

Synthesis of ZSM-5 molecular sieve from coal gasification fine slag and its adsorption mechanisms for Pb^{2+} in aqueous solution

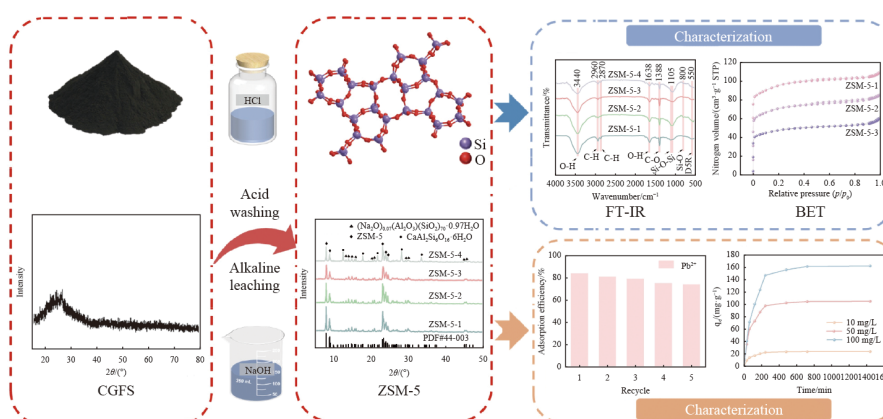
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Abstract: Coal gasification fine slag (CGFS) is a solid waste generated from entrained-flow coal gasification, characterized by fine particles and rich in silicon and aluminum elements. Using CGFS as a raw material for the preparation of ZSM-5 molecular sieves enables the high-value utilization of coal-based solid waste,

offering significant economic and environmental benefits. This study proposes a simple and economical hydrothermal synthesis process for ZSM-5 using CGFS as the raw material. In this process, impurities in CGFS were first removed by acid washing, which facilitated the subsequent alkaline extraction of silicon and aluminum species. The extracted Si-Al precursors were then converted into ZSM-5 *via* hydrothermal crystallization at 170 °C for 48 h, yielding a product with a high specific surface area of 358 m²/g. Adsorption experiments demonstrated that the synthesized ZSM-5 exhibited excellent adsorption performance for Pb^{2+} in aqueous solutions. At 25 °C, the removal efficiency for a 50 mg/L Pb^{2+} solution reached 83.7%, with an adsorption capacity of 104.625 mg/g under optimized conditions. The adsorption process is mainly governed by chemisorption mechanisms, including surface complexation, precipitation, and ion exchange. The synthesized ZSM-5 shows promising potential for application in the treatment of lead-containing wastewater.

Key words: coal gasification fine slag; ZSM-5; synthesis and performance; adsorption mechanisms; Pb^{2+}



In the coal chemical industry, coal gasification serves as a crucial technique for converting coal into clean gaseous fuels^[1–2]. Compared with traditional fuels, this process demonstrates superior advantages in efficiency, economic viability, and environmental protection^[3–4]. China's coal gasification industry, which consumes substantial coal resource annually, has become a primary driver for the development of the coal chemical sector^[5]. However, the process also generates large amounts of by-products, among

which coal gasification slag is the most significant. This slag is generally categorized into coarse and fine slag according to particle size and discharge mode^[6–7]. Coal gasification fine slag (CGFS), in particular, contains fine particles and elevated concentrations of heavy metals, presenting significant environmental challenges. Its large-scale accumulation in treatment facilities and landfills not only reduces land use efficiency but also creates serious environmental and safety risks^[8–11]. Hence, developing

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efficient and sustainable utilization methods for CGFS is essential for promoting clean coal gasification^[12–13].

The high content of silicates and aluminosilicates in CGFS enables its utilization as raw material for synthetic molecular sieves^[14]. At present, the conventional synthesis method generally involves high-temperature alkali fusion followed by hydrothermal treatment. For instance, Xu et al.^[15] prepared zeolite catalysts from gasified fine powder *via* high-temperature alkali activation with hydrothermal methods. Similarly, Shu et al.^[16] synthesized Na-P1 zeolite from CGFS through high-temperature activation with NaOH and hydrothermal treatment, demonstrating excellent adsorption performance for methylene blue. Chai et al.^[17] employed silicon-aluminum inorganic minerals derived from coal gasification fines to synthesize Y-type zeolite through hydrothermal synthesis with high-temperature alkali activation and the addition of sodium aluminate as an external aluminum source. While significant progress has been made in this field, research on ZSM-5 synthesis using CGFS remains limited. Furthermore, these conventional methods are energy-intensive and unsuitable for large-scale applications.

Molecular sieves are classified into various types according to pore size and framework topology^[18]. Among them, ZSM-5 has been widely applied in industrial processes owing to its well-defined microporous structure, high thermal stability, and shape-selective properties^[19]. In petroleum refining, ZSM-5 is commonly employed as a catalyst or catalyst support to improve reaction efficiency and product selectivity^[20–21]. In addition, it has also been used in water purification for environmental protection due to its excellent adsorption properties^[22–23]. Given these advantages, the development of ZSM-5 using low-cost solid wastes such as CGFS is highly attractive. However, the preparation of ZSM-5 using CGFS remains challenging. First, high aluminum content increases the difficulty of preparation^[24], ZSM-5 is a type of high-silica molecular sieve. During the synthesis process, an excessively high aluminum content often leads to the formation of amorphous aluminosilicates or other impurity crystalline phases in the hydrothermal products. Second, the purity of raw materials largely influences the performance of synthetic molecular sieves. As components such as Fe₂O₃, MgO, CaO and K₂O can adversely affect the structure and properties of molecular sieves^[25]. To overcome these

limitations, a commonly adopted approach is to adjust Si/Al molar ratio by adding external silicon source. For instance, Yu et al.^[26] utilized fly ash as raw material with fumed silica supplementation to synthesize ZSM-5, while Ramteke et al.^[27] employed fly ash combined with solid silicon dioxide as an additional silicon source for ZSM-5 synthesis. However, the reliance on additional silicon sources inevitably increases the overall synthesis cost, thereby limiting the economic feasibility of large-scale applications^[28].

In this study, we developed an acid-base synergistic leaching method using CGFS as the sole source of silicon and aluminum. This approach demonstrates simplicity, energy efficiency, and cost-effectiveness. Initial acid washing effectively removed impurities including Al₂O₃, MgO, CaO and K₂O from CGFS. Subsequent NaOH extraction of silicon and aluminum enabled the effective dissolution of silicon and aluminum species, which were then converted into ZSM-5 with high crystallinity and substantial specific surface area *via* hydrothermal treatment. Given that lead is a highly toxic and non-biodegradable heavy metal with long-term environmental persistence, its accumulation in aqueous systems poses a serious global concern. Accordingly, the as-synthesized ZSM-5 was investigated as an adsorbent for Pb²⁺ removal from simulated wastewater, with particular focus on elucidating its adsorption performance and underlying removal mechanisms.

1 Materials and methods

1.1 Materials

The coal gasification fine slag (CGFS) used in this study was collected at a coal-based gasifier in Anhui Province. Table 1 presents the results of proximate and ultimate analyses of CGFS, with ash yield and volatile yield of 89.26% and 2.25%, respectively. Table 2 shows the chemical composition analysis, revealing SiO₂ and Al₂O₃ contents of 60.91% and 24.74%, respectively. The chemicals used in this study included NaOH (Sinopharm Chemical Reagents Co., Ltd), HCl (Sinopharm Chemical Reagents Co., Ltd), tetrapentylammonium hydroxide (TPAOH) (McLean Biochemical Technology Co., Ltd.), and Pb(NO₃)₂ (Xilong Science Co., Ltd.).

Table 1 Proximate and ultimate analyses of CGFS

Sample	Proximate analysis $w_{ad}/\%$				Ultimate analysis $w_{ad}/\%$				
	<i>M</i>	<i>A</i>	<i>V</i>	FC	<i>C</i>	<i>H</i>	<i>N</i>	<i>S</i>	<i>O</i>
CGFS	1.59	89.26	2.25	6.90	7.47	0.07	0.00	0.16	1.45

Table 2 Chemical composition analysis of CGFS

Constituent	Content $w/\%$							
	SiO ₂	Al ₂ O ₃	Na ₂ O	MgO	Fe ₂ O ₃	CaO	K ₂ O	others
Percentage	60.91	24.74	0.72	0.95	2.57	6.16	2.03	1.92

1.2 Synthesis of ZSM-5

ZSM-5 was synthesized using CGFS as the Si-Al source through sequential acid washing, alkali extraction, and hydrothermal synthesis. The synthesis process is illustrated in Figure 1. Initially, 5 g of CGFS was mixed with 50 mL of HCl solutions at varying concentrations, maintaining a solid-to-liquid ratio of 1:10 (g/mL). The mixture was then magnetically stirred in a water bath at 80 °C for 3 h. Upon reaction completion, the slurry was subjected to vacuum-filtered, and the filter cake was rinsed with deionized water until neutrality before drying at 60 °C for 24 h. The obtained sample was designated CGFS-acid. Subsequently, 5 g of CGFS-acid was added to 50 mL of NaOH solutions with concentrations of 1, 2, 3 and 4 mol/L. The mixture was stirred at 80 °C for 2 h. After cooling and filtration, the silicon-enriched filtrate was collected. It is worth noting that the residual carbon in CGFS mainly exists as insoluble unburned char. During acid leaching and alkaline extraction, these carbonaceous particles remain in the solid phase and are removed together with the filter residue, thus preventing them from entering the subsequent crystallization system. A 15 mL of the filtered solution was combined with TPAOH solution at a molar Si/TPAOH ratio of 1:0.25. The pH was precisely adjusted to 10 by dropwise addition of concentrated H₂SO₄. The precursor was transferred to a reactor vessel and subjected to hydrothermal reaction at 170 °C for 48 h. The cooled product was filtered, dried at 60 °C for 24 h, and finally, the samples were calcined at 550 °C for 6 h in a muffle furnace to remove the template agent from the ZSM-5.

1.3 Characterizations

The crystal phases of ZSM-5 was analyzed using X-ray diffraction (XRD, Rigaku SmartLab SE, Japan). The chemical composition in CGFS were determined using an X-ray fluorescence spectrometer (XRF, WEPER XRF2510, Changsha, China). The microstructure of the ZSM-5 was observed with a scanning electron microscopy (SEM,

ZEISS Sigma 300, Germany) operated at an accelerating voltage of 3 kV, coupled with an energy-dispersive X-ray spectrometer (EDX, Oxford Xplore 50). The surface chemical state and composition of the ZSM-5 was analyzed by X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi, USA). Silicon and aluminum contents in the solution after NaOH extraction of fine residue, along with Pb²⁺ concentration, were quantified using inductively coupled plasma optical emission spectroscopy (ICP-OES, PE Avio550, Shanghai, China). The surface functional groups of ZSM-5 was analyzed by Fourier transform infrared spectroscopy (FT-IR, Nicolet iS50, USA). The spectra were recorded in the wavenumber range of 500–4000 cm⁻¹ at a resolution of 4 cm⁻¹. The specific surface area and pore structure of the ZSM-5 was evaluated using a Brunauer-Emmett-Teller surface area analyzer (BET, ASAP2460, USA) after degassing at 120 °C for 8 h.

1.4 Adsorption experiment

The adsorption performance of the synthesized ZSM-5 for Pb²⁺ in simulated wastewater was evaluated through batch adsorption experiments. ZSM-5 was added to Pb²⁺ aqueous solutions with different concentrations. The samples were agitated at 200 r/min using a water bath shaker. After filtration, a clear solution was obtained, and the residual Pb²⁺ concentration was determined by ICP-OES. The adsorption capacity (q_e), removal rate and related formulas for Pb²⁺ in the simulated wastewater by ZSM-5 was calculated as follows:

$$q_e = \frac{(C_0 - C_e)}{m} \times V \quad (1)$$

$$\text{Removal rate} = \frac{C_0 - C_e}{C_0} \times 100\% \quad (2)$$

where q_e represents the adsorption capacity of Pb²⁺ by ZSM-5 at adsorption equilibrium (mg/g), C_0 and C_e denote the initial and equilibrium concentrations of Pb²⁺, respectively (mg/L), V denotes the volume of Pb²⁺ solution (L), and m is the mass of ZSM-5 (g).

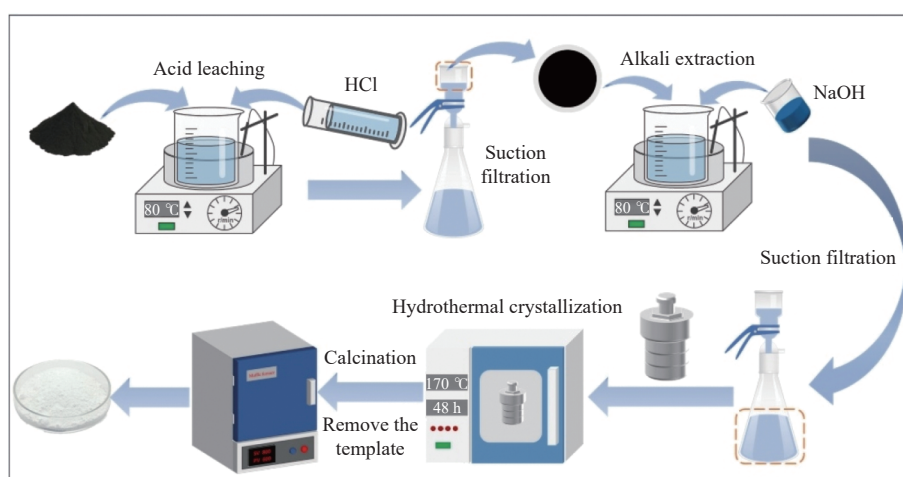


Fig. 1 Flow chart of ZSM-5 preparation process

To obtain adsorption kinetic and mechanism, the quasi-first-order kinetic model (Eq. (3)), the quasi-second-order kinetic model (Eq. (4)) and the Weber-Morris intra-particle diffusion model (Eq. (5)) were adopted.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (4)$$

$$q_t = k_{int} t^{1/2} + C \quad (5)$$

where q_e (mg/g) and q_t (mg/g) denote the equilibrium adsorption capacity and the adsorption capacity at time t , respectively. The rate constants k_1 (g/(mg·min)), k_2 (mg/(g·min)), and k_{int} (mg/(g·min^{0.5})) correspond to the pseudo-first-order, pseudo-second-order, and intra-particle diffusion models, respectively, while C represents a boundary layer effect.

Adsorption isotherms reflect the relationship between the amount of adsorption on the adsorbent surface and the solution concentration at constant temperature. This study employed three adsorption isotherm models: Langmuir (Eq. (6), (7)), Freundlich (Eq. (8)) and Tempkin (Eq. (9)).

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad (6)$$

$$R_L = \frac{1}{1 + K_L C_0} \quad (7)$$

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (8)$$

$$q_e = \frac{RT}{b_T} \ln \alpha + \left(\frac{RT}{b_T} \right) \ln C_e \quad (9)$$

where q_{max} (mg/g) denotes the sorbent's maximum adsorptive capacity, and K_L (L/mg) corresponds to the Langmuir coefficient. R_L is the separation constant and is often used to describe its adsorption capacity, K_F ((mg/g) (L/mg)^{1/n}) is the Freundlich constant and is related to the adsorption capacity, and the value of n determines whether the adsorption process is favorable. α (mg/L) is the equilibrium binding constant. b_T is the Tempkin constant.

To investigate the internal energy changes related to adsorption processes, thermodynamic parameters including ΔH° (kJ/mol), ΔS° (J/(mol·K)) and ΔG° (kJ/mol) were determined through temperature-dependent adsorption isotherm analysis using the following equations (10)–(12).

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (10)$$

$$\Delta G^\circ = -RT \ln K_c \quad (11)$$

$$\ln K_c = -\frac{\Delta G^\circ}{RT} = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (12)$$

where T (K) is the thermodynamic temperature. R (8.314 J/(mol·K)) is the ideal gas constant. K_c is the thermodynamic equilibrium constant.

2 Results and discussion

2.1 Effect of acid washing and alkali extraction on Si and Al extraction

2.1.1 Influence of acid concentrations on leaching efficiency

Coal gasification fine slag (CGFS) contains silicon and aluminum elements that are essential for molecular sieves formation, along with multiple metal oxide impurities that may compromise the quality of synthesized molecular sieves in subsequent processes^[14]. Acid washing serves as a critical pretreatment to enhance CGFS utilization by removing metal oxides impurities and increasing the Si/Al molar ratio. Under the action of HCl solution, surface impurities dissolve to form initial wedge-shaped channels, which progressively expand into particle interior^[29]. The random distribution of metal oxides in CGFS leads to irregular channel connections after acid leaching, generating dendritic microporous/mesoporous structures. This structure is conducive to subsequent alkali extraction^[30].

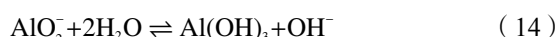
The chemical composition of CGFS after acid washing were determined by XRF analysis. As shown in Table 3, a gradual increase in SiO₂ content from 60.91% to 75.22% with rising HCl concentration, accompanied by a significant decrease in the Al₂O₃ content. This behavior can be attributed to the reaction with HCl to form soluble chlorides, which effectively remove interfering impurities such as Fe₂O₃, CaO and MgO. Specially, the Fe₂O₃ content decreases from 2.57% to 0.43% and CaO decreases from 6.16% to 3.96% after leaching, indicating that HCl leaching significantly reduces the metal impurity and provides a cleaner Si-Al source for ZSM-5 crystallization. Notably, when the HCl concentration was increased to 4 mol/L, the SiO₂ content shows minimal increase while Al₂O₃ and Fe₂O₃ exhibit slight elevation. This apparent increase stems from the preferential dissolution of other components at higher acid concentrations, resulting in relative enrichment of residual Al₂O₃ and Fe₂O₃. Considering both leaching efficiency and cost-effectiveness, an HCl concentration of 3 mol/L was selected as the optimal concentration.

Table 3 Chemical composition of CGFS after acid washing

$C_{HCl}/(\text{mol} \cdot \text{L}^{-1})$	Content w/%							
	SiO ₂	Al ₂ O ₃	Na ₂ O	MgO	Fe ₂ O ₃	CaO	K ₂ O	others
0	60.91	24.74	0.72	0.95	2.57	6.16	2.03	1.92
1	69.56	20.52	0.62	0.77	0.7	4.26	1.77	1.8
2	70.77	20.04	0.58	0.75	0.43	3.97	1.64	1.82
3	74.99	15.78	0.58	0.76	0.43	3.96	1.66	1.84
4	75.22	16.56	0.57	0.71	0.69	4.03	1.69	0.53

2.1.2 Influence of different concentrations of alkali on Si and Al extraction

The concentration of NaOH significantly influences the extraction of Si and Al from CGFS. Therefore, a systematic analysis of the dissolved Si and Al contents under different alkaline conditions is essential for optimizing subsequent synthesis experiments. In this study, CGFS-acid was extracted using NaOH solutions with concentrations ranging from 1 to 4 mol/L. As shown in Table 4, increasing NaOH concentrations enhance the solubility of Si and Al in the extraction solution, while the Si/Al molar ratio of the extracted components gradually decreases with increasing alkalinity. This phenomenon can be explained through reactions (13), (14). In addition, the residue after alkaline extraction may serve as precursor material for synthesizing C-Si composite materials^[30–34].



When the concentration of NaOH increases, reaction (13) shifts strongly to the right, converting more Al_2O_3 into NaAlO_2 and significantly increasing the amount of dissolved Al. Simultaneously, the dissolution equilibrium of reaction (14) shifts to the left, which inhibits $\text{Al}(\text{OH})_3$ precipitation and further enhances Al extraction efficiency.

2.2 Characterization of ZSM-5

2.2.1 Phase and structure analysis

The ZSM-5 synthesized from Si-Al solutions extracted with different NaOH concentrations are denoted as ZSM-5-*x* (*x* = 1, 2, 3 and 4), where *x* represents the NaOH molarity.

As illustrated in Figure 2(a), all samples except ZSM-5-4 exhibit distinct characteristic peaks without detectable impurities, indicating high material purity. For the sample ZSM-5-4, additional diffraction peaks appear at $2\theta = 15.7^\circ$, 23.7° , 26.9° and 31.1° alongside ZSM-5 characteristic peak. These extra peaks suggest the formation of impurities such as clinoptilolite (FAU-type, CaX). This phenomenon is attributed to the excessive NaOH concentration, which promotes extensive dissolution Ca elements from CGFS that were not completely removed during acid washing. Under conditions of relatively high Ca^{2+} concentration and low Si/Al molar ratio, thermodynamically stable FAU-type structures preferentially crystallize. Such competitive crystallization suppresses ZSM-5 crystal growth, ultimately reducing product purity.

The FT-IR spectrum (Figure 2(b)) further confirms the structural features of the ZSM-5, the absorption bands at approximately 3440 and 1638 cm^{-1} are attributed to O–H stretching and bending vibrations of adsorbed water, respectively. The framework vibration of ZSM-5 appears as a Si–O–Si peak at 1105 cm^{-1} ^[35], while the characteristic double five-membered ring vibration of the MFI topology is observed at 550 cm^{-1} .

2.2.2 Morphological characterization

For morphological analysis, ZSM-5-1 was selected. As illustrated in Figure 3(a)–(h), low-magnification images reveal that the ZSM-5 predominantly form spherical particles with minimal other morphological impurities, suggesting high product purity and crystallinity. At higher magnifications, the particles exhibit well-defined hexagonal prism morphology with sharp edges. Elemental mapping further demonstrates all components are uniformly distributed across the particle surfaces, confirming the homogeneity of the sample.

Table 4 The concentration of Si and Al under different NaOH concentration extraction

$C_{\text{NaOH}}/(\text{mol}\cdot\text{L}^{-1})$	Si/($\text{mg}\cdot\text{L}^{-1}$)	Al/($\text{mg}\cdot\text{L}^{-1}$)	Si/Al (mol ratio)
1	10646.0	294.61	34.70
2	17923.8	632.53	27.22
3	21139.5	844.54	24.06
4	28553.1	1293.5	21.20

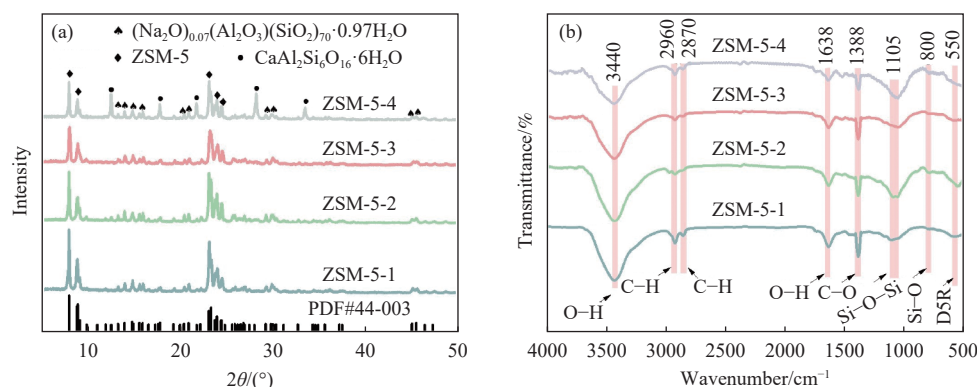
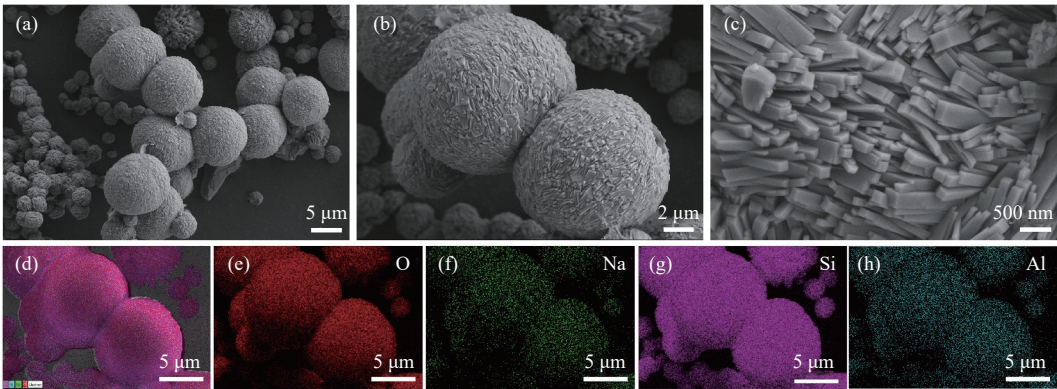


Fig. 2 (a) XRD patterns, (b) FT-IR spectra of ZSM-5



(a)–(c): SEM images; (d)–(h): EDS elemental mapping.
Fig. 3 Morphology and elemental distribution of ZSM-5-1 sample

2.2.3 Effect of NaOH concentration on specific surface area and pore structure of ZSM-5

As shown in Table 5, ZSM-5-1 exhibits the largest specific surface area (358 m²/g) among the three synthesized samples. Comparative analysis in Table 6 indicates that the ZSM-5 derived from CGFS demonstrates superior specific surface area relative to other waste-derived zeolites. In Figure 4(a), all three synthesized ZSM-5 display typical type IV isotherms with H4-type hysteresis loops,

indicating the coexistence of mesopores and micropores within the material^[36]. Figure 4(b) shows the pore size distribution of the synthesized ZSM-5, where the primary pore sizes of all samples range from 3 to 5 nm, consistent with typical mesoporous structures. These mesopores originate from alkaline etching of the framework during synthesis using concentrated NaOH solutions^[37]. With increasing NaOH concentration, the specific surface area and pore volume of the product decrease, while the average pore diameter expands.

Table 5 Pore characteristics of ZSM-5-x

Sample	$S_{BET}/(m^2 \cdot g^{-1})$	$v_{total}/(cm^3 \cdot g^{-1})$	$v_{micro}/(cm^3 \cdot g^{-1})$	$v_{meso}/(cm^3 \cdot g^{-1})$	Pore diameter/nm
ZSM-5-1	358	0.17	0.09	0.07	3.25
ZSM-5-2	325	0.15	0.09	0.07	3.59
ZSM-5-3	223	0.11	0.06	0.06	4.20

Table 6 Summary of specific surface areas of adsorbents synthesized from solid waste

Raw material	Sample	Specific surface area/(m ² ·g ⁻¹)	Reference
Coal gangue	ZSM-5	253	[38]
Biomass power plant ash	ZSM-5	250–320	[39]
Coal gasification coarse slag	4A-type zeolite	48	[40]
Rice husk ash	Zeolite Y	137	[41]
Coal gasification fine slag	Na-P1 zeolite	154	[14]
Coal fly ash	P1 zeolite	18	[42]

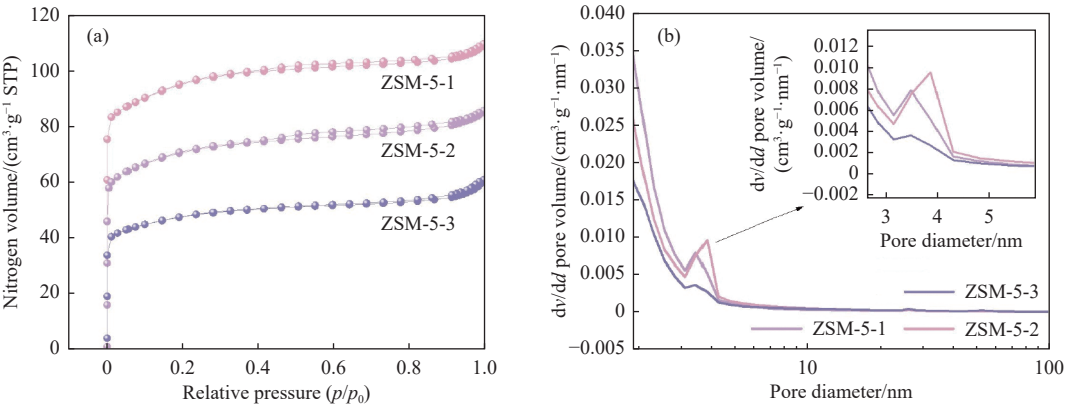


Fig. 4 (a) Nitrogen physical adsorption isotherm and (b) BJH curve of ZSM-5-x

This phenomenon can be attributed to high NaOH concentrations promoting the rapid dissolution and polymerization of Si and Al-bearing species, forming larger silica-alumina clusters. This leads to the formation of larger pore structures (mesopores or macropores) during crystallization, simultaneously reducing micropore quantity. Consequently, this structural evolution results in diminished specific surface area and increased average pore size.

2.3 Adsorption performance of ZSM-5 for Pb^{2+}

2.3.1 Adsorptive properties of the ZSM-5

ZSM-5 possesses large adsorption capacity, selective adsorption, and large pore size, making it promising for heavy metal ions removal from wastewater. In this experiment, under identical conditions (25 °C and 24 h adsorption time), 20 mg of ZSM-5 was introduced into 50 mL of a 50 mg/L Pb^{2+} solution. Adsorption experiments were conducted on three synthesized samples and untreated CGFS. As shown in Figure 5(a), untreated CGFS exhibits a low adsorption rate of only 21.8%. Among the synthesized samples, ZSM-5-1 shows the highest adsorption rate at 83.7%, indicating that a large specific surface area facilitates the adsorption process^[43]. Consequently, ZSM-5-1 was selected for subsequent adsorption experiments.

Solution pH strongly influences both the adsorption rate and extent of metal ion onto the adsorbent^[44], and affects the dissociation of functional groups^[45]. Under constant adsorption time and temperature conditions, 20 mg of ZSM-5-1 was added to a 50 mg/L Pb^{2+} solution. The relationship between removal efficiency at adsorption equilibrium and pH is shown in Figure 5(b). Within the pH value range of 2–7, lead mainly exists as Pb^{2+} and $\text{Pb}(\text{OH})^+$ species, whereas precipitation dominates at pH value > 7. As pH value increased from 2–7, the adsorption performance of ZSM-5 for Pb^{2+} progressively improved. At pH value =2.0, H^+ ions occupied most active sites on the surface of ZSM-5, limiting Pb^{2+} adsorption capacity^[46]. The adsorption

capacity and removal efficiency are 5.73 mg/g and 4.58%, respectively. With increasing pH, the concentration of H^+ decreased, weakening competitive effects and enhancing Pb^{2+} adsorption. At pH value = 7, maximum adsorption capacity 104.625 mg/g and removal efficiency 83.7% were achieved.

At 25 °C, 20 mg of ZSM-5-1 was added into 50 mL of Pb^{2+} solutions at different concentrations. The adsorption capacity and removal efficiency were measured at various adsorption time. As shown in Figure 6, ZSM-5-1 exhibits a remarkable increase in Pb^{2+} adsorption capacity for all three solution concentrations during the initial 240 min, after which the adsorption rate gradually decreases and reaches equilibrium at 720 min. The equilibrium adsorption capacity of ZSM-5-1 shows a positive correlation with the initial Pb^{2+} concentration, with higher concentrations leading to enhanced adsorption. This behavior can be attributed to the excess of available active sites on ZSM-5-1 at low metal ion concentrations, which limits effective contact between Pb^{2+} ions and adsorption sites^[47]. Figure 6(c) shows the Weber-Morris intramolecular diffusion model of ZSM-5-1 during adsorption of a 100 mg/L Pb^{2+} solution. The adsorption process can be divided into two distinct stages. The first stage involves the diffusion of Pb^{2+} from the bulk solution to the external surface of the ZSM-5. The second stage corresponds to Pb^{2+} diffusion into the internal pores of the adsorbent, dominated by intraparticle diffusion^[48]. The non-zero intercept C of the model indicates significant boundary layer diffusion effects, suggesting that both liquid film diffusion and physicochemical interactions between the molecular sieve and Pb^{2+} ions influence the adsorption process^[49]. The model indicates that the adsorption rate constant $k_{\text{int}1}$ in the first stage is higher than $k_{\text{int}2}$ in the second stage, reflecting the faster adsorption kinetics at the initial stage due to abundant available surface sites. In the second stage, as adsorption proceeds, surface adsorption sites become progressively occupied, and diffusion of Pb^{2+} into internal pores becomes rate-limiting, resulting in a slower adsorption rate^[50].

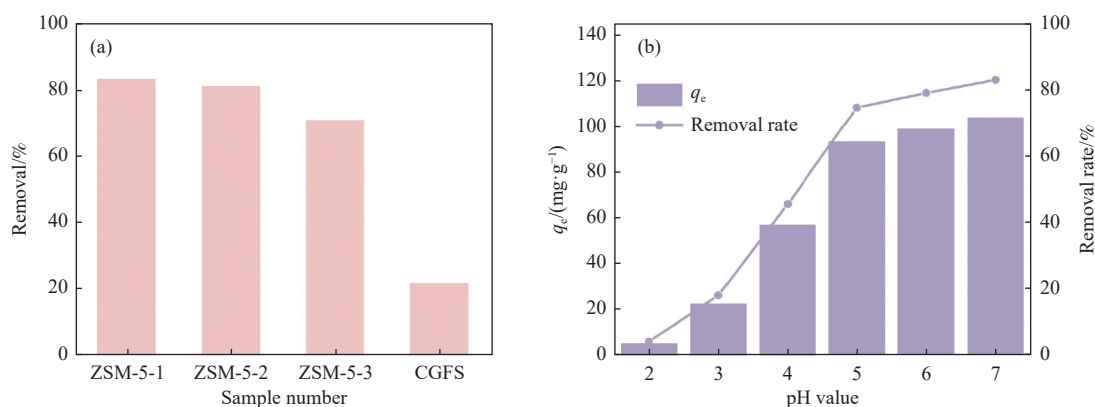


Fig. 5 (a) Adsorption of Pb^{2+} by three ZSM-5-x samples and CGFS; (b) The effect of pH value on Pb^{2+} adsorption by ZSM-5-1

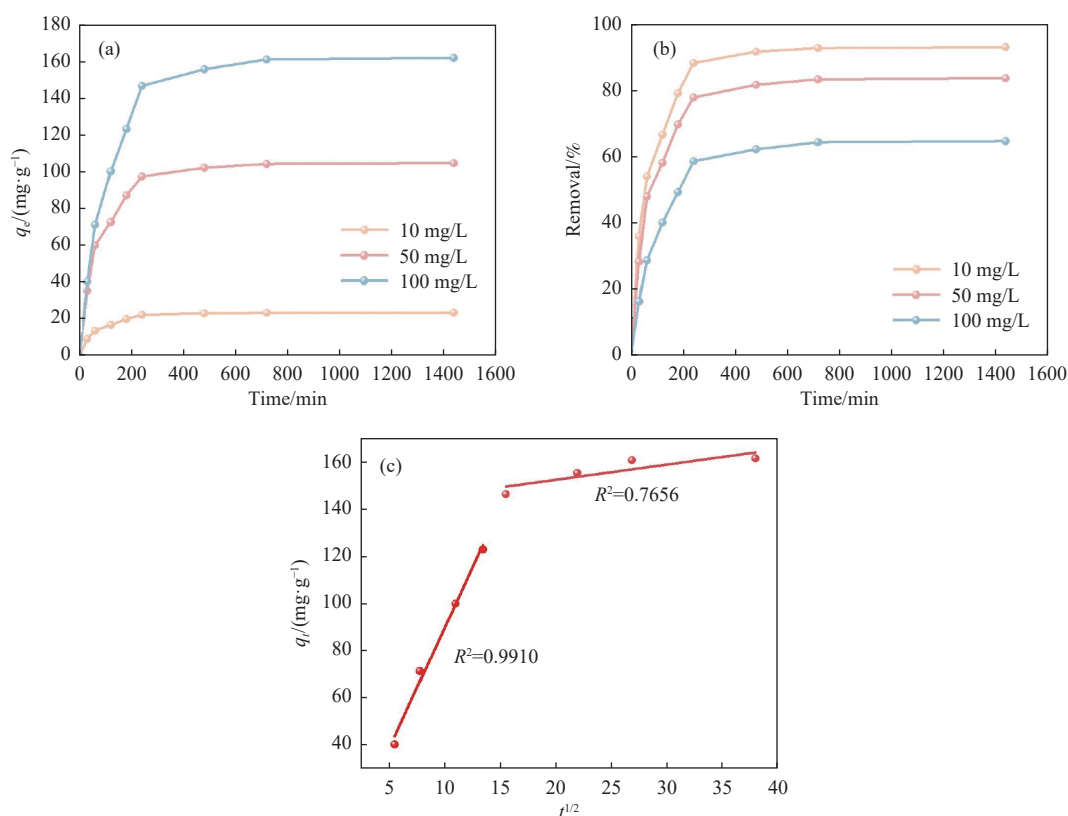


Fig. 6 (a) Effects of the initial concentration of Pb²⁺ on adsorption capacity of ZSM-5-1, (b) removal rate and (c) the Weber-Morris particle diffusion model

2.3.2 Adsorption kinetics

The adsorption behavior of Pb²⁺ onto ZSM-5-1 was evaluated using pseudo-first-order and pseudo-second-order kinetic models (Figure 7). The pseudo-second-order kinetic model exhibited a higher R^2 value, with the calculated q_e value aligning more closely with the experimentally determined equilibrium adsorption capacity (Table 7). These results indicate that the adsorption process of ZSM-5-1 is better described by the pseudo-second-order kinetic model, suggesting a chemisorption-dominated mechanism^[51]. As the Pb²⁺ concentration increases, surface adsorption sites on ZSM-5-1 are rapidly occupied, leading to site saturation. This saturation increases resistance to

further adsorption, resulting in a gradual decline in adsorption rate.

2.3.3 Adsorption Isotherms

In this experiment, 20 mg of ZSM-5-1 was added to 50 mL of Pb²⁺ solutions with different concentrations, and the adsorption experiments were conducted at 25, 30 and 35 °C. Figure 8 shows the adsorption isotherms of ZSM-5-1 for Pb²⁺ and the fitting results of the three adsorption isotherms models. For ZSM-5-1, the adsorption capacity increased with increasing initial Pb²⁺ concentration and temperature. The enhancement of adsorption at higher temperatures indicates that the process is endothermic.

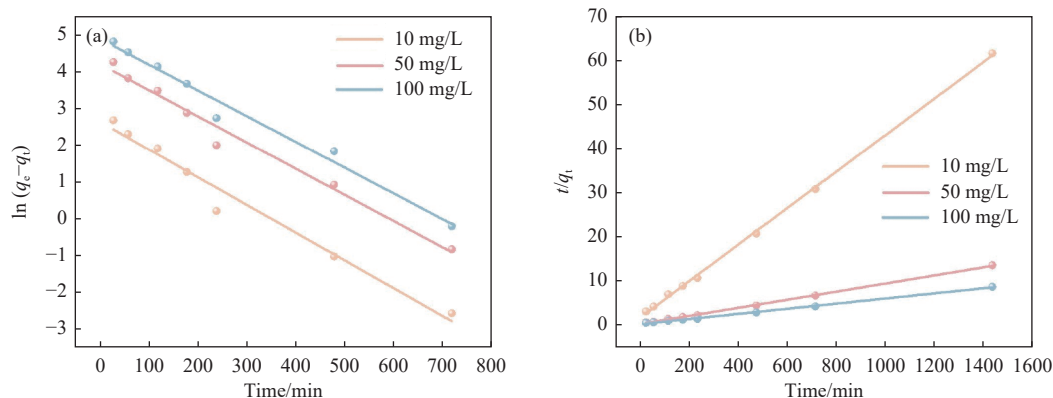


Fig. 7 (a) Pseudo-first-order and (b) pseudo-second-order adsorption kinetic curves of Pb²⁺ on ZSM-5-1

Table 7 Kinetic parameters of ZSM-5-1 adsorption of Pb^{2+}

Pb $C_0/(\text{mg}\cdot\text{L}^{-1})$	Pseudo-first-order			Pseudo-second-order		
	$q_e/(\text{mg}\cdot\text{g}^{-1})$	K_1/min^{-1}	R^2	$q_e/(\text{mg}\cdot\text{g}^{-1})$	$K_2/(\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1})$	R^2
10	13.92	0.00755	0.9699	24.11	0.04148	0.9992
50	67.24	0.00714	0.9779	108.93	0.00918	0.9990
100	132.98	0.00702	0.9780	172.12	0.00581	0.9976

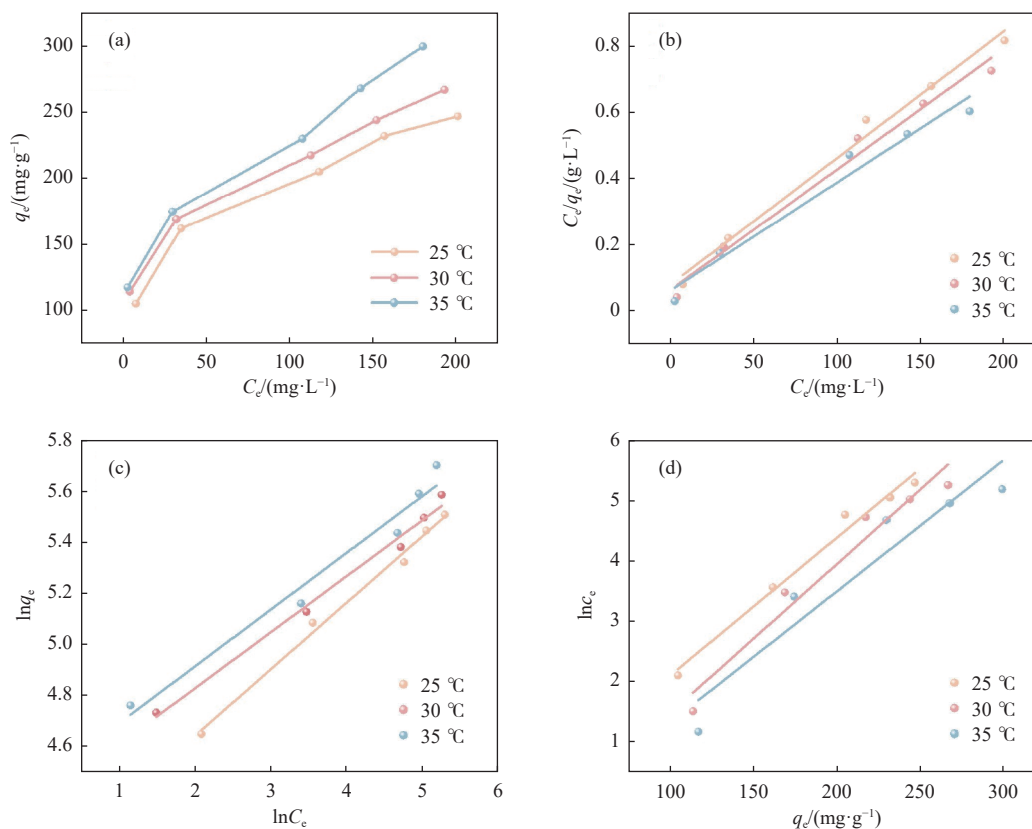


Fig. 8 (a) Adsorption isotherms of Pb^{2+} on ZSM-5-1, (b) Langmuir model, (c) Freundlich model and (d) Temkin model

All three models exhibit high linear correlation coefficients, as shown in Table 8, when fitting the adsorption process of Pb^{2+} by ZSM-5-1, suggesting that these models can adequately describe the adsorption process to some extent. However, the fitting results of the Langmuir and Tempkin models were slightly less accurate, indicating that the adsorption of Pb^{2+} by ZSM-5-1 does not occur exclusively at specific sites and does not strictly follow monolayer adsorption assumptions. In contrast, the Freundlich model exhibits a better fit, suggesting that the adsorption process may occur on heterogeneous surfaces and involve multilayer adsorption. In addition, the negative

charges on the molecular sieves surface may promote the adsorption of Pb^{2+} with excess positive charges through electrostatic effects. This demonstrates that electrostatic attraction is a key factor in the adsorption mechanism. Furthermore, the $1/n$ value of the Freundlich model is between 0 and 1, indicating that the adsorption process is thermodynamically favorable and confirming the feasibility of the adsorption process. Therefore, the adsorption process of Pb^{2+} by ZSM-5-1 involves the combined effect of multiple factors such as chemical adsorption, multilayer adsorption, and electrostatic interaction.

Table 8 Adsorption isotherm parameters of ZSM-5-1 for adsorbing Pb^{2+}

Temperature/ $^{\circ}\text{C}$	Langmuir			Freundlih			Tempkin	
	$q_{\text{max}}/(\text{mg}\cdot\text{g}^{-1})$	$K_L/(\text{L}\cdot\text{mg}^{-1})$	R^2	$K_F/(\text{mg}^{(1-1/n)}\cdot\text{L}^{1/n}\cdot\text{g}^{-1})$	$1/n$	R^2	$b_T/(\text{kJ}\cdot\text{mol}^{-1})$	R^2
25	261.78	0.0508	0.9856	61.39	0.26109	0.9908	108.34	0.9782
30	274.73	0.0615	0.9783	80.38	0.21978	0.9869	101.92	0.9413
35	305.81	0.0569	0.9591	87.08	0.22266	0.9597	117.74	0.8799

A thermodynamic analysis of the experimental process was conducted. The results are shown in Table 9. Here, $\Delta G^\ominus < 0$ indicates that the adsorption reaction proceeds spontaneously, $\Delta H^\ominus > 0$ indicates that the adsorption process is endothermic, and $\Delta S^\ominus > 0$ indicates that the degree of freedom increases during the adsorption process at the solid-liquid interface^[52].

2.3.4 Regeneration performance of ZSM-5

Adsorbent recovery and reuse are crucial for cost reduction. To explore the regeneration performance of ZSM-5-1, adsorption-desorption cycling tests were conducted using 0.1 mol/L disodium ethylenediaminetetraacetate solution for desorption. As an efficient metal chelating agent, disodium ethylenediaminetetraacetate forms stable complexes with Pb^{2+} , facilitating its desorption from the adsorbent surface. The desorption process was carried out by shaking at 25 °C for 12 h, followed by material washing and drying for subsequent adsorption cycle. As shown in Figure 9, after five repeated cycles, the Pb^{2+} removal efficiency exhibited only a slight decrease. The material demonstrates easy recovery and maintains high cyclic adsorption capacity, indicating that ZSM-5-1 possesses excellent reusability and economic potential for practical applications.

2.4 Adsorption mechanism discussion

2.4.1 XRD and FT-IR analysis of ZSM-5-1 before and after adsorption

To illustrate the feasible mechanism of ZSM-5-1 adsorbing Pb^{2+} from aqueous solution, the XRD and FT-IR spectra of ZSM-5-1 before and after Pb^{2+} adsorption were presented in Figure 10. Figure 10(a) demonstrates that after Pb^{2+} adsorption, the XRD peak intensity of ZSM-5-1 decreases slightly, while all characteristic diffraction peaks remain unchanged, indicating preservation of the molecular sieves' crystalline structure^[53]. The FT-IR analysis in Figure 10(b) revealed a shift in the -OH stretching vibration peak from 3440 to 3434 cm^{-1} with slight intensity enhancement, suggesting interactions between Pb^{2+} and

surface hydroxyl groups. Concurrently, the -OH bending vibration peak at 1638 cm^{-1} shifted to 1631 cm^{-1} , further indicating the involvement of structural water/hydroxyl groups in coordination during adsorption. Remaining -OH groups may also react with Pb^{2+} to form $\text{Pb}(\text{OH})_2$ precipitates^[54]. Notably, changes in the framework-related bands were observed. After Pb^{2+} adsorption, the 1105 cm^{-1} peak shifted to 1084 cm^{-1} , indicating that Pb^{2+} ions interact with the molecular sieves framework, possibly through electrostatic attraction or coordination with framework oxygen atoms after entering the pore channels. This interaction may lead to the formation of Si-O-Pb, Pb-O, or Pb-O-Pb species. In addition, the characteristic peak of the ZSM-5-1 at 550 cm^{-1} shifted to 544 cm^{-1} after adsorption, indicating minor framework distortion while maintaining overall structural stability. In conclusion, the adsorption of Pb^{2+} by ZSM-5-1 involves both surface hydroxyl groups interactions and localized framework disturbances. The preserved structural integrity confirms the structural stability of ZSM-5-1 and supports its suitability for repeated adsorption-regeneration cycles.

Table 9 Thermodynamic parameters of ZSM-5-1 Adsorption of Pb^{2+}

Temperature / °C	$\Delta H^\ominus$ / (kJ·mol ⁻¹)	$\Delta S^\ominus$ / (J·mol ⁻¹ ·K ⁻¹)	$\Delta G^\ominus$ / (kJ·mol ⁻¹)
25			-6.34
30	48.8	185.2	-7.27
35			-8.20

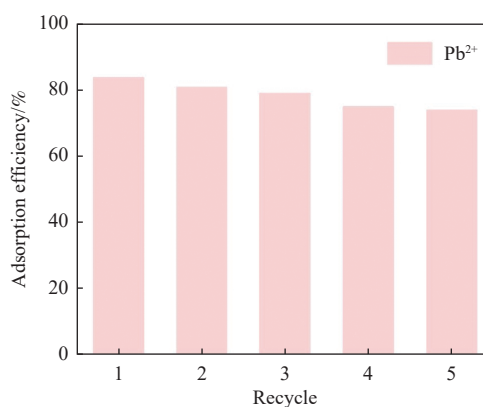


Fig. 9 Regeneration performance of ZSM-5-1

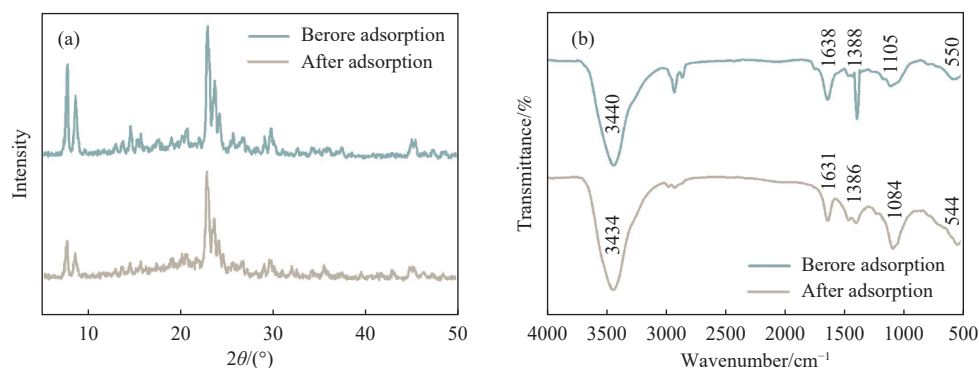


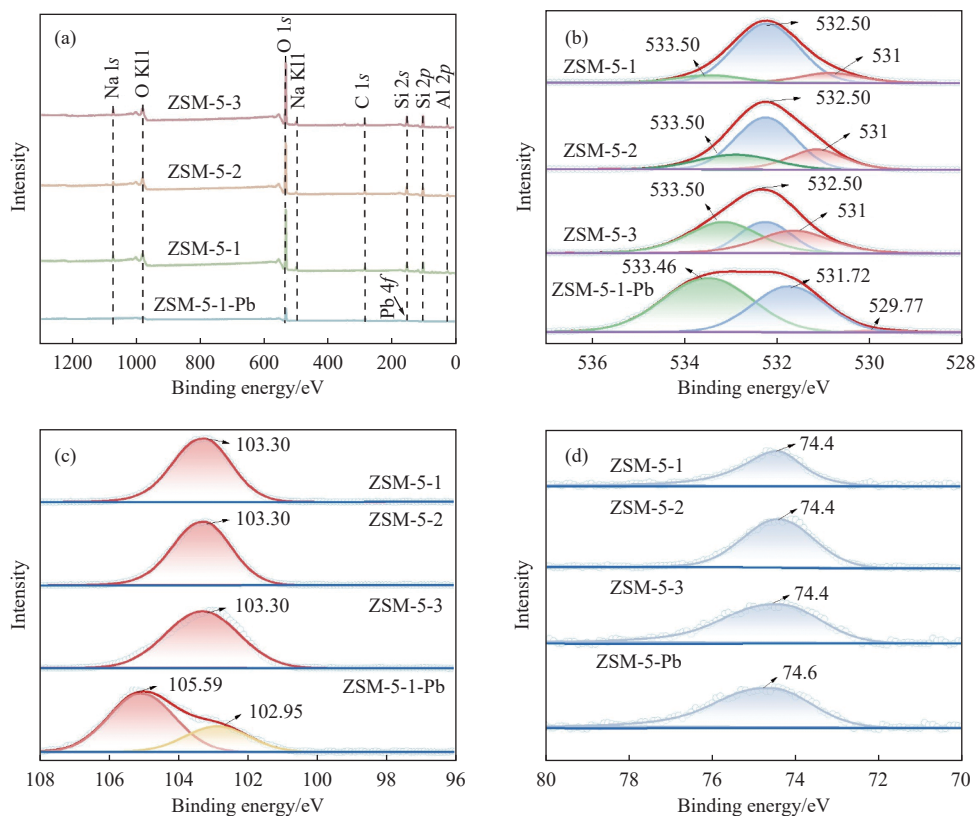
Fig. 10 (a) XRD patterns and (b) FT-IR spectra of ZSM-5-1 before and after Pb^{2+} adsorption

2.4.2 XPS analysis before and after adsorption

XPS was performed to investigate the surface chemical states of ZSM-5 before and after Pb^{2+} adsorption. As shown in Figure 11(a), a new characteristic peak corresponding to Pb^{2+} appears after Pb^{2+} adsorption on the ZSM-5. Meanwhile, the characteristic Na peak is significantly attenuated. This indicates that Pb^{2+} ions are adsorbed onto the ZSM-5 surface *via* an ion-exchange process. Figure 11(b) shows that oxygen species in the ZSM-5 exist in different chemical environments, which can be assigned to Al–O– (531 eV), Si–O–Si (532.5 eV), and Si–OH (533.5 eV), corresponding to the three deconvoluted O 1s peaks^[55–56].

After Pb^{2+} adsorption, the binding energies of these three characteristic peaks shift to lower values, suggesting that Pb^{2+} interacts with framework oxygen or surface hydroxyl groups to form Pb–O–Si or Pb–OH coordination structures, thereby reducing the binding energy. In Figure 11(c), the Si

2p spectrum of ZSM-5 before adsorption shows a single symmetrical peak at 103.3 eV. This peak is characteristic of the Si–O–Si bonds associated with Si^{4+} in the molecular sieves framework. It indicates Si exists in a uniform chemical environment within framework structure. After Pb^{2+} adsorption, the spectrum splits into two characteristic peaks at 102.95 and 105.59 eV. The higher binding energy peak (105.59 eV) likely arises from the forming of a Pb–O–Si coordination structure with surface hydroxyl groups. The lower binding energy peak (102.95 eV) may result from local framework defects induced by adsorption or exposure of extra-framework Si–OH. Figure 11(d) presents the Al 2p spectrum of ZSM-5, in which the peak at 74.4 eV corresponds to tetrahedrally coordinated framework AlO_4 species. After Pb^{2+} adsorption, the binding energy of Al 2p slightly increases to 74.6 eV, which may be attributed to partial replacement of surrounding H^+ ions by Pb^{2+} during adsorption, resulting in a modified local electronic environment around Al species.



(a): overall spectra; (b): O 1s spectra; (c): Si 2p spectra; (d): Al 2p spectra.

Fig. 11 XPS before and after adsorption

3 Conclusions

ZSM-5 was successfully synthesized from coal gasification fine slag, and their adsorption performance toward Pb^{2+} in aqueous solutions was systematically investigated. The main conclusions are summarized as follows:

(1) ZSM-5 with high specific surface area are obtained

from CGFS. Increasing the acid concentration facilitates silicon extraction and reduced impurities, while excessive alkali concentration reduces crystallinity and surface area, and even induces the formation of clinoptilolite.

(2) Adsorption experiments demonstrates that ZSM-5-1 exhibits the best performance, achieving a Pb^{2+} removal efficiency of 83.7% for a 50 mg/L solution at 25 °C, corresponding to an adsorption capacity of 104.625 mg/g.

(3) Thermodynamic and adsorption analyses indicate that Pb^{2+} adsorption process is spontaneous and endothermic, consistent with chemisorption-dominated of multilayer adsorption. Pb^{2+} removal from aqueous solution is governed by the combined effects of surface complexation, precipitation, electrostatic attraction, and ion exchange.

Declaration of Competing Interest

The authors declare no conflict of interest

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煤气化细渣合成 ZSM-5 分子筛及其对水中 Pb^{2+} 的吸附机理

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摘 要: 煤气化细渣(CGFS)是气流床煤气化的固体废弃物, 具有颗粒小、富含硅铝元素的特点, 以其为原料制备 ZSM-5 分子筛能够实现煤基固废的高值化利用, 具有较好的经济和环境效益。本研究提出了一种简便经济的以 CGFS 为原料的 ZSM-5 分子筛水热合成工艺, 通过酸洗去除 CGFS 中杂质, 继而采用碱液有效提取硅铝组分, 最终在 170 °C 水热条件下反应 48 h 合成出比表面积达 358 m^2/g 的 ZSM-5 分子筛。吸附实验结果表明, 所制备的 ZSM-5 分子筛对水溶液中 Pb^{2+} 表现出优异的吸附性能。在 25 °C 条件下, 对 50 mg/L Pb^{2+} 溶液的去除效率达 83.7%, 吸附容量为 104.625 mg/g 。吸附过程主要受表面络合、沉淀和离子交换等化学吸附机制主导。所合成 ZSM-5 分子筛在含铅废水处理领域具有良好应用潜力。

关键词: 气化渣; ZSM-5 分子筛; 合成与性能; 吸附机理; Pb^{2+}

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