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Effects of phosphate and Cr^{3+} on the sorption and transport of uranium(VI) on a silica column

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Six column experiments were performed and seven breakthrough curves (BTCs) and seven displacement (desorption) curves (DPCs) of phosphate and U(VI) were obtained, which demonstrated the effects of phosphate and Cr^{3+} on the sorption and transport of U(VI) on a silica column at pH 3 and uranium concentration 10^{-5}–10^{-6} mol/L of the influent. It was found that in the presence of phosphate sorbed preliminarily on the silica column, the amount of U(VI) sorbed on this column is significantly increased owing to the interaction of U(VI) with phosphate sorbed as compared with that in the absence of phosphate, and the breakthrough and the mean residence time of U(VI) on this column are similar to those in the absence of phosphate. While the effect of simultaneous injection of Cr^{3+} on the retardation of U(VI) on the silica column, the maximum concentration of BTC and the amount of U(VI) sorbed was found to be insignificant. Transport and sorption studies of U(VI) are important, since all uranium isotopes are radioactive, there is a need to understand the potential for migration away from radioactive waste storage and mill tailing sites.

Introduction

In the previous papers from our laboratory,[1–3] the sorption of U(VI) on SiO_2, TiO_2, and Al_2O_3 was studied by the batch technique as a function of pH and ionic strength. We focused on the effect of phosphate in the sorption. The significantly positive effect and the slightly negative effect of phosphate in the sorption of U(VI) on SiO_2 were respectively found at pH<7 and pH>7 and at the ratio of phosphate concentration to U(VI) concentration >1. This positive effect was attributed to the strong surface binding of phosphate and the subsequent formation of ternary surface complex involving uranyl and phosphate. However, in general, column experiments can better simulate field conditions than batch experiments, because (1) the transport behavior of U(VI) in porous media is controlled by interplay between physical transfer and sorption or/and precipitation reactions; (2) a higher solid/solution ratio is possible of achievement; and (3) reaction products do not accumulate. On the other hand, column experiments are often run at high flow velocity, and this leads to underestimation of slow reactions of importance in field low-flow conditions. Therefore, batch and column experiments are complementary for the understanding of the physical-chemical phenomena involved in sorption and transport of U(VI) in porous media. In our previous papers,[4] the effect of phosphate on the sorption and transport of Th(IV) on a silica column was investigated and found to be positive and significant and the effect of simultaneous injection of Cr^{3+} on Th(IV) breakthrough is not significant. This paper is an extension of our previous papers[1–4] from our laboratory. The first objective of this paper was to investigate the effect of phosphate on the sorption and transport of U(VI) on a silica column, because phosphate is ubiquitous, and silica is abundant in the earth.

In the previous paper,[1] it was found that the sorption edge of U(VI) on the silica is increased from pH 3.5, sorption%=10% to pH 6.7, sorption%=90% at initial concentration of U(VI) $3.48\cdot10^{-6}$ mol/L, ratio of solution volume to mass of SiO_2 100 mL/g, and ionic strength 0.02 mol/L KNO_3, and that the sorption edge is practically independent on the ionic strength from 0.002 to 0.2 mol/L KNO_3. However, multivalent cations, such as Cd^{2+} and Cr^{3+}, are very common in the environment as contaminants. Clearly, the competition between uranyl and other multivalent cations for sorption sites of surface is of great importance in the study of the sorption and transport behavior of U(VI). In the previous paper,[4] the effect of Cr^{3+} on the sorption and transport of Th(IV) on a silica column was investigated and found to be insignificant. The second objective of this paper was to investigate the effect of trivalent cation Cr^{3+} on the sorption and transport of U(VI) on a silica column.

The migration of uranium through siliceous porous media, the Clashach Sandstone, was investigated by conducting a series of column experiments in conjunction with the development of chemical transport model by READ et al.[5] and SIMS et al.[6] It was found experimentally that under oxidizing conditions, at pH 5–6 with NaCl concentration 0.3 mol/L and uranium concentration 10^{-6}–10^{-8} mol/L, U(VI) is strongly retarded by sorption to the Clashach Sandstone matrix, the majority of U(VI) is sorbed with a few millimeters of the inlet and no breakthrough is occurred over the timescales used in the experiments, though a slight enhancement of U(VI) migration in the presence of cadmium was found, the effect of co-injection of cadmium at concentration 10^{-4} mol/L was not remarked,

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and a proportion of U(VI) was released only very slowly by 0.3 mol/L NaCl solution. Comparative experimental and modeling studies demonstrated that the sorption behavior of Clashach Sandstone is to be close to that of amorphous silica. It was also found by READ et al.[7] that EDTA and to a greater extent, saccharic acid actively promote the migration of uranium(VI) on the Clashach Sandstone, and the breakthrough is occurred.

The sorption and the BTC of U(VI) on a quartz column are investigated by KOHLER et al.[8] with $NaNO_3$ concentration 0.01 mol/L at pH 3.9–4.8, uranium concentration 10^{-6}–10^{-5} mol/L and in the presence and absence of fluoride. It was found that in the absence of fluoride, the breakthrough is occurred at about 3 numbers of pore volume at pH 3.9, while at pH 4.26, the breakthrough is occurred at 8 numbers of pore volume, and that in the presence of fluoride, the U(VI) mobility on the quartz is increased relative to that in the absence of fluoride. In 2002, ARTINGER et al.[9] investigated the humic colloid-borne migration of uranium in sand column. The BTC indicated that U(VI) is transported as a colloid-borne species with a velocity up to 5% faster than the mean groundwater flow. Recently, KREPELOVA et al.[10] measured the BTC of U(VI) on a quartz column and found that the U(VI) migration is characterized by a kinetically controlled strong retardation. CHENG et al.[11] studied the effect of phosphate on U(VI) sorption onto goethite-coated sand by a batch technique, and found the obviously positive effect at pH<6 and the negative effect at pH>8. Very recently, the effect of phosphate and fulvic acid sorbed preliminarily and simultaneously on the sorption and transport of U(VI) on a silica column was investigated too in our laboratory.[20] The ability of a commonly-used surface complexation model to describe the adsorption of U(VI) and phosphate onto pure amorphous and crystalline Fe(III) oxides and synthetic goethite-coated sand was examined by ROMERO-GONZALEZ et al.[21] They could not use this model to consistently describe phosphate adsorption onto both amorphous and crystalline Fe(III) oxides and goethite-coated sand, though this model was able to describe U(VI) adsorption onto these adsorbents quite well in the absence of phosphate.

Experimental

Commercial silica (spectral pure) was purchased from the Shanghai Wu-Si Chemical Factory and used in previous papers.[1,14,15] The conditioning and the storage of silica were identical to those already employed.[1,14,15]

The stock solutions of $UO_2(NO_3)_2$, phosphate, and Cr^{3+} were prepared by dissolving $UO_2(NO_3)_2 \cdot 6H_2O$, KH_2PO_4, and $CrCl_3 \cdot 6H_2O$ in distilled-ionized water. The total concentration of UO_2^{2+} or phosphate was determined by spectrophotometric analysis with Arsenazo III or phosphomolybdate blue using ascorbic acid as a reducing agent.[1,14] The exact concentration of the stock solution of U(VI) was determined gravimetrically.[1] The pH of injection solution was adjusted by adding a small amount of HNO_3. Background electrolyte was KNO_3. All chemicals used here were of analytical reagent grade.

The essential procedures and the instruments of column experiments were identical to those already employed.[4,16,17] The column was slurry packed with 1.0 g of conditioned silica. The pore volume (V_0) was measured by the weight difference before and after saturation of the column with water. The column experiment was divided into three parts: (1) before column experiment, the column was percolated and conditioned preliminarily until the pH and the concentration of solute of effluents were equal to those of influent; (2) after pre-conditioning of the column, a pulse of solution was injected to the column at a constant flux, until the maximum concentration of U(VI) in effluents was reached; (3) a displacement solution (desorption solution) was introduced to displace U(VI) sorbed from the column at the constant flux until the concentration of U(VI) in effluents was approached to zero. The concentrations of U(VI) and the pH values in effluents were determined and the concentration was expressed as a relative concentration C/C_0, where C and C_0 are the concentration in the effluent and in the influent, respectively. Seven BTCs and seven DPCs (displacement curves) were obtained as a C/C_0 vs. V/V_0 plot, where V/V_0 is the number of pore volume injected and displaced through the column, V is the volume of effluent. The interruption of flow during changing of the mobile phase lasted no more than a few seconds. The mobile phases passed through the column sequentially in six column experiments are as follows:

Experiment 1: Preconditioning solution – 0.01 mol/L KNO_3 solution at pH 3.0 (also used in Experiments 2, 3, 4, 5, 6). Pulse solution – 0.01 mol/L KNO_3 solution containing $0.87 \cdot 10^{-5}$ mol/L phosphate at pH 3.05. Displacement solution – 0.01 mol/L KNO_3 solution at pH 3.00 (also used in Experiments 2, 3, 4, 5, 6).

Experiment 2: Pulse solution – 0.01 mol/L KNO_3 solution containing $9.9 \cdot 10^{-6}$ mol/L $UO_2(NO_3)_2$ at pH 3.03.

Experiment 3: Second preconditioning solution – 0.01 mol/L KNO_3 solution containing $1.09 \cdot 10^{-5}$ mol/L phosphate at pH 3.00. Pulse solution – 0.01 mol/L KNO_3 solution containing $9.9 \cdot 10^{-6}$ mol/L $UO_2(NO_3)_2$ at pH 3.09. The pulse solution was injected at $V/V_0 = 74$.

Experiment 4: Pulse solution – 0.01 mol/L KNO_3 solution containing $9.9 \cdot 10^{-6}$ mol/L $UO_2(NO_3)_2$ and $1.0 \cdot 10^{-5}$ mol/L $CrCl_3$ at pH 2.99.

Experiment 5: Pulse solution – 0.01 mol/L KNO_3 solution containing $1.05 \cdot 10^{-5}$ mol/L $UO_2(NO_3)_2$ at pH 3.02.

Experiment 6: Pulse solution – 0.01 mol/L KNO_3 solution containing $1.03 \cdot 10^{-5}$ mol/L $UO_2(NO_3)_2$ at pH 2.96.

Results

Experiment 1

The BTC(●) and the DPC(o) of phosphate, and the pH value of effluents are shown in Fig. 1. Figure 1 shows that the appearance of phosphate shortly after $V/V_0 \approx 4$, and that the pH value of effluents was approximately constant and equal to that of influent. As shown in Fig. 1, the C/C_0 value was abruptly increased before $V/V_0 = 20$, and in the range of V/V_0 from 20 to 69, the maximum C/C_0 ranged from 0.93 to 1.02. Figure 1 also shows that the DPC was sharply declined and the C/C_0 value was approached to zero at V/V_0>95.

Experiment 2

The BTC(●) and DPC(o) of U(VI) are shown in Fig. 2. Figure 2 shows the appearance of U(VI) shortly at about $V/V_0 = 4$, and that the C/C_0 value was sharply increased before $V/V_0 = 14$ and then reached a steady maximum C/C_0 ranging from 0.94 to 1.00. Figure 2 also shows an approximately constant pH value from beginning to end. In the DPC, the C/C_0 value was sharply declined and approached to zero at V/V_0>88.

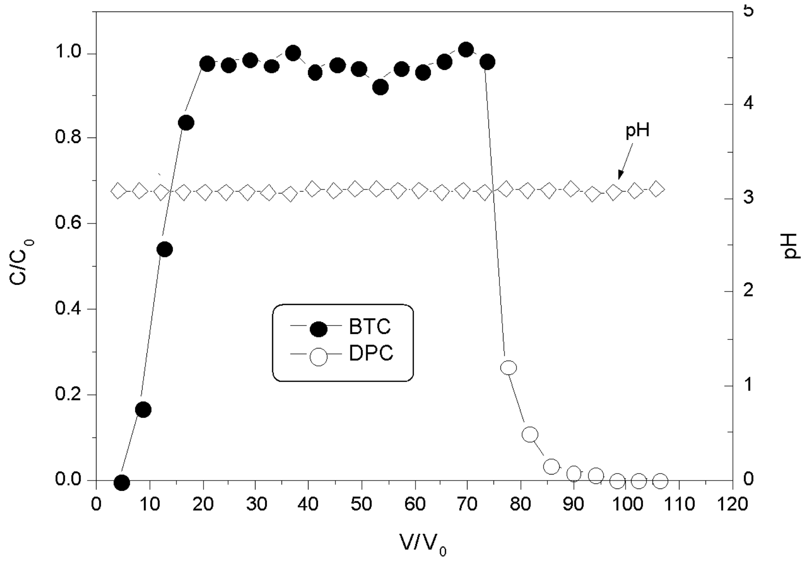

Fig. 1. BTC(●) and the DPC(○) of phosphate, and the pH value of effluents

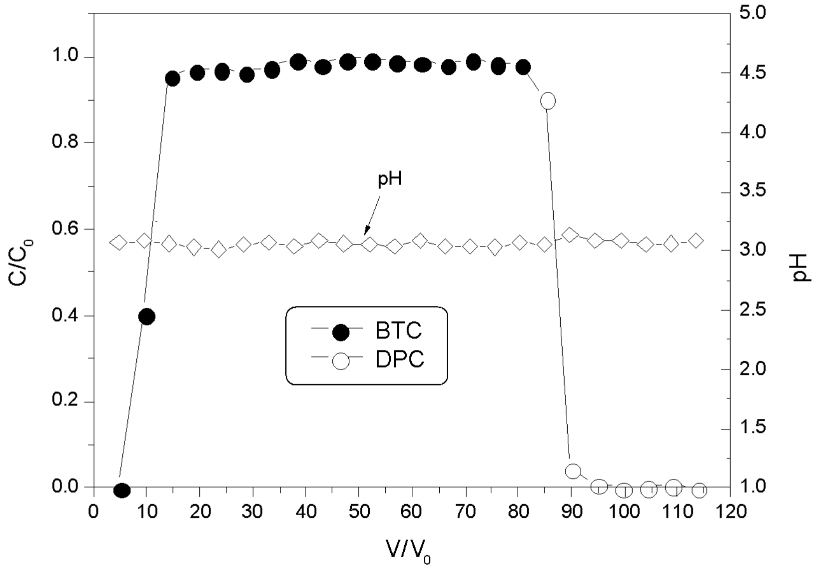

Fig. 2. BTC(●) and the DPC(○) of UO_2^{2+}, and the pH value of effluents

Experiment 3

The BTC(●) and the DPC(o) of phosphate, and the BTC(●) and the DPC(o) of U(VI) are shown in Fig. 3. As shown in Fig. 3, the C/C_0 value of phosphate was abruptly increased before $V/V_0 = 20$ and then reached maximum concentrations $C/C_0 \approx 1$ over 23 numbers of pore volume, finally the C/C_0 was sharply declined and approached to zero, the phosphate sorbed preliminarily on the column was displaced immediately. The appearance of U(VI) was occurred shortly after injection of pulse solution at $V/V_0 = 74$ and similar to that in the absence of phosphate , and the C/C_0 value was sharply increased before $V/V_0 = 94$ and then reached a steady maximum concentration over 51 numbers of pore volume and ranging from 0.93 to 1.00. Figure 3 also shows the DPC of U(VI) was sharply declined and the C/C_0 value was approached to zero after $V/V_0 = 180$ with a little tailing. In addition, the pH value of effluents was approximately constant and roughly equal to that of influent.

Experiment 4

The BTC(●) and the DPC(o) of U(VI) in the presence of Cr^{3+} in the pulse solution are shown in Fig. 4. Like the BTC(●) in the absence of Cr^{3+} (Fig. 2), Fig. 4 also shows the appearance of U(VI) shortly at about $V/V_0 = 4$ and that the C/C_0 value was sharply increased before $V/V_0 = 14$ and then reached a maximum concentration C/C_0 in the range 0.96–0.99. The DPC(o) of U(VI) was sharply declined from $V/V_0 = 72$ to 82 and then approached to zero after $V/V_0 = 85$. The pH value of effluents was also approximately constant and equal to that of influent.

Experiment 5

The BTC(●) and the DPC(o) of U(VI) on the silica column with a height 93 mm are shown in Fig. 5. As compared with the Fig. 2 with a height 76 mm, Fig. 5 shows the BTC at column height 93 mm was slightly shifted to the right, and both the DPCs at both heights were roughly parallel and sharply declined over 12 numbers of pore volume. The pH value of effluents was also approximately constant.

Experiment 6

Figure 6 shows the effect of flow rate on the BTC(●) and DPC(o) of U(VI) on a silica column. As shown in Fig. 6, the BTC(●) at slower flow rate was shifted to the right as compared with that at higher flow rate (Fig. 2). As a whole, the maximum concentrations at two flow rates were equal to each other and C/C_0 close to 1. Both the DPCs at two flow rates were roughly parallel before $V/V_0 = 64$ and sharply declined over 8 numbers of pore volume. Figure 6 shows the approximately constant pH of effluents from beginning to end.

All column parameters are listed in Table 1.

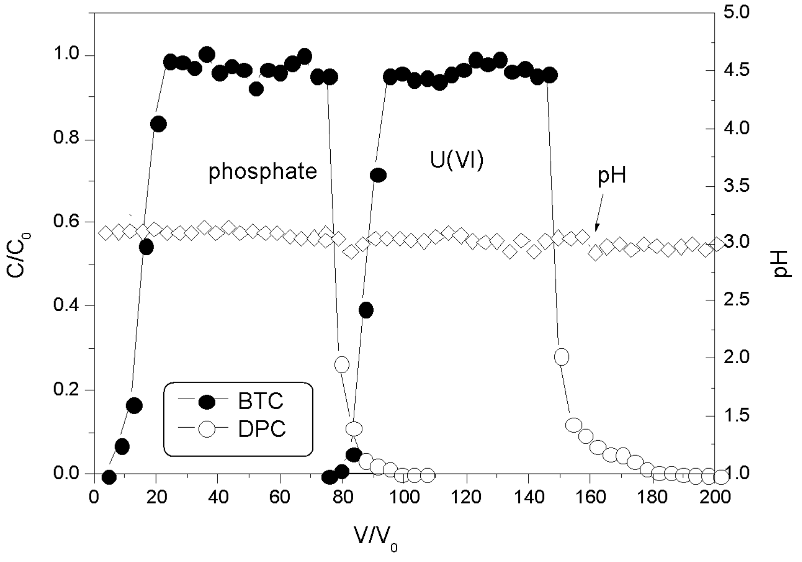

Fig. 3. BTC(●) and the DPC(○) of phosphate, the BTC(●) and the DPC(○) of U(VI) on a silica column, and the variation of pH values of effluents

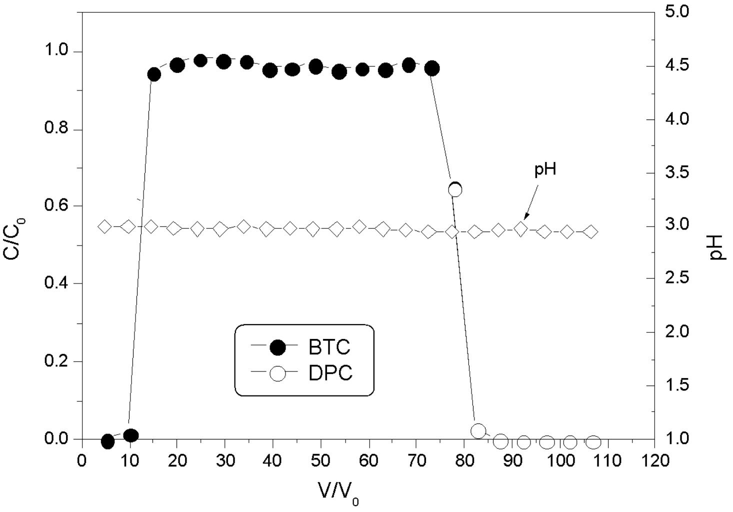

Fig. 4. BTC(●) and the DPC(○) of U(VI) on a silica column in the presence of co-injection of Cr^{3+}

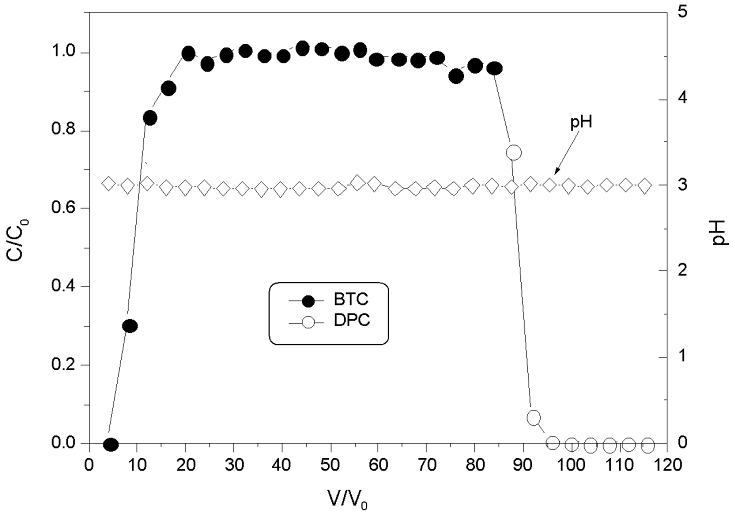

Fig. 5. BTC(●) and DPC(○) of U(VI) on a silica column with a height 93 mm

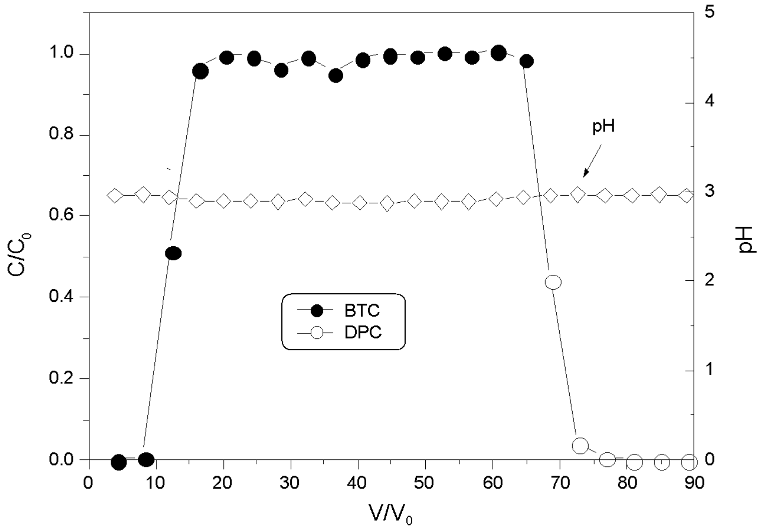

Fig. 6. BTC(●) and the DPC(○) of U(VI) on a silica column at a slower flow rate ($u = 0.33$ mL/min)

Table 1. Experimental conditions and results

Column experiment	1	2	3	4	5	6
Column height (mm)	73	76	75	73	93	74
Inner diameter of column (mm)	7	7	7	7	7	7
Mass of SiO_2 (g)	1.0	1.0	1.0	1.0	1.2	1.0
Pore volume (V_0, mL)	1.23	1.27	1.27	1.23	1.56	1.24
Flow rate (mL/min)	0.5	0.61	0.6	0.59	0.62	0.33
[1]Pulse length (V/V_0)	73	74	79	72	83	65
[2]Displacement length (V/V_0)	32	33	47	15	28	20
[3]Q_0 (10^{-7} mol/g)	1.18	1.13	1.63	1.29	1.32	1.30
[4]Q (10^{-7} mol/g)	0.58	0.60	0.37	0.41	0.55	0.26
Recovery rate ($Q/Q_0 \times 100\%$)	50	53	23	32	42	20
[5]E (min)	25	26	23	18	30	37

[1] Pulse length = the duration of injection of pulse solution to the column.

[2] Displacement length = the duration of injection of displacement solution to the column.

[3] Q_0 = the amount of U(VI) or phosphate sorbed on 1 g silica.

[4] Q = the amount of U(VI) or phosphate desorbed from 1 g silica.

[5] E = the mean residence time.

Discussion

As mentioned in the Experimental section, the column experiments were designed with a pH value 3.0 and a relatively higher concentration of U(VI) 10^{-6}–10^{-5} mol/L to be as simple as possible in order to yield unambiguous results over a limited timescales. Firstly, it was found from of the previous paper,[1] that the sorption of U(VI) onto the silica at pH 3 is about 5% and relatively lower as compared with sorption 20% at pH 4, and the sorption edge of U(VI) is practically independent on the ionic strength from 0.002 to 0.2 mol/L KNO_3. And the sorption of phosphate at pH 3 is about 7% and relatively higher as compared with sorption % less than 6 at pH 4. Secondly, at pH 3, the free UO_2^{2+} is the predominating species, the hydrolysis[18] and the complexation of U(VI) with carbonate from air can be negligible. Thirdly, the hydrolysis of Cr^{3+} at pH 3[19] and the dissolution of silica[8] also can be negligible. As shown in all Figs 1 to 6, the pH value of effluents in all column experiments is roughly constant at pH 3.0 after pre-conditioning. Thus we can definitely rule out the possibility of effect of pH on BTCs(●) and DPCs(o), and the effects of phosphate and Cr^{3+} on the sorption and transport of U(VI) on a silica column can be definitely revealed.

The moment analysis can be used to interpret the BTC. The n-th moment is defined by GABRIEL et al.[20] and KALINICHEV et al.:[21]

$$M(t^N) = \int_0^\infty t^N \frac{C(t)}{C_0} \mathrm{d}t \qquad (1)$$

where C_0 is the concentration of influent imposed for $0<t<T_0$ (pulse length). The zero order moment is used for the mass balance and the first order moment is used in the calculation of the mean residence time E. Expression for E is given by:

$$E = \frac{M(t^1)}{M(t^0)} \qquad (2)$$

The amount of sorbed U(VI) on 1.0 g silica (Q_0) and the amount of desorbed U(VI) from 1.0 g silica (Q) were calculated from the difference the amount of U(VI) in the pulse and the zero order moment of BTC and the area beneath DPC, respectively. The $M(t^0)$ and the area beneath DPC were obtained by trapezoid rule integration. The values of Q_0, Q and E for all column experiments are listed in Table 1 too.

Table 1 shows that the value of Q_{03} in the presence of phosphate sorbed preliminarily is about 1.3 times as large as Q_{02} in the absence of phosphate. This increase in Q_0 indicates that besides the surface hydroxyl groups on the silica, the phosphate ions sorbed preliminarily on the surface of silica also act as sorption sites with high affinity for U(VI). The value of Q_{06} at slower flow rate is increased by 15% and the value of Q_{05} at higher column also is increased 17% as compared with Q_{02} at faster flow rate and lower column, respectively. Both the increases in Q_0 indicate that the sorption process in the dynamic system studied here is far from equilibrium. As shown in Table 1, the recovery rates range from 23% in the presence of phosphate sorbed preliminarily on the silica column to 53% in the absence of phosphate. These recovery rates indicate that a proportion of U(VI) bound to the surface hydroxyl groups and the majority of U(VI) bound to the phosphate ions sorbed on the surface are not readily recoverable, whereas the majority of U(VI) bound to silica surface in the presence of $Cr^{3+}$are not readily recoverable too, since the recovery rate is only 32% in the presence of Cr^{3+}.

Figure 3 shows that after injection of pulse solution of U(VI), the concentration of phosphate in the effluent also sharply decreased from 74 numbers of pore volume. In other words, the increase in U(VI) concentration and the decrease in phosphate concentration in effluents are synchronous. This synchronism is the evidence of the displacement of phosphate sorbed preliminarily with UO_2^{2+} in pulse solution. In the absence of phosphate, only about 4 numbers of pore volume need to be injected before breakthrough. In the presence of phosphate, the breakthrough of U(VI) is similar to that in the absence of phosphate. As shown in Table 1, the mean residence time of U(VI) in the presence of phosphate sorbed preliminarily is roughly equal to that in the absence of phosphate too. All these results demonstrate that the phosphate sorbed on the column does not actively retard the migration of U(VI) on the silica column under these experimental conditions studied here.

Figures 4 and 2 show that in the presence of co-injection of Cr^{3+}, the BTC (Fig. 4) is slightly shifted to the right as compared with that in the absence of Cr^{3+}, and that both the maximum concentrations in the presence and absence of Cr^{3+} are close to each other and slightly less than 1. Also as shown in Table 1, considering the error of E, the value of E in the presence of Cr^{3+} is not significantly different from that in the absence of Cr^{3+}. Thus it may be concluded that the effect of Cr^{3+} on the migration of U(VI) on the silica column is not significant.

Conclusions

From the experimental results and the discussion, the conclusions are as follows:

Phosphate has a significantly positive effect on the amount of U(VI) sorption onto a silica column in a dynamic migration process at pH 3 and experimental conditions studied here. This positive effect on the amount of sorbed U(VI) is similar to that on Th(IV) sorption on the same column and at the same pH.

The breakthrough and the mean residence time of U(VI) on a silica column is not significantly influenced by the simultaneous presence of Cr^{3+} in pulse solution and by the presence of phosphate sorbed preliminary on the column. This effect of phosphate is different from that on the Th(IV) transport on the same column and at the same pH.[4] This insignificant effect of Cr^{3+} is similar to that of Th(IV) transport on the same column and at the same pH.

A proportion of U(VI) sorbed on a silica column in the absence of phosphate or Cr^{3+} is not readily desorbed with 0.01 mol/L KNO_3 at pH 3, while the majority of U(VI) sorbed on a silica column in the presence of phosphate or Cr^{3+} is not readily desorbed with 0.01 mol/L KNO_3 at pH 3.

The effect of phosphate on the sorption and transport of U(VI) onto porous media rich in SiO_2 is very important in the assessment of the long term interaction of U(VI) with the environment. While in this assessment, the effect of competing cations on the sorption and transport of U(VI) onto porous media rich in SiO_2 is less important.

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