

Removal of Pb(II) and As(V) using magnetic nanoparticles coated montmorillonite via onepot solvothermal reaction as adsorbent

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https://doi.org/10.1016/j.jece.2019.103000

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Highlights

- The amino magnetic nanoparticles can be easily caoted onto montmorillonite (Mt@MH).
- The Mt@MH was provide a high adsorption capacity towards Pb(II) and As(V).
- · Easily retrievable makes the adsorption process easy and simple.

Abstract

Clay mineral (montmorillonite) based nanocomposites was modified their surface functional groups for removal of Pb(II) and As(V) of aqueous solution. Magnetic nanoparticles with an amine functionalized surface (MH) were successfully synthesized onto the surface layer of montmorillonite (Mt) as an organomodified mineral solid via one-pot solvothermal reaction. The synthesis of amino magnetic nanoparticles coated montmorillonite (Mt@MH) was carried out by one-pot solvothermal reaction of 1,6hexanediamine, iron(III) chloride hexahydrate and Mt in ethylene glycol at ±198 °C for 6 h. The MH nanoparticles with diameter size around 30–50 nm were obtained. Characterizations were performed using powder X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), superconducting quantum interference device (SQUID), and Fourier transform infrared spectroscopy (FT-IR) analysis. The insertion of MH onto the surface layer of Mt does not only serve as an easily retrievable adsorbent but also provide a high adsorption capacity towards Pb(II) and As(V) ion. The adsorption isotherms of Pb(II) and As(V) at room temperature were well-fitted with Langmuir model providing maximum adsorption capacity for Pb(II) and As(V) of 38.15 mg/g at $pH_e^-6.5$ and 19.10 mg/g at $pH_e^-3.5$, respectively. The Mt@MH showed 2-fold higher adsorption capacity than MH and Mt. It is due to surface functionalization with the amine group (6 mmol/g) on the adsorbent. The electrostatic interaction was proposed as the primary driving forces for Pb(II) and As(V) adsorption onto the Mt@MH.

Contents lists available at ScienceDirect



Journal of Environmental Chemical Engineering

journal homepage: www.elsevier.com/locate/jece



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ARTICLE INFO

Keywords: Magnetic nanoparticles Metal ion removal Montmorillonite Solvothermal Adsorption

ABSTRACT

Clay mineral (montmorillonite) based nanocomposites was modified their surface functional groups for removal of Pb(II) and As(V) of aqueous solution. Magnetic nanoparticles with an amine functionalized surface (MH) were successfully synthesized onto the surface layer of montmorillonite (Mt) as an organomodified mineral solid via one-pot solvothermal reaction. The synthesis of amino magnetic nanoparticles coated montmorillonite (Mt@ MH) was carried out by one-pot solvothermal reaction of 1,6-hexanediamine, iron(III) chloride hexahydrate and Mt in ethylene glycol at \pm 198 °C for 6 h. The MH nanoparticles with diameter size around 30–50 nm were obtained. Characterizations were performed using powder X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), superconducting quantum interference device (SQUID), and Fourier transform infrared spectroscopy (FT-IR) analysis. The insertion of MH onto the surface layer of Mt does not only serve as an easily retrievable adsorbent but also provide a high adsorption capacity towards Pb(II) and As(V) ion. The adsorption isotherms of Pb(II) and As(V) at room temperature were well-fitted with Langmuir model providing maximum adsorption capacity for Pb(II) and As(V) of 38.15 mg/g at pHe^{-6.5} and 19.10 mg/g at pHe^{-3.5}, respectively. The Mt@MH showed 2-fold higher adsorbent. The electrostatic interaction was proposed as the primary driving forces for Pb(II) and As(V) adsorption capacity than MH and Mt. It is due to surface functionalization with the amine group (6 mmol/g) on the adsorbent. The electrostatic interaction was proposed as the primary driving forces for Pb(II) and As(V) adsorption onto the Mt@MH.

1. Introduction

Recently, in view of population growth, the problem of wastewater has been aroused more and more attention and the demand of freshwater for various uses would increase. The presence of toxic heavy metal ions such as arsenic and lead in effluents wastewater is a serious issue, it can cause a problem for both the environment and the living organism [1–3]. Various techniques have been employed for those metal ions contaminated water, including precipitation, coagulation, ion exchange, oxidation, and reverse osmosis. Among these treatments, adsorption is considered as an efficient and economical method for metal ions removal.

Clay mineral is used as an adsorbent since it has good adsorption capacity towards various metal ions such as Ni, Pb, Zn, Co, Cd, Hg, As, Cr, Mn, etc [4,5]. Clay minerals are available widespread and easily found in the environments such as in sediments, soils, or rocks. These minerals are characterized by large surface areas per unit of mass, comprised of multiple layers of hydroxylated, coordinated tetrahedral and octahedral sheets. Having such nature, their surface reactions are frequently exploited for the removal of pollutants from aqueous solutions. Clay minerals are excellent adsorbents for metal ions, owing to their high cation exchange capacity and high specific surface area associated with their small particle sizes. Therefore, this material finds favored application for the treatment of various effluents, such as industrial and processing water and wastewaters, or extracts resulting from the treatment of contaminated soils by soil washing [6–8].

In the search for better adsorbent materials, modifications of clay minerals such as intercalation, pillaring, and organo-functionalization have been found to be important techniques [7]. In recent years, there has been an increasing interest in the development of composites material which combines clay and magnetic nanoparticles for metal ion removals such as Ni(II), Cu(II), Cd(II), pb (II) and As(V) [4,9–13].

Recent developments in clay minerals have focused on montmorillonite, one type of clay minerals that have high cation exchange capacity due to the substitution of their main cations, namely Si^{4+} and Al^{3+} . The extent and location (i.e. tetrahedral or octahedral sheets) of these substitutions give rise to the unique physicochemical properties of clay minerals. In montmorillonite, changes mainly take place in the

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https://doi.org/10.1016/j.jece.2019.103000

Received 19 January 2019; Received in revised form 1 March 2019; Accepted 2 March 2019 Available online 05 March 2019 2213-3437/ © 2019 Published by Elsevier Ltd. octahedral sheets, i.e. separated from the interlayer spacing where cations are present and balancing the excess negative charge. Therefore, the interaction between the charge-balancing cations and the two tetrahedral sheets is rather weak, leading to a relatively large interlayer expansion [14,15]. Due to these physical properties, the montmorillonite was used as a material for composites in the application of cationic biopolymer [16–18] and metal ion removal [7,19].

Applications of using nano-technology for water and wastewater treatment are important [20]. Magnetic iron oxide nanoparticles intercalated to montmorillonite has advantageous use in wastewater treatment and has been receiving attention [6-8,21]. A considerable amount of literature discussing magnetite nanoparticle has been published. Magnetic nanoparticles with functionalized surface have been widely used in wastewater problems treatment processes due to their physical and chemical properties. It has been applied in water treatment including heavy metal ions removal [2,3,10,22], inorganic and organic contaminant removal [23], adsorption of toxic water-soluble molecules [24], and oily wastewater treatment [25]. However, far too little consideration has been paid to the surface functionalization of clay/magnetic nanocomposites. This research focussed on the prepared magnetic nanocomposites with the amino functional group have synergistic effect from organic (flexibility, strength) and inorganic (adsorption, magnetic properties) components. Emphases are given on the surface functional groups, surface effects and specific interaction mechanism on adsorption process.

The objective of this research was to investigate the physical and chemical properties of Mt@MH such as morphology, crystalline structure, surface functional groups, magnetic properties, and amino content. Furthermore, the effects of magnetic nanoparticle interaction and surface functionalization on Mt as well as adsorption capacity for Pb(II) and As(V) ion were also investigated.

2. Materials and methods

2.1. Materials

Montmorillonite was obtained from Sigma-Aldrich. Anhydrous sodium acetate ($C_2H_3NaO_2$), ethylene glycol ($C_6H_6O_2$), 1,6-hexanediamine (($C_6H_{16}N_2$, HDMA), iron(III) chloride hexahydrate (FeCl₃.6H₂O), lead(II) nitrate (Pb(NO₃)₂), sodium arsenate heptahydrate (Na₂HAsO₄.7H₂O), hydrochloric acid (HCl), and sodium hydroxide (NaOH) were obtained from ACROS. All the chemicals were analytical grade and used without further purification.

2.2. Preparation of magnetic nanoparticles coated montmorillonite

The synthesis of Mt@MH was carried out by a one-pot solvothermal reaction. Surface amine-functionalized magnetic nanoparticles were synthesized adopting a previously applied method [26]. Briefly, anhydrous sodium acetate (1.6 g) and iron(III) chloride hexahydrate (0.8 g) were dissolved in ethylene glycol (24 mL) with vigorous stirring at 50 °C using magnetic stirrer, resulted in an orange solution. When 1,6-hexanediamine (7 mL) was added, the solution turned into dark-orange. Magnetic nanoparticles coated montmorillonite (Mt@MH) were synthesized by soaking montmorillonite (5 g) into solution, then the solvothermal reaction was carried out at \pm 198 °C for 6 h. The originally dark-orange solution turned into black suspension after this process.

After cooling to room temperature, the black precipitate was collected from the solution by employing external magnetic field and was rinsed with deionized (DI) water followed by ethanol each for 3 times to remove the remaining chemicals. The obtained material was designated as Mt@MH and kept in DI water for future use. Schematic illustration of the preparation of Mt@MH is shown in Fig. 1.

2.3. Equilibrium adsorption isotherm of Pb(II) and As(V)

Removal of Pb(II) and As(V) of aqueous solution using Mt@MH as adsorbent were carried out in batch adsorption expperimental. Batch adsorption experiments were carried out by putting 100 mL solution of a certain concentration of lead, Pb(II) and arsenate, As(V) into 150 mL glass bottle and adjusting its pH with 1 M NaOH or 1 M HCl. Afterwards, a weighed amount of the Mt@MH was added into the glass bottle. The mixture was then placed in a shaker with a water bath (Firstek Scientific) at a predetermined temperature for certain contact time. The adsorption was carried out while shaking at room temperature for 8 h. At the end of the experiment, the solution was separated by external magnetic field and filtered by using a 0.2 µm PVDF membrane (Advantec). Finally, the filtrates were analyzed for residual metal concentration using inductively coupled plasma atomic emission spectrophotometer (ICP-AES JY2000 2, Horiba Jobin Yvon). Adsorbed metal ion was calculated from the difference between the initial and equilibrium adsorbate concentrations. The adsorption experiments were done in triplicate and the average value was taken.

In order to regenerate Mt@MH for repeated uses, the metal ionsloaded Mt@MH was desorbed by shaking in 0.1 N HCl for 24 h. After washing thoroughly with DI water, the regenerated Mt@MH was designated as the adsorbent for the next cycle of Pb(II) and As(V) adsorption. The recycle usage of such adsorbent was repeated for three times in triplicate analysis.

2.4. Characterization of Mt@MH

Field-emission scanning electron microscopy (FE-SEM, JOEL JSM-6500 F) was used to probe the surface morphology of Mt@MH. The Xray diffraction (XRD) measurement was performed on Rigaku D/MAX-B X-ray diffractometer by using Copper K-alpha (CuK α) radiation. The operation voltage and current were kept at 40 kV and 100 mA, respectively. The magnetic properties of Mt@MH was studied by a superconducting quantum interference device (SQUID, LakeShore 7307) magnetometer. Fourier transform infrared spectrometry (FT-IR, Biorad, Digilab FTS-3500) was used to identify the functional groups on the material.

The presence of amine on Mt@MH was determined by using ninhydrin assay [27]. The amine content was determined by the retrotitration method [28]. A 50 mg sample was added to 25 mL solution of 0.01 M HCl and then shaken for 2 h at room temperature. After separated, 5 mL of supernatant was titrated by 0.01 N NaOH. The concentration of amine group was calculated by Eq. (1).

$$C_{\rm NH_2} = \left[\frac{(C_{\rm HCl} \cdot V_{\rm HCl}) - (5 C_{\rm NaOH} \cdot V_{\rm NaOH})}{m_{\rm sample}} \right]$$
(1)

where C_{HCI} and C_{NaOH} are the concentration of HCl solution (mmol/L) and concentration of NaOH solution (mmol/L), respectively, V_{HCl} is the volume of HCl solution (L), V_{NaOH} is the volume of NaOH spent in the titration of non-reacted acid's excess (L) and m_{sample} is the sample mass (g).

3. Results and discussion

3.1. Characterization of the amino magnetic nanoparticles coated montmorillonite (Mt@MH)

Magnetic nanoparticles with amine functionalized surface were successfully synthesized onto surface layer of montmorillonite (Mt@ MH), in the form of a black precipitate. Amine functionalized magnetic nanoparticle (MH) was achieved by adding HMDA during the synthesis. The presence of amine groups on the surface of samples was qualitatively identified by ninhydrin which form a Ruhemann's purple colored solution (Fig. 2, inset). Based on the intensity purple color that



Fig. 1. Schematic diagram for preparation of amino magnetic nanoparticles coated montmorillonite (Mt@MH).

developed, the presence of amine groups on Mt@MH were confirmed. In addition, amine functionalization of Mt@MH surface was also proctected the iron for leaching in solution. The amine content was determined by retro-titration and found to be 6 mmol/g.

Typical FE-SEM of a representative sample show the smooth surface of Mt (Fig. 2a). The Mt@MH clearly revealed the round shaped magnetic nanoparticle along montmorillonite surface with diameter size in the range of 30-50 nm when FeCl₃.6H₂O was included in the reaction solution. In addition, the white color of Mt became black due to the presence of magnetic nanoparticles which covered space layer of Mt (Fig. 2b). As shown in the FT-IR spectra (Fig. 3), the Mt@MH exhibited the resonance bands of amine group (1645 cm^{-1} and 1450 cm^{-1}), Fe-O (598 cm^{-1}) and OH (3400 cm^{-1}) bondings [2,10]. The Mt spectra shown no spesific peak for Fe-O, it indicates that the surface modification of Mt@MH by one-step solvothermal treatment has been achieved.

Furthermore, to identify the chemical structure of as-prepared Mt@ MH, the XRD analysis has been carried out (Fig. 4). Montmorillonite has



Fig. 2. FESEM micrograph of (a) original montmorillonite (Mt), (b) amino magnetic nanoparticles coated montmorillonite (Mt@MH), (c) Mt@MH with higher magnification by solvothermal reaction at 198 °C for 6 h.



Fig. 3. IR spectra of montmorillonite (Mt) and amino magnetic nanoparticles coated montmorillonite (Mt@MH).



Fig. 4. Typical XRD pattern of iron oxide nanoparticles (MH), montmorillonite (Mt), and amino magnetic nanoparticles coated montmorillonite (Mt@MH).

typical diffraction peaks which corresponds to silica and aluminum at 20 of 26.5° and 47°, respectively [29]. The positions and relative intensities of diffraction peaks for Fe₃O₄ magnetite nanoparticles structure were at 2 θ of 36° and 43° (JCPDS, 75-1610) [2]. The diffraction pattern of Mt@MH in Fig. 4 exhibits peaks at 20 of 26.5° which corresponds to silica and at 20 of 35° and 42° which belong to Fe₃O₄. Based on this data, the Mt@MH was assumed as a magnetite (Fe₃O₄) phase which has superparamagnetic properties, and hence will facilitate in easier magnetic separation [23].

The saturation magnetization measurements of MH and Mt@MH were investigated at room temperature employing Superconducting Quantum Interference Device (SQUID) analysis. As observed in Fig. 5, the magnetization curves of both materials show ferromagnetic behavior. The magnetic saturation values of the naked MH and Mt@MH are 70 and 15 emu/g, respectively. The smaller magnetic saturation of Mt@ MH was expected, prossibly due to an appreciable amount of montmorillonite. The saturation of Mt@MH almost same which magnetic nanoparticle nanocomposite about 9–14 emu/g by previous research [30,31]. The superparamagnetic MH and Mt@MH shows a fast response to external magnetic field and allow the adsorbent well functionalized and easily separated around 100%.

3.2. Equilibrium adsorption isotherm of Pb(II) and As(V)

The as-prepared amino magnetic nanoparticles coated montmorillonite (Mt@MH) were employed to adsorb Pb(II) and As(V) under



Fig. 5. Room temperature magnetization curve of naked iron oxide nanoparticles (MH) and amino magnetic nanoparticles coated montmorillonite (Mt@MH).

room temperature at pH_e 6.5 \pm 0.2 and 3.5 \pm 0.2, respectively. The adsorption experiment was conducted for 8 h to ensure the equilibrium isotherm adsorption condition was reached. The amount of Pb(II) and As(V) ions removed from the solution was considered as being adsorbed by Mt@MH. The Langmuir adsorption model as shown in Eq. (2) was employed to fit the adsorption isotherms of Pb(II) and As(V) ions toward Mt@MH:

$$q_e = \frac{Q_o \, b \, C_e}{1 + b \, C_e} \tag{2}$$

where q_e is the amount of metal ion adsorbed at equilibrium (mg/g), Q_o is the maximum adsorption capacity (mg/g), C_e is the equilibrium concentration (mg/L) and *b* is the adsorption constant (L/mg). Figs. 6 and 7 show that the adsorption isotherm can be well-fitted with the Langmuir model as demonstrated by the square of correlation close to unity for Pb(II) (r² > 0.97) and As(V) (r² > 0.98).

It is commonly known that the pH_e of the solution is a critical factor in the removal of metal ions and also determine the main driving force for the adsorption. The maximum adsorption capacity for Pb(II) was 38.15 mg/g at pH_e \sim 6.5. The electrostatic interaction is the dominant driving force to capture lead ions in this pH_e condition when the surface of Mt@MH was negatively charged due to formation of -NH₂OH-. In this condition, the adsorption could be increased but reduce the adsorption of metal ion through complexation. The maximum adsorption capacity of 19.10 mg/g for As(V) at pH_e ~3.5 has achieved during adsorption. The amine groups on surface of Mt@MH were protonated



Fig. 6. Langmuir isotherm plot for Pb(II) adsorption onto Mt, MH, and Mt@MH at room temperature, pH_e of 6.5 \pm 0.2, adsorbent dose of 20 mg/L, reaction time for 8 h, and shaking rate of 100 rpm.



Fig. 7. Langmuir isotherm plot for As(V) adsorption onto Mt, MH, and Mt@MH at room temperature, pH_e of 3.5 \pm 0.2, adsorbent dose of 20 mg/L, reaction time for 8 h, and shaking rate of 100 rpm.

Table 1

Comparison of maximum adsorption capacities with previous reports.

Adsorbent	Q _o (mg/g)		pHe values	Reference
	Pb(II)	As(V)		
Iron oxide	-	0.66	< 7	[32]
Mixed magnetite-maghemite nanoparticles	-	3.71	2	[33]
Na-montmorillonite	7.52	-	3.6	[34]
Montmorillonite	9.58	-	5.5	[6]
Iron oxide modified Clay-Activated Carbon	74.2	5.00	4.5	[10]
Mt@MH	38.15	19.10	6.5 for Pb(II) and 3.5 for As(V)	This study



Fig. 8. Ion uptake capacity for Pb(II) and As(V) adsorption using Mt@MH as adsorbent during 5 repeated used at room temperature, Mt@MH dose of 20 mg/L, reaction time of 8 h, and shaking rate of 100 rpm.

(-NH³⁺) and complexation of metal ions was taking place and would be occur simultaneously at low pH [9]. Moreover the amine functionalized groups more active and would be favorably interact with arsenate ion species. The adsorption of metal ions on amino magnetic nanoparticles coated montmorillonite has specific type mechanism depend on pH of solution and both of electrostatic and complexation mechanism will have good capability for ion metals adsorption.

When compared MH and Mt for Pb(II) and As(V) adsorptions, the Mt@MH showed around 2-fold higher adsorption capacity due to its high contents of iron oxide and amino groups. The adsorption capacity

of Mt@MH for Pb(II) and As(V) were also studied with those reported by other investigators using different adsorbents [6,32,33] as shown in Table 1. When compared to the same equilibrium metal ions concentration, the adsorption up take capacities of Mt@MH for Pb(II) and As(V) are approximately 4-fold and 5-fold higher than montmorillonite and mixed magnetite-maghemite nanoparticles.

The prepared Mt@MH was regenerated successfully without significantly reducing its adsorption capacity. The recovered Mt@MH was kept in 0.1 N HCl with vigorous stirring for 24 h and collected by external magnetic field. The activity of the recovered Mt@MH was slightly lower than that observed in the 1st reaction. In a 5-repetition adsorption experiment, a 7% uptake capacity decrease was observed after the 4th reaction (Fig. 8). These results indicate that the Pb(II) and As(V) ions can be adsorbed on Mt@MH and the adsorption performance has not significantly deteriorated on the course of adsorption runs. The Mt@MH has been demonstrated to act as a highly active adsorbent for wastewater treatment, especially for the removal of metal ions in the wastewater effluent.

4. Conclusions

Magnetic nanoparticles with an amine functionalized surface (MH) were successfully synthesized onto the surface layer of montmorillonite (Mt) as an organomodified mineral solid (Mt@MH). The MH nanoparticle with diameter size of around 30-50 nm was obtained. This MH nanoparticles was then coated montmorillonite via One-pot solvothermal reaction as adsorbent. The adsorption isotherms of Mt@MH as adsorbent at room temperature were well-fitted by Langmuir model with maximum adsorption capacity for Pb(II) was 38.15 mg/g at pHe 6.5 and As(V) was 19.10 mg/g at $pH_e\,\tilde{}$ 3. When compared to the same equilibrium metal ions concentration from previous study, the adsorption capacities of Mt@MH for Pb(II) and As(V) are approximately 4-fold and 5-fold higher than montmorillonite and mixed magnetitemaghemite nanoparticles. The electrostatic interaction was proposed as the primary driving force for Pb(II) and As(V) adsorption onto the Mt@ MH. In addition, the ferromagnetic properties of Mt@MH can facilitate the retrieval of the spent adsorbent around 100% from the waste stream by external magnetic field. Mt@MH seems to be a novel adsorbent in comparison to clay mineral nanocomposites and mixed magnetite-maghemite nanoparticles for effective removal of Pb(II) and As(V) ions from its aqueous solutions.

Acknowledgement

The authors acknowledge the facility support from Biomolecular Engineering Laboratory, Department of Chemical Engineering, National Taiwan University of Science and Technology/Taiwan Tech, Taipei, Taiwan.

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