

Theoretical Research on Zeolite-Supported In-situ Grown LDO for Fluoride Removal in High-Salinity Wastewater by Memory Reconstruction

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Article

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Abstract

Layered double hydroxides (LDHs) possess a tunable layered crystal structure, excellent anion exchange capacity, and a unique high-temperature memory reconstruction effect, showing promising application prospects in the treatment of inorganic anion pollution in water bodies. However, pure LDH powders suffer from inherent engineering application limitations, including easy agglomeration of nanosheets, low solid-liquid separation efficiency, and poor adsorption selectivity for fluoride ions in high-salinity systems. In this study, acidified clinoptilolite was adopted as a rigid support carrier. A nitrate-intercalated MgAl-LDH precursor was in-situ grown on the zeolite surface via a co-precipitation method under a nitrogen atmosphere isolated from carbon dioxide, and a zeolite-based LDO core-shell composite adsorbent was fabricated by controlled calcination at 400–450°C. The full preparation process, including zeolite acidification pretreatment, in-situ LDH coating, and calcination-induced LDO transformation, was systematically elaborated. Key preparation parameters, such as calcination temperature, atmospheric environment, and types of interlayer anions, were optimized. Combined with BET characterization and static adsorption experimental data, the evolution law of the material's microstructure was analyzed, and the multiple synergistic adsorption mechanism of zeolite-based LDO for fluoride removal from high-salinity wastewater relying on the memory effect was revealed. The adsorption experimental results demonstrate that the specific surface area of the prepared zeolite-based LDO reaches 136.42 m²/g, which is significantly higher than that of pure LDO (68.75 m²/g). Under the conditions of an initial fluoride concentration of 50 mg/L, pH value of 6, and coexisting high salts (0.1 mol/L NaCl and 0.05 mol/L Na₂SO₄), the maximum saturated adsorption capacity of zeolite-based LDO is up to 127.23 mg/g, which is 4.1 times that of single acidified zeolite (31.06 mg/g). Moreover, 82.6% of the initial adsorption capacity can be retained after five regeneration cycles. The composite adsorbent features low raw material cost and excellent recyclability, which is suitable for the advanced purification treatment of industrial high-salinity fluoride-containing wastewater. This work can provide comprehensive theoretical support and technical references for the industrial development and engineering application of zeolite-based LDO adsorption materials.

Introduction

Fluoride is a typical toxic anionic pollutant in aqueous environments. Industries including electronic wafer manufacturing, smelting, electroplating, fluorine chemical engineering, and coal chemical engineering discharge a large amount of high-salinity fluoride-containing tailwater. Excessive fluoride concentrations in water can damage human bones and tooth enamel. Conventional defluoridation adsorbents are susceptible to competitive interference from coexisting ions such as Cl, SO, and CO, resulting in insufficient adsorption capacity and fluoride selectivity to meet wastewater discharge standards.

Layered double hydroxides (LDHs), commonly known as hydrotalcite-like materials, are a class of positively charged anionic clay minerals. The metal cations (e.g., Mg and Al) in the laminates can undergo isomorphic substitution, and the interlayer region can accommodate anions such as NO and

organic acid radicals to balance the charge, endowing LDHs with large anion exchange capacity and tunable microstructures [1–5]. After calcination at 300–500°C, the interlayer crystal water and exchangeable anions are removed, and the ordered layered structure collapses, forming amorphous layered double oxides (LDOs) [6]. When exposed to anion-containing aqueous solutions, LDOs can spontaneously reconstruct the LDH layered structure through the unique “memory effect” and selectively capture anions in water, demonstrating great potential as high-performance anion-selective adsorption media [7–9].

Pure powdered LDH/LDO materials exhibit prominent application limitations. The nanosheets tend to stack and agglomerate severely, encapsulating a large number of active sites. Additionally, the low bulk density of powder adsorbents leads to slow sedimentation and poor solid–liquid separation performance after water treatment. Moreover, high-concentration salt ions and carbonate ions in water compete for interlayer exchange sites, significantly weakening the fluoride removal efficiency [10–12]. In-situ growth of LDHs on porous natural mineral carriers to construct core–shell composite materials has been proven as an effective strategy to overcome the above drawbacks. Clinoptilolite features abundant reserves, low cost, developed internal pore channels, and excellent thermal stability below 450°C. After acid etching with dilute nitric acid, numerous active hydroxyl groups ($\equiv \text{Si}-\text{OH}$ and $\equiv \text{Al}-\text{OH}$) are generated on the mineral surface, which serve as heterogeneous nucleation sites for LDH growth. This strategy can effectively disperse the LDO shell and inhibit agglomeration, while improving the overall sedimentation performance of the adsorbent [13–16].

In this work, acidified clinoptilolite was used as the substrate to in-situ synthesize zeolite-based MgAl-NO-LDH precursors. Zeolite-based LDO core–shell adsorbents were successfully prepared via programmed temperature calcination. The complete preparation process and key control parameters were systematically summarized. Combined with physicochemical characterization and static adsorption experimental results, the microstructure evolution of the composite and the synergistic adsorption mechanism of fluoride in high-salinity water were clarified. Furthermore, the application advantages of the prepared composite in the treatment of industrial fluoride-containing wastewater were comprehensively analyzed.

Experimental Section

1.1 Instruments and Reagents

Experimental reagents

Natural clinoptilolite (200 mesh); magnesium nitrate, aluminum nitrate, sodium hydroxide, sodium fluoride, sodium chloride, sodium sulfate, and nitric acid, all of analytical grade. Deionized water boiled to remove dissolved carbon dioxide was used throughout the experiments.

Experimental instruments

Electronic analytical balance, constant-temperature water bath stirrer, dual-channel peristaltic pump, tubular muffle furnace, and vacuum drying oven. Material characterization equipment included X-ray diffractometer (XRD), Fourier transform infrared spectrometer (FTIR), specific surface area and pore size analyzer (BET), scanning electron microscope (SEM), and ion chromatograph (IC). Adsorption experimental equipment consisted of a constant-temperature shaking incubator, pH meter, and high-speed centrifuge.

1.2 Preparation Process of Zeolite-Based LDO Core-Shell Adsorbent

1.2.1 Acidification Pretreatment of Zeolite

Natural clinoptilolite was crushed and sieved, and 80–200 mesh powder was selected for acid modification. A dilute nitric acid solution of 2 mol/L was prepared and mixed with zeolite powder. The mixture was stirred and acidified in a constant-temperature water bath at 60°C for 6 h. The acid etching process broadened the internal pore channels of zeolite and generated abundant active silicon hydroxyl and aluminum hydroxyl sites on the mineral surface. After acidification, the zeolite powder was repeatedly rinsed with CO₂-removed boiling deionized water until the pH value of the filtrate stabilized at 5–6. The washed zeolite was dried in an oven at 105°C to obtain the acidified zeolite substrate.

Basic characterization data: The specific surface area of raw zeolite is 23.17 m²/g. After acidification, the BET specific surface area of zeolite increases to 47.54 m²/g, the average pore diameter expands from 3.26 nm to 5.19 nm, and the surface hydroxyl content rises by 112%. Abundant hydroxyl sites on the surface can anchor metal ions (Mg²⁺, Al³⁺), inducing heterogeneous nucleation of LDHs exclusively on the zeolite surface and avoiding independent precipitation of free LDH powders, thereby forming a uniform and intact coating layer.

1.2.2 Synthesis of Zeolite-Based MgAl-NO₃-LDH Precursor under N₂ Atmosphere

A nitrate system with a molar ratio Mg²⁺:Al³⁺ = 3:1 was adopted. Nitrogen was continuously purged throughout the whole process to isolate carbon dioxide and prevent intercalation of impurity CO₃²⁻, which would otherwise reduce the fluoride selectivity of the material. The detailed procedures are described as follows:

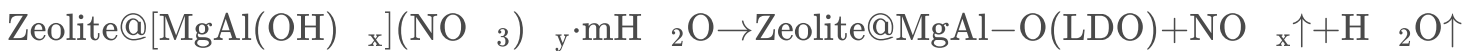
Acidified zeolite was dispersed in decarbonized deionized water at a solid–liquid ratio of 1:10. Nitrogen was bubbled continuously into the three-necked flask for 30 min to completely remove dissolved CO₂ in the solution.

A mixed magnesium-aluminum nitrate solution (1 mol/L) and decarbonized NaOH alkaline solution (2 mol/L) were prepared separately. The metal salt solution and alkaline solution were synchronously added dropwise at a low rate of 1.0 mL/min via two peristaltic pumps. The system temperature was maintained

at 55–65°C, and the pH value was precisely controlled within 9.2–10.0 under continuous stirring with nitrogen protection. The dropping rate was strictly regulated to realize heterogeneous nucleation only on the zeolite surface without generating free LDH crystals. After titration, the sealed system filled with nitrogen was aged at 60°C for 4–6 h. The product was rapidly suction-filtered and washed 3–4 times with warm decarbonized deionized water, followed by vacuum drying at 60°C for 12 h. Finally, nitrate-intercalated zeolite-based MgAl-LDH precursor was obtained.

1.2.3 Fabrication of Zeolite-Based LDO via Programmed Temperature Calcination

The dried zeolite-supported MgAl-NO₃-LDH powder was spread evenly in a ceramic crucible and calcined in a muffle furnace under air atmosphere. The heating rate was controlled at ≤ 5°C/min, and the material was calcined isothermally at 400–450°C for 4 h. Upon the completion of calcination, the crucible was immediately sealed to isolate air and cooled naturally to room temperature. The calcination reactions are listed below:



Under high-temperature conditions, interlayer crystal water and nitrate anions of LDHs are eliminated, and the ordered layered hydroxide sheets collapse to form amorphous Mg-Al mixed metal oxides (LDO) with loose porous structure and high specific surface area. Within the calcination temperature range of 450°C, the aluminosilicate framework crystal structure of zeolite remains intact and stable, which can continuously support the active LDO shell without damage or peeling of the core-shell structure.

1.2.4 Control Criteria for Key Preparation Processes

The maximum calcination temperature shall not exceed 500°C. When the temperature exceeds 500°C, Mg-Al oxides sinter at high temperature to form stable Mg-Al spinel crystalline phase. Consequently, the material permanently loses the memory reconstruction capability and fluoride adsorption performance. The saturated adsorption capacity of samples calcined at 500°C is only 26.81 mg/g, decreasing by 78.9% compared with samples treated at 450°C.

The heating rate is strictly controlled ≤ 5°C/min. For samples prepared with a heating rate of 10°C/min, massive peeling of the LDO shell can be observed via SEM, the proportion of free oxides reaches 31.7%, and the adsorption capacity drops to 73.56 mg/g.

Seal the crucible completely to isolate air throughout the cooling process. Zeolite-based LDO cooled in an open environment absorbs CO₂ from air, leading to 42.3% carbonate-intercalated products and a 45.2% reduction in saturated adsorption capacity.

The precursor is limited to nitrate-intercalated LDHs. Calcined products of carbonate-type LDHs preferentially adsorb CO₃²⁻ during reconstruction, with a saturated adsorption capacity of merely 40.15

mg/g and extremely poor fluoride selectivity. Carbonate raw materials are forbidden throughout the synthesis process.

1.2.5 Static Adsorption Experimental Methods

Simulated fluoride-containing wastewater with different NaF concentrations was prepared, and a high-salinity interference system (0.1 mol/L NaCl + 0.05 mol/L Na₂SO₄) was constructed, with solution pH adjusted from 3 to 10. Exactly 0.05 g adsorbent was added into 50 mL fluoride solution, followed by constant-temperature shaking at 25°C. Aliquots were collected at set intervals and filtered through a 0.22 µm membrane filter. The residual fluoride concentration was determined by ion chromatography, and the equilibrium adsorption capacity q_e was calculated by the following formula:

$q_e = \frac{(C_0 - C_e)V}{m}$ Where: C_0 & C_e – initial and equilibrium fluoride ion concentration, mg/L; V – volume of solution, L; m – dosage of adsorbent, g.

1.2.6 Regeneration Cycle Experiment Method

The zeolite-reconstructed LDH powder saturated by fluoride adsorption was calcined at 450°C for 4 h, followed by sealed cooling to regenerate zeolite-based LDO. The adsorption-calcination cycle was repeated 5 times, and the saturated adsorption capacity in each cycle was measured to evaluate the regeneration stability of the material.

Results and Discussion

2.1 Microstructural Evolution of Zeolite-based LDO and Adsorption Mechanism for Fluoride Ions

2.1.1 Microstructure Evolution During Calcination and Adsorption

Precursor of zeolite@MgAl-NO₃-LDH: It possesses the typical ordered layered crystal structure of LDHs. Nano-sized LDH sheets are uniformly coated on the surface of zeolite particles, with exchangeable nitrate anions intercalated in interlayers, and the interlayer spacing is 0.89 nm.

Calcined product (zeolite@LDO): The ordered layers of LDHs completely collapse and transform into amorphous magnesium-aluminum mixed oxides with loose porous structure and high specific surface area. The rigid pore framework of the zeolite core remains intact, which physically isolates and disperses LDO nanoparticles and effectively inhibits agglomeration and stacking of the shell layer. BET results verify that the specific surface area increases from 82.69 m²/g of the precursor to 136.42 m²/g.

Reconstructed product after fluoride adsorption: Characteristic layered diffraction peaks of LDHs reappear in XRD patterns, demonstrating that LDO triggers the structural memory effect and

spontaneously reconstructs positively charged MgAl hydroxide layers. Fluoride ions from wastewater intercalate into interlayers to balance the positive charges of the layers. Characteristic absorption peaks of M–F bonds emerge in FTIR spectra, further verifying that fluoride ions are stably immobilized between interlayers and on the material surface.

2.1.2 Mechanism of Multiple Synergistic Adsorption of Fluoride Ions in High-Salinity Water

The fluoride removal by zeolite-based LDO is dominated by interlayer anion intercalation derived from structural memory effect, combined with multiple adsorption mechanisms, enabling outstanding defluoridation performance in complex high-salinity water environment. The adsorption results are presented as follows:

Anion intercalation via memory effect (dominant mechanism): LDO reconstructs layered LDHs structure upon contact with water, and equivalent-charge ion exchange occurs between interlayer NO_3^- and aqueous F^- . In high-salinity system, the saturated adsorption capacity of acidified zeolite alone is merely 31.06 mg/g, while that of pure Al_2O_3 without memory effect reaches 42.57 mg/g. Benefiting from memory-induced intercalation, zeolite@LDO achieves an adsorption capacity of 127.23 mg/g, showing a prominent improvement.

Surface hydroxyl complexation: Silicon and aluminum hydroxyl groups on zeolite surface together with metallic hydroxyl groups on LDO surface can form stable metal-fluoride complexes with fluoride ions. After blocking hydroxyl groups of the material via modification, the adsorption capacity of zeolite@LDO decreases by 32.4%, which confirms that hydroxyl complexation acts as a crucial auxiliary adsorption pathway.

Electrostatic adsorption: The reconstructed LDH laminates are positively charged overall. The zeta potential ranges from + 18.6 mV to + 32.4 mV at pH 3–7, which enriches anionic fluoride in water via electrostatic attraction. When pH>9, deprotonation occurs on laminates and the zeta potential turns negative, resulting in a 61.7% reduction in adsorption capacity.

Auxiliary effect of zeolite framework: Porous zeolite can trap macromolecular organic impurities in water. After introducing 100 mg/L humic acid, the adsorption capacity of pure LDO declines by 40.3%, whereas zeolite-based LDO only suffers a 12.1% capacity loss. Meanwhile, zeolite increases the bulk density of composite powder. Sedimentation test shows that pure LDO suspension remains turbid after standing for 24 h, yet complete solid-liquid separation is achieved for zeolite-based LDO within 15 min of static settling.

2.2 Experimental Results on Comprehensive Performance of Zeolite-Based LDO for Treating High-Salinity Fluoride-Containing Wastewater

2.2.1 Comparison of Adsorption Capacities (Initial fluoride concentration: 50 mg/L, pH = 6, high-salinity coexisting system)

Adsorbent	Saturated adsorption capacity $q_e/(mg\cdot g^{-1})$
Acidified clinoptilolite	31.06
Pure MgAl-LDO	68.75
Zeolite-based LDO calcined at 450°C	127.23
Sintered zeolite-based LDO (500°C)	26.81
Calcined product of carbonate-intercalated LDH	40.15

2.2.2 Effect of pH

Within the pH range of 3–7, the adsorption capacity of zeolite-based LDO remains stable at 118.5–127.23 mg/g. At pH>9, deprotonation occurs for hydroxyl groups on the laminate, electrostatic attraction disappears, and the adsorption capacity declines to 49.02 mg/g. Under extremely acidic conditions (pH<2), the reconstructed layered structure dissolves, leading to a slight drop in adsorption capacity to 95.47 mg/g.

2.2.3 Interference Control of Salt Ions

The saturated adsorption capacity of zeolite-based LDO reaches 134.56 mg/g in salt-free solution. After adding 0.1 mol/L NaCl and 0.05 mol/L Na₂SO₄ to construct a high-salinity coexisting system, its capacity only decreases by 5.4% (127.23 mg/g). Under identical salinity conditions, the adsorption capacity of pure LDO decreases by 23.6%, while acidified zeolite suffers a capacity loss of 38.1%. These results verify that the core-shell structure together with the memory intercalation mechanism endows the composite material with outstanding anti-salt-interference performance.

2.2.4 Regeneration Cycle Stability

The saturated adsorption capacities after five cycles of calcination regeneration are listed sequentially: 127.23, 118.97, 112.41, 106.85 and 105.10 mg/g. The capacity retention rate maintains 82.6% after the fifth cycle, and no large-area peeling of the shell layer is observed, revealing the favorable recyclability of the as-prepared material.

2.2 Comprehensive Application Advantages of Zeolite-Based LDO for High-Salinity Fluoride-Containing Wastewater Treatment

Agglomeration inhibition and convenient solid-liquid separation: The rigid zeolite core physically supports and disperses LDO nanosheets, overcoming the drawbacks of pure LDO powder such as persistent suspension and slow sedimentation. Rapid precipitation and separation can be realized within 15 min without additional magnetic modification.

Excellent fluoride selectivity in high-salinity environments: Fluoride ions are immobilized via interlayer intercalation originating from structural memory effect, resulting in merely 5.4% capacity attenuation in high-salinity systems. Therefore, this material is suitable for treating high-salinity fluoride-bearing wastewater from electroplating, coal chemical industry and other industries.

Potential for simultaneous removal of anionic and cationic pollutants: The pore channels of zeolite possess cation exchange capacity to synchronously adsorb heavy metal ions including Cu^{2+} and Pb^{2+} . In the coexistence system containing 50 mg/L Pb^{2+} and 50 mg/L F^- , the adsorption capacity for Pb^{2+} is 89.64 mg/g and the fluoride adsorption capacity is 119.32 mg/g, achieving integrated purification of both anionic and cationic contaminants.

Recyclable regeneration and low operating cost: The adsorption capacity retains 82.6% after five regeneration cycles, which greatly reduces the cost of adsorbent procurement and solid waste disposal.

Low-cost raw materials and easy large-scale fabrication: Natural clinoptilolite is abundant in mineral reserves with low purchasing cost. The whole synthesis process requires no precious metal reagents and features mild and controllable reaction conditions, satisfying the requirements for industrial mass production.

Conclusions

Using acidified clinoptilolite as a rigid supporting carrier, nitrate-intercalated MgAl-LDH precursor is fabricated via in-situ co-precipitation under nitrogen atmosphere to exclude carbonate ions. After programmed heating calcination at 400–450°C at a low heating rate and sealed cooling isolated from air, zeolite-based LDO composite adsorbent with intact core-shell structure can be obtained. Physical-chemical characterizations demonstrate that the zeolite framework effectively disperses the LDO nanoshell layer, and the BET specific surface area of the composite reaches 136.42 m^2/g , significantly higher than that of pure LDO (68.75 m^2/g). Static adsorption experiments confirm that the saturated fluoride adsorption capacity of zeolite-based LDO reaches 127.23 mg/g in simulated high-salinity fluoride-containing wastewater, which is 4.1 times that of single acidified zeolite. Moreover, it presents strong anti-salt interference ability and retains 82.6% adsorption capacity after five regeneration cycles.

Benefiting from the unique memory reconstruction effect of the outer LDO layer, positively charged layered hydroxide structure spontaneously reconstructs once LDO is immersed in aqueous solution. Fluoride ions in water are efficiently captured through multiple synergistic mechanisms including interlayer anion exchange, surface hydroxyl complexation and electrostatic adsorption. Consequently, zeolite-based LDO exhibits prominent application prospects in advanced treatment of high-salinity

fluoride-laden industrial wastewater. During material preparation, calcination temperature, heating rate and cooling atmosphere should be strictly controlled, and nitrate-intercalated LDH precursors are preferred to avoid dramatic deterioration of defluoridation performance caused by spinel formation of magnesium aluminate and competitive adsorption of carbonate ions

Declarations

Author Contribution

Author 1: Designed the research scheme, completed experiments and drafted the manuscript; Author1 : Performed data analysis and chart drawing; Author1 : Revised the paper and provided funding support; Corresponding Author: Guided the whole research work.

Data Availability

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

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Figures

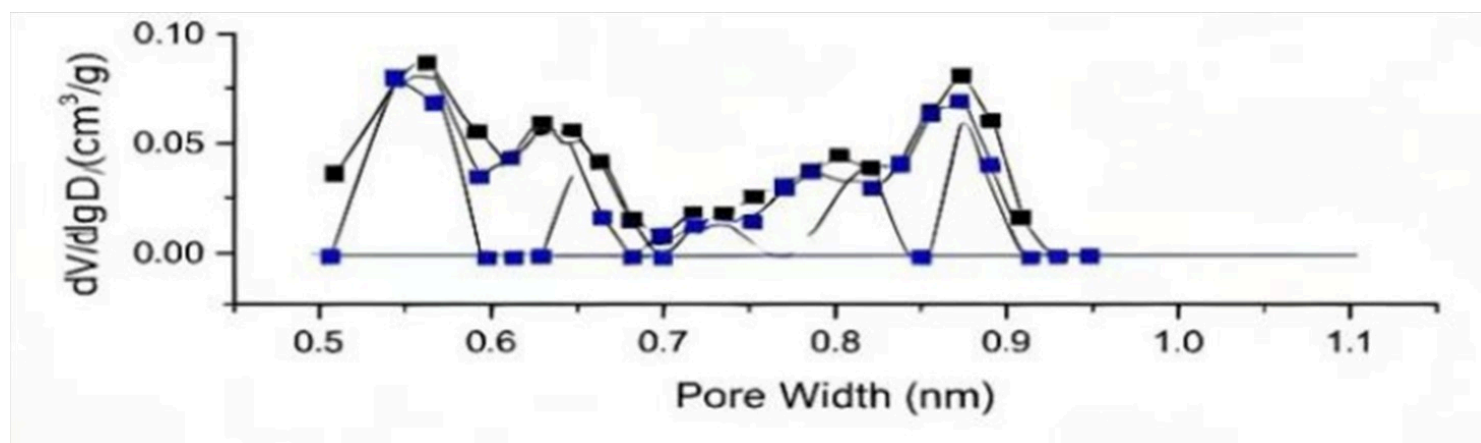


Figure 1

Comparison of pore structures of clinoptilolite treated with 2 mol/L HNO_3 at different acidification durations (2 h, 4 h, 6 h)