

# Sorption study of uranium from aqueous solution on ordered mesoporous carbon CMK-3

Bin-wen Nie · Zhi-bin Zhang · Xiao-hong Cao ·  
Yun-hai Liu · Ping Liang

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**Abstract** The ability of ordered mesoporous carbon CMK-3 has been explored for the removal and recovery of uranium from aqueous solutions. The textural properties of CMK-3 were characterized using small-angle X-ray diffraction and N<sub>2</sub> adsorption–desorption, and the BET specific surface area, pore volume and the pore size were 1143.7 m<sup>2</sup>/g, 1.10 cm<sup>3</sup>/g and 3.4 nm. The influences of different experimental parameters such as solution pH, initial concentration, contact time, ionic strength and temperature on adsorption were investigated. The CMK-3 showed the highest uranium sorption capacity at initial pH of 6.0 and contact time of 35 min. Adsorption kinetics was better described by the pseudo-second-order model and adsorption process could be well defined by the Langmuir and Freundlich isotherm. The thermodynamic parameters,

$\Delta G^\circ$  (298 K),  $\Delta H^\circ$  and  $\Delta S^\circ$  were determined to be  $-7.7$ ,  $21.5 \text{ k J mol}^{-1}$  and  $98.2 \text{ J mol}^{-1} \text{ K}^{-1}$ , respectively, which demonstrated the sorption process of CMK-3 towards U(VI) was feasible, spontaneous and endothermic in nature. The adsorbed CMK-3 could be effectively regenerated by 0.05 mol/L HCl solution for the removal and recovery of U(VI). Complete removal (99.9 %) of U(VI) from 1.0 L industry wastewater containing 15.0 mg U(VI) ions was possible with 2.0 g CMK-3.

**Keywords** Ordered mesoporous carbon · CMK-3 · Adsorption · Uranium

## Introduction

Many activities dealing with radioactive materials produce low, intermediate and high level radioactive wastes that require advanced treatment [1, 2]. Over the last few decades, a variety of technologies, such as solvent extraction [3, 4], ion-exchange [5] and adsorption, have been developed for the removal and recovery of uranium from radioactive wastes in consideration of the dual significance of the potential environmental health threat and a nonrenewable resource of nuclear energy [6]. Recently, adsorption, due to its high efficiency and ease of handling, based on carbonaceous materials such as activated carbon [7, 8], carbon nanotubes [9, 10] and carbon fiber [11] has been gradually applied to this area because of their higher thermal and radiation resistance than organic exchanger resins and better acid–base stability compared with familiar inorganic sorbents [8].

In addition, as a new member of the carbonaceous material family, the ordered mesoporous carbon CMK-3, which is synthesized through the nanocasting technique

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B. Nie · Z. Zhang · X. Cao · Y. Liu · P. Liang  
State Key Laboratory Breeding Base of Nuclear Resources and Environment (East China Institute of Technology),  
Ministry of Education, Nanchang 330013,  
People's Republic of China

B. Nie · Z. Zhang (✉) · X. Cao · Y. Liu · P. Liang  
Key Laboratory of Radioactive Geology and Exploration  
Technology Fundamental Science for National Defense,  
East China Institute of Technology, Fuzhou 344000,  
People's Republic of China  
e-mail: zhangnjut@163.com

Z. Zhang  
Engineering Research Center of Nano-Geomaterials  
of Ministry of Education, China University of Geosciences,  
Wuhan 430074, People's Republic of China

Z. Zhang · P. Liang  
State Key Laboratory of Biogeology and Environmental  
Geology, China University of Geosciences,  
Wuhan 430074, People's Republic of China

[12], has attracted considerable attention because of its unique features, such as high surface area, regular mesoporous pore structure, narrow pore size distribution, large pore volume, as well as excellent chemical and physical stabilities [13, 14]. These features make CMK-3 more attractive in the fields of biological medicine, electrochemistry, energy storage and environment [15–17]. CMK-3 and its composite have been used to remove VE [18],  $\text{VB}_{12}$  [19], phenol [20], lysozyme [21],  $\text{Pb}^{2+}$  [22] and  $\text{Hg}^{2+}$  [23]. However, to the best of our knowledge, CMK-3 has not been reported for adsorption of uranyl ions from aqueous system so far. Therefore, it would be interesting to explore the possibility of CMK-3 for the environmental purpose mentioned above.

The aim of the present investigation was to study the efficiency of ordered mesoporous carbon CMK-3 prepared by the hard-templating method for removing uranium from aqueous solutions. Various techniques were used to characterize the structure and textural property of CMK-3, including small-angle X-ray diffraction (SAXRD) and  $\text{N}_2$  adsorption–desorption. The effect of various experimental parameters including pH of the solution, ionic strength, contact time, initial concentration, and temperature, as well as adsorption kinetics, isotherm models, and thermodynamics were studied. In addition, the regeneration method of CMK-3 and attempt for uranium(VI) removal from industry wastewater were also investigated.

## Experiment

### Materials

The ordered mesoporous silica (SBA-15) was obtained from the Nanjing Cano technology Co., Ltd. For the preparation of a stock U(VI) solution, 1.1792 g  $\text{U}_3\text{O}_8$  was put into a 100 mL beaker, and 10 mL hydrochloric acid ( $\rho = 1.18 \text{ g/mL}$ ), 2 mL 30 % hydrogen peroxide were added. The solution was heated until it was nearly dry and then 10 mL hydrochloric acid ( $\rho = 1.18 \text{ g/mL}$ ) was added. The solution was transferred to a 1,000 mL volumetric flask and diluted to the mark with distilled water to produce a U(VI) stock solution (1 mg/mL). The uranium solutions were prepared by diluting the stock solution to appropriate volumes depending upon the experimental requirements. All the other reagents were of AR grade.

### Preparation of ordered mesoporous carbon CMK-3

Mesoporous carbon material (CMK-3) was prepared using the ordered mesoporous silica (SBA-15) as the hard template and sucrose as the carbon recourse [24, 25]. Typically, 1 g of SBA-15 was added into a solution of 1.25 g of

sucrose and 0.14 g of  $\text{H}_2\text{SO}_4$  in 5 g of water, the mixture was polymerized at 100 °C for 6 h and 160 °C for another 6 h. The obtained product was impregnated with 0.09 g  $\text{H}_2\text{SO}_4$  and 0.8 g sucrose later, and subjected the same thermal treatments depicted above. The pre-products were then pyrolyzed at 900 °C for 6 h to be carbonized in a nitrogen flow with a heating rate of 5 °C/min, and resultant black powders was obtained. The mesoporous carbon was acquired by filtration after the silica template was dissolved by NaOH solution. Finally, the product was washed several times with deionized water and dried at 120 °C.

### Characterization

SAXRD patterns were recorded using Cu  $K\alpha$  radiation ( $\gamma = 1.5418$ ) on ARL X'TRA diffractometer operating at 40 kV and 40 mA with 0.25° divergence slit and 0.5° anti-scatter slit between 1.0° and 16° ( $2\theta$ ) at a step size of 2°/min. The surface area and pore structure were determined by an ASAP 2020  $\text{N}_2$  adsorption–desorption device, and the surface area was calculated using the Brunauer–Emmett–Teller (BET) method [26], and the pore distribution was determined by Barrett–Joyner–Halenda (BJH) method [27].

### Adsorption experiments

The adsorption of U(VI) was studied as a function of pH, ionic strength, contact time, initial concentration and temperature. The batch sorption was performed in a reciprocating water bath shaker with concussion speed of 200 rpm. In the experiments 0.01 g of sorbent was suspended in 50 mL solution containing different U(VI) concentration and different initial pH (adjusted with 0.1 mol/L  $\text{HNO}_3$  and 0.1 mol/L NaOH). The concentration of U(VI) in the solution was determined by the arsenazo III method [23] with a 721 type spectrophotometer at 650 nm. The amount of uranyl ions adsorbed per unit mass of the CMK-3 was calculated by Eq. 1.

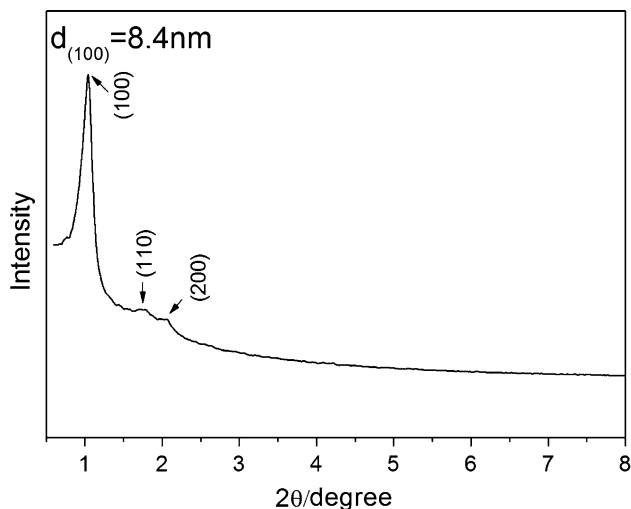
$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where,  $q_e$  is the adsorption capacity of the CMK-3 (mg/g);  $C_0$  and  $C_e$  are the uranium concentration in the initial and equilibrium solution (mg/L) respectively;  $V$  is the volume of the aqueous solution (L) and  $W$  is the mass of CMK-3 (g).

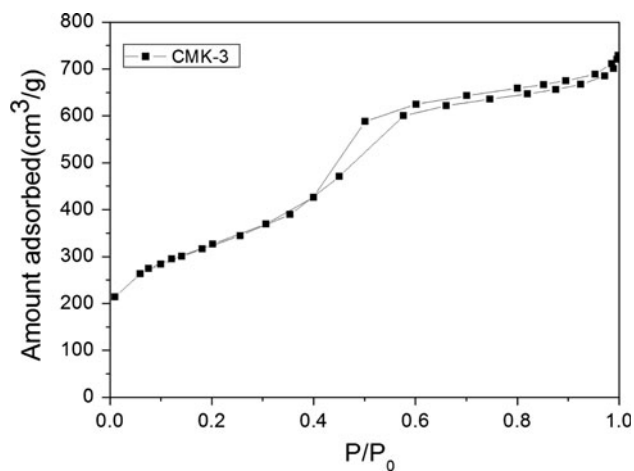
## Results and discussion

### Characterization

The ordered arrangement of the carbon nanorods in CMK-3 gives rise to the well-resolved XRD peaks shown in Fig. 1, which can be assigned to (100), (110), and (200)



**Fig. 1** The SAXRD patterns of CMK-3

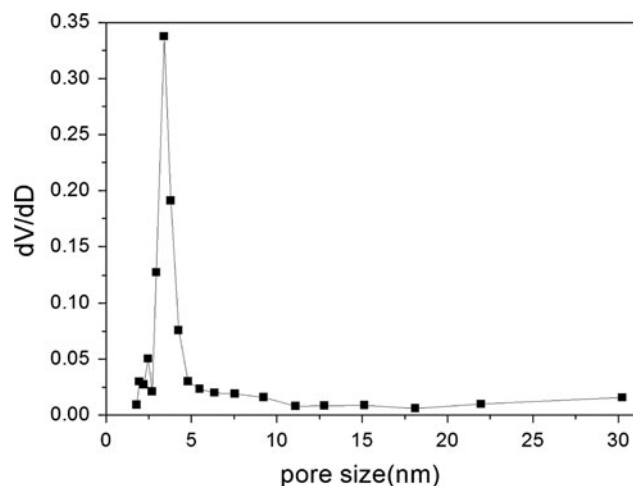


**Fig. 2** The N<sub>2</sub> adsorption–desorption isotherm of CMK-3 at 77 K

diffractions of the 2D hexagonal space group ( $p6mm$ ) similar to the case of SBA-15. The N<sub>2</sub> adsorption–desorption isotherm at 77 K and pore size distribution of CMK-3 were shown in the Figs. 2, 3 and Table 1. It was noteworthy that N<sub>2</sub> adsorption isotherms of CMK-3 was type-IV curve with clear capillary condensation steps [28]. In addition, CMK-3 exhibited high BET specific surface area (1143.7 m<sup>2</sup>/g), large pore volume (1.10 cm<sup>3</sup>/g) calculated by BJH model and a narrow pore size distribution. The pore size of CMK-3 obtained from the N<sub>2</sub> isotherm was about 3.4 nm.

#### The effect of solution pH

The pH of solution is one of the most crucial parameters for the sorption of metal ions. It can affect the surface charge, the metal speciation and surface metal binding sites. The effect of pH on the adsorption of U(VI) onto CMK-3 was carried

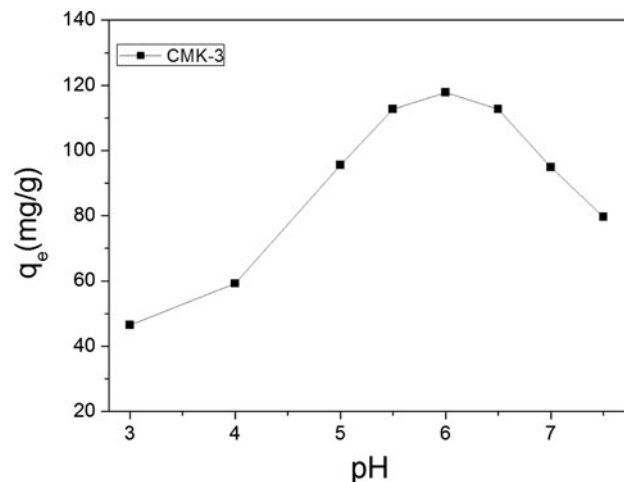


**Fig. 3** The pore size distribution of CMK-3

**Table 1** The textural parameters of CMK-3

Adsorbents	$d$ spacing (nm)	$S_{\text{BET}}$ (m <sup>2</sup> /g)	$V$ (cm <sup>3</sup> /g)	$d_{\text{p, NLDFT}}$ (nm) <sup>a</sup>
CMK-3	8.4	1143.7	1.10	3.4

<sup>a</sup> Pore size obtained from the N<sub>2</sub> isotherm on the basis of nonlocal density functional theory (NLDFT)



**Fig. 4** The effect of solution pH on U(VI) adsorption onto CMK-3

out over the pH range 3.0–7.5 using 50 μg/mL initial uranium concentration at 298 K and the results were displayed in Fig. 4. The adsorption of U(VI) on CMK-3 was greatly depended on the variation of solution pH. The uptake amount of U(VI) increased from 3 to 6, and reached the maximum adsorption capacity of 117.81 mg/g at pH 6.0 and then declined subsequently. The low adsorption capacity at lower pH value could be attributed to the increasing positivity of the adsorbents and the competition of H<sup>+</sup> ions with U(VI) on the adsorptive active sites. And with the increase of pH above

6, the hydroxide products of U(VI) such as  $\text{UO}_2(\text{OH})^+$ ,  $(\text{UO}_2)_2(\text{OH})_2$  and  $(\text{UO}_2)_3(\text{OH})_5^{2+}$  appeared and led to the decrease of the adsorption capacity [29]. Therefore, the solution pH of 6 was used for the further experiments for U(VI) adsorption on CMK-3.

#### The effect of contact time

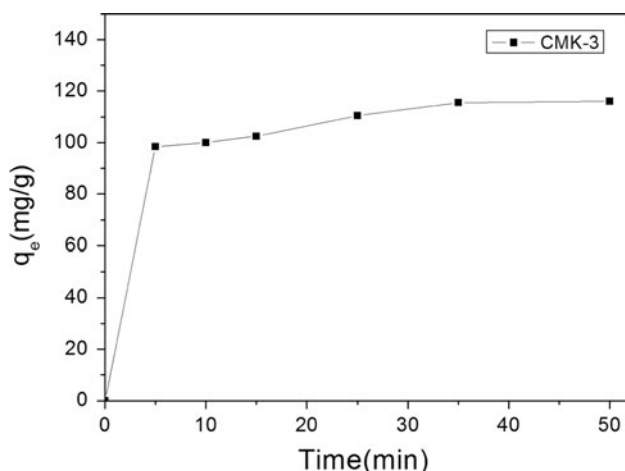
Contact time is also an important factor which can reflect the adsorption kinetics. The variation of adsorption amount with contact time was studied using 50  $\mu\text{g}/\text{mL}$  initial U(VI) concentration at pH 6.0 and 298 K. As showed in Fig. 5, the amount of U(VI) adsorbed on CMK-3 increased sharply at the beginning, and then gradually reached equilibrium after 35 min. The faster adsorption rate at the beginning would be due to the larger concentration gradient. Therefore, the contact time of 35 min was deemed sufficient to establish sorption equilibrium and used in all the subsequent experiments.

#### The effect of initial U(VI) concentration

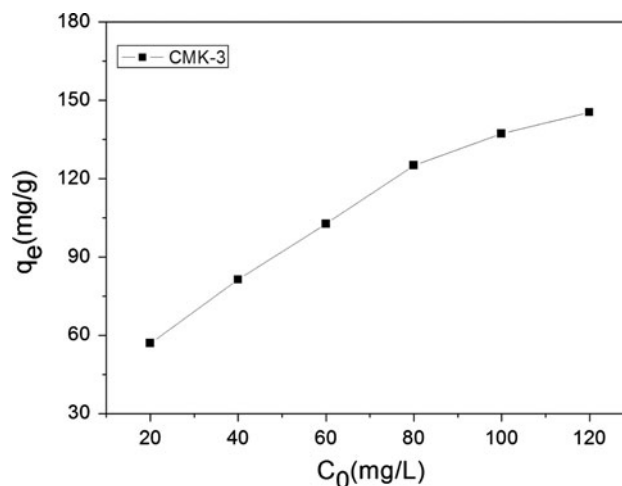
The initial concentration provides an important driving force to overcome all mass transfer resistance of uranium between the aqueous and solid phases [30]. The effect of initial U(VI) concentration on sorption was studied at 298 K and revealed in Fig. 6. The adsorptive capacity increased with the increase of the initial U(VI) concentration. According to the actual situation, the concentration of uranium in next following experiments should be controlled at 50  $\mu\text{g}/\text{mL}$ .

#### The effect of ionic strength

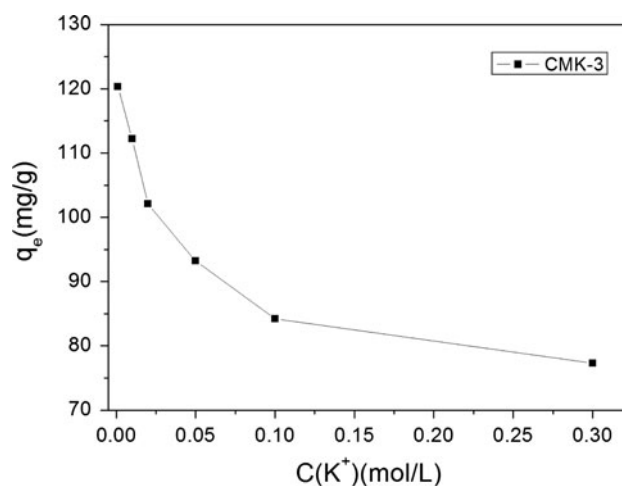
Ionic strength is another important factor reflecting adsorption. In the present study, the ionic strengths of the



**Fig. 5** The effect of contact time on U(VI) adsorption onto CMK-3



**Fig. 6** The effect of initial concentrations on U(VI) adsorption onto CMK-3



**Fig. 7** The effect of ionic strength on U(VI) adsorption onto CMK-3

solutions were adjusted by  $\text{KNO}_3$ . As shown in Fig. 7, an increase in the ionic strength from 0.01 to 0.1 mol/L had a rapid decrease in the amount of U(VI) ions adsorbed, and had little effect from 0.1 to 0.3 mol/L. The sorption capacity of U(VI) was 112.25 mg/g at the  $\text{K}^+$  concentration of 0.01 mol/L, and declined to 84.20 mg/g at 0.1 mol/L. This phenomenon could be attributed to two reasons: (1) The presence of  $\text{KNO}_3$  in the solution which screened the electrostatic interaction between the charges on CMK-3 surface and the U(VI) ions in solution and also competed with the U(VI) ions for surface adsorption sites. (2) Ionic strength of solution influenced the activity coefficient of U(VI), which limited their transfer to sorbent's surface.

#### Adsorption isotherm

The equilibrium adsorption isotherms are one of the essential data to understand the mechanism of the adsorption systems.

Langmuir and Freundlich equations are the most frequently used for describing sorption isotherms. The Langmuir model is based on assumptions of adsorption homogeneity such as equally available adsorption sites, monolayer surface coverage, and no interaction between adsorbed species. The Langmuir equation can be described by the linearized Eq. 2 [31].

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (2)$$

where  $C_e$  is the equilibrium concentration (mg/L),  $q_e$  is the amount of solute sorbed per unit weight of sorbent (mg/g),  $q_m$  is the Langmuir constant, which represents the saturated monolayer sorption capacity (mg/g).  $K_L$  is a constant related to the energy of adsorption.

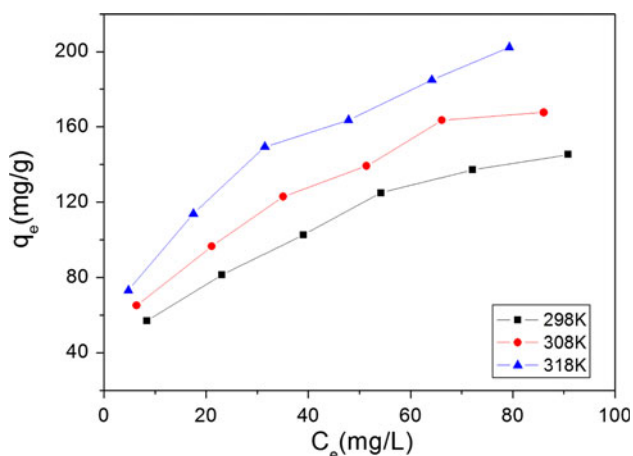
The Freundlich model can be applied to nonideal sorption on heterogeneous surfaces as well as multilayer sorption [32]. The empirical Freundlich equation can also be transformed into linearized Eq. 3.

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (3)$$

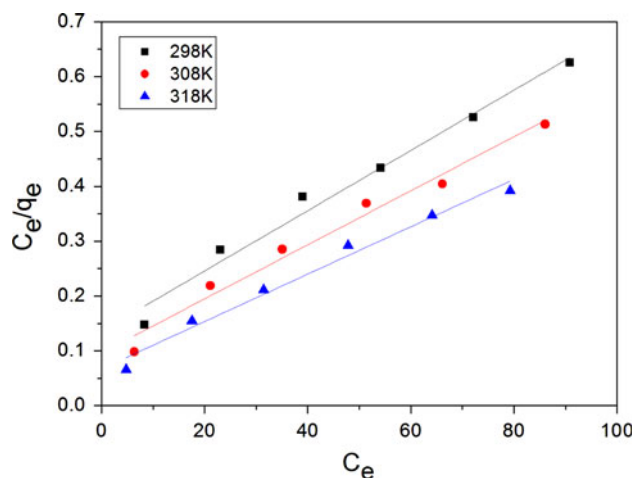
where  $C_e$  is the equilibrium concentration (mg/L),  $q_e$  is the amount of solute sorbed per unit weight of sorbent (mg/g),  $K_F$  is the Freundlich constant related to the adsorption capacity, and  $n$  is relevant to the adsorption intensity.

Figure 8 presented the effect of initial concentration on the uranium adsorption on CMK-3 at 298, 308 and 318 K.

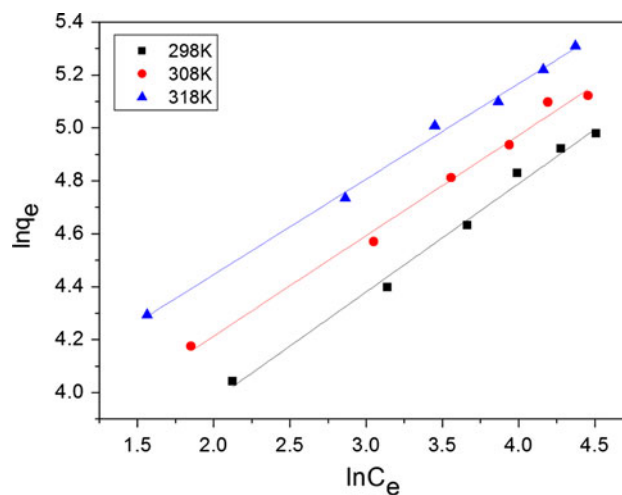
The linearized form of Langmuir and Freundlich adsorption isotherms obtained at 298, 308 and 318 K were presented in Figs. 9 and 10 respectively. And the adsorption constants evaluated from the isotherms with the correlation coefficients ( $R^2$ ) were given in Table 2. The  $R^2$  values of the Freundlich and Langmuir adsorption isotherm were over 0.98, which indicated that the sorption of U(VI) onto CMK-3 was suitable to Langmuir and Freundlich



**Fig. 8** The adsorption isotherms of U(VI) onto CMK-3 at 298, 308 and 318 K



**Fig. 9** The Langmuir adsorption isotherms of U(VI) onto CMK-3



**Fig. 10** The Freundlich adsorption isotherms of U(VI) onto CMK-3

isotherm models. Moreover, the saturated monolayer sorption capacity ( $q_m$ ) was 178.6, 204.1 and 232.6 mg/g at 298, 308 and 318 K, namely, with the increase of temperature, the saturated monolayer sorption capacity reduced, which indicated that the sorption of U(VI) onto CMK-3 was endothermic.

#### Adsorption kinetics

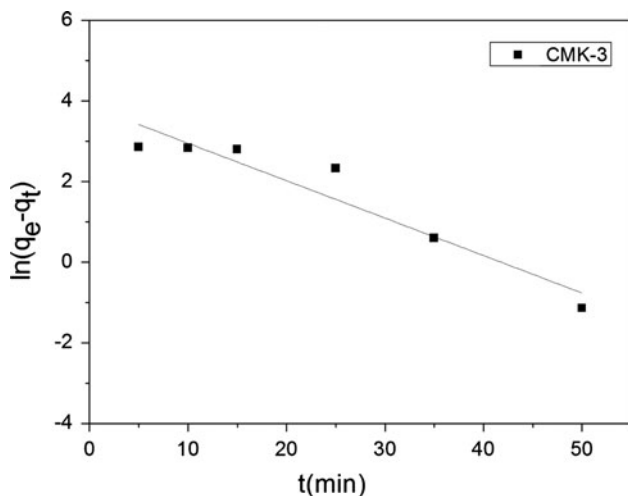
In order to explain the controlling mechanism of adsorption processes such as mass transfer and chemical reaction, pseudo-first-order and pseudo-second-order kinetic equations were applied to describe the kinetic characteristic of U(VI) onto CMK-3. The pseudo-first order kinetic model is usually given as Eq. 4 [33].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (4)$$

where  $k_1(\text{min}^{-1})$  is the rate constant of first order adsorption,  $q_e$  and  $q_t$  are the amounts of U(VI) adsorbed (mg/g) at

**Table 2** The adsorption isotherm parameters of U(VI) onto CMK-3

Adsorbents	T(K)	Langmuir adsorption isotherm			Freundlich adsorption isotherm		
		$K_L$	$q_m$ (mg/g)	$R^2$	$K_F$	$n$	$R^2$
CMK-3	298	0.043	178.6	0.98	2.51	2.51	0.99
	308	0.051	204.1	0.98	2.38	2.66	0.99
	318	0.066	232.6	0.98	2.27	2.82	0.99

**Fig. 11** The pseudo-first order adsorption kinetics of U(VI) onto CMK-3

equilibrium and time “ $t$ ”, respectively. Using Eq. 4, linear plot of  $\ln(q_e - q_t)$  versus  $t$  was plotted (Fig. 11). The  $k_1$ ,  $q_{e,cal}$  and correlation coefficient ( $R^2$ ) were calculated from the plot and presented in Table 3.

The pseudo-second order kinetic model is always given as Eq. 5 [34].

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

Where  $k_2$  ( $\text{min}^{-1}$ ) is the rate constant of second order adsorption. Using Eq. 5, linear plot of  $t/q_t$  versus  $t$  was plotted (Fig. 12). The  $k_2$ ,  $q_{e,cal}$  and correlation coefficient ( $R^2$ ) were calculated from the plot and presented in Table 3.

As showed in Table 3, the square of correlation coefficients ( $R^2$ ) of pseudo-second order equation was better than the value of the pseudo-first order equation. Moreover, the values of the amounts of U(VI) adsorbed at equilibrium,  $q_{e,cal}$  (125.0 mg/g) was very close to the experimental values,  $q_{e,exp}$  (121.7 mg/g). Therefore, the adsorption

**Table 3** The kinetic parameters of U(VI) adsorption onto CMK-3

Adsorbents	Pseudo-first-order kinetics			Pseudo-second-order kinetics		
	$q_1$ (mg/g)	$k_1$ ( $\text{min}^{-1}$ )	$R^2$	$q_2$ (mg/g)	$k_2$ ( $\text{mg g}^{-1} \text{min}^{-1}$ )	$R^2$
CMK-3	120.5	1.96	0.89	125.0	$2.98 \times 10^{-3}$	0.99

process is more favor of the pseudo-second order equation, which indicate that adsorption involves chemical reaction in adsorption in addition to physical adsorption [35].

#### Adsorption thermodynamics

Thermodynamic parameters such as enthalpy ( $\Delta H^\circ$ ), entropy ( $\Delta S^\circ$ ) and Gibbs free energy ( $\Delta G^\circ$ ) are useful in defining whether the sorption reaction is endothermic or exothermic, and spontaneity of the adsorption process [36]. The thermodynamic data were calculated using the following Eq. 6 and 7.

$$\ln k_L = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (6)$$

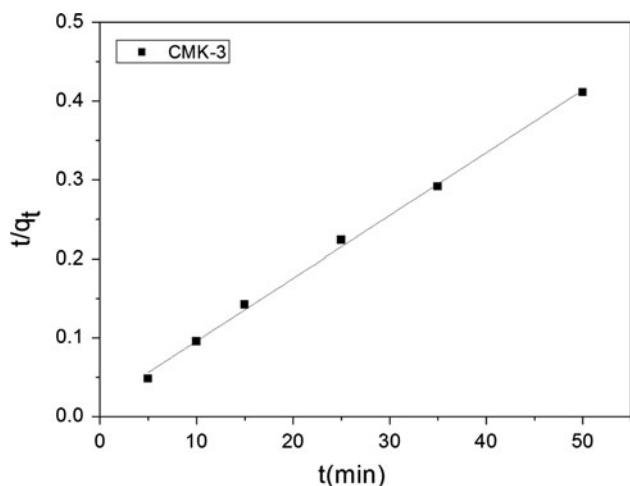
$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (7)$$

where  $k_L$  is the Langmuir constant,  $\Delta S^\circ$  is the change of entropy ( $\text{J mol}^{-1} \text{K}^{-1}$ ),  $\Delta H^\circ$  is the change of enthalpy ( $\text{kJ/mol}$ ),  $T$  is the absolute temperature in Kelvin (K) and  $R$  is the gas constant ( $8.314 \text{ J mol}^{-1} \text{K}^{-1}$ ).  $\Delta H^\circ$  and  $\Delta S^\circ$  can be calculated from the slope and intercept of the straight line (Fig. 13). The change of Gibbs free energy values are calculated from Eq. 7.

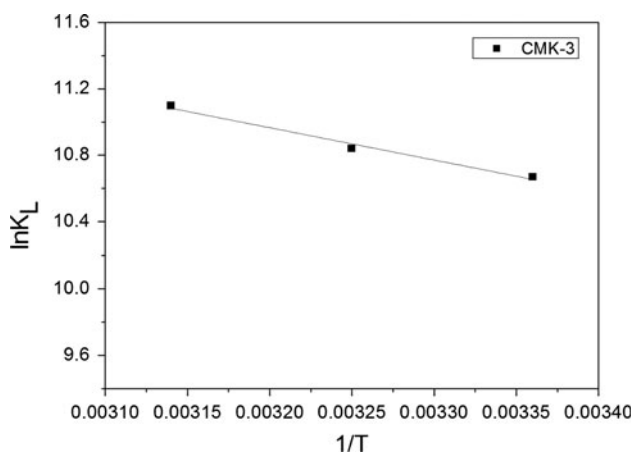
The values of thermodynamic parameters for the sorption of U(VI) at different temperature were given in Table 4. The negative value of  $\Delta G^\circ$  at different temperatures confirmed the feasibility and spontaneous nature of adsorption process. Further, the increase in the value  $\Delta G^\circ$  with the decreasing temperature indicated that higher temperature favored the sorption process. The positive value of  $\Delta S^\circ$  reflected the affinity of the CMK-3 for U(VI) and confirmed the increased randomness at the solid-solution interface during adsorption [37].

#### Desorption and regeneration

Desorption is an important process in adsorption studies due to its enhancement of the economical value. Desorption



**Fig. 12** The pseudo-second order adsorption kinetics of U(VI) onto CMK-3



**Fig. 13** The adsorption thermodynamics of U(VI) onto CMK-3

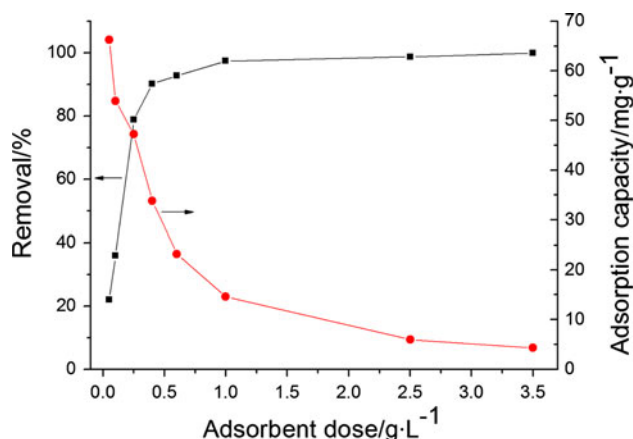
**Table 4** The adsorption thermodynamic parameters for U(VI) onto CMK-3

Adsorbents	$\Delta H^\circ$ (kJ mol <sup>-1</sup> )	$\Delta S^\circ$ (J mol <sup>-1</sup> K <sup>-1</sup> )	$\Delta G^\circ$ (kJ mol <sup>-1</sup> )		
			298 K	308 K	318 K
CMK-3	21.5	98.2	-7.7	-8.7	-9.7

studies will help to regenerate the spent adsorbent so that it can be reused to adsorb metal ions. Desorption efficiency of the spent CMK-3 was checked by 0.001–0.1 mol/L HCl solution. The results demonstrated that the adsorbed U(VI) could be desorbed completely from the spent adsorbent using 0.05 mol/L HCl, and hence to investigate the regeneration properties, the adsorption–desorption cycle was repeated four times with same adsorbent using 0.05 mol/L HCl. It was clear from Table 5 that the initial adsorption capacity, removal and desorption ratio were 117.45 mg/g, 99.31 and 97.33 %, and after four cycles decreased to 101.89 mg/g,

**Table 5** Four cycles of uranium adsorption–desorption with 0.05 mol/L HCl as desorbing agent

Cycle	Adsorption Capacity (mg/g)	Removal (%)	Desorption (%)
1	117.45	99.31	97.33
2	112.91	95.17	94.92
3	107.22	93.92	91.09
4	101.89	89.23	88.21



**Fig. 14** The U(VI) ion removal from industry wastewater by CMK-3

89.23 and 88.21 %. Therefore 0.05 mol/L HCl solution could regenerate the adsorbent effectively.

**Test with simulated nuclear industry wastewater**

The simulated U(VI) nuclear industry wastewater was recovered by CMK-3 to demonstrate its adsorption potential and utility in removing U(VI) ion from wastewater in the presence of other ions. The wastewater was prepared according to the composition of resin adsorption tail liquid from uranium hydrometallurgy plant. The wastewater contained U(VI) (15 mg/L), SO<sub>4</sub><sup>2-</sup>(10 g/L), NO<sub>3</sub><sup>-</sup>(1 g/L), Mg<sup>2+</sup>(0.2 g/L), Ca<sup>2+</sup>(0.5 g/L) and Fe<sup>3+</sup>(1.6 g/L). The effect of adsorbent dose on U(VI) removal from wastewater was investigated (Fig. 14). The percentage of U(VI) adsorption increases with increasing HDTMA<sup>+</sup>-bentonite dosage and almost complete removal (≈ 100 %) of U(VI) from the wastewater containing 15 mg/L was achieved with 2.0 g HDTMA<sup>+</sup>-bentonite in 1.0 L.

**Conclusion**

In this paper, ordered mesoporous carbon CMK-3 was prepared using SBA-15 as the hard template and sucrose as the carbon recourse with the BET specific surface area of 1143.7 m<sup>2</sup>/g, pore volume of 1.10 cm<sup>3</sup>/g and pore size of

3.4 nm obtained from  $N_2$  adsorption–desorption isotherm. The sorption performances were controlled by solution pH, contact time, initial uranium concentration, ionic strength, and temperature. The maximum capacity of CMK-3 was observed at the pH value of 6.0 and contact time of 35 min. The U(VI) sorption on CMK-3 was well fitted to the Langmuir, Freundlich adsorption isothermal and pseudo-second kinetics models. The thermodynamic parameters, such as  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$ , clearly indicated that the adsorption process was feasible, spontaneous and endothermic in nature. The adsorption–desorption study showed that U(VI) sorbed CMK-3 could be effectively regenerated by 0.05 mol/L HCl solution for the removal and recovery of U(VI) from aqueous solution. Attempts for the U(VI) removal from industry wastewater using CMK-3 revealed acceptability. Almost complete removal ( $\approx 100\%$ ) of U(VI) from the wastewater containing 15 mg/L was achieved with 2.0 g CMK-3 in 1.0 L.

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