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ABSTRACT

The utility of macroporous cation-exchange resins for the separation of uranium from thorium (relevant to the final purification of the product uranium after reprocessing of the thorium breeder fuel) was examined. Two such cation-exchange resins were studied and compared with a previously used gel-type resin. Batch experiments and column experiments were performed to generate equilibrium data and to optimize the procedure for the separation of U from Th under process conditions. Recovery and purity of the final product were the same while loading and washing rates were much higher (120 mL/h) than those used for gel-type resins (40 mL/h). Much higher throughput was achieved without losing product quality when macroporous resins were used instead of the gel-type resin.

Key Words. Cation-exchange resin; Macroporous resin; Separation of U from Th; Purification of U; Distribution ratio; Thorium breeder; Fuel reprocessing

INTRODUCTION

Capture of a neutron by ^{232}Th followed by two successive β decays gives ^{233}U, a fissile isotope of uranium. This forms the basis of the utilization of the large reserve of thorium in power reactors through breeder technology. After neutron irradiation of aluminum-clad thorium metal or oxide rods

in a nuclear reactor, the fissle ^{233}U formed from ^{232}Th must be recovered by chemical processing. At BARC, this was accomplished (1) in three main steps: 1) chemical decladding by dissolving aluminum in NaOH (containing $NaNO_3$ to prevent hydrogen evolution), 2) dissolution of the metal or the oxide in HNO_3 (in the presence of 0.05 M HF to catalyze the dissolution reaction), and 3) solvent extraction process for the separation of ^{233}U from the bulk of Th. The procedure generally adopted for the reprocessing of the nitric acid solution of the irradiated fuel using tributyl phosphate (TBP) as the extractant (Thorex Process) depends on whether only ^{233}U or both ^{233}U and Th are to be recovered. In the initial phase of the program, recovery of only ^{233}U was planned. On the basis of the distribution data collected on the relevant components, two extraction schemes were proposed (2) for the recovery of ^{233}U as U(VI) extracted in 5% TBP in Shell Sol-T (a kerosene type of solvent) as diluent. U(VI) extracted preferentially in the organic phase was then scrubbed with nitric acid to remove the coextracted Th(IV). The concentration of HNO_3 in the feed and scrub were 4 and 2 M, respectively. The schemes were tested with 20-stage air-pulsed mixer-settler units (12 to 14 stages for extraction and 6–8 stages for scrubbing). The flow ratios of feed(aq):extractant-(org):scrub(aq) in two schemes were 1:2:0.6 and 1:1:0.3. A loaded organic containing U with only 5–10% Th could be obtained when the aqueous feeds contained 200 g/L Th with 0.12% U. Finally, U(VI) was stripped from the organic phase with water. However, various constraints were encountered while operating the pilot plants during processing, and the uranium product contained a significant amount of Th as impurity (often referred to as contamination). The content varied from 20 to 100% of U depending on the process conditions.

The final purification (polishing) of U at the tail end of reprocessing was achieved by anion exchange in 8 M HCl. U(VI) forms an anionic chloro complex which is absorbed strongly on the anion-exchange resin, leaving Th(IV) in the effluent. However, as nitrate ions interfere seriously, it is necessary to remove nitrate completely from the feed solution for the ion-exchange process. This method of purification involves several steps such as evaporation of the product solution, precipitation with ammonium hydroxide, washing the precipitate to remove nitrate, and finally dissolution of the precipitate in concentrated HCl to obtain feed in an 8 M HCl medium. Though the purification is excellent, the method has certain disadvantages like corrosion of equipment, poor removal of iron, gassing in the ion-exchange column (3), etc. Various attempts were made to overcome these difficulties. Acetate was tried as an alternative to chloride (4) as acetic acid also forms a strong anionic complex with U(VI). Th(IV) and U(VI) were first precipitated as hydroxide using NH_4OH, washed

free from nitrate ions and suspended in water (200 mL/g Th). Acetic acid (50%) was then added to a pH of 2.5 (2–3 M free acetic acid). An anion-exchange column of Dowex 1×4 (acetate form) was loaded up to 60% capacity and washed with 2 M CH_3COOH to remove Th(IV). U(VI) was eluted using 0.5 M HNO_3 to obtain a product with less than 0.1% Th content. However, this method also involved several steps to obtain the feed for the anion-exchange purification. A sequential precipitation technique was tried (5). Th(IV) was precipitated from a 1 M HNO_3 medium as oxalate by slow addition of 10% aqueous oxalic acid solution (up to an excess of 0.1 N free oxalic acid concentration), filtered, and washed with 0.5% hot (60°C) oxalic acid solution. The filtrate and washings were collected, ammonium nitrate was added (40 g/L), the mixture was heated to 90°C, and hot concentrated (25%) ammonia solution was added (up to pH 9.5 ± 0.3) to precipitate U(VI) as ammonium diuranate (ADU). The precipitate was washed with a 2% solution of NH_4NO_3 containing a little NH_4OH. The ADU cake was dried and heated for 4 hours at 760°C for conversion to oxide. Detailed studies on various parameters indicated optimum acidity at about 1 M and a feed U concentration of less than 10 g/L to minimize loss of uranium in the precipitation stage. Product purity was satisfactory.

In order to develop a more convenient alternative, an ion-exchange separation method directly from the nitric acid process solution was tried. Anion exchange in nitric acid medium tried earlier did not give satisfactory results in column experiments (6). A cation-exchange procedure using a gel-type cation exchanger was studied in detail (7). It involved preferential absorption of Th(IV) from 0.5 M HNO_3 feed containing U(VI) and Th(IV), and it gave an effluent and elution (or wash) stream containing more than 99% U(VI) almost free of Th(IV). A satisfactory separation could be achieved by careful manipulation of feed and elution acidities and by controlling the thorium loading to within 60–80% of the column capacity. This method was of great advantage as the U product solution from the reprocessing stream could be directly fed to the column. But, as expected with gel-type resins, the ion-exchange process was rather slow. In order to process a large volume of the process solution in the shortest possible time, it was necessary to look for a faster cation-exchange method.

Macroporous ion-exchange resins (8) have a very large surface area (up to 800 m^2/g) and characteristic properties (high microvoid volume ratio, rapid ion-exchange kinetics, easily accessible sites, low pressure drops, etc.) that increased the chemical accessibility and the mass transfer of ions and large molecules (9) to a great extent. These resins were investigated by various authors for the actinide separation processes and were reviewed by Ramakrishna and Patil (10). Macroporous resins of coarse size are

particularly suitable for fast separation as the pressure drop in the ion-exchange column is also much reduced. Therefore, the possibility of using these resins for relatively fast separation and purification of U from Th was explored. Two such cation-exchange resins were taken for studies along with the gel-type resin used by the earlier worker mentioned above. Distribution ratios and breakthrough capacities were measured, and decontamination factors in column operations under certain condition were determined for varying Th(IV) to U(VI) ratios to optimize a procedure for obtaining U with as little Th impurity as possible. The procedure to be developed was aimed at retaining Th(IV) on the column and collecting U(VI) in the effluent and wash solution.

REAGENTS AND CHEMICALS

Ion-Exchange Resins

Amberlyst-15 (16–50 mesh) and Tulsion T-42 (15–50 mesh), both macroporous cation-exchange resins in the H^+ form (hereafter referred as A-15 and T-42) were obtained from Rohm and Haas, France, and THERMAX, India, respectively. Dowex 50×8, a gel-type resin in H^+ form and of 100–200 mesh size, was obtained from J. T. Baker Chemical, USA. Dowex 1×4 (50–100 mesh) in Cl^- form was obtained from Dow Chemical Company, USA. The resins were thoroughly washed with water and air-dried for several days.

Thorium and Uranium Stock Solutions

Stock solutions of Th(IV) and U(VI) in dilute HNO_3 were prepared from thorium nitrate and uranium oxide (as U_3O_8), obtained from Indian Rare Earths, Bombay, and Uranium Metal Plant, BARC, Bombay, respectively. Th(IV) concentration was determined (11) by EDTA titration and U(VI) concentration by redox titrimetry using the Davies–Gray method (12) as modified by Eberle et al. (13). Dilute solutions containing required concentration of Th(IV), U(VI), or their mixtures were prepared by evaporating known volumes of the stock solutions and dissolving them in the desired quantity of nitric acid.

All chemicals or reagents used were of A.R. or G.R. grade.

EXPERIMENTAL

Batch Experiments

Distribution ratios of U(VI) and Th(IV) were measured separately after equilibrating 20 mL of the aqueous solutions of U(VI) or Th(IV) with

~100 mg of the air-dried resins (in duplicate) in a thermostatic bath at 23 ± 0.1°C. The equilibration times for A-15 and T-42 were found to be 3 and 4 hours, respectively. However, a shaking time of 5 hours was used. From the initial and equilibrium metal ion concentration in the aqueous phase, the distribution ratios (D) as the ratio of equilibrium metal ion concentration in the resin (mg/g) to the concentration (mg/mL) in the aqueous phase were calculated.

Analysis of U(VI) and Th(IV)

U(VI) was determined by potentiometry as above. In the absence of U(VI), milligram amounts of Th(IV) were determined by EDTA titration using Xylenol Orange as indicator. Microgram amounts were determined by spectrophotometry (14) of Th(IV)-Arsenazo(III) after anion-exchange separation (15) using Dowex 1×4 resin in 6 M HCl medium.

Column Experiments

Column experiments were performed using a Corning glass column fitted with a stopcock at the bottom and a cup at the top. Flow rates were maintained almost constant by maintaining a constant column head pressure. Flow rates for A-15 and T-42 were 120 mL/h, while the flow rate was around 40 mL/h for Dowex 50×8 as used in earlier work (7). The high flow rate of 120 mL/h was difficult to achieve with the Dowex 50 column as Th(IV) loading progressed, presumably due to compacting of the resin. Capacities of the resins were determined by passing a 0.5 M NaCl solution and titrating the acid released with standard alkali. Columns of (9.5 ID × 140 H) mm size with 10 mL of the resin (aspect ratio 14.7) conditioned with 10 bed volumes (100 mL) of 0.5 M nitric acid medium were used for all breakthrough studies for Th(IV) as well as for thorium–uranium mixtures.

Breakthrough data indicated 1% breakthrough of Th at around 55 bed volumes. Therefore, for the study of the decontamination factor (DF) an effluent quantity up to 10 bed volumes less than that was collected in one lot and thereafter 5–10 bed volumes in separate lots were collected while maintaining a flow rate of 120 mL/h. The decontamination factors under the experimental conditions were obtained on dividing the ratio of Th to U concentrations in the feed (r_1) by the ratio of Th to U concentrations in the effluent (r_2).

Th(IV) was held up in the column and U(VI) passed out in the effluent and washings. A number of column runs were carried out to arrive at the conditions whereby more than 90% U could be obtained in the effluent and the wash solution as a product with the least Th content. Resin A-15

was used for all the exploratory work. Only those column runs which were found optimum with A-15 were repeated with T-42. Generally the feed composition was a mixture containing 1 mg/mL each of Th and U, the flow rate was 5 min/bed volume, the resin volume was 10 mL, and the medium was 0.5 M HNO_3 unless stated otherwise. Some column experiments were performed to find the effect of the concentration of HNO_3 in the wash solution. Fifty-five bed volumes of feed corresponding to about 50% of the capacity utilization were passed through identical columns and washed with 10 bed volumes of either 0.5, 1.0, or 2.0 M HNO_3. In some other column runs, after passing the feed solution, washing with 0.5 M HNO_3 was continued until more than 90% U was reported in the effluent plus wash. For each run, 10 bed volumes of the wash solution were collected separately and analyzed for U and Th. In one run the loading and washing rates were increased from 5 to 2 min/bed volume.

Scaled-up Column Operation

Some column runs were carried out using larger (22 ID × 260 H) mm columns with 100 mL of the resins (aspect ratio 11.8) while keeping the feed composition and medium the same. Washing was reduced from 50 to 30 bed volumes. The flow rates used were 5 and 8 min/bed volume, i.e., 1.2 and 0.75 L/h. One column operation with about 30% capacity utilization was carried out to see if there was any advantage regarding recovery or purity of the product U.

Regeneration of the Resin Column

The bulk of Th (along with residual U) was eluted from the resin by washing it with 10–12 bed volumes of 6–7 M HNO_3 (to be recycled in the dissolver solution). Any remaining Th was then removed by using a 2:1 mixture of 3 M ammonium acetate and 1.5 M acetic acid (pH 5.5). The column was then washed with water and reused after conditioning.

RESULTS AND DISCUSSION

Results of the Batch Experiments

Typical batch distribution data for Th(IV) and U(VI) with the two resins are given in Table 1. The D values decrease with an increase in the molarity of nitric acid as well as metal ion concentration. A decrease is expected with an increase in hydrogen ion activity, nitrate complexing of Th(IV) in the aqueous phase, and partial saturation of the resin phase due to loading of the metal ion. D values for Th(IV) are higher than those of U(VI) for both resins. The selectivity factors or α values [defined as (D_{Th})/

TABLE 1
Distribution Ratios of Th(IV) and U(VI) at Varying Concentrations of Metal Ions and HNO_3 (aqueous phase, 20 mL; resin, ~100 mg; equilibration time, 5 hours)

		Amberlyst-15		Tulsion T-42	
Metal ion	HNO_3 (M)	Initial metal concentration (mg/mL)	D	Initial metal concentration (mg/mL)	D
Th(IV)	0.3	0.990	839	0.990	1114
		1.485	281	1.485	297
		2.475	117	2.475	115
		3.713	83	3.713	75
	0.5	0.990	630	0.990	574
		1.485	237	1.485	249
		2.475	108	2.475	108
		3.713	64	3.713	64
	1.0	0.990	281	0.990	245
		1.485	167	1.485	140
		1.980	114	2.475	80
		3.713	96	3.713	51
U(VI)	0.3	0.472	491	0.630	227
		0.945	274	0.945	180
		1.574	164	1.574	135
	0.5	0.472	235	0.630	116
		0.945	156	0.945	94
		1.574	110	1.574	73
	1.0	0.472	97	0.630	40
		0.945	75	0.945	36
		1.574	58	1.574	32

(D_U)] are 4 and 6.1 for A-15 and T-42, respectively, at a concentration level of 1 mg/mL of Th(IV) and U(VI) in 0.5 M HNO_3, which indicates the feasibility of separating U from Th using these resins.

Results of Column Experiments

Breakthrough Capacities

Capacities of T-42 and A-15 were found to be 1.8 and 1.7 meq/mL, respectively, while it was 1.86 meq/mL for Dowex 50×8. The breakthrough curves for U(VI) and Th(IV) are shown in Fig. 1. The change in breakthrough capacity of Th(IV) for feed with and without U(VI) is

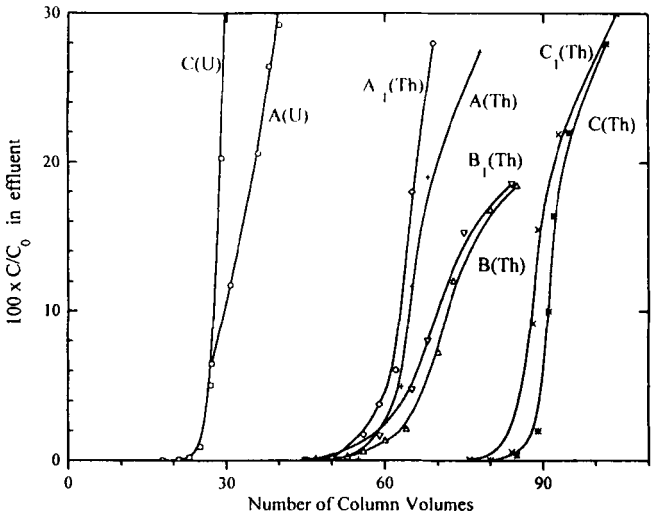

FIG. 1 Column breakthrough curves of U(VI) or Th(IV) (indicated in parenthesis on each plot) in 0.5 M HNO_3 medium containing 1 mg/mL each (alone or in mixture) using 10 mL resin in (9.5 ID × 140 H) mm column. Flow rates were 120 mL/h for Amberlyst-15 and Tulsion T-42, and −40 mL/h for Dowex 50×8. Amberlyst-15: A(Th), Th only (1 mg/mL); A(U), U only (1 mg/mL); A_1(Th), Th and U mixture (1 mg/mL each). Tulsion T-42: B(Th) Th only (1 mg/mL); B_1(Th), Th and U mixture (1 mg/mL each). Dowex 50×8: C(Th), Th only (1 mg/mL); C(U), U only (1 mg/mL); C_1(Th), Th and U mixture (1 mg/mL each).

negligible. At the 1 mg/mL level and the flow rates given in Fig. 1, 10% breakthrough capacities of Th(IV) were 65, 74, and 91 mg/mL for A-15, T-42 and Dowex 50, respectively, whereas those of U(VI) were 30 and 29 mg/mL for A-15 and Dowex 50, respectively. A decrease in breakthrough capacity of A-15 was observed when Th(IV) was at the 2 mg/mL level and the Th:U ratio was 2:1. The breakthrough capacity of T-42 is slightly higher than that of A-15, while both of them have much lower breakthrough capacities than Dowex 50×8. This may be due to the much higher flow rates (insufficient time of contact) used in the case of macroporous resins.

Effluent Profiles

The effluent profiles of the column experiments using a U–Th mixture are shown in Fig. 2. C_0 and C are the concentrations of Th(IV) or U(VI)

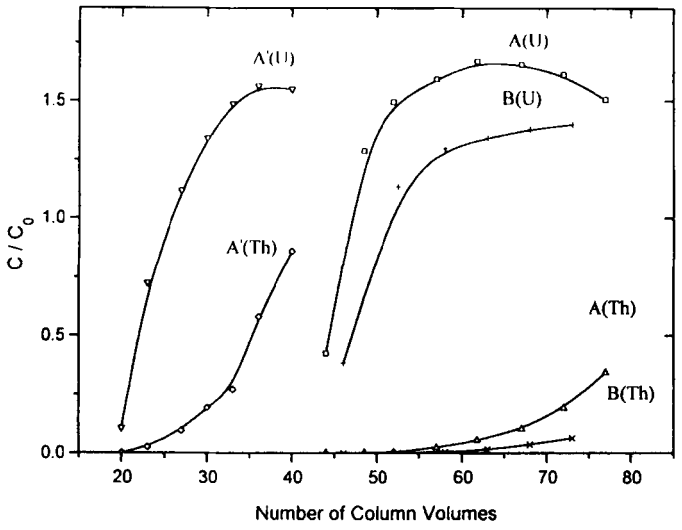

FIG. 2 Effluent profile of the binary frontal experiment using 0.5 M HNO_3 solution of U(VI) and Th(IV). C_0 and C are the concentrations in the feed and in the effluent, respectively. A(U) and A(Th): Mixture of 1 mg/mL each on Amberlyst-15. A'(U) and A'(Th): Mixture of 1 mg/mL of U(VI) and 2 mg/mL of Th(IV) on Amberlyst-15. B(U) and B(Th): Mixture of 1 mg/mL each on Tulsion T-42.

in the feed and effluent, respectively. U(VI) starts appearing in the effluent much earlier than Th(IV), and the ratio of the concentration of U in the effluent to that in the feed solution goes on increasing and exceeds unity. This is because that while loading the U–Th mixture initially both U(VI) and Th(IV) are loaded on the resin simultaneously, and at a later stage Th(IV) starts displacing U(VI) from the resin due to its greater affinity to the resin. This results in a higher U(VI) concentration in the effluent than in the feed solution. The decontamination factor and the purity of the product U in the effluent with increasing feed volume in these experiments are shown in Table 2.

Effect of the Concentration of HNO_3

Column runs 1–3 in Table 3 show that recovery of U in the effluent increased on washing with a higher concentration of HNO_3, but at the same time the retention of Th on the resin decreased very much. Therefore, it was better to use more washing with 0.5 M HNO_3 only.

TABLE 2
Decontamination of U(VI) from Th(IV) with Increasing Feed Volume [resin volume: 10 mL; column: (9.5 ID × 140 H) mm; flow rate: 5 min/bed volume; medium: 0.5 M HNO_3]

Resin and (feed)[a]	Cumulative feed volume (mL)	Total amount in effluent		r_2 ($\times 10^3$)	DF,[b] r_1/r_2	% Th in U
		U (mg)	Th (mg)			
A-15	440	186.5	0.68	3.6	276	0.36
(a)	490	251.3	0.78	3.12	319	0.31
	540	326.1	1.03	3.15	315	0.32
	590	405.7	2.17	5.34	186	0.53
	640	486.3	4.91	10.10	98	1.01
	690	569.0	10.06	17.68	56	1.77
A-15	200	21.5	0.35	16.28	122	1.63
(b)	250	57.8	1.69	29.27	68	2.93
	280	91.4	4.61	50.44	39	5.04
	310	131.7	10.47	79.48	25	7.95
	340	176.4	18.67	105.84	19	10.58
	370	223.3	36.11	161.68	12	16.17
	420	301.0	79.20	263.10	8	26.31
T-42	450	189.9	0.22	1.18	843	0.11
(a)	500	307.6	0.34	1.12	888	0.11
	550	372.5	0.67	1.79	556	0.18
	600	439.7	1.45	3.29	302	0.33
	650	508.7	3.30	6.49	153	0.64
	700	578.7	6.52	11.27	88	1.13

[a] Feed compositions. (a) Th(IV) 1.01 mg/mL and U(VI) 1.02 mg/mL. (b) Th(IV) 2 mg/mL and U(VI) 1.02 mg/mL.

[b] r_1 and r_2 are the ratios of the concentration of Th(IV) to U(VI) in the feed and the effluent, respectively.

Effect of Lower Capacity Utilization and Increased Flow Rate

Column runs 4 and 5 show the effect of lower capacity utilization. On loading less feed solution (45 instead of 55 bed volumes) on A-15 column followed by eluting with 50 bed volumes of 0.5 M HNO_3 as before, a purer product (with less Th content) was obtained, but the recovery of U was less (85%). Another 20 bed volumes of 0.5 M HNO_3 were needed to elute more of the U(VI) held up in the column for −90% recovery of U, thus giving more dilute product solution. This was to be expected because the capacity unutilized by Th(IV) tends to retain U(VI) on the resin. Run 6

TABLE 3
Recovery and Purity of Uranium after Separation from Thorium [feed: U(VI) (1.02 mg/mL) and Th(IV) (1.01 mg/mL) in 0.5 M HNO_3; column: (9.5 ID × 140 H) mm; flow rate: 120 mL/h except in Run 6]

Run	Feed volume (mL)	Wash		U recovery (%)			Th content (%)
		Molarity	Volume (mL)	In effluent	In wash	Cumulative	
				Resin: A-15			
1	550	0.5	100	57.9	16.3	74.2	0.43
2	550	1.0	100	58.4	27.7	86.1	4.58
3	550	2.0	100	58.3	35.0	93.3	22.5
4	550	0.5	100	59.1	16.5	75.6	0.45
		0.5	100	—	6.8	82.4	0.54
		0.5	100	—	3.8	86.2	0.67
		0.5	100	—	2.8	89.0	0.94
		0.5	100	—	2.6	91.6	1.00
5	450	0.5	100	41.6	18.2	59.8	0.27
		0.5	100	—	9.9	69.7	0.26
		0.5	100	—	6.6	76.3	0.27
		0.5	100	—	4.7	81.0	0.27
		0.5	100	—	3.9	84.9	0.27
		0.5	200	—	5.5	90.4	0.28
6[a]	550	0.5	500	68.5	21.5	90.0	9.31
				Resin: T-42			
7	550	0.5	500	52.5	39.0	92.5	0.1
8	550	0.5	500	55.1	36.7	91.8	0.1

[a] Flow rate: 300 mL/h.

shows increased flow rate and washing result in high Th(IV) content in the effluent. This may be due to insufficient time of contact between the influent and the resin.

From the results of runs 7 and 8 in Table 3, T-42 appears to be better than A-15 for the separation of U from Th.

Scaled-up Column Operation

Results of operation with a larger (100 mL resin) column are presented in Table 4. A much better product (with higher recovery and purity) could be achieved by washing with only 30 bed volumes. An increase in the flow rate from 750 to 1200 mL/h did not affect the quality of the product appreciably. However, with only 30% capacity utilization of the resin for

TABLE 4
Recovery and Purity of U(VI) Using Larger Column [Column: (22 ID × 260 H) mm; feed: U(VI) of 1.02 mg/mL and Th(IV) of 1.01 mg/mL in 0.5 M HNO_3]

Resin used	Feed volume (L)	Wash volume (L)	Flow rate (L/h)	U recovered (%)	Th content (%)
T-42	5.5	3.0	1.2	91.0	<0.03
	5.5	5.0	1.2	96.4	<0.03
	5.5	3.0	0.75	94.2	<0.02
	5.5	5.0	0.75	97.0	<0.02
A-15	5.5	3.0	1.2	83.7	<0.09
	5.5	5.0	1.2	89.2	<0.09
	5.5	3.0	0.75	86.7	<0.02
	5.5	5.0	0.75	92.3	<0.02
T-42[a]	3.1	2.0	1.2	87.0	<0.04
	3.1	3.0	1.2	93.5	<0.04

[a] 30% capacity utilization and washing with 1 M HNO_3.

holding Th(IV), the recovery of U(VI) in the effluent was low and most of it was obtained in the washings.

CONCLUSION

Both loading and washing rates used in this work are much higher (120 mL/h) than those used for the gel-type resin (7) (−40 mL/h). Enhanced mass transfer kinetics of the macroporous resin made it possible to achieve comparable dynamic capacities at higher flow rates by using larger particle sizes of the resins without sacrificing product quality. There is a definite advantage of high throughput of U(VI) purification with macroporous resins as compared to gel-type resins.

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