

Article

Precise Adsorption and Separation of Tin(IV) and Cadmium(II) from High-Level Liquid by Mesoporous XAD-Based Adsorbent

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Abstract

A novel mesoporous XAD-based adsorbent (A336/XAD-7) was produced by impregnating the ionic liquid A336 into the pores of XAD-7 resin and used to separate tin(IV) and cadmium(II) from high-level liquid waste (HLLW). The as-produced material was characterized by SEM-EDS, TG-DSC, and N₂ adsorption–desorption isotherms, which revealed a well-developed open pore structure, high loading capacity, and large specific surface area. Adsorption performance analysis showed that in 4 M HCl solution, the experimental saturated adsorption capacity q_{exp} of A336/XAD-7 for Sn(IV) and Cd(II) were 39.51 mg/g and 34.18 mg/g, respectively, with equilibrium reached within 120 min. Among ten co-existing metal ions (Sn⁴⁺, Cd²⁺, Co²⁺, Ni²⁺, Cu²⁺, Eu³⁺, Y³⁺, Ca²⁺, Mg²⁺, Al³⁺) in HLLW, A336/XAD-7 exhibited excellent selectivity for Sn(IV) under high acidity, with a separation factor ($SF_{Sn/others}$) of 13.13. Column experiments further evaluated the dynamic separation of Sn(IV) from simulated HLLW using A336/XAD-7, achieving an enrichment factor greater than 7. XPS spectra indicated that the adsorption mechanism involved anion exchange between A336/XAD-7 and the complex anions SnCl₆^{2−} and CdCl₄^{2−}. This work demonstrates the application potential of A336/XAD-7 for HLLW treatment and provides valuable guidance for the efficient separation of other metal ions.

Keywords: ionic liquid; separation; tin; cadmium; adsorption mechanism; wastewater treatment



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1. Introduction

As a reliable, stable and efficient low-carbon energy source, nuclear energy can not only help reduce fossil fuel consumption and carbon dioxide emissions [1–3], but also contribute to global climate change mitigation and sustainable development [4–6], it is also playing an important role in the construction of low-carbon economy and green energy network [7,8]. The fission of ²³⁵U or ²³⁹Pu in nuclear reactors produces more than 40 elements (e.g., Ru, Rh, Pd, Tc, Se, Cd, Ag, Cs, Sr, Sn, Cd, etc.) and more than 400 nuclides, which is regarded as a man-made “mine” [9,10]. As one of the major fission products, Sn and Cd exist in considerable quantities, and their contents vary depending on the type and burnup of the reactor. Fission-generated nuclear waste contains variety isotopes, most of which are non- or weakly radioactive. For example, ¹²⁷Sn has a half-life of 2.1 h and ¹¹⁵Cd a half-life of 53.46 h [11]; thus, after storage for only a few days or even hours, Sn and Cd decay to levels meeting the basic safety standard (BSS) for industrial applications.

Consequently, high-level liquid waste (HLLW) from nuclear reactions can be regarded as a man-made resource pool rich in Sn and Cd. In summary, efficient separation and recovery of tin and cadmium from HLLW are of great significance, enabling their use as strategic reserve resources and promoting the resource utilization of radioactive waste.

Various methods have been investigated for treating wastewater containing Sn and Cd, including chemical precipitation [12], electrochemical [13–15], solvent extraction [16,17], and adsorption (including ion exchange) [18–20]. Among these, adsorption is the more commonly used method for separating Sn and Cd from wastewater. On the other hand, adsorption and ion exchange methods are less energy-intensive, easy to automate, and allow material reuse, thus offering more prominent advantages in terms of environmental protection and cost. A wide variety of adsorbents exist, such as metal oxides [21,22], carbon materials [23–27], imprinted materials [28], and resins [29–32], which provide considerable design and modification flexibility to meet different separation needs. Cheng et al. [33] prepared a bifunctional resin with ion exchange and complexation by introducing iminodiacetic acid complexing groups into a weakly acidic cation exchange resin. This modification enhanced the adsorption capacity for Cd(II) by two-fold and enabled adsorption under more acidic conditions (pH 0.5–2.5), with equilibrium reached within 5 min. Yan et al. [28] synthesized organic–inorganic hybrid Cd²⁺ imprints via hydrolysis and condensation under alkaline conditions, using Cd(II) and cetyltrimethylammonium bromide (CTAB) as templating agents in a mixture of tetraethyl silicate and 3-(2-aminoethylamino)propyltrimethoxysilane. The resulting hybrid imprints exhibited optimal Cd(II) adsorption at pH 5–6, with a very fast adsorption rate (equilibrium reached in 5 min). In the presence of coexisting Cd(II) and Zn(II), the selectivity coefficient of the imprinted material for Cd(II) exceeded 100. Additionally, a Cd(II)-imprinted chitosan resin showed an adsorption capacity of 0.795 mmol/g for Cd²⁺ at pH 5.0 and 45 °C, with an equilibrium time of 10 h.

Owing to its excellent chemical stability, high selectivity, and high loading capacity, di-(2-ethylhexyl)phosphoric acid (D₂EHPA) was loaded onto macroporous silica by Qin et al. [34] to prepare the adsorbent D₂EHPA/SiO₂-P for the selective separation of Sn(IV) from liquid crystal displays. In a 6 M H₂SO₄ solution, this adsorbent exhibited remarkable selectivity for Sn(IV), with separation factors ($SF_{\text{Sn/other metals}}$) exceeding 1479 mL/g. The organic adsorbent also demonstrated excellent acid resistance stability, making it highly promising for the recovery of Cd(II) and Sn(IV) from strongly acidic media.

Functional ionic liquids exhibit outstanding performance in the adsorption and removal of metal ions due to their structural tunability and multiple coordination effects. By precisely regulating the cationic skeleton and anionic species, specific coordination sites such as carboxyl, amino, and thiol groups can be introduced. These sites enhance electrostatic attraction, chelation, and hydrophobic interactions with heavy metal ions, enabling efficient selective capture and enrichment of target metal ions. This adsorption system combines high adsorption capacity, fast kinetics, and excellent reusability, providing a green and efficient technological pathway for the deep purification of heavy metal-containing wastewater, resource utilization of hazardous waste, and in-situ environmental remediation. Therefore, it holds significant theoretical value and application potential in environmental governance and industrial clean production [35].

To our knowledge, the recovery of tin and cadmium from high-level radioactive waste solutions using ionic liquids has not been reported. Ionic liquids offer advantages such as excellent thermal stability, structural tunability, and chemical stability. In this work, the A336/XAD-7 adsorbent was produced by vacuum impregnation and further evaluated via batch experiments for the efficient removal of Sn(IV) and Cd(II) from the liquid phase under various influencing conditions, including acidity, contact time, temperature, and the presence of competing ions. On this basis, column experiments in a hydrochloric acid

system were carried out to further explore the feasibility of separating Sn(IV) and Cd(II) using the A336/XAD-7 adsorbent. The interaction mechanism of selective adsorption of Sn(IV) and Cd(II) by A336/XAD-7 was analyzed by combining multiple characterization methods. This work not only provides a new method for the selective separation of Sn(IV) and Cd(II) but also contributes to realizing the reutilization of metal resources.

2. Materials and Methods

2.1. Materials

Analytical grade chemical reagents were used in this study without additional purification. Methyltriethylammonium chloride (A336) was obtained from Aladdin (Shanghai, China) Co. XAD-7 resin, magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), aluminum nitrate nonahydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), copper nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$), yttrium nitrate hexahydrate ($\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), europium nitrate hexahydrate ($\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), cobalt nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), cadmium nitrate tetrahydrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), tin(IV) chloride pentahydrate ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$), and dichloromethane (CH_2Cl_2) were supplied by Shanghai Macklin (Shanghai, China) Co. Hydrochloric acid (HCl) was provided by Sinopharm Chemical Reagent (Shanghai, China) Co. Appropriate amounts of $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ and $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ were dissolved in hydrochloric acid to prepare a stock solution containing 1000 mg/L of Sn(IV) and Cd(II).

2.2. Preparation of A336/XAD-7

The ionic liquid A336 was loaded onto the XAD-7 support via a vacuum impregnation method [36]. The synthesis process is illustrated in Figure 1 and involved the following steps: (i) XAD-7 was placed in a conical flask, and an appropriate amount of methanol was added. The mixture was gently shaken for 1 h and then allowed to stand until the supernatant became clear. The solid and liquid phases were separated, and the above procedure was repeated three times. The resulting solid was then dried in a vacuum oven at 45 °C for 48 h for subsequent use. (ii) 5 g of A336 was dissolved in an appropriate amount of dichloromethane (CH_2Cl_2) in a round-bottom flask. Then, 10 g of XAD-7 was added, and the mixture was stirred to ensure uniform dispersion. The flask was subsequently attached to a rotary evaporator. (iii) At room temperature and pressure, the flask was rotated at 100 rpm for 1 h, followed by standing for 1 h. (iv) The water bath was heated to 36 °C, and the flask was kept rotating at 100 rpm for 1.5 h. Meanwhile, the cooling water circulator was turned on during heating. (v) After completing the above steps, the vacuum pump was turned on. A336 was impregnated into the pores of XAD-7 under a slow pressure reduction until the diluent CH_2Cl_2 was completely volatilized. (vi) The produced A336/XAD-7 was placed in a vacuum drying oven and dried for later use.

2.3. Characterization

The surface and cross-sectional morphology as well as the elemental distribution of the bare XAD-7 support and the A336/XAD-7 adsorbent (both fresh and after adsorption of Sn(IV) and Cd(II)) were characterized by field emission scanning electron microscopy (ZEISS Sigma 300, Oberkochen, Germany). The specific surface area and pore diameter of XAD-7 and A336/XAD-7 were determined using a fully automated surface area analyzer (Micromeritics Instrument Ltd., Norcross, GA, USA, TriStar II 3020). Thermogravimetric (TG) data of XAD-7 and A336/XAD-7 were collected using a simultaneous thermal analyzer (NETZSCH, Selb, Germany, STA 449 F3). An X-ray photoelectron spectrometer (Thermo Fisher Scientific, Waltham, MA, USA, ESCALAB 250XI+) was employed to examine the changes in chemical bonding before and after adsorption. XPS data analysis was performed

using Advantage 5.9929. The structural analysis of XAD-7 and the samples before and after adsorption was conducted by Fourier-transform infrared spectroscopy (Shimadzu, Kyoto, Japan, IRTracer-100). For Fourier-transform infrared spectroscopy (FT-IR) analysis, the samples were thoroughly mixed and ground with potassium bromide (KBr) at a mass ratio of 1:100.

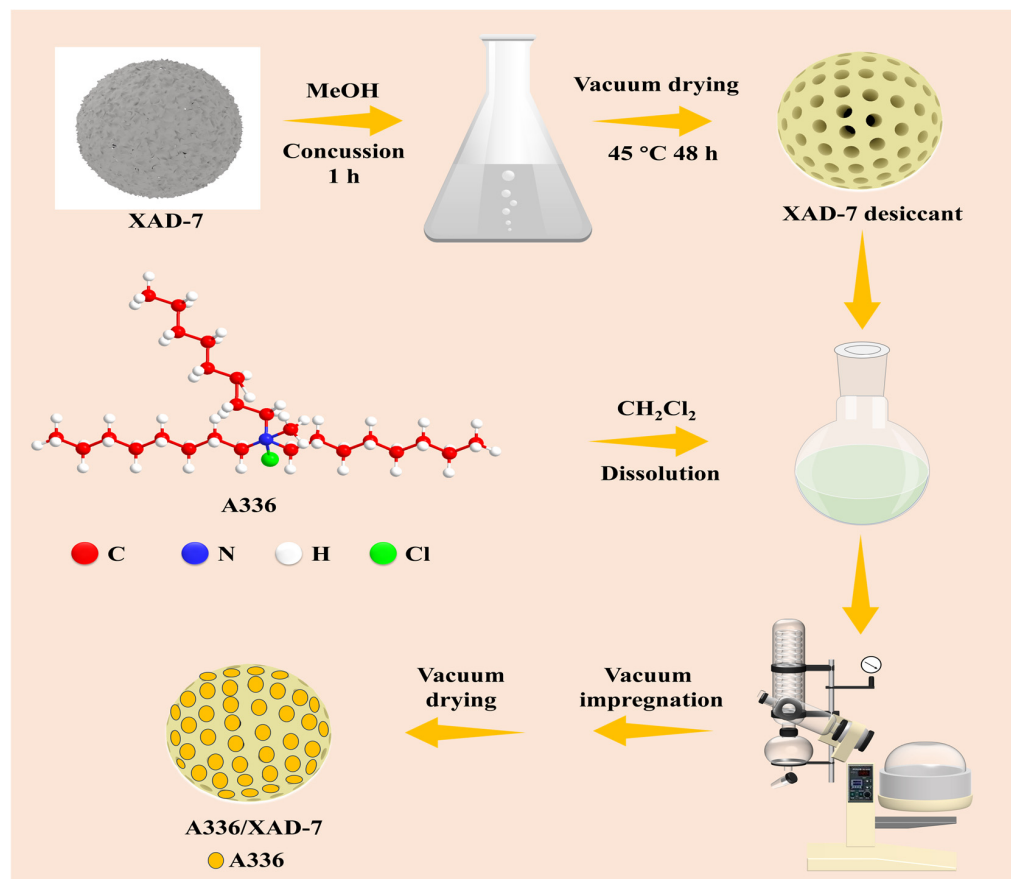


Figure 1. Schematic illustration of the preparation process for A336/XAD-7 adsorbent.

2.4. Batch Adsorption Experiments

The adsorption of Sn(IV) and Cd(II) by A336/XAD-7 was first investigated at a fixed solid-to-liquid ratio of 0.1 g/10 mL in sealed glass bottles, with the mixtures shaken in a water bath at 140 rpm. After 6 h of shaking, the solid phase was separated from the liquid phase using a syringe and a membrane filter with an average pore size of 0.45 µm. The concentrations of metal ions in the aqueous phase were determined by ICP-AES (Shimadzu, ICPS-7510). The adsorption capacity Q (mg/g), distribution coefficient K_d (mL/g), adsorption efficiency E (%), and separation factor $SF_{A/B}$ were calculated using Equations (1)–(4) [37].

$$\text{Adsorption amount } Q \text{ (mg/g)} : Q = \frac{(C_0 - C)}{m} \times V \quad (1)$$

$$\text{Distribution coefficient } K_d \text{ (mL/g)} : K_d = \frac{(C_0 - C)}{C} \times \frac{V}{m} \quad (2)$$

$$\text{Adsorption rate } E \text{ (%) } : E = \frac{(C_0 - C)}{C_0} \times 100\% \quad (3)$$

$$\text{Separation factor } SF_{A/B} : SF_{A/B} = K_{dA}/K_{dB} \quad (4)$$

where C_0 and C (mg/L) are the initial and equilibrium concentrations of Sn(IV) and Cd(II), respectively; V (mL) is the volume of the solution; and m (g) is the mass of the adsorbent.

2.5. Column Experiment

0.2 g of dried A336/XAD-7 was loaded into a chromatographic column (0.5 cm × 10 cm) using the wet packing technique. Simulated wastewater containing Sn(IV) and Cd(II) was then fed into the packed column at a flow rate of approximately 0.5 mL/min, and the entire effluent was collected using a fraction collector.

3. Results

3.1. Characterization

The SEM-EDS analysis results of XAD-7, A336/XAD-7, Cd-loaded A336/XAD-7, and Sn-loaded A336/XAD-7 are shown in Figure 2. Figure 2a presents a field emission scanning electron microscope image of an XAD-7 sphere. Figure 2b shows that, at higher magnification, abundant honeycomb-like pores can be observed on the surface of XAD-7. These numerous pores provide the prerequisite and foundation for ligand infusion and immobilization. Figure 2c shows the XAD-7 resin after infusion of A336. After the composite was formed by the vacuum impregnation method, the surface of A336/XAD-7 still exhibited a porous structure, but the porosity was significantly reduced, indicating that A336 had been infused into XAD-7 and that the ligand infusion did not destroy the original structure of the resin support (XAD-7). Figure 2d shows that, compared with the bare XAD-7 support, the surface of A336/XAD-7 still possessed a rich honeycomb-like pore structure. In addition to the major elements C and O, elements N and Cl were also observed. Since N and Cl are present only in A336, this confirms that the ligand was successfully infused into the pores of the XAD-7 resin support. Figure 2e,f show that Sn and Cd were detected only in A336/XAD-7 after the adsorption of Sn(IV) and Cd(II), respectively, indicating that Sn(IV) and Cd(II) were successfully adsorbed by A336/XAD-7 [38].

According to the BET test results shown in Figure 3a, both the XAD-7 and A336/XAD-7 isotherms are typical Type IV adsorption isotherms with H3-type hysteresis loops, indicating that the materials possess well-developed mesoporous structures. As summarized in Table 1, compared with pristine XAD-7, the specific surface area and pore volume of A336/XAD-7 decreased significantly, and the average pore size also decreased. These observations confirm that A336 was successfully impregnated and immobilized within the pores of XAD-7 (Figure 3b) [39].

The thermal stability of XAD-7 and A336/XAD-7 was evaluated by TG-DSC (Figure 3c). Upon heating to 300 °C, XAD-7 exhibited a weight loss of 5%, which is attributed to the loss of bound water within the material, indicating super high-temperature resistance. As the temperature increased further, additional weight loss occurred, and a mass loss of nearly 100% was observed when the temperature reached approximately 500 °C. In contrast, the weight loss of A336/XAD-7 occurred mainly above 200 °C, which can be ascribed to the pyrolysis of A336 [40].

Table 1. BET results for XAD-7 and A336/XAD-7 obtained by nitrogen adsorption.

Material	BET Surface Area (m ² /g)	Pore Volume (cm ³ /g)	Pore Size (nm)
XAD-7	580.07	1.27	24.93
A336/XAD-7	75.69	0.61	18.55

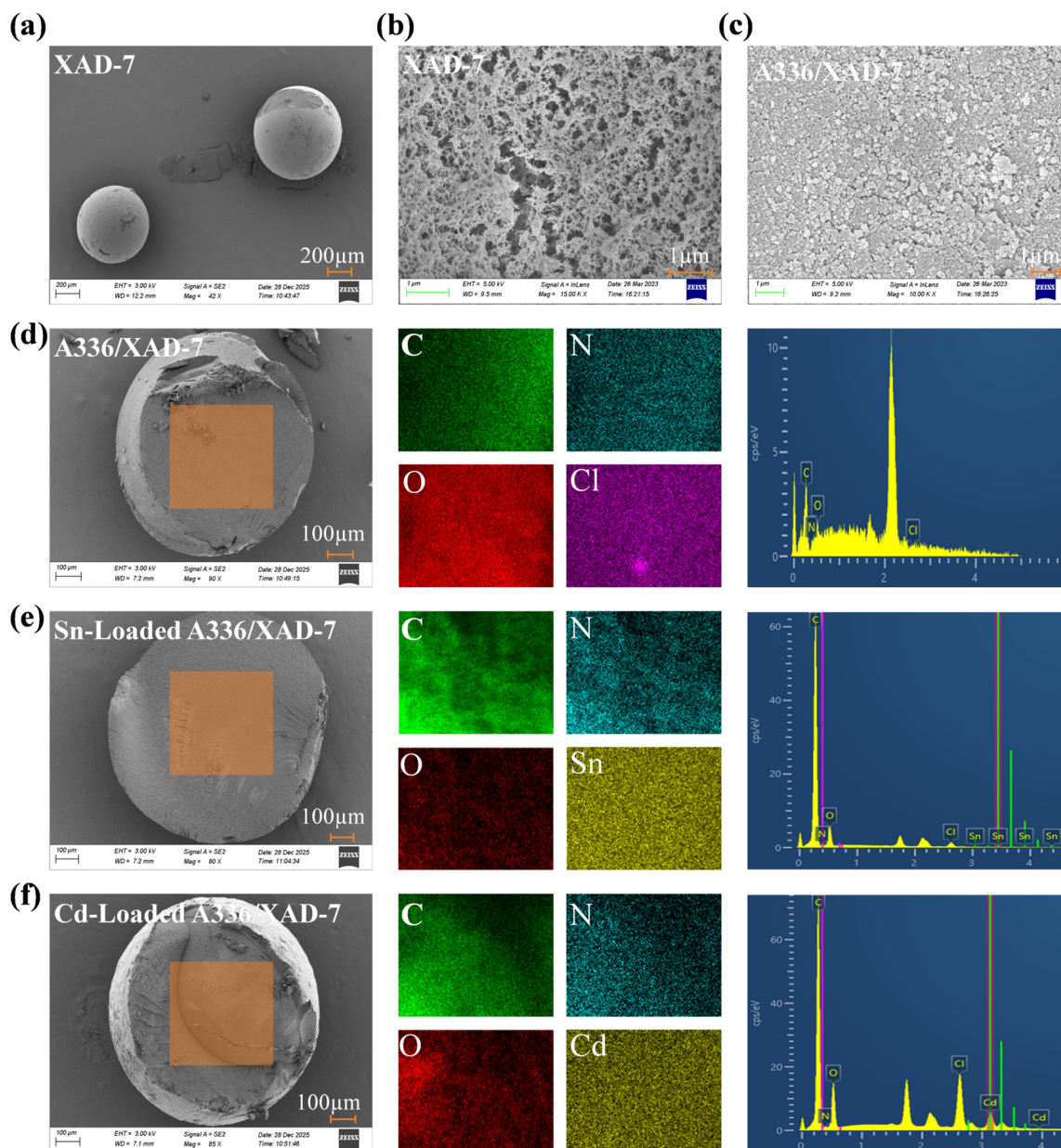


Figure 2. SEM images of (a,b) XAD-7; (c) A336/XAD-7; (d–f) EDS mapping spectra of A336/XAD-7, Sn-Loaded A336/XAD-7 and Cd-Loaded A336/XAD-7.

FT-IR is a common technique for analyzing material structures. As shown in Figure 3d, the absorption band at 3460 cm^{-1} arises from the H–O–H vibration; the bands in the range of $2850\text{--}2950\text{ cm}^{-1}$ originate from the stretching vibration of C–H in –CH_3 ; the band at 1730 cm^{-1} corresponds to the vibration of C=O in –COOR groups of XAD-7; and the band at 1145 cm^{-1} arises from the vibration of C–O in –COOR [41]. Additionally, a characteristic peak of C–N at 1057 cm^{-1} is observed [42], which indicates that A336 has been successfully impregnated in XAD-7. $500\text{--}900\text{ cm}^{-1}$, which are due to the vibration of XAD-7 polyacrylate skeleton.

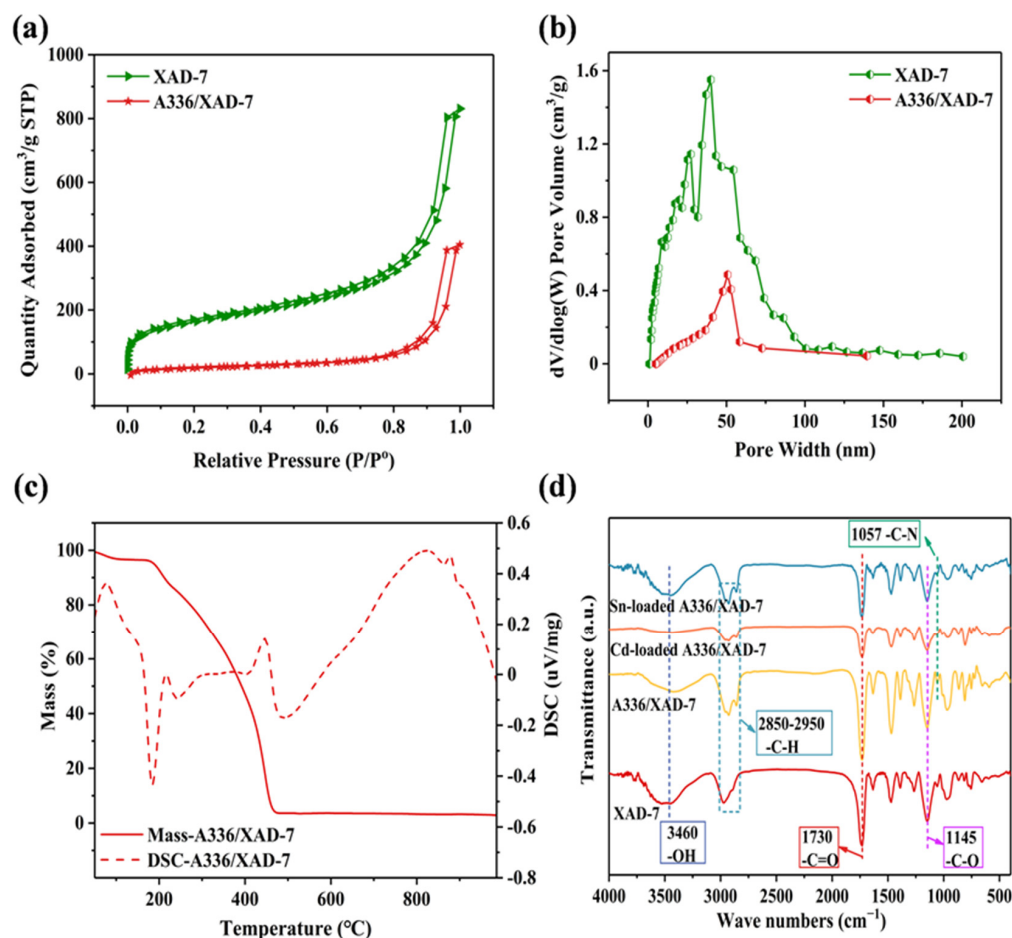


Figure 3. (a) N₂ adsorption–desorption isotherms and (b) pore size distribution analysis of XAD-7 and A336/XAD-7; (c) TG-DSC analysis; (d) FT-IR spectra.

3.2. Batch Adsorption Experiments

3.2.1. Effect of Impregnation Ratio on Adsorption

The adsorption performance of the A336/XAD-7 adsorbent toward Sn(IV) and Cd(II) at different impregnation ratios is shown in Figure 4a,b. The adsorption experimental data indicated that the adsorption capacity for both Sn(IV) and Cd(II) first increased and then decreased with increasing impregnation ratio, reaching a maximum at an impregnation ratio of 1:2 (*w/w*). This trend is attributed to the limited loading capacity of the XAD-7 resin. As the impregnation ratio increased, the viscosity of the reactant solution increased, causing the excess A336 that was not successfully loaded to adhere to the surface of the XAD-7 resin. This resulted in resin agglomeration and a reduction in adsorption performance [41].

3.2.2. Effect of Acidity on Adsorption Capacity of Tin and Cadmium

Acidity is one of the key factors affecting adsorption efficiency. The acidity of the aqueous phase influences the surface charge, ionization degree, and surface metal binding sites of the adsorbent, and optimizing the acidity can enhance adsorption performance. Therefore, the effect of acidity on adsorption was investigated, as shown in Figure 5. Figure 5a,b show that the adsorption capacity of A336/XAD-7 for Sn(IV) and Cd(II) increases with increasing acidity. Figure 5c,d present the species distribution curves for the Sn(IV)/Cd(II)–Cl[−]–H⁺. As the chloride ion concentration increases (i.e., pCl decreases), SnCl₆^{2−}, CdCl₄^{2−} become the dominant species in highly acidic chloride systems. These anionic complexes readily undergo ion exchange with the chloride ions in A336, which explains why the adsorption capacity increases with increasing acidity [43,44].

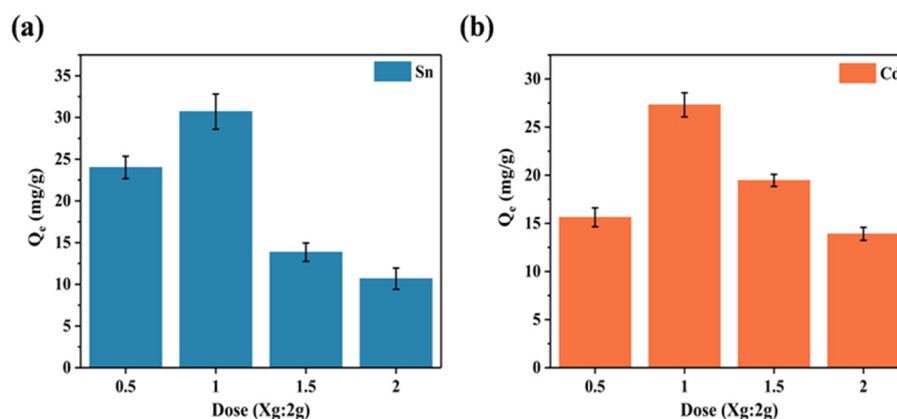


Figure 4. Effect of impregnation ratio on the adsorption capacity of (a) Sn(IV) and (b) Cd(II). Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, 1 M HCl, temperature = 298 K, contact time = 6 h.

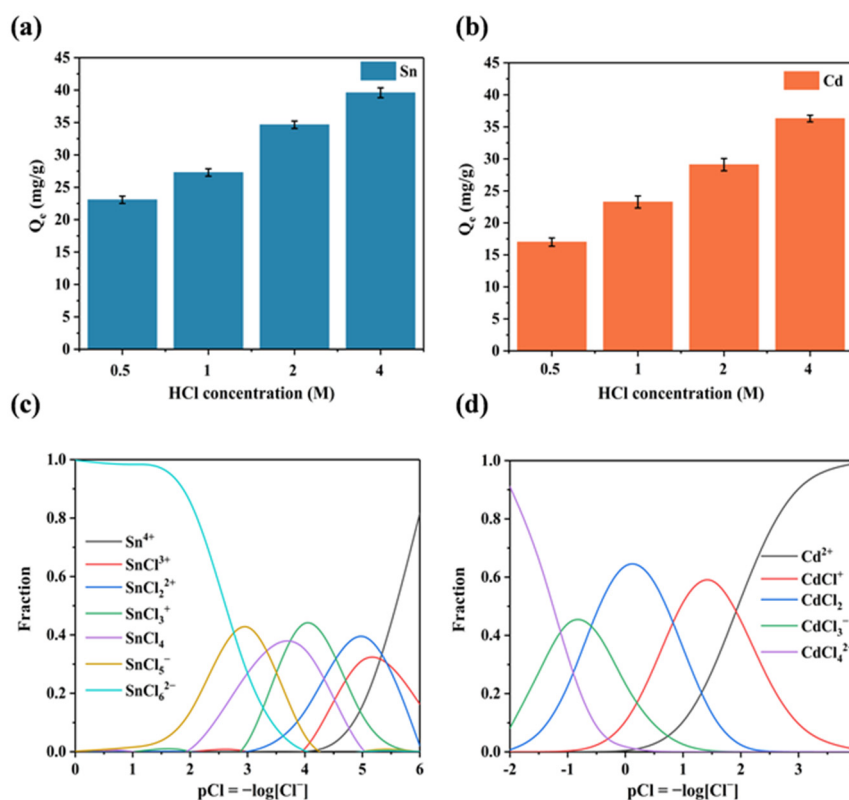


Figure 5. Effect of HCl concentration on the adsorption capacity of (a) Sn(IV) and (b) Cd(II); (c,d) species distribution curves for Sn(IV) (c) and Cd(II) (d). Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, temperature = 298 K, contact time = 6 h.

3.2.3. Kinetic Study

The contact time between the adsorbent and target ions in solution is an important factor affecting adsorption efficiency. Figure 6a,c show the effect of contact time on the adsorption performance of A336/XAD-7 toward Sn(IV) and Cd(II). A rapid adsorption phase lasting 120 min was observed, during which abundant active sites on the surface of A336/XAD-7 were available for the adsorption of Sn(IV) and Cd(II), resulting in a faster adsorption rate in the initial stage. However, between 120 and 480 min, most of the surface active sites had already been occupied by Sn(IV) and Cd(II). Further adsorption could only occur through diffusion of the metal ions into the interior of the adsorbent, so the adsorption at this stage took place mainly within the pores of the resin.

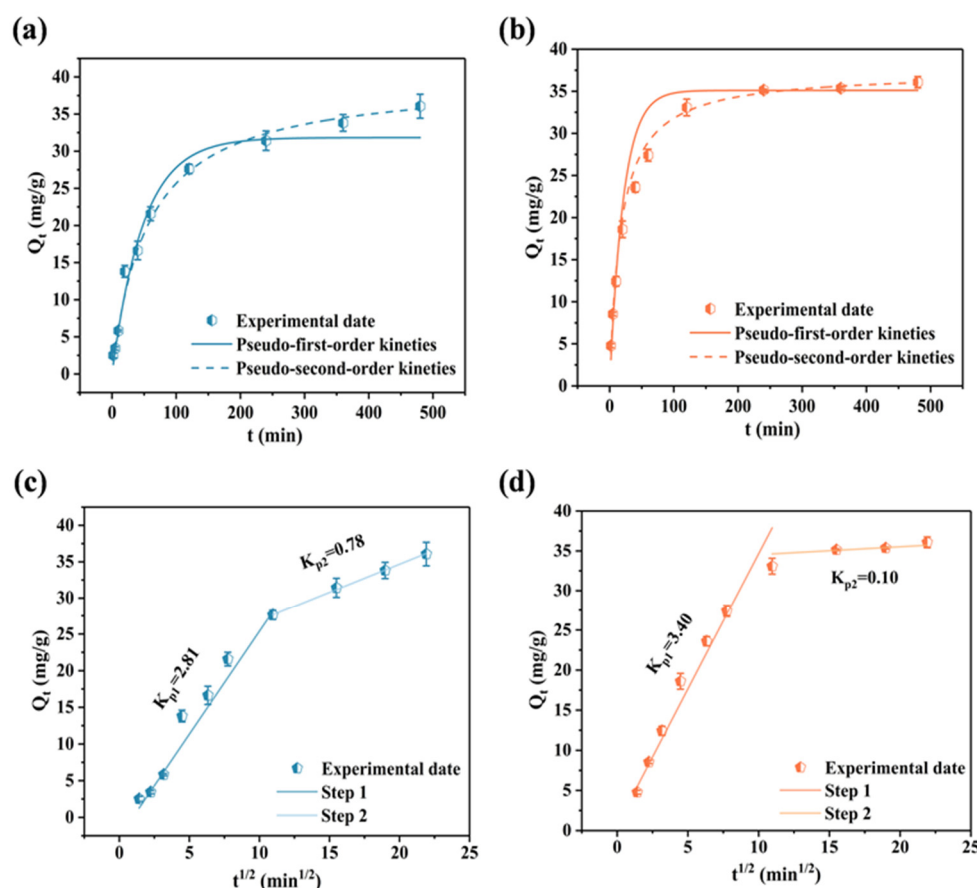


Figure 6. Fitting results of the pseudo-first-order, pseudo-second-order, and lintra-particle diffusion kinetic models for (a,b) Sn(IV) and (c,d) Cd(II). Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, 4 M HCl, temperature = 298 K.

Pseudo-first-order, pseudo-second-order, and lintra-particle diffusion models were used to fit the experimental kinetic data. The resulting kinetic parameters are summarized in Table 2. Comparison of the correlation coefficients (R^2) for the adsorption models in Table 2 indicates that the adsorption behaviors of Sn(IV) and Cd(II) by A336/XAD-7 are more consistent with the pseudo-second-order kinetic model, suggesting that the adsorption is mainly controlled by chemical interactions [45]. The lintra-particle diffusion model fitting for the adsorption of Sn(IV) and Cd(II) by A336/XAD-7 is shown in Figure 6b,d, and the adsorption process can be divided into two stages. In the first stage, the K_{p1} values for Sn(IV) and Cd(II) were 2.73 and 2.91, respectively, indicating high adsorption rates. When the adsorption equilibrium approached the second stage, the K_{p2} values for Sn(IV) and Cd(II) were 0.71 and 0.32, respectively, indicating that the adsorption process slowed down and neared equilibrium. Furthermore, none of the fitted curves passed through the origin, suggesting that intraparticle diffusion was not the sole rate-limiting step controlling the adsorption process.

Table 2. Adsorption kinetic parameters of A336/XAD-7 for Sn(IV) and Cd(II).

Element	Pseudo-First-Order			Pseudo-Second-Order		
	k_1 (1/min)	q_e (mg/g)	R^2	k_2 (g/mg·min)	q_e (mg/g)	R^2
Sn	0.02	32.36	0.98	0.001	37.25	0.99
Cd	0.03	34.22	0.96	0.001	37.41	0.99

3.2.4. Isotherm Study

The adsorption isotherms of Sn(IV) and Cd(II) on A336/XAD-7 at 298 K, 308 K, 318 K were investigated by increasing the initial concentrations of Sn(IV) and Cd(II) in the solution, as shown in Figure 7a,b. The adsorption capacity increased significantly with increasing Sn(IV) and Cd(II) concentrations at 298 K until a saturation plateau was reached at an equilibrium concentration (C_e) of approximately 150 mg/L. The adsorption data for Sn(IV) and Cd(II) by A336/XAD-7 were fitted and analyzed using the Langmuir and Freundlich adsorption isotherm models, and the corresponding model parameters are summarized in Table 3. Based on the correlation coefficients (R^2), the Langmuir model provided a better fit, indicating that the adsorption of Sn(IV) and Cd(II) onto the resin is a homogeneous monolayer chemisorption process [45].

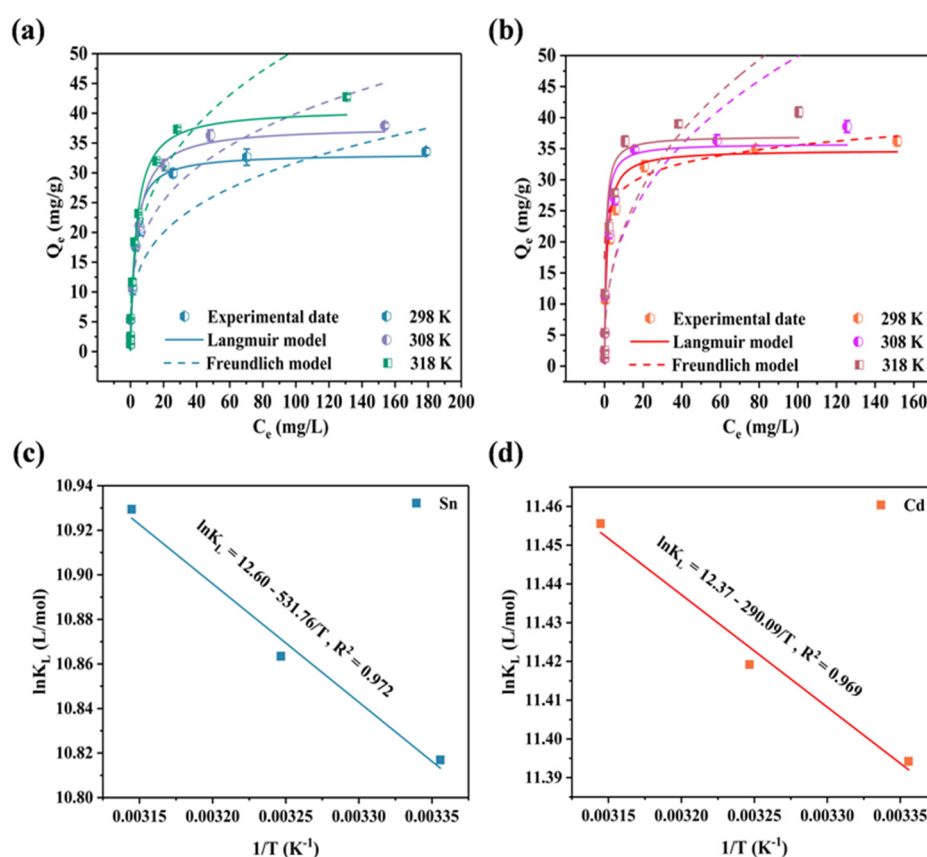


Figure 7. (a,b) Adsorption isotherms of Sn(IV) and Cd(II) onto A336/XAD-7 in 4 M HCl and (c,d) plots of $\ln K_L$ versus $1/T$ for Sn(IV) and Cd(II). Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, 4 M HCl, contact time = 6 h, temperature = 298 K, 308 K, 318 K.

Table 3. Parameters obtained from fitting the adsorption isotherm data at 298 K.

Element	T (K)	Langmuir Model			Freundlich Model		
		q_{max} (mg/g)	K_L (L/mg)	R^2	K_F ($mg^{1-n} \cdot L^n/g$)	n	R^2
Sn	298	33.23	0.42	0.95	8.49	0.29	0.90
	308	37.79	0.44	0.95	10.84	0.28	0.90
	318	40.77	0.47	0.95	11.79	0.32	0.93
Cd	298	34.79	0.79	0.99	22.97	0.10	0.79
	308	35.05	0.81	0.99	11.81	0.23	0.82
	318	37.03	0.84	0.94	9.10	0.39	0.80

3.2.5. Thermodynamics Study

To better describe the thermodynamics of Sn(IV) and Cd(II) adsorption by the A336/XAD-7 adsorbent, the experimental data were analyzed using the Van't Hoff equation, and the main thermodynamic parameters obtained are presented in Figure 7c,d and Table 4. The thermodynamic parameter ΔH^0 for the adsorption of Sn(IV) and Cd(II) by A336/XAD-7 was found to be 4.42 kJ/mol and 2.41 kJ/mol, respectively, indicating that the adsorption process is endothermic. The ΔG^0 values for Sn(IV) and Cd(II) at different temperatures were all negative, suggesting that the adsorption reactions are spontaneous. Furthermore, the positive ΔS^0 values indicate that the adsorption process of A336/XAD-7 toward Sn(IV) and Cd(II) is entropy-driven.

Table 4. Thermodynamic parameters for the adsorption of Sn(IV) and Cd(II) by the A336/XAD-7 adsorbent.

Element	T (K)	K_L (L/mol)	ΔH^0 (J/mol)	ΔS^0 (J/mol·K)	ΔG^0 (J/mol)
Sn	298	4.99×10^4	4.42×10^3	104.74	-2.68×10^4
	308	5.22×10^4			-2.78×10^4
	318	5.58×10^4			-2.89×10^4
Cd	298	8.88×10^4	2.41×10^3	102.81	-2.82×10^4
	308	9.11×10^4			-2.92×10^4
	318	9.44×10^4			-3.03×10^4

3.2.6. Effect of Acidity on Selective Adsorption of Sn and Cd

As shown in Figure 8a, A336/XAD-7 exhibited outstanding selective adsorption for Sn(IV) and Cd(II) in 0.5–4 M HCl solutions. With increasing HCl concentration, the adsorption capacity for Sn(IV) continuously increased, whereas the adsorption capacity for Cd(II) first increased and then decreased, reaching a maximum at 1 M HCl. Furthermore, Figure 8b presents the separation coefficients of various metal ions on A336/XAD-7 in 4 M hydrochloric acid. The K_d value of Sn(IV) is extremely high, nearly reaching 250 mL/g, which indicates that the resin has a much stronger adsorption affinity for Sn(IV) than for other metals in 4 M HCl. The K_d value of Cd drops sharply to approximately 20 mL/g, while the K_d values of all other metals (Co, Ni, Cu, Eu, Y, Ca, Mg, Al) are close to zero, meaning the resin barely adsorbs these metals. The separation factor of Sn for the other nine ions is shown in Table 5. The separation factor $SF_{Sn/other\ metals}$ is greater than 13.13, demonstrating that the A336/XAD-7 resin possesses an exceptionally high selectivity for Sn(IV) in 4 M HCl [46].

Table 5. Separation Factor of Sn relative to the other nine ions.

X	Al	Mg	Ca	Y	Cu	Ni	Co	Cd
$SF_{Sn/X}$	585.08	827.55	1063.92	1006.23	324.06	450.04	228.05	13.14

3.2.7. Desorption Study of A336/XAD-7

A336/XAD-7 exhibited outstanding adsorption performance for Sn(IV) and Cd(II) in hydrochloric acid media. Its desorption performance is also an important indicator for evaluating the practical applicability of the adsorbent material. As shown in the batch shaking-bed experiment results (Figure 9), 3 M HNO₃ and 0.1 M HNO₃ exhibited the best desorption efficiency for A336/XAD-7 loaded with Sn(IV) and Cd(II), respectively.

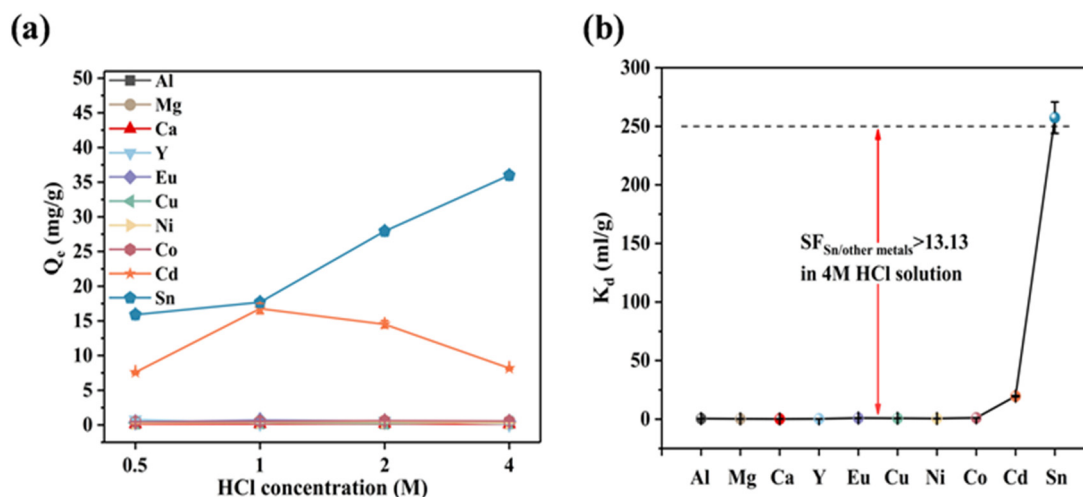


Figure 8. (a) Effect of HCl concentration on the adsorption of ten coexisting ions by A336/XAD-7. (b) Separation coefficients of ten coexisting ions adsorbed by A336/XAD-7 adsorbent. Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, temperature = 298 K, contact time = 6 h.

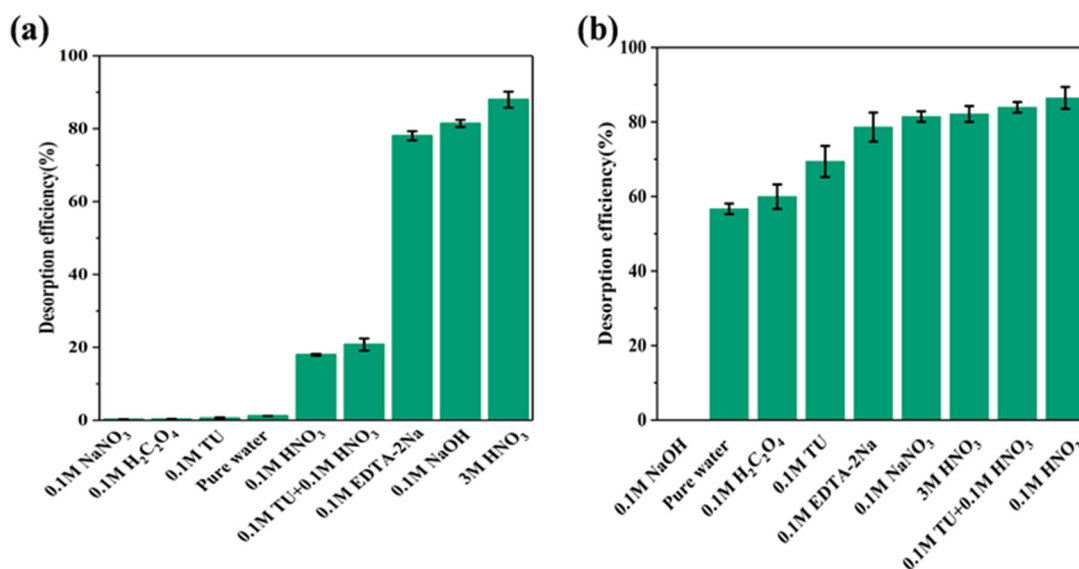


Figure 9. Desorption performance of (a) Sn(IV) and (b) Cd(II) with different eluent. Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, temperature = 298 K, contact time = 6 h.

3.2.8. Study on the Recycling Performance of A336/XAD-7

From an economic perspective, the recovery of adsorbents in practical applications can cut costs and reduce resource waste, so research on the recyclability of adsorbents is of vital importance. To explore the practical applicability of this composite material, four batches of cyclic adsorption experiments were carried out in this work (Figure 10) to further investigate its reusability. After each adsorption process, the materials were desorbed with 3 M HNO₃ and 0.1 M HNO₃ respectively, followed by washing, filtration and drying for the subsequent cyclic adsorption test. As presented in Figure 10, the removal efficiencies of Sn(IV) and Cd(II) by A336/XAD-7 declined by 9.85% and 7.48% respectively with the rising number of adsorption cycles, and such reduction in adsorption efficiency can be attributed to the incomplete desorption of Sn(IV) and Cd(II) from the adsorbent surface. The obtained results further verify that A336/XAD-7 possesses favorable stability and regeneration performance.

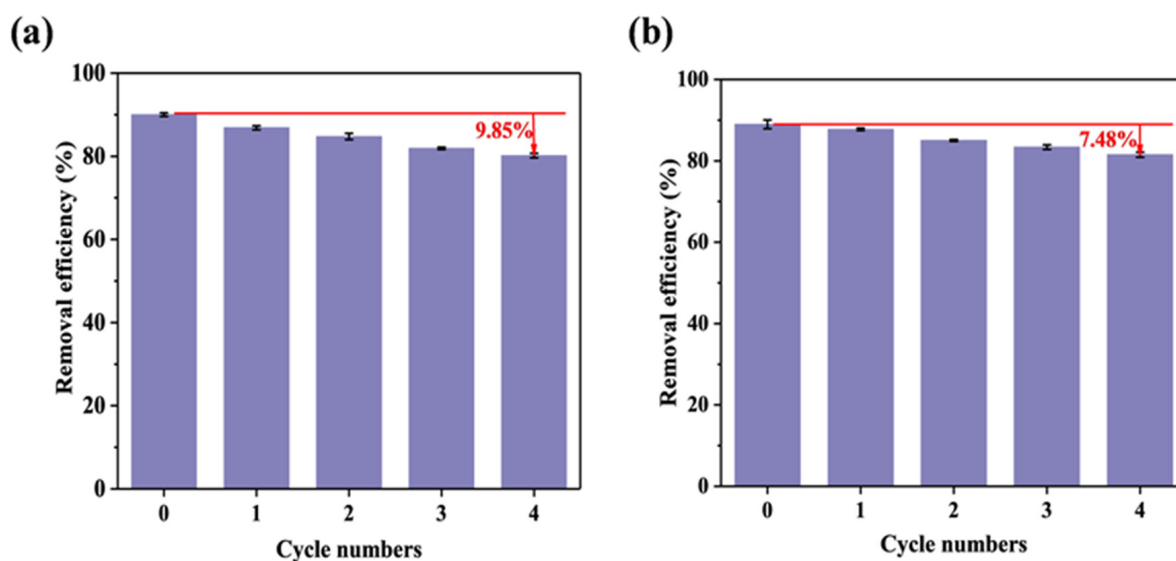


Figure 10. Reusability of A336/XAD-7 on (a) Sn(IV) and (b) Cd(II) removal. Experimental conditions: $C_0 = 500$ mg/L, solid-to-liquid ratio = 0.1 g/10 mL, temperature = 298 K, contact time = 6 h.

3.3. Adsorption Mechanism

To confirm the functional group composition of A336/XAD-7 and the binding mechanism between the resin and metal ions, the material was characterized by FT-IR and X-ray Photoelectron Spectroscopy (XPS). The FTIR spectra before and after adsorption are shown in Figure 11a, the C-N peak at 1057 cm^{-1} changed remarkably. After the adsorption of Sn(IV) and Cd(II), the intensity of the peak shape altered obviously, which indicates that the NR_4^+ of A336 had direct interactions (ion exchange) with Sn(IV) and Cd(II). Nevertheless, the variation range of the C-N peak after Cd(II) adsorption was smaller than that after Sn(IV) adsorption, demonstrating that the interaction strength between Cd and the resin is weaker than that between Sn(IV) and the resin [42]. The fingerprint peaks at $500\text{--}900\text{ cm}^{-1}$ showed a slight overall shift after metal ion loading, which further proved the occurrence of adsorption interactions.

The relationship between metal ions and chemical bonds was further analyzed by XPS. The survey spectra, as well as the Sn 3d, Cd 3d, and N 1s narrow-scan spectra before and after adsorption by A336/XAD-7 were recorded, fitted, and analyzed in detail, and the results are presented in Figure 11b–d. As shown in Figure 11b,c, the characteristic peaks of the main elements (C, N, O, and Cl) of the A336/XAD-7 resin were successfully observed in the XPS survey spectra. Compared with the A336/XAD-7 resin after Sn and Cd adsorption, the Sn 3d peak [47] and Cd 3d peak [48] were present only in the resin after adsorption, indicating that Sn and Cd were effectively adsorbed. The N 1s narrow-scan results are shown in Figure 11d. Two forms of nitrogen were present in A336/XAD-7: NR_4^+ , with a characteristic peak at 402.12 eV [49,50]; and C-N , with a characteristic peak at 399.43 eV , originating from trioctylamine. The NR_4^+ binding energies of Sn- and Cd-loaded resins decreased to 401.85 eV and 401.83 eV , indicating that the electrostatic environment around the NR_4^+ is changed after anion exchange. Furthermore, Cl characteristic peaks were observed in the XPS survey spectra both before and after adsorption, suggesting that the entire adsorption process requires the participation of Cl^- for charge balance [51]. The proposed adsorption mechanism is illustrated in Figure 12.

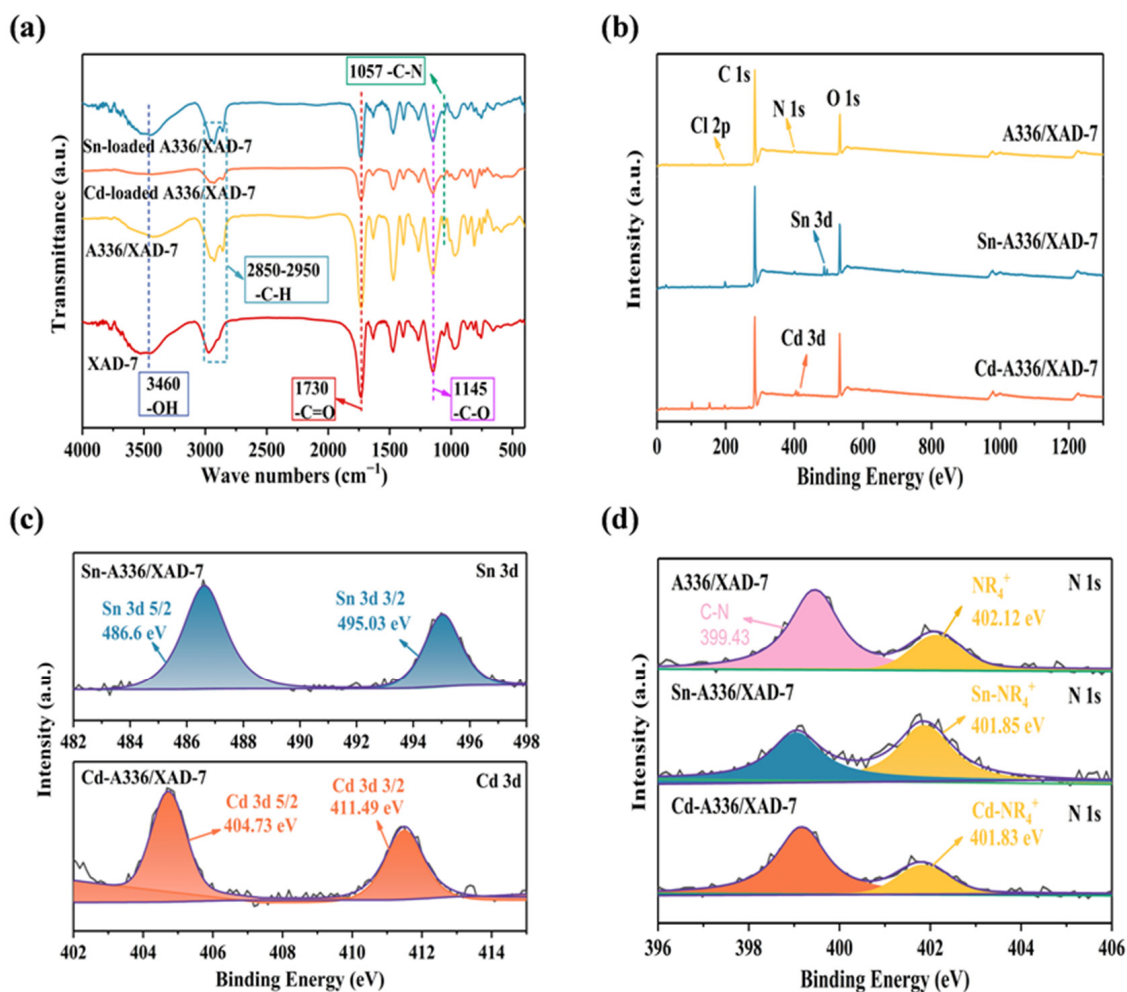


Figure 11. (a) FT-IR spectra of XAD-7, A336/XAD-7, Sn-loaded A336/XAD-7, and Cd-loaded A336/XAD-7; (b) XPS spectra of A336/XAD-7, Sn-loaded A336/XAD-7, and Cd-loaded A336/XAD-7; (c) Sn 3d and Cd 3d XPS spectra; (d) N 1s XPS spectrum.

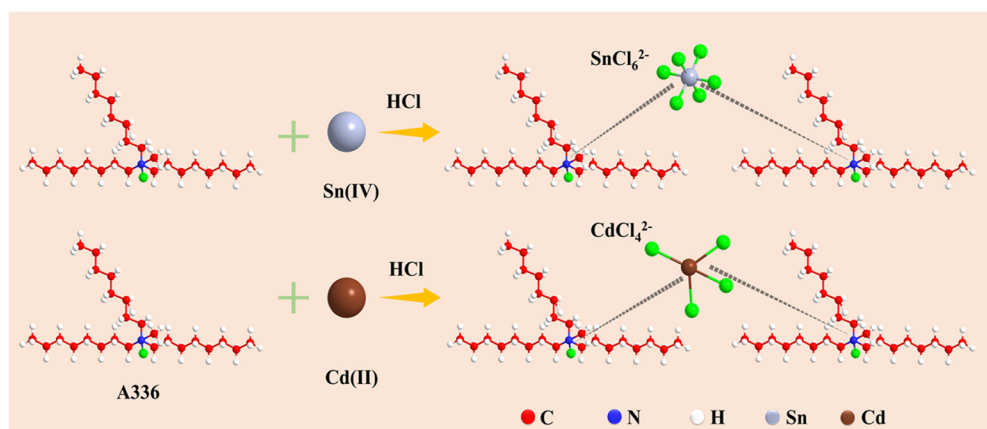


Figure 12. Adsorption mechanism of A336/XAD-7 towards Sn(IV) and Cd(II).

3.4. Dynamic Column Experiment with Simulated Wastewater

Based on the static experimental results, a sequential column experiment was conducted to separate Sn(IV) from a simulated HLLW solution using A336/XAD-7, and the results are shown in Figure 13. In Stage A, the column was pre-equilibrated with 4 M HCl. In Stage B, Sn(IV) and Cd(II) were selectively adsorbed from the simulated HLLW

solution containing 4 M HCl and other coexisting ions. Most of the metal ions (including Al^{3+} , Cu^{2+} , Ca^{2+} , Mg^{2+} , Ni^{2+} , etc.) flowed out rapidly with the feed solution, indicating that A336/XAD-7 did not adsorb these impurity ions. An interesting phenomenon was observed: at this stage, the relative concentration (C/C_0) of Cd(II) did not peak at 1 but continued to rise to 1.23 and remained at that level for some time until the concentrations of Sn(IV) and Cd(II) gradually became equal to those in the feed solution ($C/C_0 = 1$). This can be explained by the fact that as the feed solution continued to flow through the column, fresh Sn(IV) in the feed gradually occupied the adsorption sites previously taken by Cd(II), thereby displacing Cd(II) from the resin and causing it to exit with the effluent. Consequently, the effluent concentration of Cd(II) became higher than that in the feed solution. In Stage C, the residual ions (Al^{3+} , Cu^{2+} , Ca^{2+} , Mg^{2+} , Ni^{2+}) in the column were eluted with ultrapure water. In Stage D, Sn(IV) was desorbed using 3 M HNO_3 , achieving an enrichment factor greater than 7 ($\text{EF} = C/C_0$, where C represents the ion concentration in the effluent, C_0 is the initial ion concentration of the feed solution flowing into the column). These results demonstrate that A336/XAD-7 is an excellent adsorbent for the separation of Sn(IV) from Cd(II)-containing solutions.

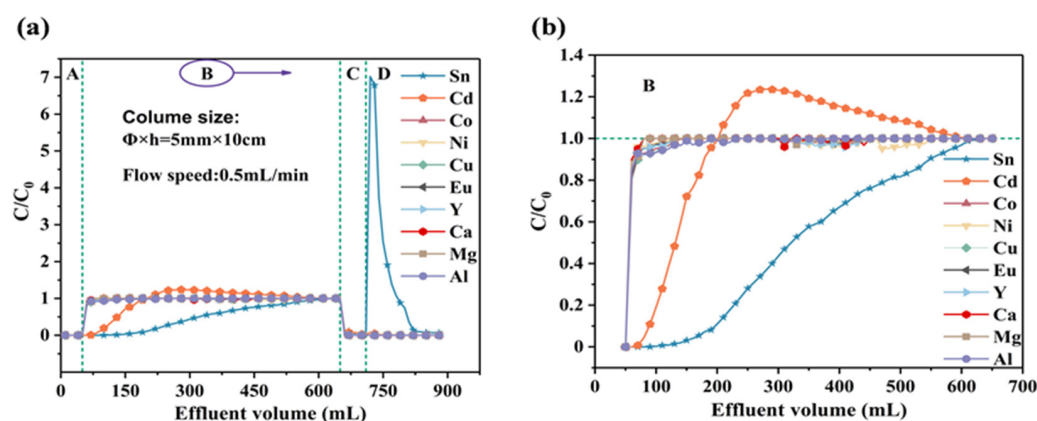


Figure 13. (a) Column separation of Sn(IV) using A336/XAD-7 packed column at 298K. (b) Stage B of separation of Sn(IV) by column. Experimental Conditions: $m = 2.06$ g; A: 4 M HCl; B: simulated wastewater; C: ultrapure water; D: 3 M HNO_3 , temperature = 298 K.

4. Conclusions

In this study, a novel XAD-7-based adsorbent was prepared by impregnating A336 onto XAD-7 for the selective separation of Sn(IV) and Cd(II) from high-level liquid waste (HLLW). Kinetic and isotherm studies showed that the adsorption equilibrium of the A336/XAD-7 adsorbent for Sn(IV) and Cd(II) was reached within 120 min, and the adsorption process followed the pseudo-second-order kinetic model and the Langmuir isotherm model. The A336/XAD-7 adsorbent exhibited excellent selectivity for Sn(IV) and Cd(II) among ten coexisting metal ions in HLLW. Nearly complete desorption of Sn(IV) and Cd(II) was achieved using 3 M HNO_3 and 0.1 M HNO_3 , respectively. Column experiments successfully separated Sn(IV) and Cd(II), and it was observed that Sn(IV) could displace Cd(II) that had already been adsorbed onto the resin. This indicates that Sn(IV) and Cd(II) were separated without any desorbing agent during the adsorption stage of the column experiment. XPS analysis suggested that the adsorption mechanism involved ion exchange between the chloride ions carried by A336 and the anionic complexes. Overall, this study demonstrates the A336/XAD-7 as cost-effective adsorbent materials for the selective separation of Sn(IV) and Cd(II) from HLLW. In this study, simulated high-level waste liquid was used to carry out experiments, but the effect of ionizing radiation on the stability of Xad-7 resin and A336 was not discussed; in the future, γ -ray or β -ray irradiation experi-

ments will be carried out to verify the application feasibility of the adsorption material in actual scenarios.

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