

Removal of chloride ions from desulfurization wastewater by ZnMgAl-LDHs

Leyi Zhang

School of Architecture and Engineering, Chongqing Industry Polytechnic College, Chongqing 401120, China, email: xixunko39@163.com

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ABSTRACT

Desulfurization wastewater containing high chloride ions will cause serious harm to pipelines, concrete buildings and the environment. Therefore, it is necessary to remove the high concentration of chloride ions in desulfurization wastewater. In this work, Zn-Mg-Al ternary hydrotalcite (ZnMgAl-LDHs) was prepared by chemical precipitation method and used to remove chloride ions from desulfurization wastewater. Batch adsorption experiments showed that the removal efficiency of chloride ions by ZnMgAl-LDHs reached 92% when the dosage was 8 g/L and pH was 7.0. Additionally, sulfate, carbonate, silicate and phosphate inhibited the removal of chloride ions by ZnMgAl-LDHs. Notably, the adsorption of chloride ions by ZnMgAl-LDHs was mainly controlled by chemical interactions, such as ion exchange and complexation. After three times of NaOH regeneration, the removal efficiency of chloride ions by ZnMgAl-LDHs can effectively remove chloride ions from desulfurization wastewater.

Keywords: Layered double hydroxides; Desulfurization wastewater; Chloride ions; Adsorption performance; Mechanism

1. Introduction

Wet desulfurization technology produce high concentration of chloride ions (Cl-) wastewater, which have a certain impact on the later water reuse [1]. When the desulfurization system is running, the Cl⁻ in the slurry in the absorption tower will gradually enrich, which will cause great harm to the desulfurization equipment and the surrounding environment, and will also corrode the water pipeline [2]. High Cl⁻ wastewater can also erode the concrete structure of the building, causing damage to the building structure [3]. Therefore, how to remove high Cl- wastewater from aqueous solution become an urgent problem to be solved. At present, the methods for treating high Cl- wastewater include chemical precipitation, biophysical and steam concentration evaporation [1]. However, the removal efficiency of Cl⁻ by these methods is not too high. Nowadays, the adsorption method is considered to be a promising method with low investment and good effect because of its simple operation and wide applicability [4,5].

Lavered double hydroxides (LDHs) method is based on the fact that the interlayer anions of LDHs can be exchanged with various anions (including inorganic anions, organic anions, homologous anions, heteropoly acid ions and anions of coordination compounds) [6,7]. LDHs can reabsorb anions under certain conditions to achieve the purpose of removing anions [7]. Hu and Lv [8] used Mg-Al-LDHs to remove chloride ions and the removal efficiency of Mg-Al-LDHs for low-concentration chlorine-containing wastewater could reach 97%. Lei et al. [9] used magnesite as raw material to prepare magnesium aluminum hydrotalcite to treat chlorine-containing wastewater. The result suggested that the increase of interlayer spacing was beneficial to the removal of chloride ions, and the optimum removal efficiency of chlorine-containing wastewater with lower concentration was 85.96%. The LDHs method has a promising effect on the treatment of chlorine-containing wastewater and could be reused, but it is mainly used in experimental research. With the development of new LDHs, the LDH method will have a wide application prospect in the field of water treatment.

Herein, ZnMgAl-LDHs prepared by chemical precipitation method was used to remove chloride ions from desulfurization wastewater. The effects of various influencing factors (removal time, initial Cl⁻ concentration, solution pH, co-existing ions, dosage) on the removal performance of chloride ions by ZnMgAl-LDHs were investigated through single factor adsorption experiment, and the suitable Cl- removal process conditions were determined. Moreover, the reusability of ZnMgAl-LDHs was investigated by adsorption-desorption experiments. The structural changes of LDHs before and after chloride ion removal were investigated by X-ray diffraction (XRD), Fouriertransform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Combined with the adsorption kinetics and isothermal adsorption characteristics of chloride ion removal, the mechanism of chloride ion removal by ZnMgAl-LDHs was preliminarily discussed.

2. Materials and methods

2.1. Reagents

 $Mg(NO_3)_{2'}$ Al $(NO_3)_{3'}$ Zn $(NO_3)_{2'}$ NaOH and NaCl were analytical reagent (AR) and purchased from Sinopharm Reagent Group Co., Ltd., (Shanghai, China). The water used for the experiments was deionized water (DW).

2.2. Preparation of ZnMgAl-LDHs

The ZnMgAl-LDHs were prepared by co-precipitation [10]. According to proportional of 2:1:1, $Zn^{2+}:Mg^{2+}:Al^{3+}$ was dissolved in 100 mL DW to obtain mixed solution. Approximately 5.0 g NaOH was dissolved in 100 mL of DW. The two solutions were mixed in flask and the pH was adjusted to 12.0. During the reaction, the flask was continuously ventilated with N₂ and transferred to water bath at 60°C for 6 h. The reaction was cooled to room temperature and washed 3 times by DW. The obtained product was dried in a vacuum freeze dryer for 24 h to obtain ZnMgAl-LDHs.

2.3. Adsorption experiments

In adsorption experiments, 8 g/L ZnMgAl-LDHs and 500 mg/L Cl⁻ solution were mixed. Besides the dosage experiments, the ZnMgAl-LDHs was dosed at 8 g/L in the remaining adsorption experiments. The initial pH solution was adjusted by 0.1 mol/L of HNO₃ or NaOH. The mixture placed in an oscillator at 150 rpm to research the effects of dosage 1~10 g/L, solution pH 2~10, co-existing ions (sulfate, carbonate, silicate and phosphate), reaction time 10~600 min, temperature 15°C~35°C and initial concentration 500~2,000 mg/L on the Cl⁻ adsorption on ZnMgAl-LDHs. After the reaction was completed, 10 mL of the solution was centrifuged for 10 min. The Cl⁻ concentration of the adsorbed solution was measured by a chloride ion content meter. Finally, the removal efficiency [Eq. (1)] and adsorption capacity [Eq. (2)] of Cl⁻ were calculated.

$$R = \frac{C_0 - C_e}{C_0} \times 100\%$$
(1)

$$q = \frac{\left(C_0 - C_e\right) \times V}{m} \tag{2}$$

where *R* indicates the removal rate of Cl⁻, %; C_0 indicates the initial concentration of Cl⁻, mg/L; C_e is the residual concentration of Cl⁻ in the solution after adsorption equilibrium, mg/L; *q* indicates the adsorption capacity, mg/g; *V* is the volume of solution, mL; *m* is the mass of adsorbent, mg.

2.4. Structural characterization of materials

The crystalline structure was analyzed by an X-powder diffractometer (XRD, D8 ADVANCE, Bruker, Germany). The surface morphological features were analyzed by scanning electron microscope (SEM, JEOL JSM-7500F, Japan). Zeta potential analyzer (Zetasizer, Malvern, UK) analyses the change in surface potential at different pH. The functional groups on the surface were determined by Fourier-transform infrared spectroscopy (FTIR, Nicolet-460, Thermo Fisher, USA) with a scanning wavenumber of 4,000~400 cm⁻¹.

2.5. Analytical model

2.5.1. Adsorption kinetic

The pseudo-first-order model [Eq. (3)], the pseudosecond-order model [Eq. (4)], Elovich model [Eq. (3)] and the intraparticle diffusion model [Eq. (5)] were used for fitting analysis.

$$q_t = q_e \left(1 - e^{-k_1 t} \right) \tag{3}$$

$$q_{t} = \frac{q_{e}^{2}k_{2}t}{1 + q_{e}k_{2}t}$$
(4)

$$q_t = K_d t^{1/2} + C_i$$
(5)

where q_e and q_t are the adsorption capacity at the equilibrium time and time "t" time (mg/g), respectively; k_1 and k_2 represent adsorption rate constant of the pseudo-first-order (min⁻¹) and the pseudo-second-order (g/mg·min), respectively; K_d is the intraparticle diffusion rate constant (mg/m·min^{1/2}); C_i is the boundary layer constant.

2.5.2. Adsorption isotherm

The Langmuir isotherm model [Eq. (6)] and the Freundlich isotherm model [Eq. (7)] were used to fit these data.

$$q_{e} = \frac{q_{\max}K_{L}C_{e}}{1 + K_{L}C_{e}}, R_{L} = \frac{1}{1 + K_{L}C_{0}}$$
(6)

$$q_e = K_f C_e^{1/n} \tag{7}$$

where q_e is the adsorption capacity at equilibrium (mg/g); C_e is the Cl⁻ concentration at adsorption equilibrium (mg/L);

 q_{max} , K_L and R_L are the maximum adsorption capacity of Cl⁻ (mg/g), Langmuir equilibrium constant (L/mg) and separation factor, respectively; K_f and n represent the Freundlich affinity coefficient (mg¹⁻ⁿ·Lⁿ/g) and Freundlich constant related to the surface site heterogeneity, respectively.

2.5.3. Adsorption thermodynamics

The thermodynamic investigation of Cl⁻ removal at different temperatures (288, 298 and 308 K). The thermodynamic parameters for the removal process were calculated using Eqs. (8) and (9).

$$\ln K^{\circ} = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT}$$
(8)

$$\Delta G^{\circ} = -RT\ln K^{\circ} \tag{9}$$

where *R* is the gas constant (8.314 kJ/mol). K° is the equilibrium constant and *T* is the Kelvin temperature (K). The ΔG° , ΔH° and ΔS° represent the standard free energy (kJ/mol), enthalpy (J·k/mol), and entropy (kJ/mol), respectively.

3. Results and discussion

3.1. Dosage

Fig. 1a showed the effect of dosage on the chloride ions adsorption. As the dosage increased from 1 to10 g/L, the removal efficiency of chloride ions by ZnMgAl-LDHs increased from 18.65% to 93.04%, while the adsorption capacity of chloride ions per unit mass gradually decreased. This was mainly due to the adsorption sites provided by the adsorbent increased as the dosage increased, thus facilitating the removal of chloride ions [11]. At dosage > 8 g/L, the removal efficiency was stable, and the adsorption capacity decreased. This was mainly because the dosage increase to a certain extent caused the adsorbent agglomeration, hindering the Cl⁻ diffusion in the solution to the adsorbent surface [5]. Finally, the adsorption capacity of chloride ions per unit mass of adsorbent decreased. Consequently, a dosage of 8 g/L was used in subsequent experiments.

3.2. Co-existing ions

Additionally, the effect of co-existing ions on the removal of chloride ions was investigated separately in



Fig. 1. Effects of dosage (a), co-existing ions (b) and initial solution pH (c) on the Cl⁻ removal by ZnMgAl-LDHs. Zeta potential of ZnMgAl-LDHs at different pH (d).

this work (Fig. 1b). Sulfate, carbonate, silicate and phosphate had a significant inhibitory effect on the removal of chloride ions. Studies shown that when chloride ions coexisted with the above anions, LDHs preferentially reacted with the above ions, thereby reducing the adsorption of chloride ions by LDHs [12].

3.3. Initial pH

As shown in Fig. 1c, the worst removal efficiency and adsorption capacity was observed at pH 2. The removal efficiency and adsorption capacity increased rapidly with the initial pH increase. The maximum removal efficiency and adsorption capacity were reached at pH 7.0. The zeta potential of ZnMgAl-LDHs is shown in Fig. 1d. As the solution pH increased, the zeta potential value changed from positive to negative, which indicated that the surface electrical properties of ZnMgAl-LDHs changed from positive to negative. According to calculation, the zero potential point of ZnMgAl-LDHs was 7.39. This indicated that the adsorption of chloride ions by ZnMgAl-LDHs was not affected by electrostatic effects [13].

3.4. Adsorption kinetics

Adsorption kinetics determines the adsorption rate of adsorbents, and then determines the adsorption efficiency. At different concentrations, the adsorption of chloride ions by ZnMgAl-LDHs increased rapidly at the initial stage, then slowed down until the adsorption equilibrium (Fig. 2a). At 360 min, the adsorption reaction had reached equilibrium. Therefore, the adsorption of chloride ions by ZnMgAl-LDHs could be divided into two stages: fast and slow adsorption [14].

The pseudo-first-order model (Fig. 2b, R^2 less than 0.900) was not suitable for fitting the adsorption of chloride ions on ZnMgAl-LDHs, while the pseudo-second-order model (Fig. 2c) fitted the adsorption of chloride ions well ($R^2 = 0.999$), indicating that the adsorption of chloride ions was mainly controlled by chemisorption [15]. The intraparticle diffusion model (Fig. 2d) fitted the adsorption data well ($R^2 = 0.948$ ~0.992), indicating that intraparticle diffusion was one of the rate-limiting steps in the adsorption process [3,7]. The fitted curve did not cross the origin, which would indicate that intraparticle diffusion was not the only controlling process and that the adsorption rate



Fig. 2. Effect of adsorption time on the Cl⁻ removal by ZnMgAl-LDHs (a). The adsorption model of pseudo-first-order (b), pseudo-second-order (c), and intraparticle diffusion (d).

was also influenced by membrane diffusion [16]. Therefore, the adsorption of chloride ions by ZnMgAl-LDHs can be divided into 2 stages: (i) rapid diffusion of chloride ions from the solution to the surface of ZnMgAl-LDHs; (ii) the chloride ion diffused through the pores and adsorbed on the active sites in the pores to achieve adsorption equilibrium.

3.5. Adsorption isotherm

The adsorption of chloride ions by ZnMgAl-LDHs was consistent with the Langmuir model ($R^2 = 0.903 \sim 0.957$), indicating that the adsorption was approximately unilamellar. This result suggested that the complexation reaction between the O-containing functional groups on the LDHs and chloride ions [17]. Furthermore, the maximum adsorption capacity was 104 mg/g at 25°C. According to the separation factor R_1 (Fig. 3b), it can be judged whether the adsorbent can effectively adsorb the adsorbate. $0 < R_1 < 1$ was conducive to adsorption and $R_1 > 1$ was not conducive to adsorption. $R_1 = 1$ and $R_1 = \overline{0}$ were linear adsorption and irreversible adsorption, respectively [13]. In this work, $R_1 = 0.008 \sim 0.062$ in the range of initial chloride ion concentration, this suggested that the adsorption process of ZnMgAl-LDHs on chloride ions was favorable [12]. R_{r} decreased with the increase of initial concentration of chloride ion, indicating that the greater the initial concentration of chloride ion, the more conducive to the adsorption process. Moreover, the high reaction temperature was able to reduce the R_L value, which indicated that the removal of chloride ions from aqueous solutions by ZnMgAl-LDHs could be promoted under high temperature conditions [12]. According to the Langmuir model, the maximum adsorption of chloride ions by ZnMgAl-LDHs can reach 104 mg/g at 25°C.

As shown in Table 3, ΔG° was negative, indicating that the adsorption of chloride ions by ZnMgAl-LDHs was spontaneous [11]. The value of ΔG° decreased with increasing temperature, indicating that the high temperature facilitated the adsorption process [18]. The value of ΔS° was greater than 0, which indicated that the confusion at the adsorbent-water phase interface increased during the adsorption process [18]. Combining the results of adsorption kinetics study, it could be assumed that the chemical adsorption was the dominant mechanism in the chlorine ions removal by ZnMgAl-LDHs.

3.6. Adsorption mechanism analysis

3.6.1. Scanning electron microscopy

The SEM images of ZnMgAl-LDHs were displayed in Fig. 4. Before adsorption, the surface was rough, porous



Fig. 3. Isothermal adsorption model of Cl⁻ removal by ZnMgAl-LDHs (a). The R_L value of Cl⁻ removal on ZnMgAl-LDHs (b). Plot of $\ln K^\circ$ vs. 1/T for the estimation of thermodynamic parameters (c).

	Pseudo-first-order model			Pseudo-second-order model			
	$q_{e,cal} (mg/g)$	$k_1 ({\rm min}^{-1})$	R^2	$q_{e,cal} (mg/g)$	k_2 (g/mg·min)	R^2	
500 mg/L	20.09	7.81 × 10 ⁻³	0.864	60.24	0.840×10^{-3}	0.999	
800 mg/L	48.19	8.30×10^{-3}	0.917	97.56	0.320×10^{-3}	0.999	
1000 mg/L	42.52	7.08×10^{-3}	0.869	107.78	0.344×10^{-3}	0.999	
	Intraparticle diffusion model						
	k_{d1} (mg/m·min ^{1/2})	<i>C</i> ₁	R^2	k_{d2} (mg/m·min ^{1/2})	<i>C</i> ₂	R^2	
500 mg/L	2.375	19.27	0.978	0.018	56.05	0.986	
800 mg/L	4.000	21.36	0.992	0.041	88.17	0.980	
1000 mg/L	4.250	29.41	0989	0.039	96.64	0.948	

 $T(^{\circ}C)$

15°C

25°C

35°C

Table 1	
Kinetic model fit	ting parameters

Table 2 Fitting parameters of adsorption isotherm

Table 3		
Thermodynamic parameters of	of Cl [_] removal or	n ZnMgAl-LDHs

 ΔH° (J·k/mol)

175.450

 ΔG° (kJ/mol)

-2.385

-3.821

-5.458

	Langmuir model			Freundlich model		
	q _{max} (mg∕g)	K _L (L/mg)	R^2	$K_f(\mathrm{mg}^{1-n}\cdot\mathrm{L}^n/\mathrm{g})$	п	<i>R</i> ²
15°C	96	0.030	0.957	40.001	8.000	0.680
25°C	104	0.041	0.903	46.002	8.500	0.566
35°C	112	0.059	0.933	52.127	8.546	0.603

and loose, showing an irregular hexagonal plate sheet structure with clear edges and corners, which was typical LDHs morphology. Moreover, the surface of LDHs before adsorption was relatively fluffy [16]. After adsorption, the layers were tiled and stacked, the agglomeration phenomenon was more obvious and the collapse occurs, but the basic structure remains unchanged.

3.6.2. X-ray diffraction

The crystal structure of ZnMgAl-LDHs was investigated using XRD (Fig. 5a). According to the Inorganic Crystal Structure Database (ICSD), the XRD spectra was the typical of the LDHs phase (PDF#89-0460) [15]. There were strong reflection peaks on (003), (006) and (009) planes, and wide asymmetric peaks on (110) and (113) planes, indicating that the materials prepared had the structural characteristics of ZnMgAl-LDHs [19]. The interlayer spacing was calculated to be 0.767 nm according to the Bragg formula [13]. After adsorption, the diffraction peaks all appeared to weaken, while the original structure was maintained. This indicated that the ZnMgAl-LDHs did not undergo hydrolysis [17]. Additionally, the layer spacing of the ZnMgAl-LDHs after adsorption was 0.754 nm, which may be due to the ion exchange between NO_{-}^{-} and chloride ions and the reduction of the layer spacing [15].

3.6.3. Fourier-transform infrared spectroscopy

As shown in Fig. 5b, the surface of ZnMgAl-LDHs was rich in functional groups. 514 and 607 cm⁻¹ were bending

vibration peaks of M–O (M represented Zn, Mg and Al) [7]. 1,382 cm⁻¹ was a stretching vibration peak of N–O asymmetry of NO₃ [17]. The 1,641 cm⁻¹ was the H–O–H stretching vibration peak of the hydroxyl functional group of the interlayer water molecule [14]. The O-H stretching vibration band of ZnMgAl-LDHs was at 3,423 cm⁻¹ and the peak intensity was large [12]. This indicated that ZnMgAl-LDHs had a large number of hydroxyl groups on the surface, and the hydroxyl groups can complex with chloride ions in coordination reactions, which can effectively remove chloride ions from the wastewater [19]. After adsorption, the wavenumber of the -OH group shifted to 3,435 cm⁻¹ and the intensity weakened, which indicated that the chloride ions reacted with the -OH groups [16]. In addition, the N–O group showed a weakening after adsorption. This was presumed to be due to the ion exchange between the nitrate in the layer of ZnMgAl-LDHs and chloride ions, resulting in a weakening of the N-O groups [15]. Notably, the intensity and wavenumber of M-O groups did not change, suggesting that the ZnMgAl-LDHs was structurally stable and not hydrolyzed [9]. According to the analysis, the adsorption of chloride ions by ZnMgAl-LDHs could be involved in the complexation with -OH groups and the ions exchange with NO₃.

3.7. Reproducibility of ZnMgAl-LDHs

Reusability is considered a key indicator to evaluate the efficient performance of an adsorbent. In this work, different chemical reagents (e.g., deionized water, HCl, NaOH, NaHCO₃, HNO₃ and NaCl) were used as regenerant. The regeneration of ZnMgAl-LDHs was the best when NaOH was used as the regenerant (Fig. 6a). The removal efficiency of Cl⁻ on regenerated ZnMgAl-LDHs were

 ΔS° (kJ/mol)

48.250



Fig. 4. Scanning electron microscopy images of ZnMgAl-LDHs before (a) and after (b) adsorption of Cl-.



Fig. 5. X-ray diffraction (a) and Fourier-transform infrared spectroscopy (b) spectra of ZnMgAl-LDHs before and after Cl⁻ removal.



Fig. 6. Regeneration performance of ZnMgAl-LDHs under different regenerant (a) and the removal efficiency of chloride ions by ZnMgAl-LDHs under NaOH as regenerant (b). The X-ray diffraction of ZnMgAl-LDHs after three regenerations (c).

92.58%. Therefore, NaOH was used as the regenerant for 3 regeneration experiments. After 3 regeneration experiments (Fig. 6b), the Cl⁻ removal efficiency on regenerated ZnMgAl-LDHs was 80.18%. The decrease in removal efficiency could be attributed to the adsorbent loss or active adsorption site reduced [13]. However, after 3 regeneration experiments, the removal efficiency remained above 80%, indicating that ZnMgAl-LDHs held the potential to remove Cl⁻ form wastewater. Notably, previous literature demonstrated that the removal of chloride ions by Mg-Al-LDO obtained by calcination of Mg-Al-LDHs remained above 90% after 11 repetitive experiments, indicating that calcination could considerably improve the usability of LDHs [11]. The regeneration of ZnMgAl-LDHs appeared to be very fragile compared to the strong repetition capacity of Mg-Al-LDO. Nevertheless, the adsorption capacity of ZnMgAl-LDHs (104 mg/g) for chloride ions was much higher than that of Mg-Al-LDO (57 mg/g), indicating that ZnMgAl-LDHs was a promising material for chloride ion removal. Simultaneously, this provided a new direction and purpose for our future work. The XRD spectra was carried out on the ZnMgAl-LDHs after three repetitive experiments, and the results showed that the structure of ZnMgAl-LDHs was still evident, which demonstrated the high stability of the ZnMgAl-LDHs. However, the intensity of the characteristic peaks in the ZnMgAl-LDHs displayed a very noticeable decrease after all three repetitive experiments, which was attributed to the collapse of the ZnMgAl-LDHs after adsorption [13].

4. Conclusion

In this work, the ternary ZnMgAl-LDHs was prepared by chemical precipitation method. The surface of ZnMgAl-LDHs was rough, porous and loose. The Cl⁻ removal process on ZnMgAl-LDHs proved to the pseudo-second-order kinetics and Langmuir model, suggesting that the removal reaction was a uniform chemical adsorption. Furthermore, the maximum adsorption capacities were 104 mg/g. The adsorption mechanism was involved in complexation and ions exchange. Moreover, the removal efficiency of chloride ions remained above 80% after three times of ZnMgAl-LDHs regeneration. This work indicated ZnMgAl-LDHs was a promising material for the removal of chloride ions from desulphurization wastewater.

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Conflicts of interest

The authors declare that i have no competing financial interests.

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