2-}/NO_3^-) was successfully changed by the α-$SiW_{12}O_{40}^{4-}$ anion in partial. The result corresponds with result of the XRD analysis in which the typical diffraction peak of the (003) plane of the MgAl LDH slightly shifted into the lower 2θ angle.

Next, the synthesized MgAl LDH and MgAl POM were employed as an adsorbent for removal of Cd (II) ion from aqueous solution as the model of industrial wastewater. The adsorption study was conducted in batch method where typically 0.1 g of adsorbent was contacted with 10 mL of 10 mg/L Cd(II) solution then stirred for certain time. The remaining concentration of the Cd(II) after adsorption process was determined by UV-Vis spectrometry method after complexation with phenanthroline. In order to investigate the adsorption capacity at the equilibrium, the adsorption experiment was carried out at different contact time ranging from 0 to 90 minutes. The obtained results were analyzed and depicted in Fig 3. It can be observed that the adsorption of Cd(II) on both MgAl LDH and MgAl POM gradually increased by increasing the contact time. The adsorption process seemed to reach the equilibrium after 50 min contact time. At the equilibrium, MgAl LDH can adsorb Cd(II) up to 5.9 mg/L while the MgAl POM can adsorb up to 7.87 mg/L. This finding clearly showed that the intercalated MgAl LDH by α-$SiW_{12}O_{40}^{4-}$ has higher adsorption capacity toward Cd(II) ion rather than the pristine MgAl LDH. As described in the preceding discussion, this result probably induced by the increase of the interlayer distance of the LDH after intercalated by α-$SiW_{12}O_{40}^{4-}$ ion.

CONCLUSION

In this work, we provide an insight into the adsorption of Cd(II) ion into the intercalated MgAl LDH by α-$SiW_{12}O_{40}^{4-}$ ion. The obtained results indicated that the α-$SiW_{12}O_{40}^{4-}$ anion was successfully inserted into the interlayer of the MgAl LH partially. It was proofed by the XRD and FTIR analysis in which the XRD pattern of the typical diffraction peak (003) MgAl POM showed a shifting toward a lower 2θ value and the FTIR spectra of the interlayer anion vibration band decreased after intercalation. Moreover, the synthesized MgAl POM exhibited higher adsorption capacity to the Cd(II) ion in the aqueous solution rather than the pristine MgAl LDH.

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