

## Influence of anionic species on uranium separation from acid mine water using strong base resins

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### Abstract

The presence of uranium and other elements in high concentrations in acid mine drainage at Poços de Caldas Uranium Mine (Brazil) is a matter of concern. The acid water pH is around 2.7, the uranium concentration is in the range of 6–14 mg L<sup>-1</sup>, sulfate concentration near 1400 mg L<sup>-1</sup>, fluoride 140 mg L<sup>-1</sup> and iron 180 mg L<sup>-1</sup>. In this solution, where sulfate is present in elevated concentrations, uranium is basically in the form of UO<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub><sup>4-</sup>. This study investigated the separation of uranium from the other anions present in the acid water under batch and column mode using ion exchange technique. The pH studied was 2.7 and 3.9. Two strong base anionic resins were tested. The influence of ions, commonly found in acid waters like sulfate and fluoride, on ion exchange process was also assessed. Equilibrium studies were carried out to determine the maximum adsorption capacities of the resins. The resins showed a significant capacity for uranium uptake which varied from 66 to 108 mg g<sup>-1</sup> for IRA 910U and 53 to 79 mg g<sup>-1</sup> for Dowex A. The results also showed that SO<sub>4</sub><sup>2-</sup> is the most interfering ion and it had a deleterious effect on the recovery in the pH range studied. Fluoride did not affect uranium removal.

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**Keywords:** Uranium; Ion exchange; Acid mine drainage; Sulfate; Fluoride

### 1. Introduction

Acid drainage, commonly referred to as acid mine drainage (AMD), has become an economic and environmental burden. It is a very ubiquitous problem in areas where there has been a history of coal or hard rock mining. This phenomenon is caused by sulfide oxidation that is generally associated with metallic ores and fossil fuels. The world's increasing demand for metals and fossil fuels also increased the amount of mining tailing and wastes, which leaves sulfide in nature in a state of perturbation. The result of this perturbation is sulfite oxidation which produces acid drainage often enriched with heavy metals [1,2].

In the southeast of Brazil, in the state of Minas Gerais (Poços de Caldas Municipality) a uranium mine generates acid water which contains radionuclides (uranium, thorium and radium) and other elements like manganese, zinc, fluoride and iron in concentrations above the permissible levels for discharging. The acid water is treated with lime to reduce the level

of contaminants and to comply with the recommended levels for discharging according to Brazilian legislation [18]. The current water treatment comprises the precipitation of the metals, including uranium, which generates a radioactive sludge that has to be disposed of. Removal and recovery of uranium from such acid water are imperative not only from the point of view of its economic value, but also to reduce its amount for disposal as radioactive waste as a consequence of water treatment [2].

Among the proven technologies for removing different types of dissolved substances, ion exchange process has been identified as one of the most appropriate for low levels of contaminants [2–6]. Ion exchange is suitable for the removal and/or recovery of most metals, including uranium. Although it allows the separation of uranium from aqueous solutions, the presence of competitive ions in concentrations much higher than uranium, generally decreases the operational loading capacity of the exchangers which is quickly exhausted [4,7].

Uranium uptake has also been investigated by adsorption on a variety of economically priced sorbents. Materials like zeolite apatite, bone char, calcined phosphate and oxy/hydroxides are examples of low price sorbents [8–11]. The main problems in using these materials are their relatively low adsorption capacity

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compared to the resins, the difficulty in removing the adsorbed uranium and the presence of impurities on the structure of the adsorbents. In addition, the use of these materials for uranium removal from effluents with high concentrations of different anions and cations, presents insufficient selectivity which is required for complex mixtures. According to Katsoyiannis [12] only anion exchange and titanium dioxide are able to remove uranium to below  $1 \mu\text{g L}^{-1}$ .

Uranium(VI) is present in oxic waters in hexavalent form mainly as uranyl ion ( $\text{UO}_2^{2+}$ ) or as hydroxyl complexes. However it is well known that it forms stable complexes with a large variety of ligands such as carbonates, phosphates and sulfates. The majority of the studies for uranium removal are based on the presence of species like  $\text{UO}_2(\text{CO}_3)_2^{2-}$  and  $\text{UO}_2(\text{CO}_3)_3^{4-}$  in groundwater. In acid mine drainage, where stable anionic uranium complex is mainly present as  $\text{UO}_2(\text{SO}_4)_3^{4-}$ , the literature is scarce [5,13–17].

The motivation for this study was the need to separate uranium from the other contaminants in order to: (i) minimize the generation of radioactive sludge, (ii) prevent human contamination due to uranium chemical toxicity and (iii) provide funds for the mine rehabilitation through the recuperation of uranium. The present work investigated the sorption affinity, selectivity and adsorption capacity of two SBA resins, such as IRA 910U and DOWEX A, for naturally occurring uranium. The liquid sample used was a real acid mine water. In addition, studies were carried out to investigate the effect of some competitive ions on the uranium sorption process such as sulfate and fluoride.

## 2. Materials and methods

### 2.1. Anion-exchange resins

Two commercial strong-base anion exchange resins were obtained from Rohm and Haas (IRA 910U) and Dow Chemical Company (Dowex Marathon A). The resins were activated with  $\text{NaCl } 1 \text{ mol L}^{-1}$  and rinsed with distilled water before use. The resin IRA 910U is type 2, benzyldimethylethanolammonium group, chloride form, macroreticular polystyrene–DVB polymer, theoretical capacity of  $1.0 \text{ equiv. L}^{-1}$  ( $\text{Cl}^-$  form) and specific for uranium recovery. The resin Marathon Dowex A is type 1, trimethylbenzylammonium group, styrene-DVB, chloride form, gel, theoretical capacity of  $1.3 \text{ equiv. L}^{-1}$  ( $\text{Cl}^-$  form).

### 2.2. Sample

The liquid sample was collected nearby the uranium mine in the southeast of Brazil (State of Minas Gerais, Poços de Caldas Municipality) and consists of acid water generated at waste rock piles. The water was chemically and radiochemically characterized and the analyses are shown in Table 1.

### 2.3. Batch adsorption experiments

The experiments were carried out by varying the mass of adsorbent in a range of  $0.075\text{--}0.5 \text{ g}$  (dry basis) and the solution volume was fixed in  $500 \text{ mL}$ . The suspension was shaken

Table 1  
Chemical and radiochemical analyses of the acid mine water

Determination	Acid mine water <sup>a</sup>	Permissible level
U	12.0	0.02
Th	0.8	b
<sup>226</sup> Ra	$3.5 \text{ Bq L}^{-1}$	b
Mn	173.0	1.0
Ca	158.0	b
Mg	8.9	b
Al	170.4	b
Zn	41.0	5.0
Fe	180	15.0
$\text{SO}_4^{2-}$	1400	b
$\text{F}^-$	110	10.0
$\text{SiO}_2$	57.0	b
pH	2.7	6.0–9.0

<sup>a</sup> Unless when indicated units are expressed in  $\text{mg L}^{-1}$ , except pH.

<sup>b</sup> Permissible level not defined by Brazilian legislation CONAMA 357/2005 [18].

at 150 rpm for 24 h, at room temperature ( $25 \pm 0.5^\circ\text{C}$ ). The pH values adopted were 2.7 (natural acid water pH) and 3.9, adjusted with lime. After a period of 24 h, the suspension was filtered, and the solution assayed for uranium. Each data point corresponds to a separate batch test. Uranium uptake was obtained by determining the metal concentrations before and after contact with the resin. To determine the distribution coefficient,  $K_D$  ( $\text{mL g}^{-1}$ ),  $0.05 \text{ g}$  of resin were shaken with  $1000 \text{ mL}$  of uranium solution in a sealed erlenmeyer for 24 h. The exchanger was then separated from the solution by filtering and the filtrate was analyzed for uranium. The distribution coefficients were estimated by the ratio of the metal ion concentration in the resins to that in solution after sorption in the following way:  $K_D = ((C_0 - C)/C) \times V/m$ , where  $C_0$  and  $C$  are the uranium concentration in solution before and after contact;  $V$  the solution volume ( $\text{mL}$ ) and  $m$  is the dry resin mass ( $\text{g}$ ) [13,14,17].

### 2.4. Column experiments

Trials were carried out in a glass column with a  $1.2 \text{ cm}$  inner diameter which operated with the acid water sample at pH 2.7 and 3.9 which was adjusted with lime, and at temperature  $25 \pm 0.5^\circ\text{C}$ . Five ( $5 \text{ mL}$ ) of each resin ( $1.6 \text{ g}$  of IRA 910U and  $1.9 \text{ g}$  of Dowex A) dry basis, previously treated with  $\text{NaCl}$ , were wet-packed into distinct glass columns. The operation was performed by downstream flow at a constant flow rate of  $2 \text{ mL/min}$  ( $24 \text{ BV/h}$ ) using a peristaltic pump. Samples were collected periodically from the column effluent and analyzed to determine uranium, sulfate and fluoride content.

### 2.5. Chemical analyses

Uranium determination was by the neutron activation technique which consists of exposing a chemical element to a neutron flux producing a new radioactive nuclide. The irradiation was performed in the Triga Marki IPR-R1 reactor. The radioactive nuclide measurement was carried out after 2 days of cooling based on  $^{239}\text{Np}$  nuclide. The detection limit of this technique

is  $1 \mu\text{g mL}^{-1}$ . Potentiometric determination, by ion selective electrode method, was used for fluoride analyses. The detection limit of the method is  $0.1 \text{ mg L}^{-1}$ . Sulfate was determined using barium sulfate turbidimetric method [19,20].

### 3. Results and discussion

#### 3.1. Batch adsorption experiments

Uranium in acid solution is basically in the form of  $\text{UO}_2^{2+}$  but the presence of a high content of sulfate ( $1.4 \text{ g L}^{-1}$ ) enhances complexes formation such as  $\text{UO}_2(\text{SO}_4)_3^{4-}$  which is the predominant species in the present solution. The following equations represent the equilibrium between sulfate and uranium species in acid medium [16,17]. The constants  $K_1$ ,  $K_2$  and  $K_3$  are called equilibrium or stability constants and provide a measure of the stability of the respective complexes. A large equilibrium constant indicates a highly stable complex:



The influence of pH was studied in batch experiments. The pH values adopted for uranium removal were 2.7 (original pH) and 3.9, adjusted with lime. The increase in pH above 4.0 causes the uranium precipitation as calcium diuranate and this fact restricted the maximum pH adopted in this investigation to 3.9 [2]. Fig. 1 shows uranium uptake by the two resins investigated. Uranium equilibrium concentration ( $\text{mg L}^{-1}$ ) is plotted against uptake by the resins ( $\text{mg U g}^{-1}$  resin).

The maximum loading capacity ( $Q_{\text{max}}$ ) of the resins was determined by adjusting the experimental data to the Langmuir type equation (solid lines) [21]. Table 2 shows the  $Q_{\text{max}}$  and also the distribution coefficients at pH 2.7 and 3.9.

The pH slightly influences the sorption of uranium by the resins. Loading capacity at pH 3.9 presented by both resins

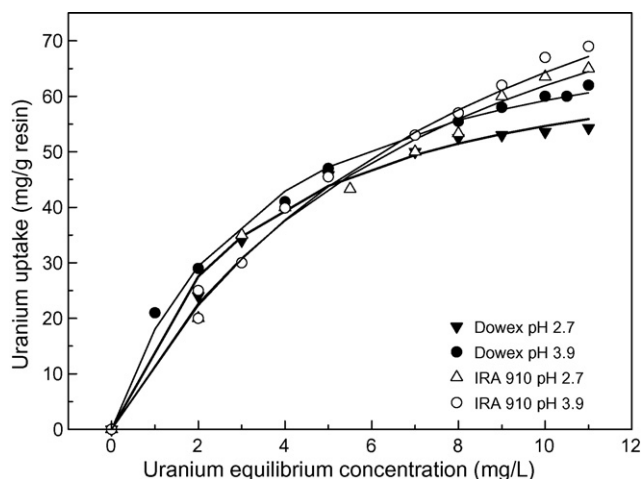


Fig. 1. Uranium isotherms for resins IRA 910U and DOWEX at pH 2.7 and pH 3.9,  $T = 25 \pm 0.5^\circ\text{C}$ . Solid lines represent the fit to the Langmuir equation.

Table 2

Maximum loading capacities and distribution coefficients for uranium

Resin	$K_D$ ( $\text{mL g}^{-1}$ )		$Q_{\text{max}}$ ( $\text{mg U g}^{-1}$ resin)	
	pH 2.7	pH 3.9	pH 2.7	pH 3.9
IRA-910U	6667	6887	100	108
Dowex-A	5889	6435	72	79

is no more than 10% higher than the loading capacity at pH 2.7. According to batch experiments, IRA 910U is the most suitable material due to the highest values for  $Q_{\text{max}}$ , around  $100 \text{ mg g}^{-1}$  ( $0.54 \text{ equiv. L}^{-1}$ ) compared to the values around  $75 \text{ mg g}^{-1}$  ( $0.46 \text{ equiv. L}^{-1}$ ) presented by the resin Marathon Dowex-A. The  $Q_{\text{max}}$  values determined correspond to the operational capacity of the resins. The operational capacities in the present study are less than the theoretical values,  $1.0 \text{ equiv. L}^{-1}$  for IRA 910U and  $1.3 \text{ equiv. L}^{-1}$  for Dowex A, which means that competitive ions also take ion exchange sites. Nascimento et al. [2] studied uranium removal from a similar acid water sample by using a strong base anion resin and the operational capacity determined was  $0.24 \text{ equiv. L}^{-1}$  of the complex  $\text{UO}_2(\text{SO}_4)_3^{4-}$ . That value is lower than the values obtained in the current study which confirm the best selectivity of the present resins.

The values of the  $K_D$  at pH 3.9 for the two resins are slightly greater than at pH 2.7. However, they are still smaller than the values reported in the literature. Gu et al. [5], determined the  $K_D$  for  $0.06 \text{ mM U(VI)}$  in the presence of  $10 \text{ mM}$  sulfate at pH 8.5. The distribution coefficients calculated for five polystyrenic strong base anion exchange resins with trialkylammonium group varied from 30,200 to 146,800. At that pH, the author considered that the predominant anionic species were  $\text{UO}_2(\text{CO}_3)_2^{2-}$  and  $\text{UO}_2(\text{CO}_3)_3^{4-}$  and the presence of sulfate does not interfere on the process. Vaarama et al. [13] determined uranium  $K_D$  for strong base resins using drinking water at pH 8.0 in the presence of  $39.4 \text{ mg L}^{-1}$  of sulfate. They observed that  $K_D$  decreased with the increase of uranium concentration from  $0.054$  to  $12.4 \text{ mg L}^{-1}$  and with the decrease of pH. The latter behavior is due to the alteration of uranium speciation with lower pH which favors the predominance of cationic uranyl ion. Although there was a decrease in  $K_D$  in lower pH (around 5), it was still higher than ten thousand, which indicates a fairly high processing capacity. The great majority of the investigations are carried out with alkaline pH and the predominance of uranyl carbonate species.

According to Table 1 the main competitive ions in acid water are  $\text{SO}_4^{2-}$  and  $\text{F}^-$ . Regardless of the high anionic concentrations, mainly  $\text{SO}_4^{2-}$ , IRA 910U and Dowex A readily adsorbed all uranium from the solution. This behavior is especially true for strong base anionic resins. The good performance showed by strong basic anionic resins may be attributed to the relatively high affinity to uranium even though the  $\text{SO}_4^{2-}$  concentrations are orders of magnitude higher than the concentration of uranium in solution. In addition, higher valence ions like  $\text{UO}_2(\text{SO}_4)_3^{4-}$  are preferred because the electrostatic attraction is proportional to the ionic charge which implies that complex formation enhances the uptake of the metal species by the anion exchanger [21].

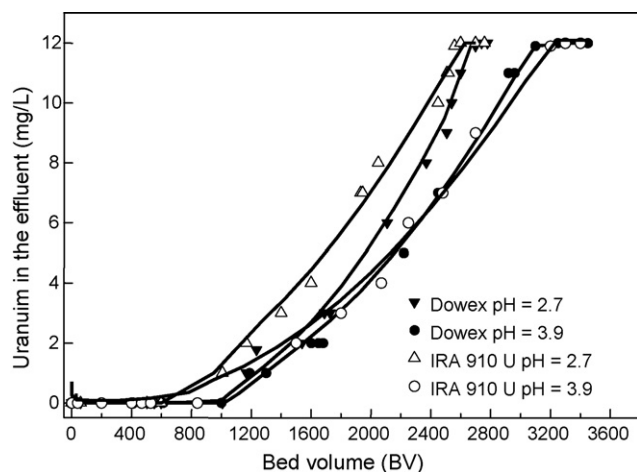


Fig. 2. Adsorption profile for uranium at different pH. Flow rate =  $2 \text{ mL min}^{-1}$ ,  $T = 25 \pm 0.5^\circ \text{C}$ . Bed volume = volume of processed acid water.

### 3.2. Continuous adsorption experiments

Column experiments were carried out to determine the performance of the exchangers and compare with batch experiments. Fig. 2 shows the adsorption profiles for uranium at pH 2.7 and 3.9 and flow rate of  $2 \text{ mL min}^{-1}$  where the concentration of the contaminant ( $\text{mg L}^{-1}$ ) is plotted against cumulative bed volume (BV). Bed volume refers to the volume of solution equivalent to the resin volume in place. The flow rate was set at  $2 \text{ mL min}^{-1}$  according to preliminary investigation with different flow rates of 1, 2 and  $4 \text{ mL min}^{-1}$  (data not shown). The loading capacities were calculated by integrating the area above the curves in Fig. 2, and they represent the maximum amount of solute the column can store. The calculated loading capacity for uranium at pH 3.9 was  $65 \text{ mg g}^{-1}$  ( $0.41 \text{ equiv. L}^{-1}$ ) for Dowex A and  $79 \text{ mg g}^{-1}$  ( $0.43 \text{ equiv. L}^{-1}$ ) for IRA 910U. At pH 2.7 these values were  $53 \text{ mg g}^{-1}$  ( $0.33 \text{ equiv. L}^{-1}$ ) for Dowex A and  $66 \text{ mg g}^{-1}$  ( $0.36 \text{ equiv. L}^{-1}$ ) for IRA 910U. All the loading capacities are slightly lower than the ones obtained by batch tests (Table 2). The maximum loading capacity depends on the saturation of the resin. When saturation is achieved there is the formation of the plateau which is typical of Langmuir type equation. In this study the loading capacities for the batch tests were overestimated because, as one can see in Fig. 1, the saturation was not completely achieved under the experimental conditions applied since the plateaus were not completely defined. The higher capacity presented for IRA 910U, although not so expressive, may be related to the difference between the groups responsible for the ion exchange. Similar behavior was presented by the resins at different flow rates (data not shown).

The pH effect on the loading capacity for column experiments was more accentuated than that for batch tests. This fact may be correlated with the formation of  $\text{Fe}(\text{OH})_3$  in the column when operating at pH 2.7, as a consequence of the low solubility product of iron(III), which may contribute to the decrease of the loading performance. At pH 3.9 the acid water was free of iron as it was precipitated as iron hydroxide during pH adjustment. The pH effect, shown in Fig. 2, may be correlated with

the breakthrough volume, e.g. the volume fed until the uranium concentration in the column effluent is equal to  $1 \text{ mg L}^{-1}$ . The breakthrough volume at pH 3.9 is higher for the two resins and equal to 1000 BV. This means that around 5 L of acid water may be processed before breakthrough point is achieved. At pH 2.7 the breakthrough volume is around 500 BV for both resins which corresponds to 2.5 L processed. Vaarama et al. [13] studied the removal of uranium from drinking water by using aminophosphonate and SBA resins. They concluded that, at pH 5, aminophosphonate resin works much better than SBA. For aminophosphonate resin, no breakthrough of uranium was observed up to 2000 BV ( $1 \text{ BV} = 7.85 \text{ mL}$ ) which corresponds to nearly 16 L of solution processed. Despite the better processing capacity found by the aminophosphonate exchanger, the presence of the species  $\text{UO}_2(\text{SO}_4)_3^{4-}$  makes the SBA resins more suitable for acid water systems.

Assuming that the charge of uranium complex is  $4^-$ , the theoretical exchange capacities of the exchangers for uranium are:  $1 \text{ equiv. L}^{-1} \times 0.25 \text{ mol U/equiv.} \times 0.005 \text{ L} = 1.25 \times 10^{-3} \text{ mol U}$  for IRA 910 and  $1.3 \text{ equiv. L}^{-1} \times 0.25 \text{ mol U/equiv.} \times 0.005 \text{ L} = 1.63 \times 10^{-3} \text{ mol U}$  for Dowex A. Theoretically, approximately  $185 \text{ mg U g}^{-1}$  of IRA and  $200 \text{ mg U g}^{-1}$  of DOWEX A can be adsorbed. Considering the operational capacity of the resins as being 79 and  $65 \text{ mg g}^{-1}$ , respectively, this means that around 43% and 32% of the theoretical capacity was used. Huikuri and Salomen [15] reported that only 5.4% of SBA exchanger were used during uranium removal from groundwater.

Due to the dominance of the sulfate and fluoride anions in the acid water the sorption of  $\text{SO}_4^{2-}$  and  $\text{F}^-$  was monitored during the column experiments at pH 3.9. Fig. 3 shows that the two resins do not retain the fluoride and that the values determined in the effluent are within the analytical error (5%). No extraction of fluoride was observed in any case, thus underlining the high selectivity of the resins. Ladeira and Morais [4] studied the influence of fluoride on the uranium uptake by the resin IRA 910U in a carbonate medium where uranium is present as  $\text{UO}_2(\text{CO}_3)_3^{4-}$ . The authors concluded that uranium uptake was inhibited in the presence of fluoride concentrations above  $5.0 \text{ g L}^{-1}$ . The present work corroborates with this statement since the fluoride

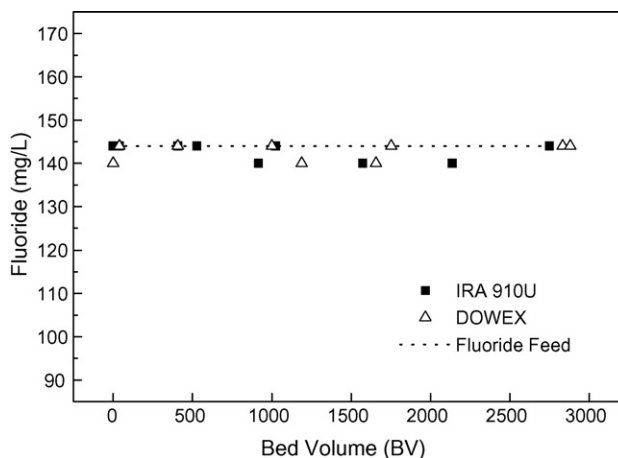


Fig. 3. Adsorption profile for fluoride. Flow rate =  $2 \text{ mL min}^{-1}$ ,  $T = 25 \pm 0.5^\circ \text{C}$ , pH 3.9. Bed volume = volume of processed acid water.



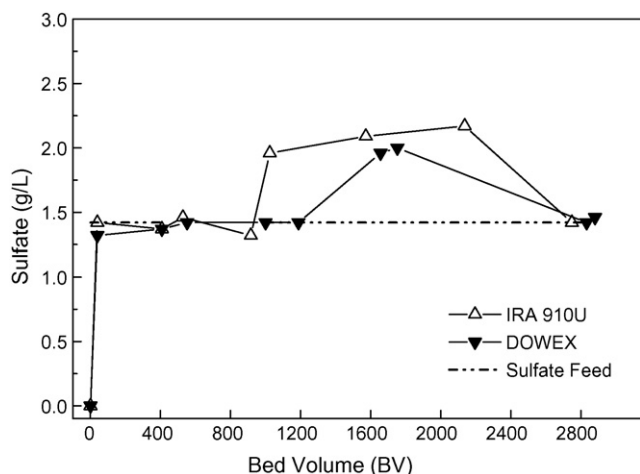


Fig. 4. Adsorption profile for sulfate. Flow rate =  $2 \text{ mL min}^{-1}$ ,  $T = 25 \pm 0.5^\circ \text{C}$ , pH 3.9. Bed volume = volume of processed acid water.

concentration in this study is around  $149 \text{ mg L}^{-1}$  and much lower than the limit suggested by the aforementioned authors.

Fig. 4 shows that sulfate is the most competitive ion for uranium. During the first contact of the solution with the resins all the sulfate was adsorbed, e.g. sulfate in the effluent solution was not detected in bed volume number one despite being present in the feed in concentrations around  $1.4 \text{ g L}^{-1}$ . Up to 900 BV, sulfate content in the column effluent remains constant and equal to the feed. From this point on, the resins released sulfate and the content in the effluent solution was greater than in the feed. This may be, in part, due to the sulfate displacement by the uranium. The resin IRA 910U released more sulfate than DOWEX A and consequently loaded more uranium, as demonstrated by its higher loading capacity ( $79 \text{ mg g}^{-1}$ ). Although both polymeric resins are appropriate for acid water treatment, the benzyl-dimethyl ethanolammonium group responsible for IRA 910U loading capacity is slightly more suitable for uranium recovery than the trimethylbenzylammonium group because of its higher selectivity. Due to the high content of competitive anions the selectivity of the resins is considered in this study as the most important factor.

In this study it has been demonstrated that ion exchange is technically efficient to extract uranium from the acid mine. Assuming an efficiency of the ion exchange process of 90%, it could be determined that 27 t of  $\text{U}_3\text{O}_8$  per year can be extracted from the acid water. At the moment, this amount of uranium is being disposed of as waste and it represents a total of US\$ 918000.00 a year considering the uranium price of US\$ 34.00 kg. Fernandes et al. [22] reported that 27 t of uranium correspond to 30% of the annual average uranium production of the Poços de Caldas mine before it was closed. In terms of economic feasibility, they estimated that the cost for an ion exchange plant would be around US\$ 1600.00. The former authors have also pointed out that, as the leaching plant will be replaced by the natural process of acid water drainage and the precipitation plant is already available at the Poços de Caldas facility, the capital cost involve the ion exchange plant only. This statement contributes to rein-

force the fact that ion exchange can be an acceptable practice not only in terms of radiological and toxicological dose reduction but also in economic terms.

#### 4. Conclusion

SBA resins seem to be very attractive sorbents for uranium since recovery is important from the economic point of view and its removal is necessary to minimize the generation of radioactive wastes. Batch and column experiments were carried out and the maximum sorption capacity of two strong base resins for uranium was in the range of  $65\text{--}79 \text{ mg g}^{-1}$ . These values showed that the operational capacities of the resins were 30–40% of the theoretical values which means that adsorption of competitive ions like sulfate are occurring. Based on these studies carried out with a real acid water sample it was demonstrated that, although loadings are not considered so high, uranium can be removed efficiently, and elevated recoveries may be achieved. Elution experiments are under investigation.

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