



Removal of U(VI) from aqueous solution using AO-artificial zeolite

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Abstract

Amidoxime (AO) based adsorbents have been proposed as low cost and efficient adsorbent for U(VI). Herein, we present a simple strategy for preparing AO modified artificial zeolite. The composition and morphology of the materials have been confirmed via XRD, FT-IR, TGA and SEM. The AO-artificial zeolite was synthesised as an efficient adsorbent for adsorbing U(VI). The critical factors affecting U(VI) adsorption from aqueous solution were exploited, such as pH, contact time, temperature and adsorbent dosage. This study reveals AO-artificial zeolite, along with a low-cost, environmentally friendly and facile synthesis, can be regarded as a promising material for uranium-containing wastewater treatment.

Keywords U(VI) · Artificial zeolite · Adsorption · Amidoxime based adsorbent

Introduction

With the rapid development of human modernization, nuclear power is developed vigorously because of the low greenhouse gas emissions and high energy density [1]. Uranium is the most major fuel for the generation of electricity in the nuclear power industry [2]. However, uranium also has chemical and radioactive toxicity, on condition that a large quantity of uranium discharged into our environment, it would be accumulated in human beings through the food chain and lead to health problems [3, 4]. Therefore, numerous methods have been used to removal of U(VI) from aqueous solutions, such as extraction [5, 6], coprecipitation [7], flotation [8], membrane dialysis [9], adsorption [10, 11] and so on. Among these methods, adsorption has attracted much attention due to its easy operation and high efficiency.

Recently, various kinds of adsorbents, such as amine [12], carboxylates [13], carbon materials [14], polymers [15, 16], magnetic materials [17] and metal organic frame works [18], have been developed for the removal of U(VI) from

aqueous solution. For most adsorbents, however, the defect of disperse in aqueous during adsorption process restrict their further industrial applications [19]. Thus, adsorbents suitable for commercialized need to be developed for uranium adsorption. Artificial zeolite has attracted considerable attention because of its regular mesoporous pore structure and chemical and physical stabilities. Nevertheless, only a few functional groups can provide the adsorption active sites for uranium. Among them, amidoxime (AO) group has acidic oxime and amino groups, and the presence of the functional groups shown great potential to form complexes with U(VI) [20, 21]. Thus, it is important to modify artificial zeolite with AO group for U(VI) adsorption.

Hence, the AO functionalized artificial zeolite are prepared. The X-ray diffraction (XRD), fourier transform infrared spectra (FT-IR), scanning electron microscopy (SEM), N₂ adsorption–desorption, and thermogravimetric analysis (TGA) are used to characterize the properties of AO-artificial zeolite. Additionally, the effects of water chemistry on the adsorption of U(VI) are investigated. It is expected that this research will provide a potential adsorbent for the removal of U(VI) from aqueous solutions in real applications.

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Materials and methods

Materials

All the chemicals are analytical grade and used without any further purification. U(VI) stock solution is made from $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (A.R. grade). Arsenazo-III (Aladdin, China) solution is prepared by dissolving 1 g Arsenazo-III in 1000 mL of deionized water. Artificial zeolite particle is purchased from Tianjin Fuchen Chemicals, China. Polyacrylonitrile power is purchased from Daqing Petrochemical Acrylic Plant, China. Dimethyl sulfoxide (DMSO) and polyvinylpyrrolidone (PVP) are purchased from Shanghai Houcheng Chemicals.

Adsorbent preparation

4.0 g of polyacrylonitrile power is dissolved by 200 ml of dimethyl sulfoxide (DMSO). After stirring for 1 d, 8.0 g of hydroxylamine hydrochloride is added. The mixture is stirred for 5 d at 25 °C. 2 g of polyvinylpyrrolidone (PVP) and artificial zeolite are dispersed into the mixture. Then the homogeneous solution is stirred for 1 d. The AO-artificial zeolite product is washed with ethyl alcohol and deionized water 3 times and dried under vacuum at 45 °C for 24 h [22].

Batch experiment

The batch experiments are conducted in a series of 50 mL polyethylene centrifuge tubes. AO-artificial zeolite, NaNO_3 solution, uranium stock solution and deionized water are added to the centrifuge tubes. HNO_3 or NaOH solution is employed to adjust pH. The centrifuge tubes are shaken in a constant temperature oscillator for 3 days, after that the centrifuge tubes are centrifuged at 10000 r/min for 30 min. Supernatant is taken out to confirm the adsorption capacity of uranium using the Arsenazo-III spectrophotometric method at 652 nm. The adsorption efficiency and capacity of U(VI) on AO-artificial zeolite is calculated as follows:

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

$$q_e = \frac{(C_0 - C_e)}{m} \times V \quad (2)$$

where C_0 and C_e are the initial and final concentration of U(VI), respectively; m is the mass of the adsorbent.

All of the experimental data were the averages of duplicate or triplicate experiments, and the relative errors of the data were less than 5%.

Characterization

TGA of the samples are recorded on a TA Q50 DTA/TGA apparatus in the range of 25 to 700 °C (USA). XRD measurements are obtained on a Philips X'Pert Pro Analytical diffractometer equipped with Cu $K\alpha$ radiation (scan rate 0.02°/s, $10^\circ \leq 2\theta \leq 90^\circ$). FT-IR are obtained using a Thermo Nicolet 6700 FT-IR spectrometer (USA) using the KBr pressed disk method. SEM is obtained using an S-4800 (Hitachi) microscope.

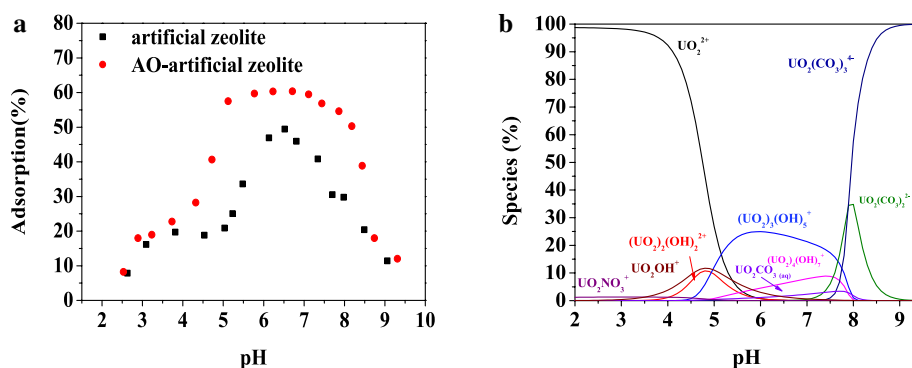
Results and discussion

Adsorption behavior studies

Effect of pH

The species of U(VI) and the surface charge of adsorbent in aqueous solutions can be affected by pH, thus the effect of pH is significant for U(VI) adsorption [23]. As shown in Fig. 1, it is clear that the amount of U(VI) adsorbed on artificial zeolite and AO-artificial zeolite increased with pH gradually changes from 2.5 to 6.0, whereas the adsorption percentage of U(VI) onto artificial zeolite and AO-artificial zeolite decrease with pH further increasing. Furthermore, the adsorption percentage of U(VI) onto AO-artificial zeolite

Fig. 1 Effect of pH on the U(VI) adsorption onto AO-artificial zeolite **a**; relative species distribution of U(VI) in the presence of CO_2 **b**. $m/V = 3.6$ g/L, $I = 0.01$ M NaNO_3 , $T = 298$ K, $[\text{U(VI)}] = 20$ mg/L, $P_{\text{CO}_2} = 3.8 \times 10^{-4}$ atm



is remarkably higher than that onto artificial zeolite. There are many unbonded N and O protons in AO-groups, and these atoms may be combine with U(VI) to form chelates, the experimental result suggesting that the formation of the chelate increased the adsorption of U(VI) [24]. At lower pH, the protonation of AO-artificial zeolite and the competition between H^+ and U(VI) limited the adsorption of U(VI). With the pH increase, the protonation and the competition become weakened, it is easy to adsorb U(VI). Fig. 1b describes the species distribution of U(VI) depend on the pH values. At $pH < 5.0$, UO_2^{2+} was the main existing species of U(VI). $5.0 < pH < 7.0$, the dominant species was $(UO_2)_3(OH)_5^+$. A further increase in pH value will generate negatively charged species, including $UO_2(CO_3)_2^{2-}$ and $UO_2(CO_3)_3^{4-}$, the adsorbent is not easy to adsorb negatively charged ions, the adsorption capacity declines [25].

Effect of Contact time and adsorption kinetics

Equilibration time is one of the parameters that could reflect the adsorption efficiency. The result of the effect of contact time on the adsorption of U(VI) by AO-artificial zeolite is shown in Fig. 2. The adsorption percentage of U(VI) shows an upward tendency with the contact time goes on, and reaches the maximum value within 72 h. Three different kinetic models are employed to evaluate the rate controlling process. The descriptions of pseudo-first-order, pseudo-second-order and Weber–Morris model are shown in Eqs. (3), (4) and (5), respectively:

$$q_t = q_e(1 - e^{-K_1t}) \quad (3)$$

$$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \quad (4)$$

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

where q_e and q_t are the adsorption capacity at equilibrium time and time t , respectively. k_1 and k_2 are the equilibrium rate constant of pseudo-first-order adsorption and pseudo-second-order, respectively. K ($mg \cdot g^{-1} \cdot min^{1/2}$) and C are the rate constant and adsorption constants of the Weber–Morris modal. The fitted results are summarized in Tables 1 and 2. By comparing the correlation coefficient, the adsorption of U(VI) onto AO-artificial zeolite can be fitted by pseudo-second-order kinetic model. This result is consistent with many other adsorbents [26, 27], the chemical complexing reaction dominates the adsorption process of U(VI) [28].

The Weber–Morris model explains that the adsorption process includes three steps (Fig. 2d). The first steep slope

Table 1 Kinetic parameters of pseudo-first-order and pseudo-second-order models

	Pseudo-first-order model	Pseudo-second-order model	
k_1 (g/mg·min)	35.4407	k_2 (g/mg·min)	0.0175
q_e (mg/g)	3.2751	q_e (mg/g)	6.9623
R^2	0.6889	R^2	0.9830

Fig. 2 a Effect of contact time on the adsorption of U(VI) onto AO-artificial zeolite; b the fitting plot of the pseudo-first-order equation; c the fitting plot of the pseudo-second-order equation; d Weber–Morris model. $pH = 5.00$, $m/V = 3.6$ g/L, $I = 0.01$ M $NaNO_3$, $T = 298$ K, $[U(VI)] = 20$ mg/L

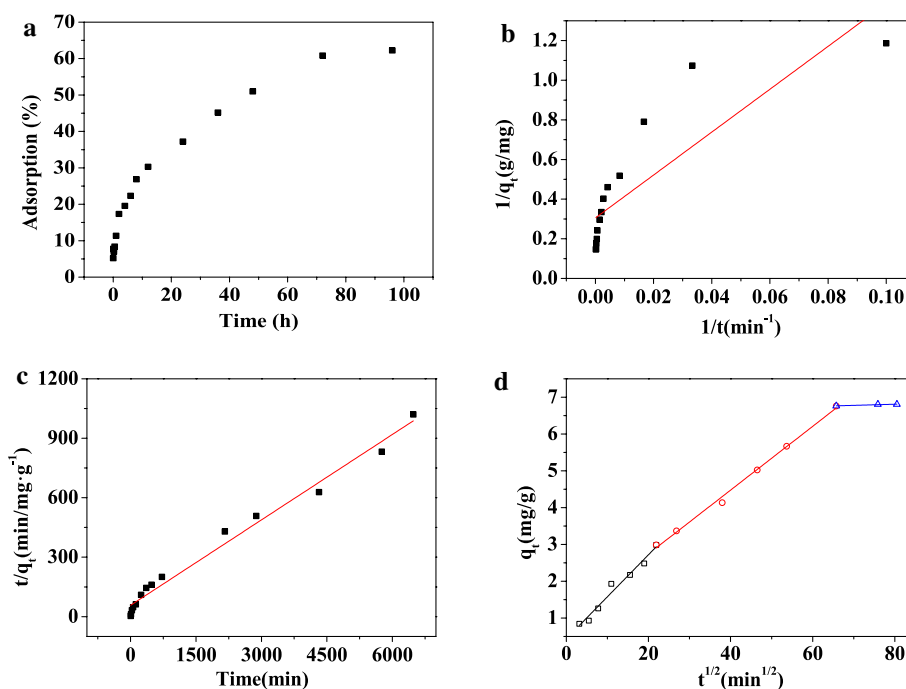
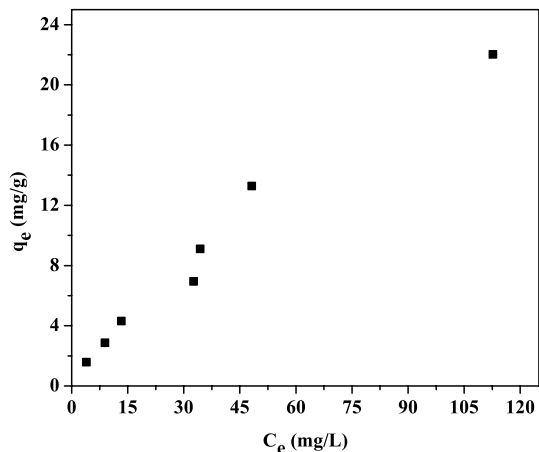


Table 2 Kinetic parameters of the Weber–Morris model

First stage		Second stage		Third stage	
$k_{ad1}(\text{g}/\text{mg} \cdot \text{min})$	0.1141	$k_{ad2}(\text{g}/\text{mg} \cdot \text{min})$	0.0866	$k_{ad3}(\text{g}/\text{mg} \cdot \text{min})$	0.0031
$C_1 (\mu\text{g}/\text{g})$	0.4371	$C_2 (\mu\text{g}/\text{g})$	1.0107	$C_3 (\mu\text{g}/\text{g})$	6.5601
R_1^2	0.9704	R_2^2	0.9955	R_3^2	0.8145

**Fig. 3** Effect of initial concentration of U(VI) on the adsorption on AO-artificial zeolite. pH = 5.00, $m/V = 3.6 \text{ g/L}$, $I = 0.01 \text{ M NaNO}_3$, $T = 298 \text{ K}$

represents the external transfer step. The next stage is the intra-particle diffusion process, and the final step means the final equilibrium step [29]. The results suggest that the second stage is the rate limiting step in the process of U(VI) adsorption onto AO-artificial zeolite.

Effect of initial U(VI) concentration

The initial concentration of U(VI) provides driving forces to proceed the adsorption process [30]. Figure 3 shows the influence of the initial U(VI) concentration on the adsorption of AO-artificial zeolite for U(VI). To better understand the adsorption processes of AO-artificial zeolite to U(VI), The adsorption data were fitted by three isotherms. The Langmuir isotherm supposes that adsorption is a monolayer adsorption, and can be expressed [31]:

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

The Freundlich isotherm is given as [32]:

$$\ln q_e = \ln K_F + \frac{1}{m} \ln C_e \quad (7)$$

where K_F and n are the Freundlich constant. The Freundlich isotherm model assumes that the adsorption site is heterogeneous. The Dubinin–Radushkevich (D–R) isotherm assumes that the adsorption energy of adsorbent is nonuniform [33]. The model can be expressed [34]:

$$\ln q_e = \ln q_k - B\eta^2 \quad (8)$$

$$\eta = RT \ln \left(1 + \frac{1}{C_e} \right) \quad (9)$$

$$E = \frac{1}{\sqrt{2B}} \quad (10)$$

where $B (\text{mol}^2/\text{kJ}^2)$ is the D–R constant, $q_k (\text{mol}/\text{g})$ is the D–R adsorption capacity, ε is the Polanyi potential, T is the temperature (K), and $E (\text{kJ}/\text{mol})$ is the free energy change [35].

The calculated linear analysis results are summarized in Table 3. The adsorption of U(VI) onto the AO-artificial zeolite obeys the Freundlich isotherm model. Therefore, the adsorption process of AO-artificial zeolite for U(VI) was considered heterogeneous and a multilayer process.

From the fitting results, the free energy change of D–R isotherm model was about 8.5 kJ/mol, indicating that the adsorption of U(VI) onto AO-artificial zeolite is mainly

Table 3 Adsorption isotherm parameters of U(VI) adsorption onto AO-artificial zeolite

Model	Freundlich model		Langmuir model		D–R model	
Parameter	n	1.2561	q_{max} (mg/g)	47.7783	B (mol^2/kJ^2)	0.0069
	K_F (L/g)	0.5246	K_L (L/mg)	7.1364×10^{-3}	q_k (mg/g)	1.0467×10^{-3}
	R^2	0.9844	R^2	0.6325	E (kJ/mol)	8.5126
					R^2	0.5475

chemical adsorption, the result is in agreement with other experimental results [36, 37].

Effect of adsorbent dosage

Adsorbent dosage determines the effective number of binding sites, so it is an important parameter in the adsorption process. The effect of adsorbent dose on U(VI) adsorption was investigated using dose range from 0.02 to 5 g/L of AO-artificial zeolite.

The data was represented in Fig. 4, the adsorption capacity of U(VI) increased from 6 to 70% at adsorbent dose from 0.02 to 5 g/L. This may be due to the increase of the available adsorption sites numbers on increasing AO-artificial zeolite dosage. However, as adsorbent dosage increases the adsorption capacity decreased. There are fewer adsorption sites on the surface of AO-artificial zeolite at lower concentration. The aggregation of particles takes place with the increase of adsorbent dosage, as a result the adsorption capacity q_e decreases. This phenomenon is also present in other adsorption systems of different adsorbents [38–40].

Characterization analysis

XRD analysis

XRD patterns of artificial zeolite and AO-artificial zeolite are shown in Fig. 5. As evident, the XRD patterns of AO-artificial zeolite do not show obvious fresh peaks

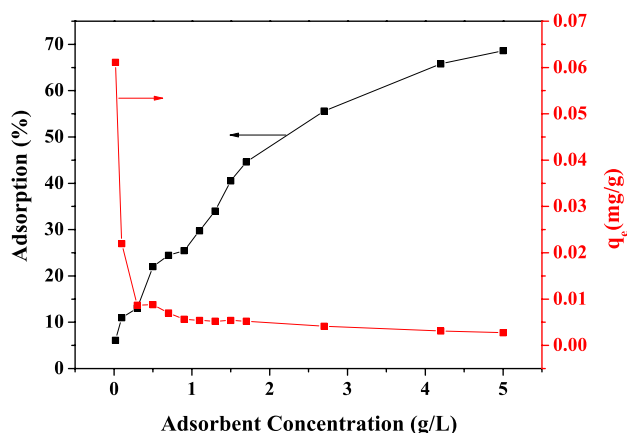


Fig. 4 Effect of adsorbent dosage on the adsorption of U(VI) onto AO-artificial zeolite. pH = 5.00, $I = 0.01$ M NaNO_3 , $T = 298$ K, $[\text{U(VI)}] = 20$ mg/L

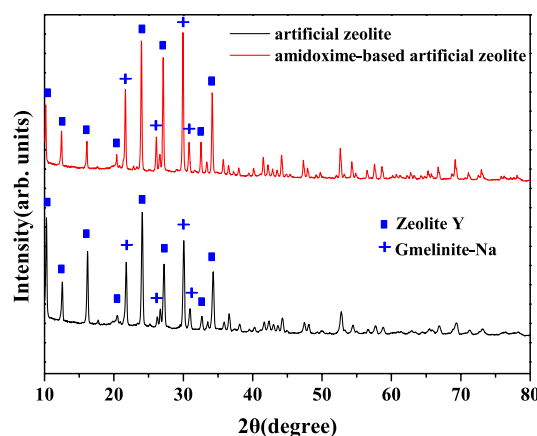


Fig. 5 XRD diffraction patterns of artificial zeolite and AO-artificial zeolite

compared with artificial zeolite, indicate that the AO-artificial zeolite maintain the original structures of artificial zeolite.

N_2 adsorption–desorption isotherm

The N_2 adsorption–desorption isotherms of artificial zeolite and AO-artificial zeolite are shown in Fig. 6. The Brunauer–Emmett–Teller (BET) surface area of artificial zeolite and AO-artificial zeolite are calculated to be 31.91 and 24.37 m^2/g , respectively. And the pore diameters are calculated to be 11.74 and 9.77 nm, respectively. This suggests that the pores of AO-artificial zeolite are partially blocked than that of artificial zeolite in the process of modification. Whereas the functional groups in AO-artificial zeolite are in favour of binding U(VI) to form chelates, which causes the AO-artificial zeolite shows better adsorption properties than artificial zeolite.

Thermogravimetric analysis

Thermogravimetric analysis is used to study the thermal behaviors of artificial zeolite and AO-artificial zeolite. As can be seen from Fig. 7, the first weight loss stage from 25 to 200 °C attributes to the elimination of physically adsorbed moisture on the surface of the particles [41]. The second weight loss occurs from 200 to 550 °C, which can be assigned to thermal decomposition of organic components. As for U(VI)-loaded AO-artificial zeolite, the weight loss observed in the range from about 250 to 600 °C may be assigned to the thermal decomposition of U(VI). Meanwhile, the weight residuals of artificial zeolite after thermal analysis is similar to that of AO-artificial

Fig. 6 Nitrogen adsorption–desorption isotherm of artificial zeolite (a) and AO-artificial zeolite (b)

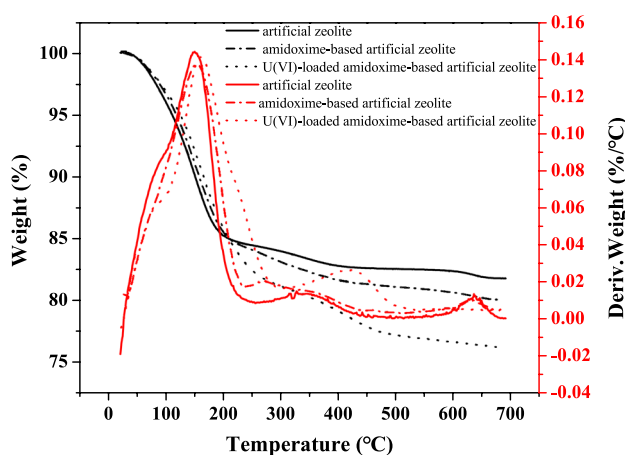
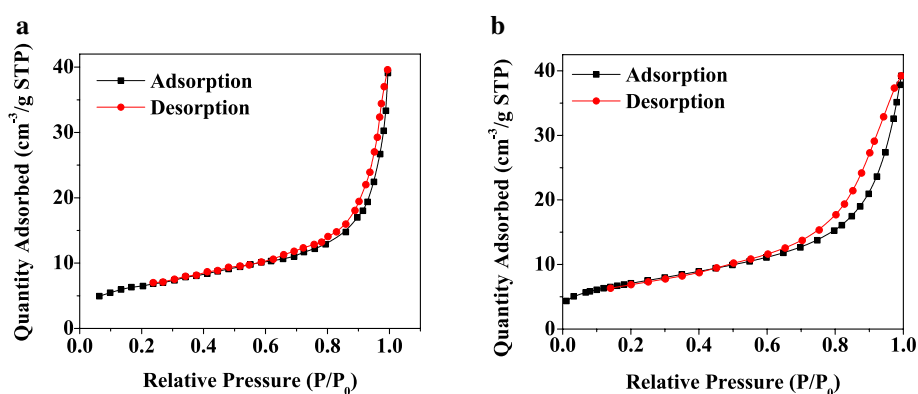


Fig. 7 Thermogravimetric analysis of artificial zeolite and AO-artificial zeolite

zeolite, the thermal stability is basically unchanged during the incorporation of AO functional groups process [42].

FT-IR analysis

FT-IR analysis is further conducted to verify the conversion of functional group in the reaction process. As shown in Fig. 8, three characteristic peaks suggest that the functional groups are grafted onto artificial zeolite successfully. The peaks at 1657 cm^{-1} could be ascribed to the stretching vibration of the $\text{C}=\text{N}$, the peaks at 1386 cm^{-1} could be ascribed to the stretching vibration of NH_2 , the peaks at 945 cm^{-1} could be assigned to the bending vibration of $\text{N}-\text{O}$. This suggests that the amidoxime group ($\text{HON}=\text{C}-\text{NH}_2$) is covered the surface of AO-artificial zeolite. In the U(VI)-loaded AO-artificial zeolite, the peak at 750 cm^{-1} confirms the binding of uranium to AO-modified zeolite. The changes in peak positions and intensity around $550\text{--}1000\text{ cm}^{-1}$ region can be assigned to asymmetric stretching vibration of uranyl ion and stretching vibrations of weakly bonded oxygen ligands with uranium [43, 44].

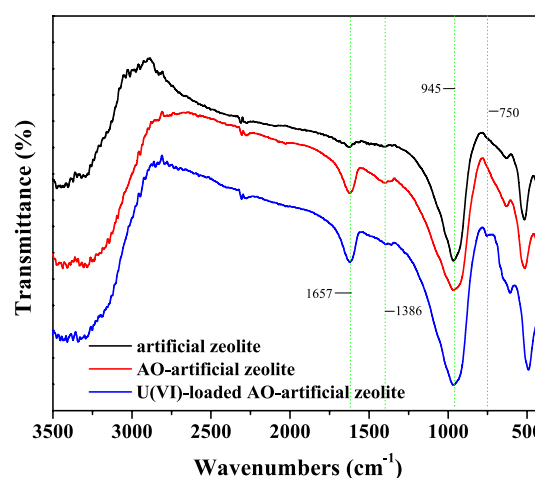


Fig. 8 FT-IR spectra of artificial zeolite, AO-artificial zeolite and U(VI)-loaded artificial zeolite

SEM analysis

The surface morphologies changes before and after artificial zeolite modified by AO are analyzed via SEM. As shown in Fig. 9a–d, after modification, the surface of AO-artificial is much rougher than artificial zeolite. Fig. 9e and f are the SEM image of AO-artificial zeolite after U(VI) adsorption. The surface of AO-artificial zeolite become rougher, a remarkable amount of particles are disposed on AO-artificial zeolite surface, which demonstrated that U(VI) is adsorbed onto the surface of AO-artificial zeolite.

Comparison with other adsorbents

The adsorption capacities of the composites for uranium were compared with other adsorbent materials reported from literature in Table 4. The comparison showed that the adsorption capacity of AO-artificial zeolite is not the best,

Fig. 9 SEM images of **a** and **b** artificial zeolite; **c** and **d** AO-artificial zeolite; **e** and **f** U(VI)-loaded AO-artificial zeolite

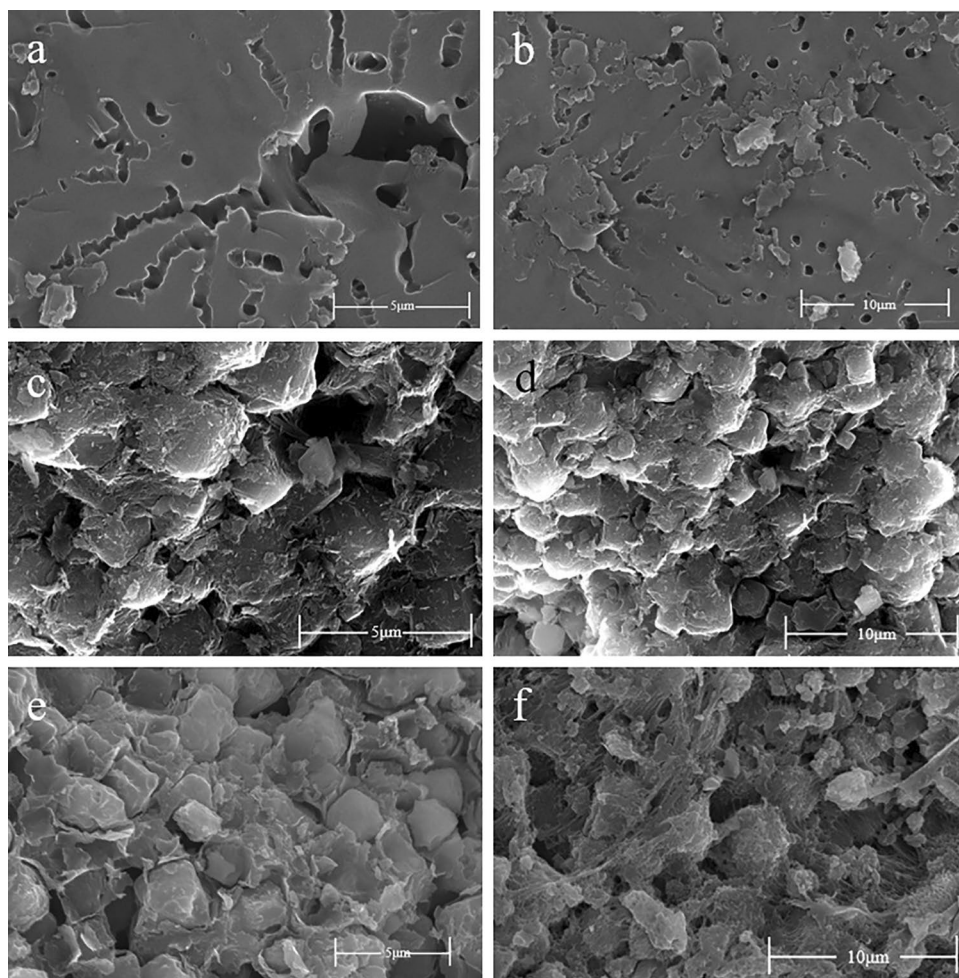


Table 4 Comparison with different adsorbents from literature

Adsorbent	Adsorption capacity (mg/g)	References
Methacryloxypropyltrimethoxysilane coated magnetic	1.1	[45]
Modified magnetic Fe ₃ O ₄	12.33	[46]
Used tires	10.1	[47]
Natural clinoptilolite zeolite	2.12	[48]
CuO/NaX nanocomposite	6.77	[49]
β -cyclodextrin functionalized silica gel	6.45	[50]
Magnetite nanoparticles	4.76	[51]
Tendurek volcanic tuff	2.38	[52]
MnO coated sand	4.76	[53]
Amidoximated polyglycidyl methacrylate microbeads	1.07	[54]
AO-artificial zeolite	6.96	This work

nonetheless it is a relatively inexpensive material in all of them. It owns high cost performance, and can be used as an alternative material for commercial applications.

Conclusions

In this study, the adsorption of U(VI) onto AO-artificial zeolite is investigated. The consequence of adsorption indicated that AO-artificial zeolite had high removal

amount towards U(VI), which is much higher than that of artificial zeolite, adsorption capacity of U(VI) is significantly enhanced. Furthermore, AO-artificial zeolite overcome the defect of disperse in aqueous during adsorption process, which could offer a promising opportunities for further improvements in the removal of U(VI) from aqueous solution.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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