

Magnetic sorption materials for recovery of organic anions from aqueous solutions

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Abstract. Layered double hydroxides (LDHs) belong to the class of two-dimensional anionic clays with a layered structure similar to brucite. The combination of sorption and magnetic properties of layered materials is promising. In this work, magnetic materials based on LDHs (MLDHs) were synthesized and their sorption and magnetic properties were studied. The synthesis of LDHs was carried out by a co-precipitation method followed by calcination. Intercalated Mg,Fe(III)-, Mg,Fe(III),Al- and Mg,Al-LDHs were prepared using LDHs by rehydration in solutions of iron(III) citrate (*Cit*) or oxalate (*Ox*) followed by the calcination at 400°C for 2-4 h. LDHs and their thermal decomposition products were characterized by FTIR spectroscopy, laser and X-ray diffraction and thermogravimetry. The sorption experiment was carried out under static conditions using glutamic acid (Glu) as a model organic compound. The maximum sorption capacity of Mg,Fe(III)-LDH and Mg,Al-Fe*Cit*-LDH calcined at 400°C was 6.6 and 8.8 mg/g Glu, respectively. The sorption kinetics of intercalated and calcined Mg,Al- and Mg,Fe(III),Al-LDH and calcined at 500°C Mg,Fe(III)-LDH was described by the pseudo-second order model. The magnetic properties of the LDHs and thermal decomposition products were retained after the adsorption of glutamic acid, making it possible to easily separate these MLDHs from the solution by magnetic field.

1 Introduction

Layered double hydroxides (LDHs) showed good performance in the field of efficient adsorbent research. LDHs belong to the group of two-dimensional inorganic layered nanomaterials. The positive charged layers contain two or more metal elements, and the anions between the layers can balance charge [1-3]. LDHs have a number of advantages such as compositional flexibility, excellent anion exchange capacity, and memory effect, and can be easily customized for functional hybrid materials and nanocomposites. LDHs are broad used in many fields, but the powdered form limits their application as adsorbent

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in water purification [4]. Therefore, the researchers combined Fe_3O_4 as a magnetic medium with LDHs to make magnetic composite materials. In terms of recycling, magnetic LDH nanocomposites (MLDH) as the adsorbents are more conducive to separation and recovery than traditional LDHs, thereby shortening the water treatment cycle and improving overall efficiency. The MLDHs have attractive broad prospects in water treatment.

Recently new approach for synthesis of MLDH composite was developed. It based on co-precipitation method using a Fe_3O_4 suspension followed drying at 60°C [4] or calcination at 500°C [5]. As it was shown by SEM and TEM images core-shell Fe_3O_4 @LDHs nanoparticles could be obtained by this approach. Jung et al. [5] synthesized magnetic core-shell Fe_3O_4 @ Mg_xAl -LDH ($x=2, 3$ and 4) composites by co-precipitation method (using the magnetite Fe_3O_4 microspheres synthesized by a solvothermal method), among which the calcined Fe_3O_4 @ Mg_4Al -LDH has the best adsorption capacity for iodide. The maximum iodide adsorption capacity was 105.04 mg/g due to its wide interlayer spacing and largest BET surface area [5]. The recyclability test of Fe_3O_4 @LDH showed that the removal performance for iodide is maintained at $>80\%$ even during the first to the fourth cycles. The adsorption capacity maximum of MLDH for removal of a phytohormone indole-3-butyric acid (IBA) was 522.6 mg/g [4]. Kinetics and adsorption isotherm followed the pseudo-second-order and Liu isotherm model respectively. The adsorbed sample can be easily magnetically separated and regenerated with NaNO_3 . The adsorption process was spontaneous and exothermic, including two path stages: surface adsorption of lamellar and interlayer anion exchange. Zhao et al. [6] prepared a magnetic ZnAl-LDH composite material, which has good adsorption energy for Cr(VI) in water due to its electrostatic attraction. Li et al. [7] synthesized a core/shell/shell Mg,Al-LDH composite material with improved magnetic separation performance for the separation and removal of pollutants such as phosphate and fluoride in water. Behbahani et al. [8] prepared Mg,Al-LDH supported on Fe_3O_4 - SiO_2 stabilized and modified with CoS-FeS chalcogenide to determine trace disulfide blue in aqueous solution and the content of bromocresol green anionic dye. The development of MLDHs for environmental applications still need fundamental investigations to understand the mechanisms of adsorption. Despite a number of advantages of the core-shell Fe_3O_4 @LDH approach, it also has disadvantages, such as the time-consuming process required to synthesize Fe_3O_4 , prepare the suspension and age Fe_3O_4 @LDH composite with heating for 24 h, as well as Fe_3O_4 particle size heterogeneity and substitution by competing ions in the adsorption process.

In this work, we used another approach for the preparation of oxide nanostructured materials [9]. The proposed method was based on chemical modification of anion-substituted LDHs. It combined the simplicity of chemical methods and the possibility to prepare nanostructures directly in the matrix. During chemical reactions of anions in the interlayer space of LDHs, the reaction zone was spatially constrained by the hydroxide layers, giving rise to the conditions similar to those in 2D nanoreactors, such as Langmuir–Blodgett films and self-assembling monolayers. It was found that chemical modification of intercalated LDHs results in the formation of iron oxide nanoparticles with different morphology and composition, depending on the composition of initial precursors and conditions of chemical modifications.

In this work magnetic composite materials based on Mg,Fe(III)-, Mg,Fe(III),Al- and Mg,Al-LDH were synthesized by co-precipitation method followed by calcination at 400 , 500 and 600°C for 2-4 h. Intercalated Mg,Fe(III)-, Mg,Fe(III),Al- and Mg,Al-LDH were prepared using obtained LDH by rehydration in solutions of iron(III) citrate (*Cit*) or oxalate (*Ox*) followed the calcination at 400°C for 2-4 h. LDHs and their thermal decomposition products were characterized by FTIR spectroscopy, laser and X-ray diffraction, and thermogravimetric analysis, and their adsorption and magnetic properties also were studied. Glutamic acid (Glu) was used as a model organic compound for glutamate anion adsorption

study by MLDHs. The results were useful to evaluate the ability of MLDHs in adsorption process of organic compounds and preparing of magnetic composite materials.

2 Material and method

2.1 Preparation of MLDHs

L-Glutamic acid (> 98.5 wt.%) and ammonium iron(III) citrate were purchased from AppliChem ITW Reagents and Sigma-Aldrich Inc. respectively. $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, NaOH , Na_2CO_3 , $\text{NH}_3 \cdot \text{H}_2\text{O}$, $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ ($\geq 98.5\%$), ethanol (96%, rectified), iso-propanol (50%) and other reagents were of analytical grade.

The Mg,Al-LDH in carbonate form was synthesized by the co-precipitation method in an alkaline solution described elsewhere [10]. Magnesium and aluminum or/and iron(III) hydroxides were co-precipitated by mixture of NaOH and Na_2CO_3 from chlorides under controlled pH 9 ± 0.5 [11]. Samples with Mg:Al ratio 2:1, Mg:Fe(III) 2:1 and 4:1, Mg:Fe(III):Al 4:1:1 were synthesized. Mg,Al- and Mg,Fe(III),Al-LDH were calcined at 400°C for 2 h (Mg,Al-400 and Mg,Fe(III),Al-400 layered double oxide samples) and Mg,Fe(III)-LDH were calcined at 400, 500 and 600°C for 4 h (Mg_xFe-400, Mg_xFe-500 and Mg_xFe-600 layered double oxide samples, $x = 2, 4$) to remove H_2O and CO_2 .

The anion exchange was performed using the Chibwe's procedure [12]. Obtained Mg,Al-400 and Mg,Fe(III),Al-400 layered double oxide samples were exposed to water solution containing 0.1 mol/L of iron(III) citrate (*Cit*) or oxalate (*Ox*) complex under stirring and heating at $55\text{-}60^\circ\text{C}$ for 4 h. As a result intercalated Mg,Al-FeO_x-, Mg,Al-Fe*Cit*-, Mg,Fe(III)-Fe*Cit*- and Mg,Fe(III),Al-FeO_x-LDH were synthesized by rehydration method due to memory effect of the layered double oxides. MLDHs were prepared using intercalated Mg,Al- and Mg,Fe(III),Al-LDH by their calcination at 400°C for 2-4 h (Mg,Al-FeO_x-400, Mg,Al-Fe*Cit*-400 and Mg,Fe(III),Al-FeO_x-400 samples).

2.2 Characterization

The MLDHs, LDHs and their thermal decomposition products were structurally characterized using powder X-ray diffraction (XRD) on a diffractometer with CuK_α radiation (Dron-3, Bourevestnik, JSC) and Spectrum 100 Series, Fourier transform infrared (FTIR) spectrometer (Perkin Elmer). Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were carried out using TGA/DSC 3+ Star System (Mettler Toledo) set-up to determine mass changes and heat effects during pyrolysis and oxidation of the samples. Particle size distribution analysis was performed using SALD-2300 instrument (Shimadzu). The Glu concentrations were measured by UV spectrophotometry (UV-2600, Shimadzu).

2.3 Adsorption experiments

Weighed MLDHs or their thermal decomposition products samples (50 mg) were dispersed in 50 mL of 10 mg/L Glu. Under stirring at a room temperature, aliquots (3-5 mL) of the dispersion solution were removed at different times. Then the Glu contents remaining in solution were determined by spectrophotometry using ninhydrin reaction [13] and monitoring the absorbance at 563 nm. The amount of the adsorbed Glu by MLDH at time t (adsorption capacity q_t , mg/g) and the removal rate of Glu (recovery, %) were calculated using determined concentrations of Glu in solution during adsorption experiment.

3 Results and discussion

3.1 Characterization of MLDHs and thermal decomposition products

The FTIR spectra in ATR mode of the calcined Mg,Al-, Mg,Fe(III)- and Mg,Fe(III),Al-LDHs did not contain OH stretching ($3500\text{--}3300\text{ cm}^{-1}$) bands and also O—C=O asymmetric stretching (1353 cm^{-1}) band of carbonate ions (Fig.1). COO⁻ and C-O stretching ($1200\text{--}1800\text{ cm}^{-1}$) and deformation ($500\text{--}800\text{ cm}^{-1}$) bands, and weak C-H deformation ($1350\text{--}1450\text{ cm}^{-1}$) bands of iron(III) citrate or oxalate complex were observed in FTIR spectra of intercalated Mg,Al-FeOx-, Mg,Al-FeCit-, Mg,Fe(III)-FeCit- and Mg,Fe(III),Al-FeOx-LDHs (Fig. 1).

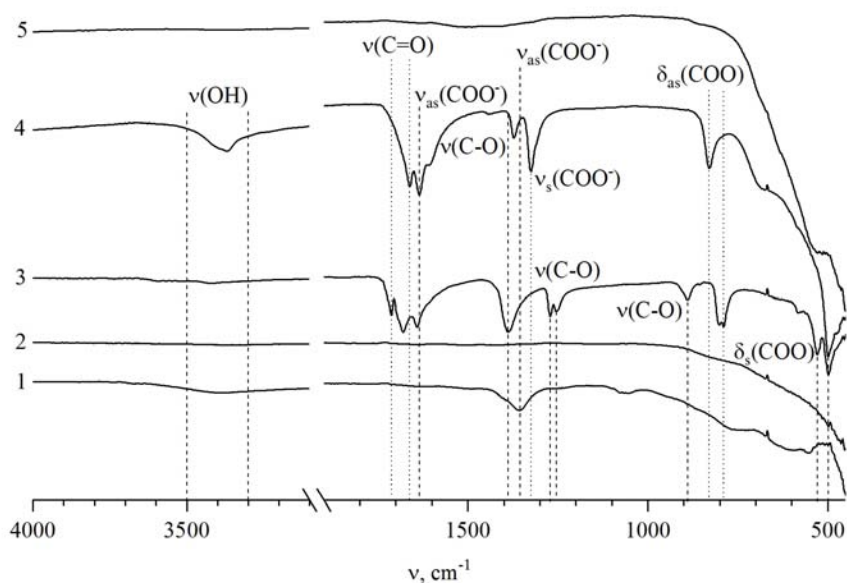


Fig. 1. FTIR: 1 – Mg,Fe(III),Al-LDH; 2 – Mg,Fe(III),Al-400; 3 – $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]$; 4 – Mg,Fe(III),Al-FeOx-LDH; 5 – Mg,Fe(III),Al-FeOx-400.

The decrease of the average particle diameter of Mg,Al- and Mg,Fe-LDHs after calcination from 35 to $30\text{ }\mu\text{m}$ and from 8 to $3\text{ }\mu\text{m}$ respectively can be explained by the destruction of the layered structure, followed by cracking of crystals, and the change in the electric charge density on the surface of the particles. Subsequent rehydration of the calcined Mg,Al-LDH using an aqueous solution of iron (III) citrate complex leads to further solid phase dispersion and a decrease of the average particle diameter of Mg,Al-FeCit-LDH to $9\text{ }\mu\text{m}$ (Table 1). However, the rehydration of Mg,Fe(III)-LDH leads to an increase in the average particle size up to $15\text{ }\mu\text{m}$ and the disappearance of a large fraction of Mg,Fe(III)-FeCit-LDH. Apparently, there is aggregation of the small particles through electrostatic interaction of anionic complexes like $[\text{Fe}_2\text{C}_6\text{H}_4\text{O}_7]_2(\text{H}_2\text{O})_2^{2-}$ with charged surface of LDH. It has been shown also, that particle size of Mg,Fe(III),Al-FeOx-LDH after calcination drastically reduces. As we believe this may be due to the destruction of the layered structure and the formation of Fe_3O_4 nanoparticles.

Table 1. Particle size of LDHs and thermal decomposition products by the laser diffraction method.

Sample	Average particle diameter (μm)	Median diameter (μm)	Modal diameter (μm)
Mg,Al-LDH	36.0	43.8	49.3
Mg,Al-400	30.2	39.1	79.3
Mg,Al-Fe <i>Cit</i> -LDH	9.2	11.0	19.0
Mg,Al-Fe <i>Ox</i> -LDH	17.4	18.5	19.0
Mg,Al-Fe <i>Ox</i> -400	1.2	0.3	0.1
Mg,Fe(III)-LDH*	8.2	9.8	19.0
Mg,Fe(III)-400*	2.7	2.6	0.2; 19.3
Mg,Fe-500*	3.2	3.4	5.8
Mg,Fe-600*	4.4	4.3	5.7
Mg,Fe(III)-Fe <i>Cit</i> -LDH	15.1	18.6	19.0
Mg,Fe(III),Al-LDH	18.1	41.5	79.3
Mg,Fe(III),Al-400	17.1	30.2	49.3
Mg,Fe(III),Al-Fe <i>Ox</i> -LDH	12.5	13.8	7.3
Mg,Fe(III),Al-Fe <i>Ox</i> -400	1.9	0.6	0.1

* Mg:Fe(III) ratio 2:1

X-ray diffraction analysis ($\text{CuK}\alpha$, λ 1.5418 Å, 2 θ scanning step 0.01°) showed the complete destruction of the layered structure of Mg,Fe(III)-LDH calcined at 500 and 600 °C and the formation of mixed oxides with the structure of magnetite, spinel and periclase. The characteristic sharp (003), (006) and (012) peaks of Mg,Al-LDH have decreased and widened in XRD pattern of Mg,Al-400 (Fig. 2). This indicates the partial destruction of the metal-hydroxide structure and the formation of amorphous metastable mixed oxides after calcination of Mg,Al-LDH at 400 °C. X-ray amorphous products containing Fe₃O₄ magnetite phase (JCPDS 65-3107) are formed after calcination of intercalated at 400°C Mg,Al-Fe*Ox*-, Mg,Al-Fe*Cit*-, Mg,Fe(III)-Fe*Cit*- and Mg,Fe(III),Al-Fe*Ox*-LDH (Fig. 2). The basal spacing, obtained from (220), (311), (400), (433), (511) and (440) diffraction peaks, indicated in XRD pattern of partially decomposed Mg,Al-Fe(III)*Cit*-400 sample and attributed to magnetite phase (Fig. 2, pattern 3), was 2.95, 2.51, 2.10, 1.69, 1.60 and 1.48 Å, respectively.

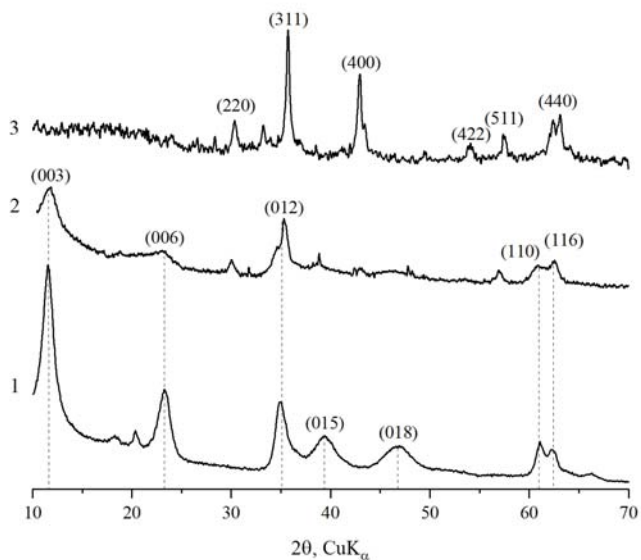
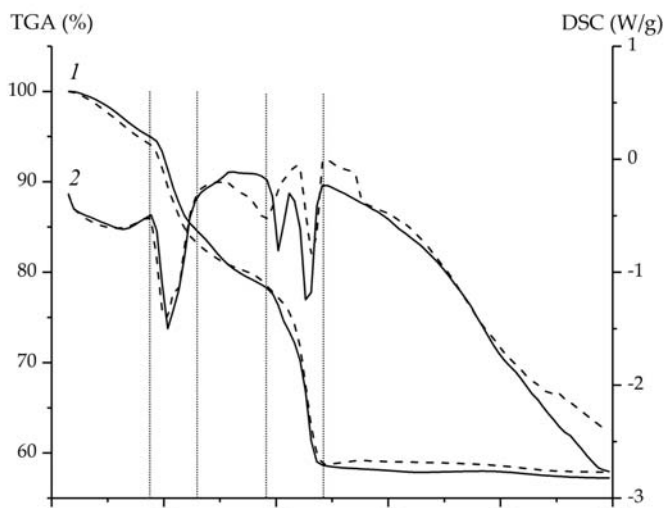


Fig. 2. XRD patterns ($\text{CuK}\alpha$, λ 1.5418 Å, 2θ scanning step 0.01°): 1 - Mg,Al-LDH, 2 - Mg,Al-400, 3 -



Mg,Al-FeCit-400.

Fig. 3. TGA (1) and DSC (2) curves for Mg,Al-FeOx- (solid) and Mg,Fe(III),Al-FeOx-LDH (dashed) samples. Program: the temperature range of 20-1000°C; heating rate 5°C/min in the inert gas (nitrogen) with gas flow rate of 50 mL/min.

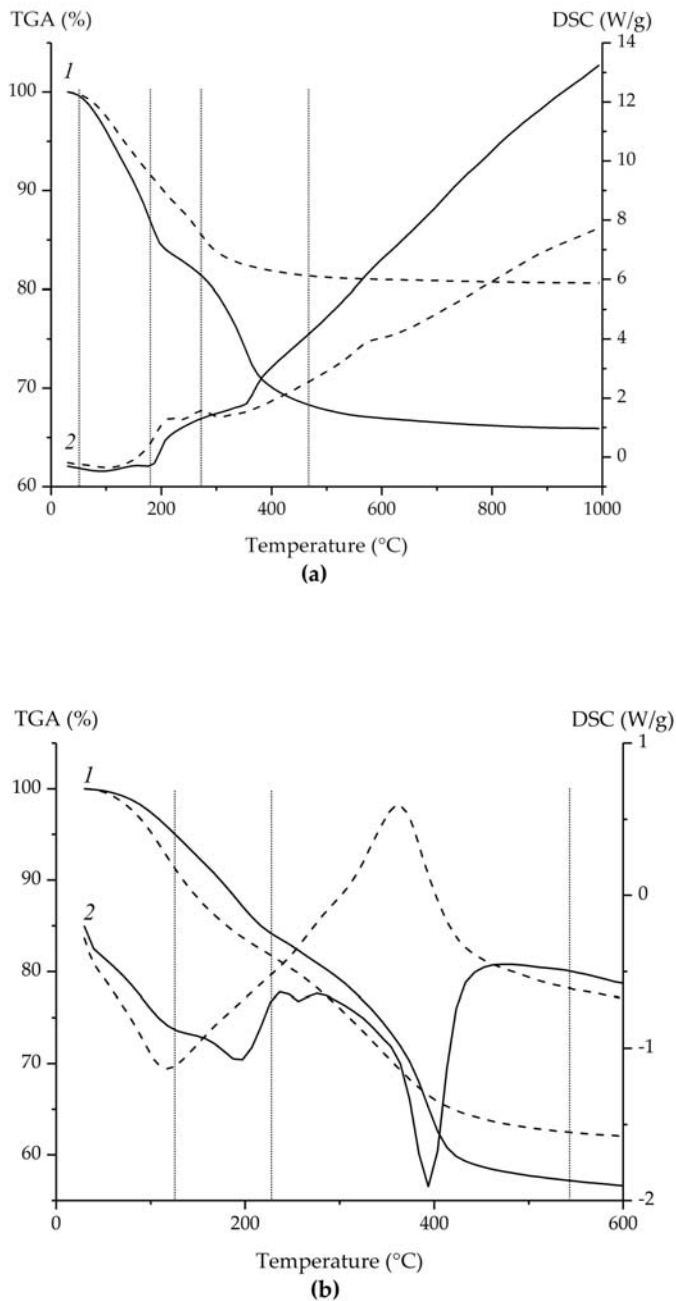


Fig. 4. TGA (1) and DSC (2) curves for: (a) Mg,Fe(III)- (solid) and Mg,Fe(III)-FeCit-LDH (dashed); (b) Mg,Al- (solid) and Mg,Al-FeCit-LDH (dashed) samples. Program: the temperature range of 20-1000°C; heating rate 5°C/min in the oxidizing atmosphere (synthetic air) with gas flow rate of 50 mL/min.

Using differential thermal analysis endothermic effects were observed at 195-250°C (removal of interlayer water and dehydration of metal-hydroxide layers) and 395-480°C (decomposition of the brucite-like layer and oxalate complex, as well as loss of interlayer carbonate ions) in DSC curves (inert gas) of the intercalated Mg,Al- and Mg,Fe(III),Al-LDHs (Fig. 3). During the thermal decomposition of Mg,Fe(III)-LDH (oxidizing atmosphere), three mass loss steps have been observed in TGA curves: the low-temperature region (50–180°C) corresponds to the removal of interlayer and adsorbed water, region at 180–275°C is attributed to the removal of interlayer water, and the high-temperature region (275–465°C) corresponds to the decomposition of metal hydroxide layers and interlayer carbonate anions (Fig. 4a). In the range from 30 to 600°C the total mass loss for Mg,Fe(III)-LDH was 33.1% and for Mg,Fe(III)-Fe*Cit*-LDH was 18.0%. In TGA and DSC curves of the oxidative thermolysis of Mg,Al-Fe*Cit*-LDH (Fig. 4b) in the range of 50-220°C (18% mass loss) dehydration processes were observed (endothermic peak at 125°C), and in a wide range of 220-535°C (20% mass loss) the processes could be attributed to decarboxylation and oxidative destruction of citrate anion (exothermic peak 335°C).

The magnetic permeability of calcined LDH Mg,Fe(III)-500, Mg,Fe(III)-600, Mg,Al-Fe*Ox*-400 and Mg,Fe(III),Al-Fe*Ox*-400 is positive and greater than for typical ferromagnets. Magnetic properties in an external strong magnetic field are most noticeable for Mg,Fe(III)-600 and Mg,Fe(III),Al-Fe*Ox*-400 sample. It should be noted that Mg,Fe(III)-, Mg,Al-, Mg,Al-Fe*Ox*, Mg,Fe(III),Al- and Mg,Fe(III),Al-Fe*Ox*-LDH samples and calcined Mg,Fe(III)-400 samples do not exhibit magnetic properties. This confirms the destruction of the layered structure and the formation of magnetite, which is responsible for the magnetic properties.

3.2 Adsorption properties and kinetics

The sorption experiment using MLDH and thermal decomposition products was carried out under static conditions with initial solution 10 mg/L glutamic acid at room temperature. The kinetics for removal of Glu was studied and two commonly used models were applied to fitting the experiment results: 1) Lagergren's first-order kinetic model and 2) Ho's pseudo second-order [14]. The maximum equilibrium sorption capacity (q_e) was 6.6 and 8.8 mg/g (recovery 76 and 88%) for Mg,Fe(III)-400 and Mg,Al-Fe*Cit*-400 with Mg:Fe(III) ratio 4:1 and 2:1 respectively, that could be explained by rehydration and partial restoration of the layered structure in solution (Table 2). It has been shown on the example of Mg,Al-LDH that sorption capacity depends on intercalated anion in iron(III) complex: sorption capacity of Mg,Al-Fe*Cit*-400 has increased 2.7 times compared to Mg,Al-Fe*Ox*-400. The sorption capacity of Mg,Fe(III)-LDH samples calcined at 500°C decreases compared to those calcined at 400°C. The sorption capacity of Mg,Al,Fe(III)-Fe(III)*Ox*-400 and Mg,Al-Fe(III)*Ox*-400 is 3.8 and 3.2 mg/L respectively.

The pseudo second-order kinetic model fits the experimental data better, indicating that the rate-limiting step is a chemical adsorption process between Glu and the intercalated Mg,Al- and Mg,Fe(III),Al-LDH calcined at 400°C (Mg,Al-Fe*Ox*-400 and Mg,Fe(III),Al-Fe*Ox*-400) and the calcined at 500°C Mg₄Fe(III)-500 (Table 2). Meanwhile, the first-order and the second-order models fit well the experimental data of Glu adsorption process by the calcined Mg,Fe(III)-400, and only the first-order model fits for Glu adsorption by Mg,Al-Fe*Cit*-400. Probably the sorption kinetics of intercalated and calcined samples depends on LDH:magnetite ratio in the obtained MLDH.

The magnetic properties of intercalated Mg,Al-Fe*Ox* and Mg,Fe(III),Al-Fe*Ox*-LDH, calcined at 400°C, and Mg,Fe(III)-LDH, calcined at 500-600°C, were preserved after glutamic acid adsorption, which could make it easy to separate them from solutions in magnetic field.

Table 2. Kinetic parameters for adsorption experiments.

Sample	q_e , mg/g	Pseudo first-order			Pseudo second-order		
		q_e , mg/g	k_1 , min ⁻¹	R ²	q_e , mg/g	k_2 , g·(g·min) ⁻¹	R ²
Mg ₄ Fe-400	6.65	6.91	$8.8 \cdot 10^{-3}$	0.9886	9.25	0.73	0.9874
Mg ₄ Fe-500	1.52	-	-	-	1.94	5.52	0.9407
Mg ₂ Fe-400	1.51	1.64	$3.4 \cdot 10^{-3}$	0.9034	1.48	9.89	0.9312
Mg ₃ Fe-500*	49.28	54.63	$1.3 \cdot 10^{-4}$	0.9945	51.35	0.19	0.9963
Mg,Al-FeOx-400	3.20	6.90	$7.1 \cdot 10^{-3}$	0.7132	3.55	3.70	0.9189
Mg,Al-FeCit-400	8.77	9.80	$6.7 \cdot 10^{-3}$	0.9659	-	-	-
Mg,Fe(III),Al-FeOx-400	3.85	-	-	-	3.67	21.26	0.9556

* [11]

4 Conclusion

The present study investigated the sorption and magnetic properties of MLDHs for glutamic acid removal. The co-precipitation method followed by iron (III) complex intercalation and calcination have been used to synthesize MLDHs and thermal decomposition products.

Mg,Fe(III)- and intercalated Mg,Al-FeCit-LDH calcined at 400°C (MLDH) might be potential magnetic materials for the removal of glutamic acid from aqueous solution. It was found that glutamic acid could be removed effectively with a removal rate of 76% (Mg,Fe(III)-400) and 88% (Mg,Al-FeCit-400). The kinetic data fit well the pseudo second-order kinetic model with good determination coefficient for intercalated and partly thermal decomposed MLDH (Mg,Al-FeOx-400 and Mg,Fe(III),Al-FeOx-400) and the calcined at 500°C LDH (Mg,Fe(III)-500) samples.

It was shown that the magnetic properties of obtained MLDH (intercalated Mg,Al-FeOx and Mg,Fe(III),Al-FeOx-LDH calcined at 400°C) and Mg,Fe(III)-LDH calcined at 500-600°C were preserved after glutamic acid adsorption. Thus they can be separated from aqueous solution due to its magnetic properties.

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