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Effect of pH, ionic strength and temperature on sorption characteristics of Th(IV) on oxidized multiwalled carbon nanotubes

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Abstract Oxidized multiwalled carbon nanotubes (MWCNTs) were characterized by SEM and FTIR. The sorption of Th(IV) on MWCNTs was studied as a function of contact time, pH, ionic strength, Th(IV) concentration and temperature. The results indicate that the sorption of Th(IV) on MWCNTs is strongly dependent on pH and weakly dependent on ionic strength. The sorption thermodynamics of Th(IV) on MWCNTs was carried out at 293.15, 313.15 and 333.15 K, respectively, and the thermodynamic parameters (standard free energy changes (ΔG^0), standard enthalpy change (ΔH^0) and standard entropy change (ΔS^0)) were calculated from the temperature dependent sorption isotherms. The sorption of Th(IV) on MWCNTs is a spontaneous and endothermic process. The oxidized MWCNTs may be a promising candidate for the preconcentration and solidification of Th(IV), or its analogue actinides from large volumes of aqueous solutions.

Keywords MWCNTs · Thorium (IV) · Sorption · Thermodynamic data

Introduction

The environmental behavior of lanthanides and actinides has aroused great interest in terms of the radioactive waste disposal [1–4]. To assess the radionuclides behavior, radionuclide phenomena such as sorption, migration and diffusion in natural minerals and oxides are of paramount importance. The fate and transport of radionuclides in the environment is generally controlled by sorption reactions, complexation, colloid formation, etc. [5–10]. Thorium is only stable at its valence +IV in solution, and is usually used as a chemical analogue of other tetravalent actinides such as Np(IV), U(IV), and Pu(IV), which are difficult to study and to keep in the tetravalent form [11]. In recent decades, sorption of Th(IV) on different minerals and oxides have been studied extensively [12–18]. The results of the previous studies indicated that sorption of Th(IV) is strongly pH dependent and weakly ionic strength dependent [19–24]. However, the sorption capacity of Th(IV) on the minerals and oxides is still not high enough, which is crucial for the removal of Th(IV) from large volumes of aqueous solution.

Since the carbon nanotubes (CNTs) were discovered by Iijima [25] in 1991, the novel materials have come under intense multidisciplinary study because of their unique physicochemical properties [26–28]. CNTs include single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) depending on the number of layers comprising them. Because of their highly porous and hollow structure, large specific surface area, light mass density, and strong interaction between carbon and molecules, CNTs have been found as an efficient adsorbent for organic pollutants and heavy metal ions [29–34]. The earlier studies indicated that CNTs may be a promising adsorbent used in the environmental protection. But report

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concerning application of CNTs for radionuclide sorption was sparse. Wang et al. [35] firstly studied the sorption of ^{243}Am(III) on MWCNTs and found that MWCNTs were a promising candidate for preconcentration and solidification of actinides from large radioactive solutions. The previous studies [36–38] demonstrated that the sorption of Sr(II), Am(III), and Eu(III) onto oxidized MWCNTs was affected by pH values and the sorption was mainly dominated by strong chemisorption and chemicomplexation. Belloni et al. [39] reviewed the application of MWCNTs in the removal of radionuclides from aqueous solutions, and conclude that MWCNTs can play a role in the fireld of nuclear waste management. However, the study of Th(IV) sorption on MWCNTs is still scarce [16], especially the effect of temperature on Th(IV) sorption.

Herein, we reported the application of MWCNTs in the removal of Th(IV) from aqueous solutions. The effect of pH, ionic strength and temperature on Th(IV) sorption was investigated. The thermodynamic parameters were calculated from the temperature dependent sorption isotherms, and the sorption mechanism was discussed.

Experimental

Materials

MWCNTs were prepared by using chemical vapor deposition (CVD) of acetylene in hydrogen flow at 760 °C using Ni–Fe nanoparticles as catalysts [35]. Oxidized MWCNTs were prepared by oxidization with 3 M HNO_3 [35]. The surface area of oxidized MWCNTs is 197 m^2/g by using the N_2-BET method. The point of zero charge, pH_{pzc}, i.e., the pH above which the total surface of the carbon is negatively charged, was measured at pH ~ 5 [35, 40].

1,000 mg L^{-1} Th(IV) stock solution was prepared as follow: ThO_2 (purity >99.9%) was dissolved in 10 mL 3 M HNO_3 and then transferred into a 500 mL vessel. The solubility of ThO_2 was greatly improved by adding 2–3 drops HF (1:20) in the preparation of the thorium stock solution. The stock solution was diluted with Milli-Q water to obtain the desired concentrations. The concentration of Th(IV) was analyzed by spectrophotometry at wavelength 650 nm by using Th(IV) arsenazo(III) complex. The amount of Th(IV) sorption was calculated from the difference between the initial concentration and the equilibrium one.

Batch sorption experiments

Sorption kinetics was firstly carried out to achieve the equilibrium time. The experiments were performed at $T = 293.15 \pm 2$ K in 0.01 M $NaClO_4$ solution at stirring rate of 300 rpm. The stirring rate of 300 rpm was chosen to make MWCNTs homogenously to be dispersed in solution. Sorption isotherms were investigated by using batch technique in polyethylene centrifuge tubes under ambient conditions at 293.15 ± 2, 313.15 ± 2, and 333.15 ± 2 K, respectively. The blank experiments demonstrated that the sorption of Th(IV) on the test tube walls was negligible. The stock solutions of $NaClO_4$ and MWCNTs were pre-equilibrated for 2 h before the addition of Th(IV) stock solution. The samples were gently shaken for 24 h (which was enough to achieve equilibrium). Duplicate batches were examined to achieve the sorption curves. The relative errors of the data were about ±5%.

Results and discussion

SEM and FTIR analysis

Figure 1 shows the scan electron microscope (SEM) image of oxidized MWCNTs. The oxidized MWCNTs have very smooth surfaces and cylindrical shapes with an external diameter of 10–30 nm. Due to inter-molecular force, the MWCNTs of different sizes and directions form an aggregated structure. The Fourier Transform infrared spectroscopy (FTIR) (Fig. 2) indicates that the acid treatment process introduces many functional groups onto the surfaces of MWCNTs: carbonyl groups (1,385 cm^{-1}), carboxyl groups (1,580 cm^{-1}), and hydroxyl groups (3,423 cm^{-1}), which provide a large number of chemical adsorption sites [41]. Meanwhile, the hydrophilic properties of these functional groups improve the dispersivity of MWCNTs in aqueous solution [42].

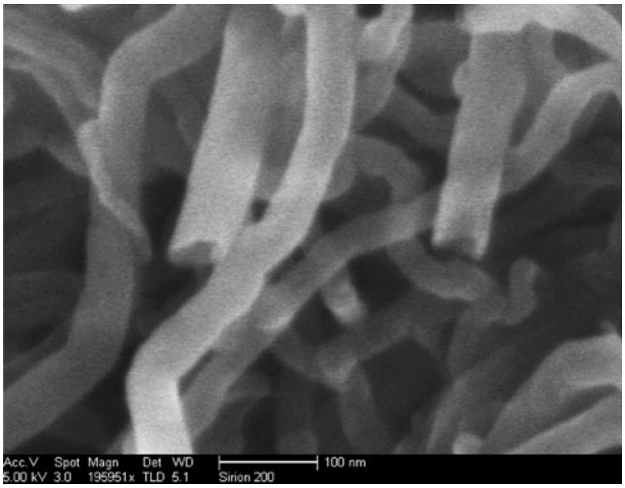

Fig. 1 SEM image of oxidized MWCNTs

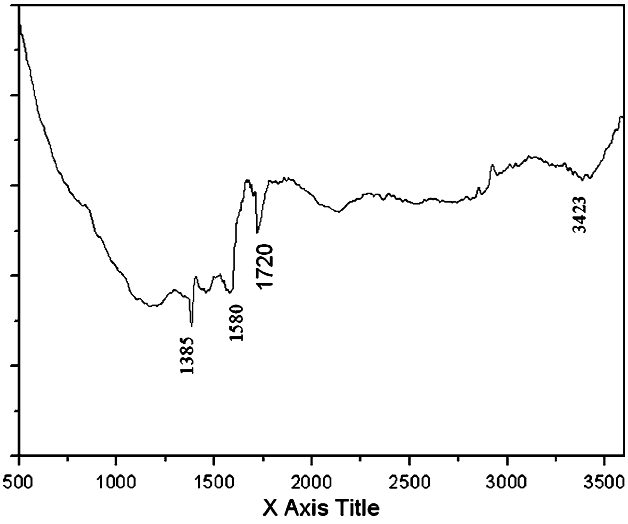

Fig. 2 FTIR spectrum of oxidized MWCNTs

Kinetic sorption study

Figure 3 shows the effect of contact time on Th(IV) sorption to MWCNTs. One can see that the sorption is rapid in the first 15 min of contact time, and 1 h is enough to achieve sorption equilibrium. The initial steep sorption curve suggests that the sorption occurs rapidly on the surface of MWCNTs. The sorption becomes slow subsequently because of the longer diffusion range of the Th(IV) ions (ionic radius ≈0.102 nm) into the inner channel (diameter ≈3.6 nm) of MWCNTs [35]. According to the above results, the shaking time was fixed for 24 h for the following batch sorption experiments.

Effect of pH and ionic strength

The sorption of Th(IV) on MWCNTs in 0.01 M $NaClO_4$ solution as a function of pH is shown in Fig. 4. Sorption of Th(IV) is strongly dependent on pH values. Sorption of Th(IV) increases from ~10 to ~95% at pH 1–4, and then maintains its maximum level with increasing pH values. The results of Th(IV) sorption on hematite [43] indicated that half of Th(IV) was sorbed on hematite at pH = 2 and ~100% of Th(IV) was sorbed on hematite at pH = 4. Jakobsson [44] studied the sorption of Th(IV) on TiO_2 and found that the sorption of Th(IV) started from pH 0.5 and increased quickly to ~100% at pH = 3, and concluded that reversible formation of an inner sphere complex with a strong pH dependence was the main sorption mechanism.

The sorption of Th(IV) on MWCNTs at pH 2.0 and 3.0 as a function of $NaClO_4$ concentrations is shown in Fig. 5. One can see that the sorption of Th(IV) decreases a little with increasing $NaClO_4$ concentration. With increasing Na^+ concentrations in solution, the competitive sorption of Th(IV) with the cations adsorbed on MWCNTs increases and thereby the sorption of Th(IV) on MWCNTs decreases. Guo et al. [45] studied the sorption of Th(IV) on alumina and found that the sorption decreased drastically with increasing ionic strength at Th(IV) concentration of 7.5×10^{-5}–2×10^{-4} mol/L; however, the sorption of Th(IV) increased with increasing ionic strength at Th(IV) concentration $<7.5 \times 10^{-5}$ mol/L. The pH and ionic strength dependent sorption of Th(IV) on MWCNTs suggests that the sorption of Th(IV) on MWCNTs can be attributed to surface complexation and cation exchange under our experimental conditions [46–48].

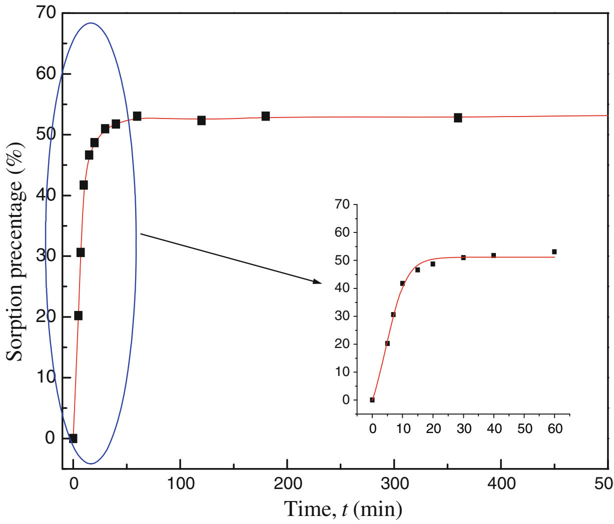

Fig. 3 Effect of contact time on Th(IV) sorption to oxidized MWCNTs (C_0(Th(IV)) = 3.2×10^{-5}mol L^{-1}, pH = 2.70 ± 0.02, $I = 0.01$ M $NaClO_4$, $m/V = 0.3$ g L^{-1}, $T = 293.15 \pm 2$ K)

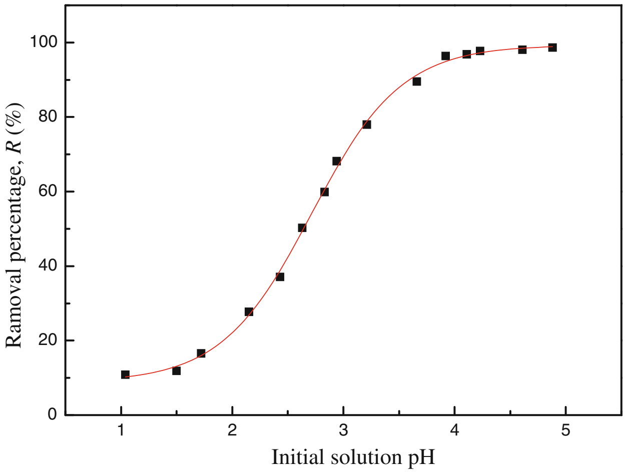

Fig. 4 Effect of pH on Th(IV) adsorption onto oxidized MWCNTs (C_0(Th(IV)) = 3.2×10^{-5}mol L^{-1}, $I = 0.01$ M $NaClO_4$, $m/V = 0.3$ g L^{-1}, $T = 293.15 \pm 2$ K)

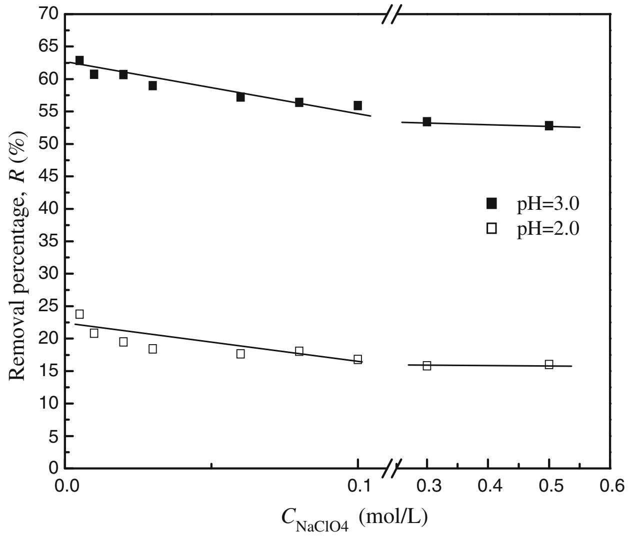

Fig. 5 Effect of ionic strength on Th(IV) adsorption onto oxidized MWCNTs (C_0(Th(IV)) = 3.2×10^{-5}mol L^{-1}, $m/V = 0.3$ g L^{-1}, $T = 293.15 \pm 2$ K)

Sorption isotherms and thermodynamic parameters

The sorption isotherms of Th(IV) on MWCNTs at 293.15, 313.15 and 333.15 K are shown in Fig. 6a. One can see that the sorption isotherm is the highest at $T = 333.15$ K and is the lowest at $T = 293.15$ K. The results indicate that high temperature is advantageous for Th(IV) sorption on MWCNTs. This phenomenon is attributed to a steep simultaneous decrease of real sorption of solvent or a negative temperature coefficient of the solubility of solute [49]. Three different models, viz. Langmuir, Freundlich and D-R isotherm equations are conducted to simulate the sorption isotherms of Th(IV) on MWCNTs.

The Langmuir model assumes that sorption occurs in a monolayer with all sorption sites identical and energetically equivalent. Its linear form can be described as [50]:

$$\frac{C_{\mathrm{eq}}}{C_{\mathrm{s}}} = \frac{1}{bC_{\mathrm{s\,max}}} + \frac{C_{\mathrm{eq}}}{C_{\mathrm{s\,max}}} \tag{1}$$

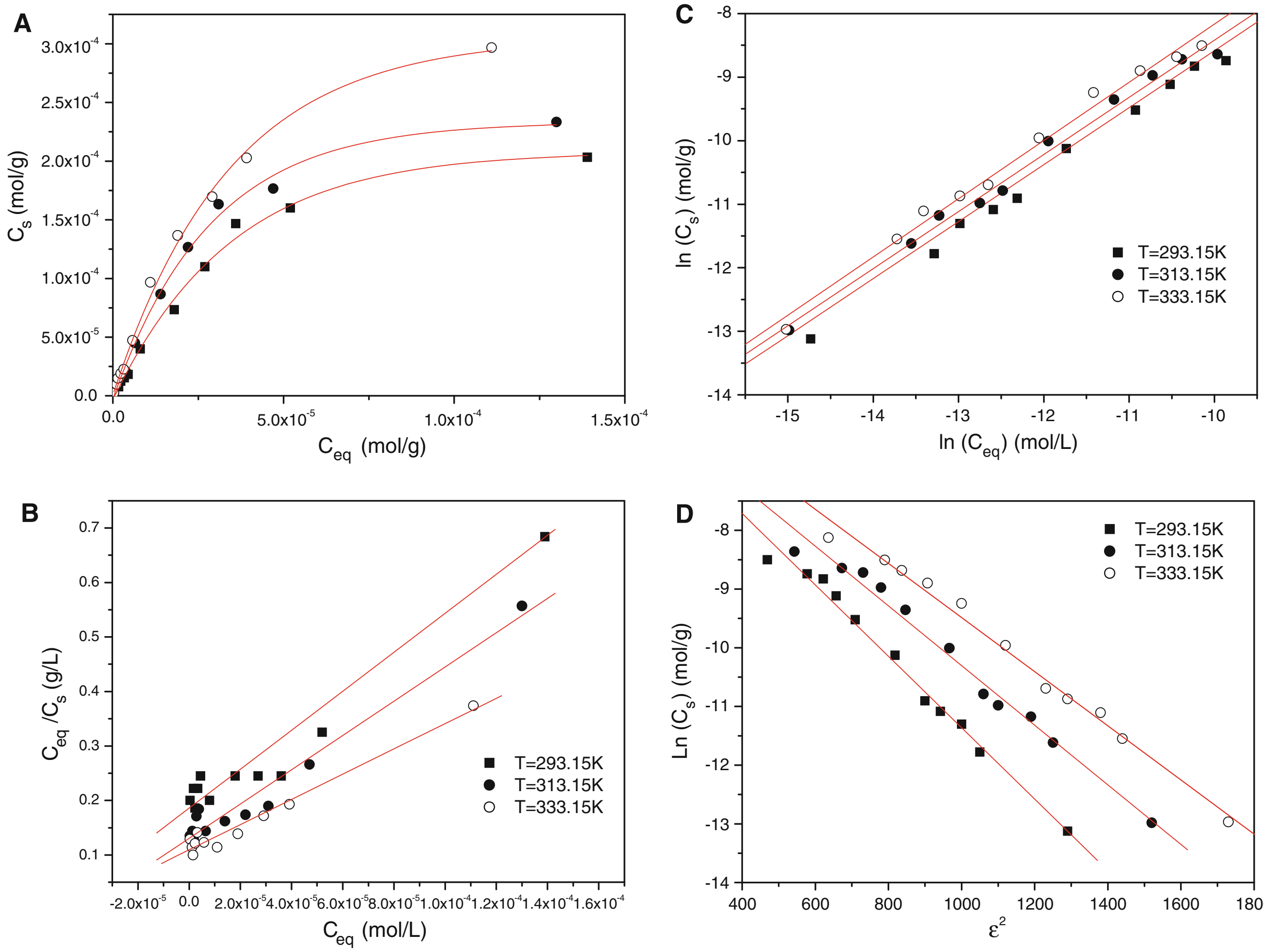

Fig. 6 Sorption isotherms of Th(IV) on MWCNTs at three different temperatures. ($m/V = 0.3$ g L^{-1}, initial pH = 3.00 ± 0.02, $I = 0.01$ M $NaClO_4$). **a** Sorption isotherms, **b** Langmuir model, **c** Freundlich model, **d** D-R model

where C_{eq} is the equilibrium concentration of Th(IV) remained in solution (mol L^{-1}); C_s is the amount of Th(IV) adsorbed on MWCNTs after equilibrium (mol g^{-1}); $C_{s\,max}$ is the maximum sorption capacity at complete monolayer coverage (mol g^{-1}), and b (L mol^{-1}) is a constant that relates to the heat of sorption.

The Freundlich expression is an exponential equation with the assumption that the sorption occurs on the heterogeneous sorbent surface. The equation is represented as [51]:

$$\ln C_s = \ln k_F + n \ln C_{eq} \tag{2}$$

where k_F (mol^{1-n} L^n g^{-1}) represents the sorption capacity when metal ion equilibrium concentration equals to 1, and n represents the degree of sorption dependent with equilibrium concentration.

The D-R model is more general than Langmuir model since it does not have the restriction of surface properties or constant sorption potential. The general expression is expressed as follow [51]:

$$\ln C_s = \ln C_{s\,max} - \beta\varepsilon^2 \tag{3}$$

where C_s and $C_{s\,max}$ are defined above, β is the activity coefficient related to mean sorption energy (mol^2 kJ^{-2}), and ε is the Polanyi potential, which is expressed as:

$$\varepsilon = RT \ln\left(1 + \frac{1}{Ceq}\right) \tag{4}$$

where R is ideal gas constant (8.314 J mol^{-1} K^{-1}), and T is the absolute temperature in Kelvin (K).

E (kJ mol^{-1}) is defined as the free energy change, which requires to transfer 1 mol ions from aqueous solution to the solid surfaces. It can be calculated from the following equation:

$$E = \frac{1}{\sqrt{2\beta}} \tag{5}$$

The magnitude of E is used to evaluate the sorption mechanism. Sorption is dominated by chemical ion exchange if E is in the range of 8–16 kJ mol^{-1}, whereas physical forces influences the sorption if E is lower than 8 kJ mol^{-1} [52].

The experimental data of Th(IV) sorption (Fig. 6a) are regressively simulated with Langmuir, Freundlich and D-R models and the results are shown in Fig. 6c–d, respectively. The relative values calculated from the three models are listed in Table 1. As can be seen from Fig. 6c–d, the three models simulate the sorption isotherms well, which is supported by the good correlation coefficients (all $R^2 > 0.97$ in Table 1). The fact that the sorption of Th(IV) on MWCNTs according with Langmuir model indicates that the binding energy on the whole surfaces of MWCNTs is uniform. This phenomenon also indicates that chemosorption is the principal sorption mechanism in sorption process. The E values obtained from Eq. 5 are 9.1 ($T = 293.15$ K), 10.0 ($T = 313.15$ K) and 10.4 kJ mol^{-1} ($T = 333.15$ K), which are in the sorption energy range of chemical ion-exchange reactions. The values of $C_{s\,max}$ obtained from the Langmuir model are the highest at $T = 333.15$ K and the lowest at $T = 293.15$ K, which indicates that the sorption is enhanced with increasing temperature. The value of n calculated from the Freundlich model is from unity, indicating that a nonlinear sorption of Th(IV) takes place on MWCNTs. It is necessary to note that the sorption capacities $C_{s\,max}$ derived from the D-R model are higher than those derived from the Langmuir

Table 1 The parameters for the three isotherm models at different temperatures

Correlation parameters	$T = 293.15$ K	$T = 313.15$ K	$T = 333.15$ K
Langmuir			
$C_{s\,max}$ (mol g^{-1})	2.99×10^{-4}	3.18×10^{-4}	4.32×10^{-4}
b (L mol^{-1})	1.80×10^4	2.41×10^4	2.12×10^4
R^2	0.97	0.98	0.98
Freundlich			
k_F (mol^{1-n} L^n g^{-1})	1.77	1.76	2.70
n	0.925	0.899	0.916
R^2	0.99	0.99	0.99
D-R			
β (mol^2 kJ^{-2})	6.01×10^{-3}	5.11×10^{-3}	4.60×10^{-3}
$C_{s\,max}$ (mol g^{-1})	5.18×10^{-3}	5.50×10^{-3}	7.99×10^{-3}
E (kJ mol^{-1})	9.1	10.0	10.4
R^2	0.99	0.99	0.99

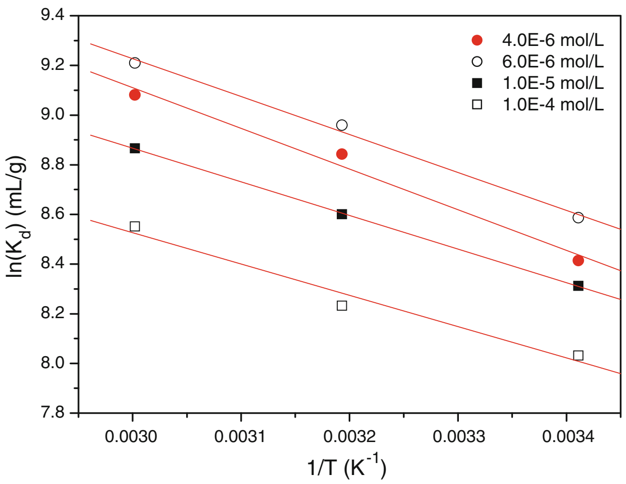

Fig. 7 Linear plot of LnK_d versus C_{eq} at different temperatures. $m/V = 0.3$ g/L, initial pH = 3.00 ± 0.02, $I = 0.01$ M $NaClO_4$

Table 2 Thermodynamic parameters of Th(IV) sorption on MWCNTs

C_0 (mg L^{-1})	ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})	ΔG^0 (kJ mol^{-1})		
			293.15 K	313.15 K	333.15 K
4.0×10^{-6}	13.62	116.6	−20.58	−22.91	−25.24
6.0×10^{-6}	12.69	114.8	−20.96	−23.26	−25.56
1.0×10^{-5}	11.25	107.5	−20.26	−22.41	−24.56
1.0×10^{-4}	10.48	102.3	−19.53	−21.57	−23.62

model. This may be attributed to the different assumptions considered in the formulation of the isotherms. The parameters calculated from the analysis of the three isotherm models indicate that sorption of Th(IV) on attapulgite is a favorable and chemisorption process [53].

The distribution coefficients as a function of temperature at different initial concentrations at $T = 293.15$, 313.15 and 333.15 K are shown in Fig. 7. The thermodynamic parameters of ΔH^0, ΔS^0 and ΔG^0 of Th(IV) sorption on MWCNTs can be calculated from the temperature dependent sorption isotherms. The values of standard enthalpy change (ΔH^0) and standard entropy change (ΔS^0) can be calculated from the slope and y-intercept of the plot of lnK_d versus $1/T$ (Fig. 7) using the following equations [41]:

$$K_d = \frac{C_0 - C_{eq}}{C_{eq}} \times \frac{V}{m} \quad (6)$$

$$\ln K_d = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (7)$$

Gibbs free energy changes (ΔG^0) of specific adsorption are calculated from:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (8)$$

Relevant parameters calculated from Eqs. 7 and 8 are given in Table 2. The values of thermodynamic parameters provide an insight into the mechanism concerning the interaction of Th(IV) on MWCNTs. The values of ΔH^0 are positive, which suggests that the sorption process is endothermic. One possible interpretation of the endothermic process is that Th(IV) ions are well solvated in water [40]. In order for Th(IV) ions to adsorb, they are denuded of their hydration sheath to some extent, and this dehydration process needs energy. It is assumed that this energy of dehydration exceed the exothermicity of Th(IV) ions attaching to MWCNTs. The removal of water molecules from Th(IV) ions is essentially an endothermic process, and the endothermicity of the desolvation process exceed the enthalpy of sorption to a considerable extent [54]. The Gibbs free energy change (ΔG^0) is negative as expected for a spontaneous process. The decrease of ΔG^0 with increasing temperature indicates more efficient sorption at higher temperature. Th(IV) ions are readily desolvated at higher temperature, and thereby their sorption to MWCNTs becomes more favorable. The positive values of entropy change (ΔS^0) reflect the affinity of MWCNTs toward Th(IV) ions in aqueous solutions and might suggest some structure changes.

Conclusion

The sorption of Th(IV) on oxidized MWCNTs was studied under ambient conditions. The effect of contact time, pH, ionic strength and temperature on Th(IV) sorption to MWCNTs was investigated. The pH and ionic strength dependent sorption suggests that the sorption of Th(IV) on MWCNTs is mainly dominated by surface complexation and ion exchange. The oxygen-containing functional groups of MWCNTs contribute to Th(IV) sorption on MWCNTs. The fast sorption of Th(IV) on MWCNTs suggests that MWCNTs can be used in the removal of Th(IV) ions from large volumes of aqueous solutions. The high removal of Th(IV) from solution to MWCNTs suggests that MWCNTs are suitable materials in the preconcentration of Th(IV) ions in real work in nuclear waste management.

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