Sorption of U(VI) and As(V) on SiO_2, Al_2O_3, TiO_2 and FeOOH: A column experiment study

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Abstract. One important factor which affects migration of U(VI) in the subsurface is the sorption at the solid/solution interface. Many factors control the sorption of U(VI) on mineral surfaces and one significant candidate among them is the distribution of aqueous species. In this study, column experiments were carried out to investigate the transport of uranium, arsenate and uranium-arsenate together (0.5:0.5 µM/l) in columns packed with SiO_2, Al_2O_3, TiO_2 and FeOOH at pH 6.5. Transport behavior of U(VI) and As(V) through SiO_2 and TiO_2 packed columns are identical in the inlet solutions containing either U(VI) or As(V) separately, or both together. In the presence of equimolar U(VI) and As(V), a substantial increase in As(V) mobility and a slight decrease in U(VI) transport through Al_2O_3 were observed. When Al_2O_3 is replaced with FeOOH, a significant change in the pattern of mobility was shown by As(V); whereas U(VI) showed only minor changes. The changes in transport behavior of both elements can be attributed to the competitive sorption between uranyl and arsenate species or due to the formation of uranyl-arsenate species. The immobilization of uranyl and arsenate with the aforementioned minerals are in the order $FeOOH>TiO_2>Al_2O_3>SiO_2$ under our experimental conditions. This study thus gives potential information about the transport behavior of uranyl and arsenate in natural systems, especially when both elements are present.

Introduction

Uranium is a naturally occurring radioactive and chemically toxic metal, which is widespread in nature. The release of uranium to the environment is not only from natural sources but also from anthropogenic sources like uranium mill tailings, nuclear industry, coal combustion, phosphate fertilizers etc (Merkel 2002; Merkel and Hasche-Berger 2006). Under oxidizing environments, uranium is readily soluble in water in the +VI oxidation state, but as well depending on pH. In porous mediums such as soils, aquifers or mine wastes, the transportation and immobilization of U(VI) is mainly governed by sorption/desorption reactions at the solid-solution interface. Batch sorption experiments were widely carried out to study the

sorption of U(VI) on different mineral surfaces like clay minerals (Pabalan and Turner 1996; Krepelova et al. 2006; Bachmaf and Merkel 2011), quartz (Lieser et al. 1992; Sylwester et al. 2000; Nair and Merkel 2011), zeolite (Aytas et al. 2004; Camacho et al. 2010), goethite (Hsi and Langmuir 1985; Missana et al. 2003) etc. under various conditions. Although batch sorption experiments are capable of providing useful information about solid-solution interaction, they poses prominent difference from the transport conditions in subsurface such as; relatively low solid-solution ratio, influence of reaction kinetics on sorption, duration of the experiment, lack of hydrodynamic mass-transport limitations which can happen in porous media, abrasion of the mineral particles etc. Hence column experiments are recommended to understand the reactive transport of radionuclides in porous media.

One of the important factors which drives the sorption behavior of U(VI) under different transport conditions is the aqueous speciation. Presence and absence of various ligands changes the U(VI) speciation in different aquatic conditions. Phosphate is one of the candidates which forms strong complexes with U(VI) and is very stable as well as insoluble in geological settings (Liu and Byrne 1997). Arsenate is analogous to phosphate and is a well-known contaminant to the environment. Long term exposure of As leads to a number of serious diseases including skin, bladder and lung cancers (Smith et al. 2000). Under oxidizing conditions, arsenic occurs arsenate with +V oxidation state [As(V)]. The formation of uranyl-phosphate complexes and its impact on the U(VI) sorption behavior have been studied extensively (Brendler et al. 1996; Payne et al. 1996; Cheng et al. 2004). On the other hand, the formation of uranyl-arsenate complex and its influence on sorption are rather less investigated (Rutsch et al. 1999; Gezahegne et al. 2012). Existence of natural minerals like Trögerite, $H_2(UO_2AsO_4)_2$. $8H_2O$ and $UO_2(HAsO_4)$. $4H_2O$ are good evidence for the affinity between uranium and arsenic to form as well aquatic species.

Reactive transport of U(VI) with various ligands and different minerals have been studied and reported elsewhere (Barnett et al. 2000; Cheng et al. 2007; Zhang et al. 2009). However, less information is available on the transportation of U(VI) and As(V) in systems containing both elements. Changes in sorption behavior of U(VI) and As(V) in columns containing bentonite (Bachmaf et al. 2009) and iron-coated sand (Schulze and Merkel 2011) were studied and reported. Less or no information is available on the transportation of U(VI) and As(V) together with minerals such as SiO_2, Al_2O_3, TiO_2 and FeOOH, which are very common in natural environments.

The objective of this study was to investigate the reactive transport of uranium (0.5 µM/l), arsenate (0.5 µM/l)) and uranium-arsenic together (0.5:0.5 µM/l) in columns packed with SiO_2, Al_2O_3, TiO_2 and FeOOH at pH 6.5. The study is also extended to investigate the influence of uranyl-arsenate species on the transport of U(VI) and As(V) with aforementioned minerals where the influent contains both elements at pH 6.5.

Materials and Methods

Materials

The stock solutions for U(VI) and As(V) (0.5 μM/l) were prepared from $UO_2(NO_3)_2.6H_2O$ (Chemapol, Czech Republic) and As_2O_5 (Alfa Aesar, Karlsruhe Germany) respectively by dissolving in deionised and purified water (TKA, Germany). To avoid precipitation or sorption of U(VI), the water was pre-acidified with HNO_3 to pH 2.5. The minerals Al_2O_3 and TiO_2 were obtained from Sigma–Aldrich (Germany). The assay, specific surface area and particle size of Al_2O_3 and TiO_2 are 99.7%, 0.6 m²/g and 10 μm as well as ≥ 99.9%, 2.5 m²/g and < 5 μm respectively. Goethite was prepared in the laboratory as described by Schwertmann and Cornell (2000) and the surface area of the mineral was measured using BET (44.77 m²/g). The silica sand (SiO_2) used for the experiment (brand name F32) was obtained from Quarzwerke Frechen, Germany. The average grain size and surface area of SiO_2 are 0.24 and 102 cm²/g respectively. XRD analysis revealed that F32 contains 98.6±0.26% quartz and 1.4±0.26% calcite. The chemical analysis shows that F32 has 99.7% SiO_2, 0.2% Al_2O_3 and 0.03% Fe_2O_3. The purification process of F32 and the removal of calcite and iron oxides were explained in Nair and Merkel (2011). All chemicals used for the column experiment were of ACS reagent grade or better.

Column Experiment

Column experiments were conducted at room temperature (22±1°C) using 12 PTFE columns with 2 cm inner diameter and 30 cm length. 0.2 μm PTFE filters were placed at both end of the column to keep the porous materials in place. 10 gm of SiO_2 and Al_2O_3, 0.5 gm of TiO_2 as well as 0.1 gm of FeOOH were mixed well with PTFE beads (grain size: 0.29-0.35 mm) and dry-packed in PTFE columns with a porosity of 0.40. The 12 columns were filled with corresponding minerals as shown below;

(1) U(VI) – SiO_2, (2) As(V) – SiO_2, (3) U(VI) – As(V) – SiO_2, (4) U(VI) – Al_2O_3, (5) As(V) – Al_2O_3, (6) U(VI) – As(V) – Al_2O_3, (7) U(VI) – TiO2, (8) As(V) – TiO2, (9) U(VI) – As(V) – TiO2, (10) U(VI) – FeOOH, (11) As(V) – FeOOH & (12) U(VI) – As(V) – FeOOH

All tools such as columns, tubes, connecting valves, collecting bottles etc. used for the experiments were made of PTFE in order to avoid the sorption of U(VI) on it especially at low U(VI) concentration and at high pH. To minimize the dead volume, short tubes with small inner diameter were used to connect the valves to the corresponding columns (Fig. 1).
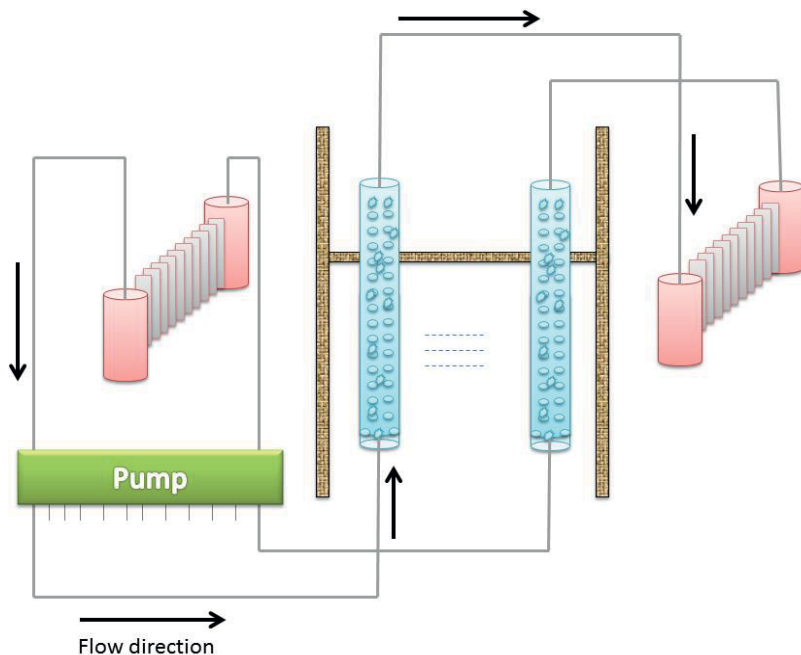
Fig.1. Schematic representation of the column experiment (see text for details and note that the size of the different parts is not to scale).

The solutions were pumped from bottom to top with a uniform flow rate of 100 μL/min using a high precision peristaltic pump ISMATEC IPC 24 (Ismatec SA, Switzerland). Due to high sorption of U(VI) and As(V) onto FeOOH, the flow rate was increased to 250 μL/min in the columns 10, 11 and 12 from 146th day of the experiment onwards. In each column experiment, the de-ionised water (DI) was pumped slowly from bottom to top for 12 hours in order to eliminate the air spaces within the porous media and to pre-condition the same. Following to this, U(VI), As(V) and U(VI):As(V) solutions were flushed through corresponding columns until the breakthrough curves (*BTC*) reached equilibrium. The pH of the inlet solution was adjusted to 6.5 using 0.1 M $NaOH/HNO_3$. One of the main reasons to select this pH was the assumed dominance of the proposed uranyl-arsenate complex at this pH range. After reaching the equilibrium *BTC*, the inlet solutions were changed back to DI water to study the desorption behavior. Desorption experiment using DI was only effective to certain extend with SiO_2 filled columns. In order to flush out the U(VI) and As(V) from the columns, the pH of the inlet solution was increased to 10 using LiOH. During the sorption and desorption experiments, samples were collected every day for the first week and later with an interval of three days per week. pH of the collected samples were noted (6.5 ± 0.2) and analysed for uranium and arsenic by using ICP-MS (XSeries 2, Thermo Fisher Scientific). To study the fate of sorbed U(VI) and As(V) on minerals, the mass balance

calculation was carried out by calculating the amount of U(VI) and As(V) sorbed and desorbed from the columns over the sorption and desorption periods.

Results and Discussion

U(VI)/As(V)/U(VI) – As(V) – SiO$_2$ Column

Figure 2 shows the transport and *BTC* of U(VI) and As(V) through columns packed with SiO$_2$. Arsenate has been identified in the outlet solution without any delay (from the 2nd day of the experiment) and showed practically no retardation in the transport behavior. As(V) has less or no affinity towards SiO$_2$ and has been reported elsewhere (Darland and Inskeep 1997; Xu et al. 1988). U(VI) movement was retarded in the U(VI) – SiO$_2$ column. The equilibrium *BTC* for U(VI) was reached after 200 pore volumes. The affinity of the U(VI) on silica is due to the hydrolysed uranyl species which are dominant at pH 6.5. More or less similar sorption as well as *BTC* for U(VI) and As(V) were observed (Fig. 2) for the experiment U(VI) – As(V) – SiO$_2$ column. There was no significant indication about the influence of uranyl-arsenate species in the transport of either U(VI) or As(V). Initially, DI water was pumped through the SiO$_2$ columns as part of desorption experiment. More than 65% of the sorbed U(VI) came through the outlet flow and shows that the sorbed surface species are not strong enough to retain on SiO$_2$ when compared with the other three minerals. Almost all U(VI) was recovered (~95%) from SiO$_2$ after pumping the solution with higher pH. Similar sorption behaviour of U(VI) and As(V) onto SiO$_2$ was observed in batch experiments.

U(VI)/As(V)/U(VI) – As(V) – Al$_2$O$_3$ Column

The sorption of uranyl and arsenate on Al$_2$O$_3$ and their transport behaviour are shown in figure 3. The retardation of U(VI) with Al$_2$O$_3$ is comparable to those of SiO$_2$ and reached the equilibrium after 350 pore volumes. This can be explained by the high reactivity of aluminol surface sites to U(VI) than the silanol surface sites in SiO$_2$ (Borovec 1981; Kohler et al. 1992). The sorption retardation of arsenate is more prominent than U(VI) and the *BTC* equilibrium was achieved after 650 pore volumes. The presence of arsenic oxy-anions (HAsO$_4^{2-}$, H$_2$AsO$_4^{-}$) enhances the sorption of arsenate on aluminol sites. The strong affinity of arsenate on alumina at acidic to neutral pH range has been studied and reported (Arai et al. 2001; Goldberg and Johnston 2001).
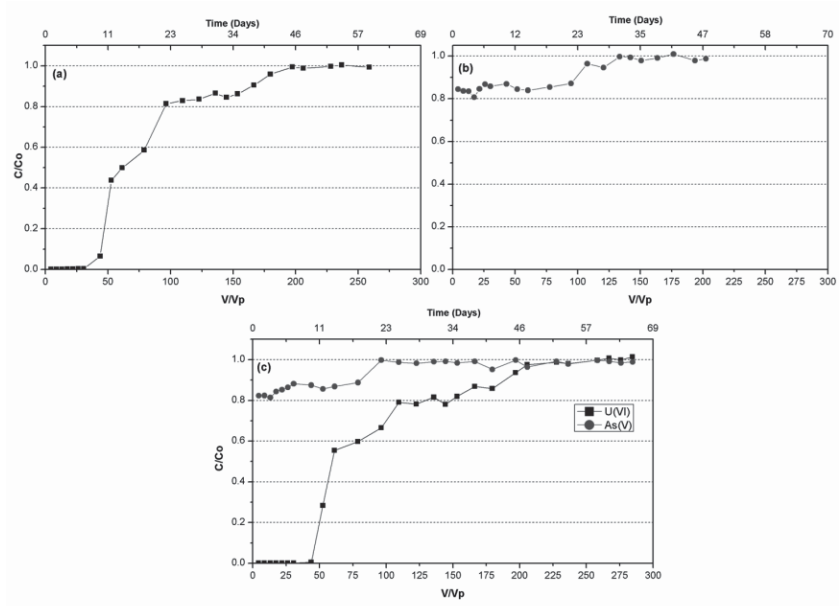
Fig.2. Experimental breakthrough curves (BTCs) of U(VI) and As(V) in SiO$_2$ columns. (a) U(VI) – SiO$_2$, (b) As(V) – SiO$_2$, (c) U(VI) – As(V) – SiO$_2$ [0.5 µM/l U, 0.5 µM/l As, 10 g SiO$_2$, pH 6.5, temp.:23°C].
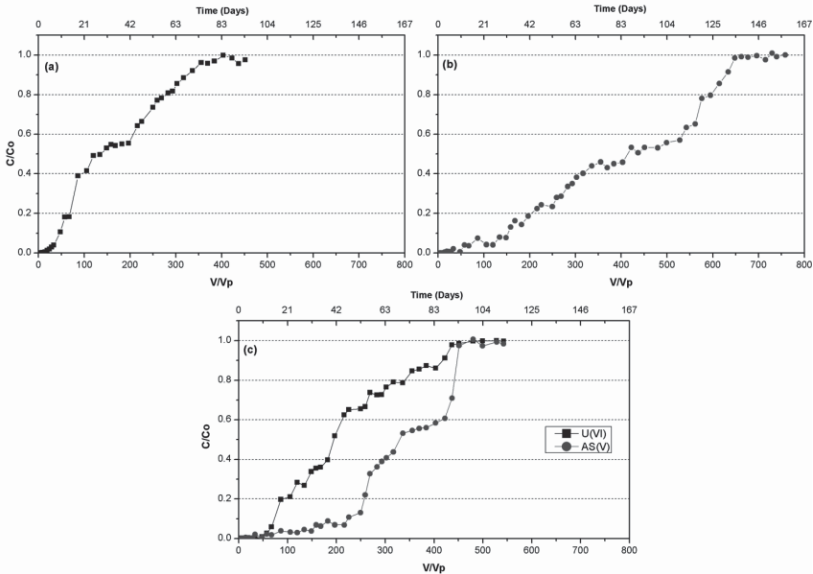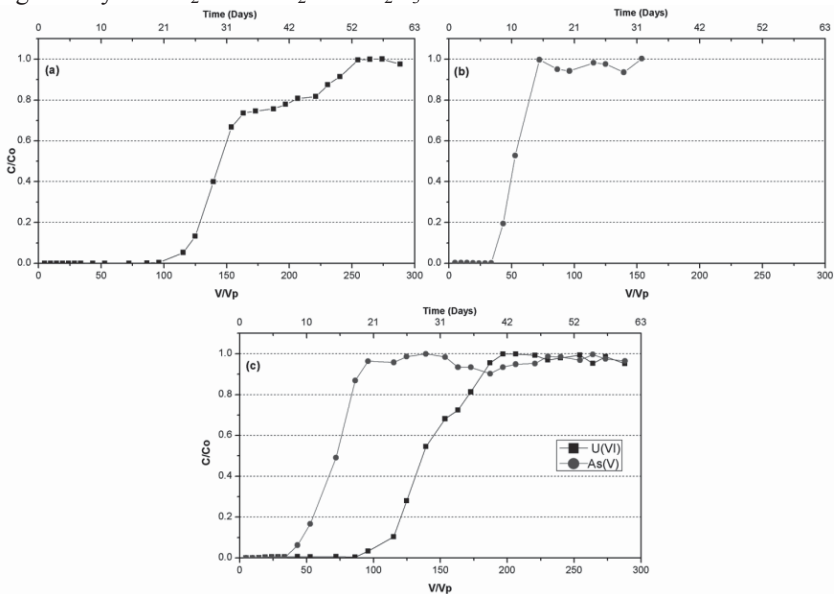

Fig.3. Experimental breakthrough curves (BTCs) of U(VI) and As(V) in Al$_2$O$_3$ columns. (a) U(VI) – Al$_2$O$_3$, (b) As(V) – Al$_2$O$_3$, (c) U(VI) – As(V) – Al$_2$O$_3$ [0.5 µM/l U, 0.5 µM/l As, 10 g Al$_2$O$_3$, pH 6.5, temp.:23°C].

The presence of both U(VI) and As(V) in the inlet solution changes the sorption behavior of both elements onto Al_2O_3. The sorption of U(VI) slightly increased and the *BTC* achieved the equilibrium after 450 pore volumes whereas the sorption of As(V) retarded and reached equilibrium similar to U(VI) (~450 pore volumes). In the initial stage of the transport, As(V) shows more retardation (up to ~250 V/Vp) and increased to reach the equilibrium. This retardation could be due to the better sorption of uranyl-arsenate species ($UO_2AsO_4^-$) compared to the $H_2AsO_4^-$ ion. The change in transport behavior of both elements is due to the competitive sorption between the uranyl and arsenate species or due to the formation of uranyl-arsenate complex. Almost 70% of the U(VI) and As(V) were desorbed from Al_2O_3. This indicates that the sorption is partially irreversible or slow desorption of both elements under the above said experimental conditions.

U(VI)/As(V)/U(VI) – As(V) – TiO₂ Column

Transport of U(VI) and As(V) through TiO_2 packed columns is shown in figure 4. The concentration of U(VI) was identified in the outlet after 100 pore volumes and reached the break through equilibrium after 250 pore volumes. U(VI) shows strong affinity to TiO_2 than SiO_2 and Al_2O_3.
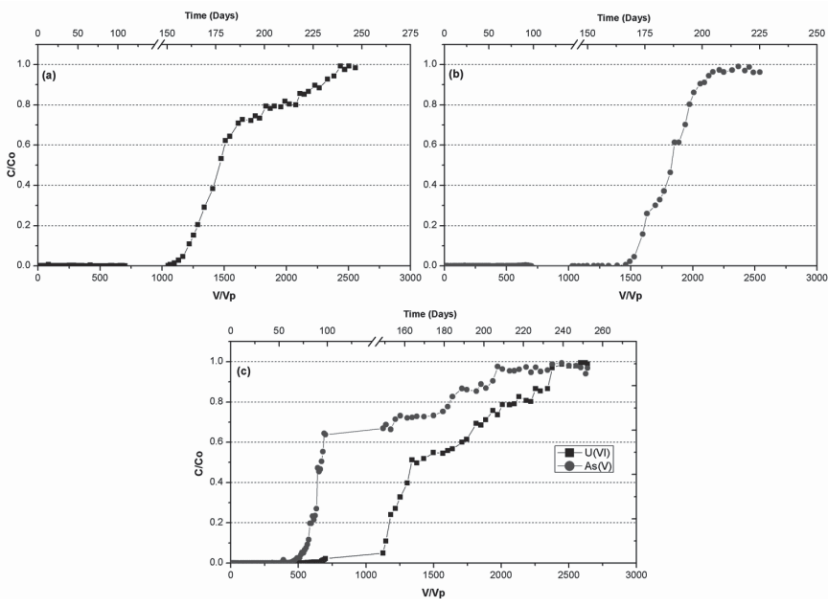

Fig.4. Experimental breakthrough curves (BTCs) of U(VI) and As(V) in TiO₂ columns. (a) U(VI) – TiO₂, (b) As(V) – TiO₂, (c) U(VI) – As(V) – TiO₂ [0.5 µM/l U, 0.5 µM/l As, 0.5 g TiO₂, pH 6.5, temp.:23°C].

The *BTC* of As(V) reached the equilibrium after 70 pore volumes and showed less affinity when compared with U(VI). Previous studies reported that As(V) shows higher sorption onto TiO_2 at higher pH and lower towards low pH values (Lee and Choi 2002; Dutta et al. 2004; Pena et al. 2005). When both elements were present in the inlet solution, the concentration of As(V) was detected initially (40 V/Vp) and is followed by U(VI) (100 V/Vp) in the outlet solution. As(V) showed *BTC* equilibrium at 70 pore volumes and at 200 pore volumes for U(VI). The sorption of As(V) on TiO_2 for both columns (As(V) – TiO_2 and U(VI) – As(V) – TiO_2) was almost similar, whereas U(VI) showed a slight decrease in the sorption behavior in U(VI) – As(V) – TiO_2 when compared with U(VI) – TiO_2 column. The recovery rate for uranyl and arsenate from all three columns were similar to that from Al_2O_3 (~70%) columns.

U(VI)/As(V)/U(VI) – As(V) – FeOOH Column

Experimental *BTC*s and the transport behavior of U(VI) and As(V) through goethite packed columns are presented in figure 5. The change in flow rate (from 100 μL/min to 250 μL/min) of the inlet solution can be seen in between ~700 V/Vp to ~1100 V/Vp in figure 5. Arsenate in the outlet solution was detected after 1500 pore volumes and reached the equilibrium at 2100 V/Vp. The retardation in transport of arsenate is due to the presence of $H_2AsO_4^-$ species, which sorbs effectively with the positively charged goethite surface. Arsenate has strong affinity to FeOOH and is previously reported elsewhere (Stollenwerk 2003). U(VI) concentration in the outlet was first detected at 1000 pore volumes. The transportation of uranyl was gradual and reached the equilibrium at 2400 pore volumes. The high sorption is attributed to the $(UO_2)_2CO_3(OH)_3^-$ species which is dominant at this pH range. Previous studies reported that U(VI) has great affinity to FeOOH over a wide range of pH (Sherman et al. 2008; Guo et al. 2009). The transport behavior of arsenate in U(VI) – As(V) – FeOOH column differs from that of As(V) – FeOOH column. As(V) started to appear in the outlet after 500 pore volumes and the *BTC* reached equilibrium at 2000 pore volumes. U(VI) has similar transport behavior as of U(VI) – FeOOH column and shows better sorption affinity to goethite than arsenate. The arsenate curve showed a sudden increase (at ~500 V/Vp) and followed a gradual movement of the element till the equilibrium. The sudden increase could be due to less sorption of arsenate species ($H_2AsO_4^-$) when competing with the uranyl species $((UO_2)_2CO_3(OH)_3^-)$. The gradual transport of arsenate after the sharp increase could be attributed to the better sorption of $UO_2AsO_4^-$ on the goethite surface. Uranyl ions showed better affinity to goethite than arsenate, even though the latter is known to be a good sorbing element. The recovered U(VI) and As(V) through the effluent solution was less than 20% and proved that the sorption is irreversible or desorption is very slow.

Fig.5. Experimental breakthrough curves (BTCs) of U(VI) and As(V) in FeOOH columns. (a) U(VI) – FeOOH, (b) As(V) – FeOOH, (c) U(VI) – As(V) – FeOOH [0.5 µM/l U, 0.5 µM/l As, 0.1 g FeOOH, pH 6.5, temp.:23°C].

Conclusions

Column experiments were conducted to study the transport of uranyl and arsenate, which revealed that the mobility of U(VI) and As(V) is highly dependent on the speciation of these elements as well as the sorbents. The immobilization of uranyl and arsenate with the aforementioned minerals are in the order of FeOOH>TiO_2>Al_2O_3>SiO_2. Transport behavior of U(VI) and As(V) through SiO_2 and TiO_2 packed columns are identical in the inlet solutions containing either U(VI) or As(V) separately, or both together. In the presence of equimolar U(VI) and As(V), a substantial increase in As(V) mobility and a slight decrease in U(VI) transport through Al_2O_3 were observed. While Al_2O_3 is replaced with FeOOH, a significant change in the pattern of mobility was shown by As(V); whereas U(VI) showed only minor changes. The changes in transport behavior of both elements can be attributed to the competitive sorption between uranyl and arsenate species or due to the formation of uranyl-arsenate species. Mass balance calculations indicated that the sorption of U(VI) and As(V) is strong with the above said minerals and the effectiveness of these minerals to retain the elements are in the order of FeOOH>TiO_2=Al_2O_3>SiO_2 under our experimental conditions. This study thus gives potential information about the transport behavior of uranyl and arsenate in natural systems, especially when both elements are present.

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