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Original Article

Removal of Uranium from Aqueous Solution by Alginate Beads

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ABSTRACT

The adsorption of uranium (VI) by calcium alginate beads was examined by batch experiments. The effects of environmental conditions on U (VI) adsorption were studied, including contact time, pH, initial concentration of U (VI), and temperature. The alginate beads were characterized by using scanning electron microscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, and Fourier transform infrared spectroscopy. Fourier transform infrared spectra indicated that hydroxyl and alkoxy groups are present at the surface of the beads. The experimental results showed that the adsorption of U (VI) by alginate beads was strongly dependent on pH, the adsorption increased at pH 3–7, then decreased at pH 7–9. The adsorption reached equilibrium within 2 minutes. The adsorption kinetics of U (VI) onto alginate beads can be described by a pseudo first-order kinetic model. The adsorption isotherm can be described by the Redlich-Peterson model, and the maximum adsorption capacity was 237.15 mg/g. The sorption process is spontaneous and has an exothermic reaction.

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1. Introduction

Uranium can enter into the environment from uranium mining, manufacture, and application, which will cause hazards to the ecological environment and human health due to chemical toxicity and radioactivity [1]. A variety of treatment technologies, including physical, chemical, and biological methods have been used to remove uranium from an aqueous solution [2]. Adsorption is a commonly used method to remove radionuclides from wastewater due to its low cost, high efficiency, and abundant adsorbents [3–6].

Biosorption, using biopolymers as adsorbent, is considered to be a potential process due to its high selectivity, high efficiency in low concentrations, wide-ranging operating pH and temperature, facile recycling, or regeneration. Different kinds of biopolymers, including natural and synthetic, have been reported for adsorption of uranium, such as salt alginate, agar, chitosan, polysulfone, polyacrylamide, polyurethane, silica, polyvinyl alcohol, etc. [7]. Alginate with the crosslinking agent CaCl₂ has been commonly used due to its high biocompatibility and simple gelation [7].

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Biopolymeric materials and microbial cells can accumulate metal ions by precipitating or binding the metal ions owing to the presence of carboxyl, hydroxyl, amino, and other negatively charged sites. Microorganisms (bacteria, fungi, and algae) can adsorb metal ions and are regarded as an environmentally-friendly adsorbents for their high adsorption capacity and abundantly available active sites [7–10]. The polysaccharides are biopolymers of monosaccharides, which have many advantages such as hydrophilicity, biocompatibility, and biodegradability. Polysaccharides such as agarose [11] and chitosan [12,13] are excellent biosorbents for uranium adsorption, because hydroxyl or amino groups on the polymer chains can act as chelation sites.

Alginic acid or alginate is a linear polysaccharide existing in brown seaweeds. The alginic acid or alginate with monovalent ions (alkali metals and ammonium) is soluble, which limits its application for removing heavy metals and radionuclides from an aqueous solution. Soluble alginate may be converted into an insoluble hydrogel by an ion-exchange reaction with multivalent metal ions. The insoluble alginate can be used as the immobilizing material for entrapment of biomass, including agricultural wastes [14], *Penicillium citrinum* [15], humic acid [16], yeast [17], cellulose [18], and bacteria [19] for the removal of uranium from an aqueous solution.

Calcium alginate beads have been used as adsorbent for the removal of heavy metal ions and radionuclides, for example, for recovery of uranium from aqueous solutions [20]. The maximum sorption capacity of immobilized agricultural waste beads was 17.59 mg U/g at pH = 4 [14]. The maximum sorption capacity of immobilized *P. citrinum* beads was 256 mg U/g when pH = 6, uranium concentration was 50 µg/mL, and contact time was 7 hours [15]. The maximum sorption capacity of calcium alginate microsphere was 400 mg U/g at pH = 4, 25°C with removal efficiency of 91%. The adsorption process is spontaneous, endothermic, and can be well described by the Langmuir isotherm model [20]. The formaldehyde crosslinking can enhance the adsorption capacity of the immobilized yeast by calcium alginate [17,19]. Fourier transform infrared (FTIR) spectroscopy analysis suggested that adsorption mechanism of U (VI) by glutaraldehyde crosslinking humic acid-immobilized sodium alginate porous membrane was U (VI) complexation with hydroxyl groups and ion exchange with carboxyl [16,18].

The objective of this study was to examine the removal of uranium from aqueous solution by calcium alginate beads. The effects of different factors, such as contact time, pH of solution, initial concentration of uranium ions, and temperature on the uranium adsorption were all determined. The results can be applied to evaluate the feasibility of application of calcium alginate beads for the removal of uranium from wastewater.

2. Materials and methods

2.1. Chemicals

All reagents used were analytical graded. All solutions were prepared in pure water. The $\text{UO}_2(\text{NO}_3)_2$ stock solution was

prepared from standard uranium solution [GBW (E) 080173], which was diluted to the required concentrations before being used. Sodium alginate and calcium chloride were purchased from West Long Chemical Co., Ltd (Shantou City, Guangdong Province, China).

2.2. Preparation of the calcium alginate beads

The molecular formula of sodium alginate is $(\text{C}_6\text{H}_7\text{NaO}_6)_x$, and the chemical structure is shown in Fig. 1. The sodium alginate was dissolved in deionized water (w/v, 1%), and dropped into 1% CaCl_2 solution through a 5 mL syringe to get uniform beads. The beads were separated from CaCl_2 solution after hardening for 24 hours, then washed with deionized water several times [19]. After drying at room temperature, the typical size of the calcium alginate beads was about 2.8–3.0 mm in diameter.

2.3. Experimental procedures

Experiments were carried out in a shaker. In a typical procedure, 0.01 g of calcium alginate beads and 9 mL of $\text{UO}_2(\text{NO}_3)_2$ solution was added into polyethylene test tubes to achieve the desired concentrations for different experimental conditions. The pH was adjusted by 0.1M HNO_3 or NaOH . After shaking for a certain time, the mixtures were centrifuged and filtered prior to determining the concentrations of U (VI). The batch adsorption experiments were performed in triplicates, and the average values were used.

2.4. Analytical methods

The concentration of U (VI) in solution was determined by inductive coupled plasma mass spectroscopy (XII, Thermo Electron, Waltham, Massachusetts, USA).

The sorption capacity of U (VI) (q_t , mg/g) by alginate beads was calculated according to the following equation:

$$q_t = \frac{(C_0 - C_t) \times V}{m} \quad (1)$$

where C_0 and C_t (mg/L) is concentration of U (VI) at initial and at time t in the aqueous solution, respectively; V is the volume of the solution (L); and m is the mass of dry state calcium alginate beads (g).

The removal efficiency of U (VI) was calculated by the following equation:

$$R = \frac{(C_0 - C_t)}{C_0} \times 100\% \quad (2)$$

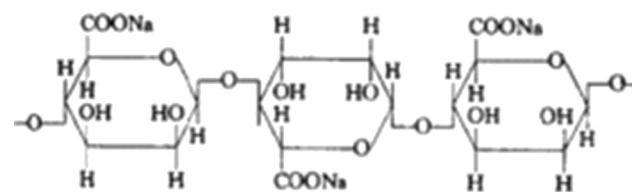


Fig. 1 – The chemical structure of sodium alginate.

2.5. Characterization of alginate beads

Calcium alginate beads were characterized by scanning electron microscopy (FEI Quanta 200; FEI Company, Hillsboro, Oregon, USA), transmission electron microscopy (H-7650B; FEI Company), X-ray photoelectron spectroscopy (XPS, PHI Quantera SXM; Ulvac-PHI Inc., Chigasaki, Kanagawa, Japan) and FTIR spectroscopy (Perkin Elmer GX; FOCAS Institute, Dublin, Ireland). FTIR analysis was performed using a KBr beam splitter with 4000–400 cm^{-1} wave number with a 4 cm^{-1} resolution.

3. Results and discussion

3.1. Characterization of alginate beads

The microstructures of alginate beads were observed by scanning electron microscopy and transmission electron microscopy and are shown in Figs. 2A and 2B. It can be seen from Fig. 2A that the wet alginate beads dispersed in solution with globular appearance. Fig. 2B indicates that the dry alginate beads had a rough surface and some pores existing in section. The energy-dispersive X-ray spectroscopy (EDS) spectrum of Fig. 3B confirmed that U (VI) was adsorbed by alginate beads based on the presence of the characteristic peak of U (VI).

Fig. 4 presents the XPS and FTIR spectra of alginate beads. The C 1s XPS spectrum of alginate beads shows three peaks in Fig. 4A, at 286.84 eV (18.92%), 285.32 eV(42.77%), and 283.64 eV(38.31%), which correspond to -C-O-, -C-C-, and -C \equiv C-bonds, respectively. As can be seen from Fig. 4B, there was a broad peak at \sim 3,400 cm^{-1} , which is a characteristic of the O-H or N-H due to stretch of hydroxyl or amine group. The two characteristic bands at 1,270 cm^{-1} and 1,425 cm^{-1} are attributed to hydroxyl groups. The peak at 1,047 cm^{-1} corresponds to alkoxy groups. The peak at 2,171 cm^{-1} corresponds to the

stretch of C \equiv C. The peak at 1,622 cm^{-1} was assigned to the vibration of adsorbed water on alginate beads. Therefore, alkynyl (-C \equiv C-), hydroxyl (-OH), and alkoxy (-C-O-C-) groups may be present at the surface of the alginate beads. The position of the peaks shifted after adsorption of U (VI), which can be seen in Fig. 4B. The results indicated that alkynyl, hydroxyl, and alkoxy groups participated in the adsorption process and provided unshared pair electrons played important effect on adsorption of U (VI).

3.2. Effect of contact time

Fig. 5 shows the effect of the contact time on U (VI) adsorption onto alginate beads. The adsorption capacity of U (VI) by alginate beads increased sharply at the first 2 minutes, the equilibrium adsorption capacity (q_e) was 10.98 mg U/g, and the removal efficiency was 65.17%. In the following experiments, 12 hours was selected to guarantee the equilibrium of U (VI) sorption by alginate beads.

To analyze the kinetic behaviors of U (VI) sorption onto the alginate beads, pseudo first-order, pseudo second-order, the Elovich model, and the intraparticle diffusion kinetic models were used to fit the experimental data [14]. The kinetic models used in this study are as follows:

$$\ln\left(1 - \frac{q}{q_e}\right) = -k_1 t \quad (3)$$

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

$$q_t = a + b \ln t \quad (5)$$

$$q_t = k_F t^{0.5} \quad (6)$$

where q_e and q_t (mg/g) are the adsorption capacities at equilibrium and at time t , respectively; k_1 (min^{-1}) and k_2 [g/(mg/

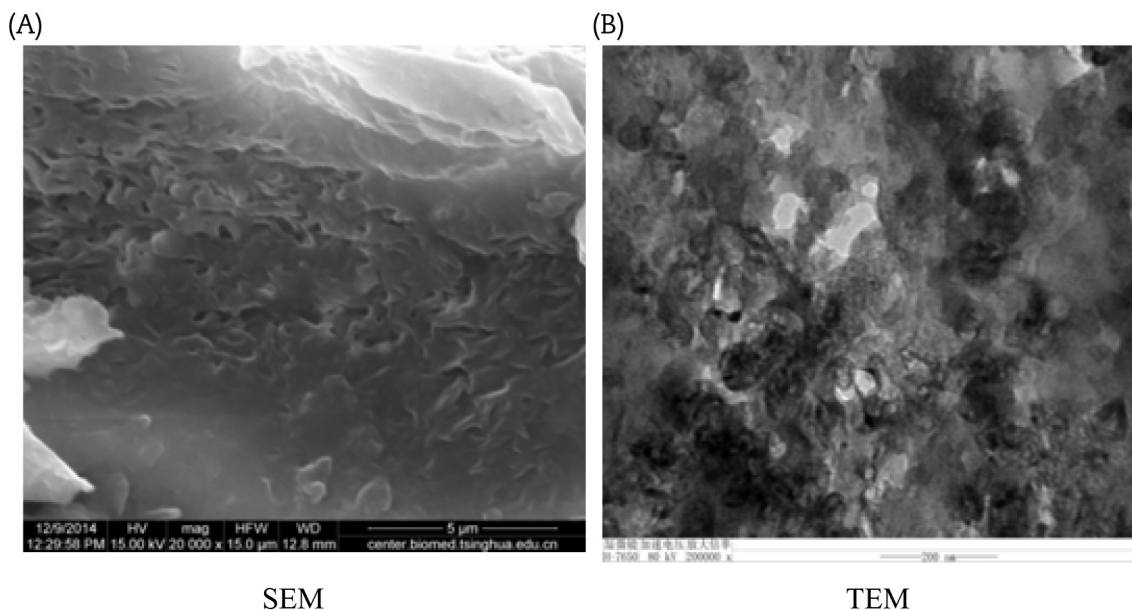


Fig. 2 – Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images of alginate beads.

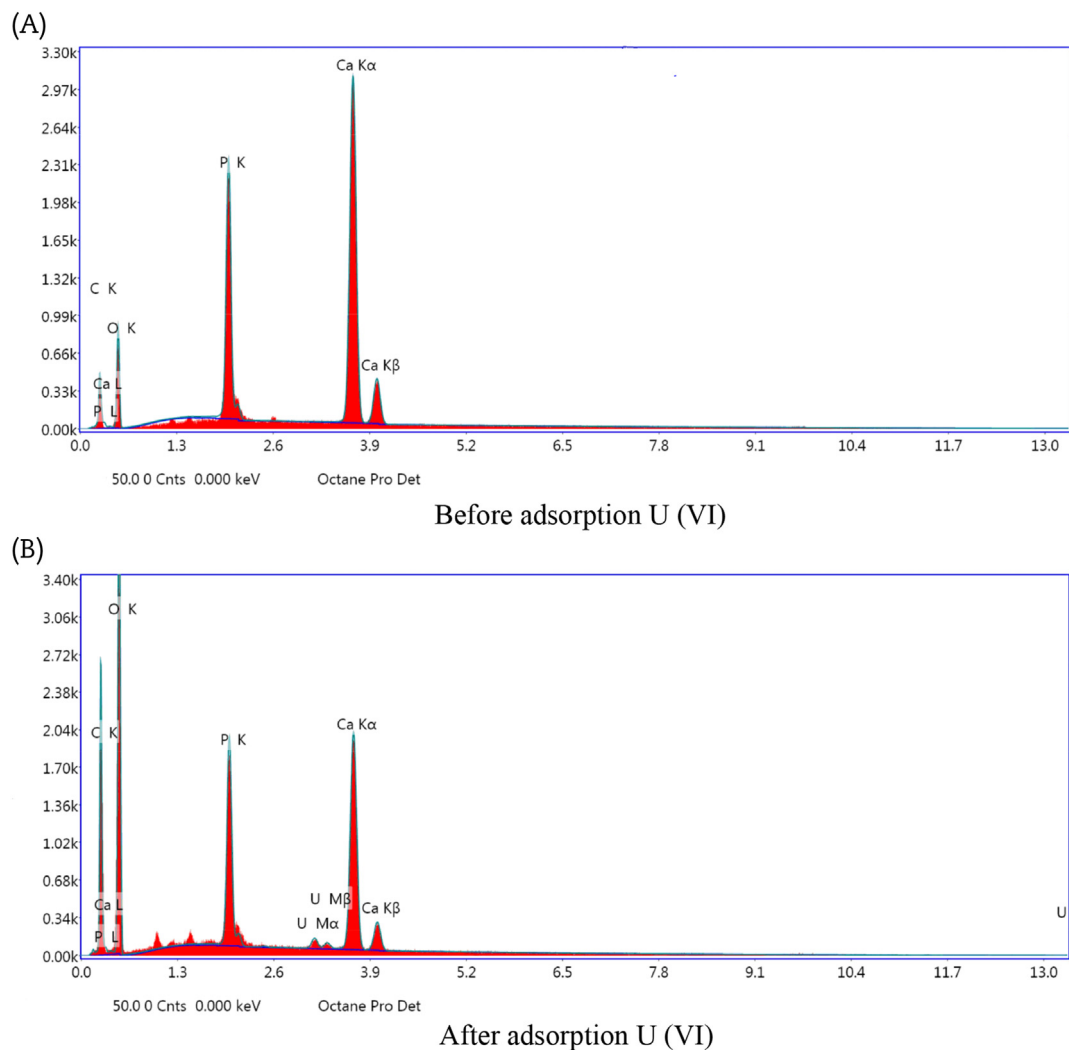


Fig. 3 – Energy-dispersive X-ray spectroscopy (EDS) spectrum of alginate beads.

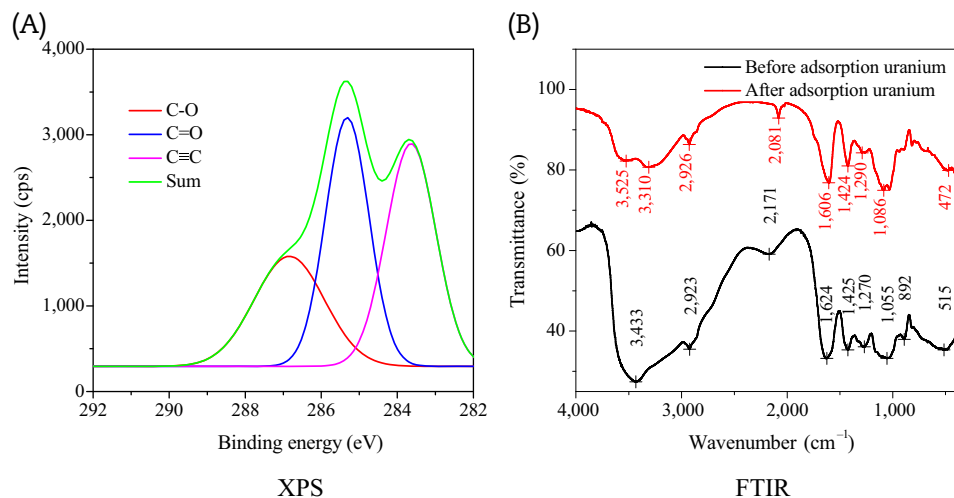


Fig. 4 – X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared (FTIR) spectra of alginate beads.

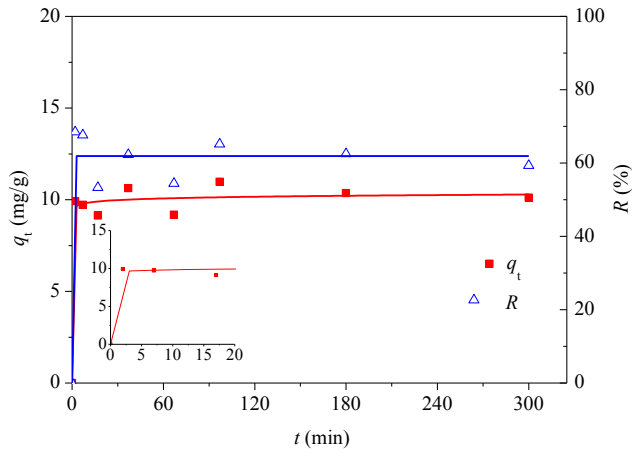


Fig. 5 – Effect of contact time on U (VI) adsorption, $C_0[\text{U}] = 20 \text{ mg/L}$, $\text{pH} = 5.0$, $m/V = 1.1 \text{ g/L}$, and $T = 25^\circ\text{C}$.

min)] are the rate constants of pseudo first-order model and pseudo second-order model, respectively, a and b are the rate constants of the Elovich model, and k_F is the rate constant of intraparticle diffusion model.

As can be seen from Fig. 6, the pseudo first-order model was more appropriate. The theoretical value (q_e) for the pseudo first-order model (10.02 mg U/g) was consistent with the experimental value (q_e) (10.98 mg U/g). The results suggested that the adsorption process was governed by chemisorption and activation energy changed obviously during the adsorption process.

3.3. Effect of pH

The value of pH is an important parameter affecting the bio-adsorption process. In this study, the pH value was adjusted to a range of 3–9. The adsorption of U (VI) onto alginate beads as a function of pH is shown in Fig. 7.

The adsorption of U (VI) increased at pH 3–7, then decreased at pH 7–9. The maximum removal efficiency was 82.24% at

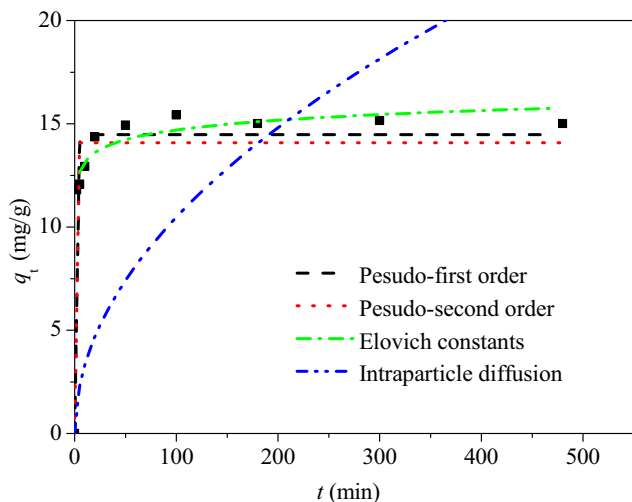


Fig. 6 – Kinetic model of U (VI) adsorption.

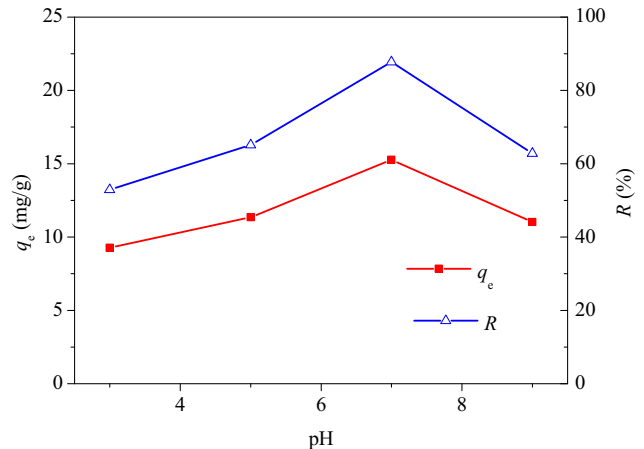


Fig. 7 – Effect of pH on U (VI) adsorption, $C_0[\text{U}] = 20 \text{ mg/L}$, $t = 12 \text{ h}$, $m/V = 1.1 \text{ g/L}$, and $T = 25^\circ\text{C}$.

$\text{pH} = 7$. The variation of U (VI) adsorption by alginate beads with pH may be ascribed to the surface charges and the distributed species of U (VI) in solution. The surface of alginate beads became negative at $\text{pH} > \text{pH}_{\text{pzc}}$ (pH of zero surface charge) because of chemical deprotonation reaction. The species of U (VI) are mostly influenced by pH solution. Free uranyl ions (UO_2^{2+}) are the dominant species at $\text{pH} < 5$. In the range of pH at 5.0–7.0, U (VI) hydrolysis complexes (UO_2OH^+) and multinuclear hydroxide complexes ($[(\text{UO}_2)_3(\text{OH})_5]^{+}$) are the dominant species. At $\text{pH} > 7.0$, carbonate uranyl ions ($[\text{UO}_2\text{CO}_3]$, $(\text{UO}_2)(\text{CO}_3)_2^{2-}$, $(\text{UO}_2)(\text{CO}_3)_3^{4-}$) are the dominant species [21]. The charge of U (VI) species changed from positive to negative at $\text{pH} > 7$. Therefore, the electrostatic repulsion of U (VI) and alginate beads becomes strong with increasing pH at $\text{pH} > \text{pH}_{\text{zpc}}$, leading to the decrease of U (VI) adsorption onto alginate beads.

3.4. Effect of concentration

The variation in adsorption behavior of alginate beads with initial U (VI) concentration is shown in Fig. 8. It can be seen

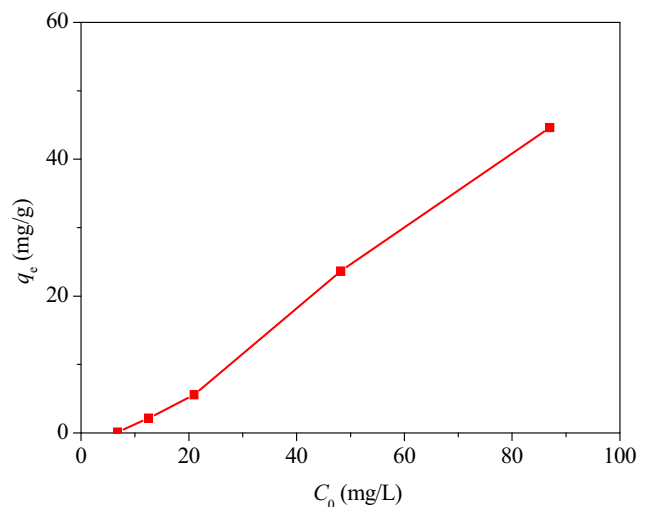


Fig. 8 – Effect of initial concentration on U (VI) adsorption, $\text{pH} = 5.0$, $t = 12 \text{ h}$, $m/V = 1.1 \text{ g/L}$, and $T = 25^\circ\text{C}$.

that the adsorption capacity of U (VI) onto alginate beads increased with increasing concentrations of U (VI) at 6.76–78.0 mg/L, because the increase in the initial concentration provides a larger driving force to overcome the whole mass transfer resistance between the solid and liquid phases. The results may lead to more collisions between U (VI) ions and active sites on the alginate beads, thus resulting in higher adsorption capacity.

For adsorption isotherm modeling, herein, the Langmuir, Freundlich, Temkin, the Redlich-Peterson, Slips, and D-R models analyzed the adsorption of U (VI) ions onto alginate beads. The general forms are expressed as follows [22]:

$$q_e = \frac{q_m k_L C_e}{1 + k_L C_e} \quad (7)$$

$$q_e = k_F C_e^{1/n} \quad (8)$$

$$q_e = A + B \ln C_e \quad (9)$$

$$q_e = \frac{k_{RP} C_e}{1 + \alpha_{RP} C_e^\beta} \quad (10)$$

$$q_e = \frac{k_s C_e^{1/b}}{1 + \alpha_s C_e^{1/b}} \quad (11)$$

$$q_e = q_m \exp\left\{-k \left[RT \ln\left(1 + \frac{1}{C_e}\right)\right]^2\right\}, E = \frac{1}{(2k)^{1/2}} \quad (12)$$

where C_e (mg/L) indicates the equilibrium concentration of U (VI) in an aqueous solution, q_e (mg/g) is the adsorption capacity after sorption equilibrium, q_m (mg/g) is the maximum adsorption capacity, k_L (L/mg) is the most principal coefficient of the Langmuir model involved with Langmuir sorption constants, and k_F [(mg/g)/(mg/L)^{1/n}] and n are parameters of Freundlich model involved with Freundlich affinity coefficient and intensity of dependence of sorption concerning equilibrium concentration, respectively. A and B are parameters of Temkin model involved with Temkin constants and sorption energy; k_{RP} (L/g), α_{RP} (L/mg)^β and β are parameters of the Redlich-Peterson model involved with adsorption capacity, energy of adsorption, and empirical exponent, respectively; k_s (L^bmg^{1-b}/g), α_s (L/mg)^b, and b are parameters of Slips model involved with sorption constants of Langmuir and Freundlich; and $k \times 10^{-3}$ (mol²/kJ²) and E (kJ/mol) are parameters of D-R model involved with D-R constants and average free energy of molecules, respectively.

The results were simulated by six models and presented in Fig. 9, which shows that the Redlich-Peterson model is favorable for U (VI) adsorption onto alginate beads. The maximum adsorption capacity was calculated to be 237.15 mg U/g.

3.5. Effect of temperature

The effect of temperature on the adsorption of U (VI) by alginate beads was performed at 20–35°C. The results are shown in Fig. 10. It can be seen that the adsorption capacity and removal efficiency obviously decreased when temperature increased.

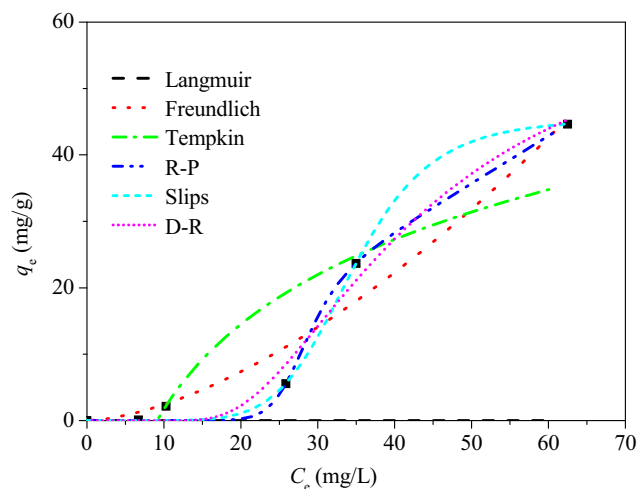


Fig. 9 – Isotherm model of U (VI) adsorption.

The thermodynamic parameters of ΔH , ΔS , and ΔG of U (VI) adsorption onto alginate beads can be calculated from the temperature-dependent sorption isotherms. The values of enthalpy change (ΔH) and entropy change (ΔS) can be calculated using the following equations:

$$K_d = \frac{(C_0 - C_e)}{C_e} \times \frac{V}{m} \quad (13)$$

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (14)$$

Gibbs free energy changes (ΔG) of specific adsorption can be calculated from following equation:

$$\Delta G = \Delta H - T\Delta S \quad (15)$$

The thermodynamic parameters are shown in Table 1. The values of thermodynamic parameters can provide an insight into the mechanism concerning the interaction of U (VI) and alginate beads. The values of ΔH are negative, which suggested that the sorption process has an exothermic reaction. The negative value of ΔG indicated that sorption is

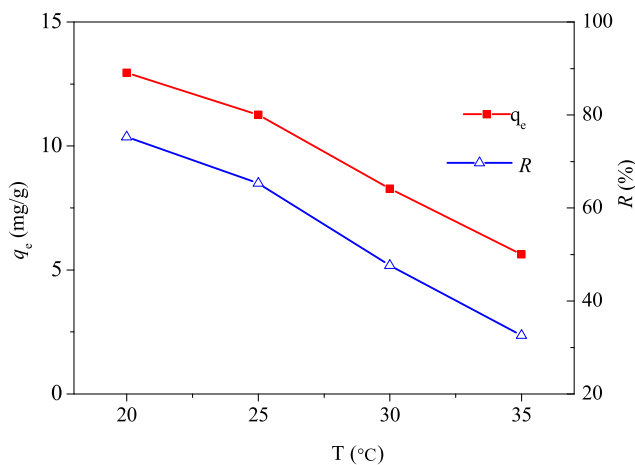


Fig. 10 – Effect of temperature on U (VI) adsorption, $C_0[U] = 20$ mg/L, pH = 5.0, t = 12 h, and m/V = 1.1 g/L.

Table 1 – The thermodynamic parameters of U (VI) adsorption onto alginate beads.

Parameter				R ²
T(°C)	ΔG (kJ/mol)	ΔS [kJ/(mol.K)]	ΔH (kJ/mol)	
20	–23.81	0.29	107.67	0.99
25	–22.38			
30	–20.95			
35	–19.52			

spontaneous. The negative values of ΔS revealed that the randomness decreased at the alginate beads-solution interface during the adsorption process.

4. Conclusions

The adsorption of U (VI) by alginate beads was examined in this study. The results showed that the adsorption of U (VI) onto the alginate beads is influenced by several factors, such as contact time, pH, initial U (VI) concentration, and temperature. The adsorption kinetics of U (VI) onto alginate beads can be described by pseudo first-order kinetic model. The adsorption isotherm can be described by the Redlich-Peterson model. The hydroxyl and alkoxy are the main functional groups for adsorption of U (VI). It is suggested that calcium alginate beads are potential adsorbents for removal and recovery of U (VI) from wastewater.

Conflicts of interest

There are no conflicts of interests in the paper.

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