

Design of Column Separation Processes for Recovery of Molybdenum from Dissolved High-Density LEU Target

Chemical Science and Engineering Division

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Design of Column Separation Processes for Recovery of Molybdenum from Dissolved High-Density LEU Target

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DESIGN OF COLUMN SEPARATION PROCESSES FOR RECOVERY OF MOLYBDENUM FROM DISSOLVED HIGH-DENSITY LEU TARGET

1 INTRODUCTION

The Global Threat Reduction Initiative (GTRI) Conversion Program develops technology necessary to convert civilian facilities that use high enriched uranium (HEU) to low enriched uranium (LEU) fuels and targets. The conversion from conventional HEU dispersion targets to LEU for ^{99}Mo production requires approximately five times the uranium in a target to maintain the ^{99}Mo yield per target. Under GTRI, Argonne National Laboratory (Argonne) is developing two frontend options for the current processes to allow the use of LEU-foil targets. In both processes, the aim is to produce a product after the frontend that will be compatible with current purification operations and that will, with the same number of targets irradiated, provide the same or a higher yield of ^{99}Mo at the end of processing. The goal of the frontend process is to deliver a product solution that is of the same or higher Mo purity than the current solution and is of equal or better compatibility with current purification process. In the first frontend option, the LEU foil (contained in a thin [10–15 μm] Ni fission-recoil barrier) is removed from the annular target and dissolved in nitric acid. In the dissolution, the uranium, nickel, and all fission and activation products are dissolved; the solution will be ~ 7 mM Mo and ~ 450 g-U/L, and the nitric-acid concentration after dissolution will be ~ 1 M. The volume of solution is assumed to be 500 mL and the pressure drop, ΔP , constraint of 0.8 atm is based on the use of a vacuum/gravity fluid transfer system. Therefore pressure loss is limited to <1 atm; hence a value of 0.8 is chosen.

Titania-based sorbents have been identified as supports separating Mo from concentrated uranium solutions. They offer high capacity and remarkable K_d values, and are slightly affected by the presence of uranium. Their superior performance in the presence of high concentrations of uranium contrasts that of alumina (Stepinski et al. 2008, 2009). The Mo K_d for alumina decreases dramatically in the presence of high concentrations of uranium, making it unsuitable for the recovery of Mo from highly concentrated uranium solutions.

Adsorption of metal ions on inorganic supports such as alumina or titania is often slow and tends to take several hours or days to reach equilibrium in batch contacts. Slow adsorption kinetics relative to mass transfer rates lead to slow development of a constant mass transfer pattern. Non-equilibrium adsorption/desorption can introduce large errors to column designs, sorbent capacity, and estimation of system parameters. In this study, a non-constant pattern mass-transfer-zone (NCP-MTZ) method was applied to the design and optimization of Mo separation using a titania column. In this approach, batch tests were conducted to estimate isotherm parameters, and breakthrough column experiments were utilized to determine particle diffusivity (D_p) for each sorbent. Once the intrinsic parameters were determined, VERSE (Versatile Reaction and Separation—developed by Purdue University, Indiana) simulations were carried out to estimate mass transfer zone (MTZ) at various linear velocities and loading times. Determination of MTZ lengths allows design of process separation column. Key designs were verified experimentally, through loading and breakthrough experiments, to verify D_p , MTZ, and column designs.

2 RESULTS AND DISCUSSION

2.1 ESTIMATION OF THE EFFECTIVE ISOTHERM PARAMETERS OF Mo IN 450-g/L URANYL NITRATE SOLUTION AT 1 M H⁺

TiO₂ (80 μm) and Al₂O₃ (75–150 μm) were evaluated for recovery of Mo from uranyl nitrate solutions containing 450 g-U/L and 1 M HNO₃ (Figure 1). Mo capacity at feed concentration, q_{cf} , is five times higher for titania sorbent than for alumina, indicating the need for a five-fold increase in alumina-based column size.

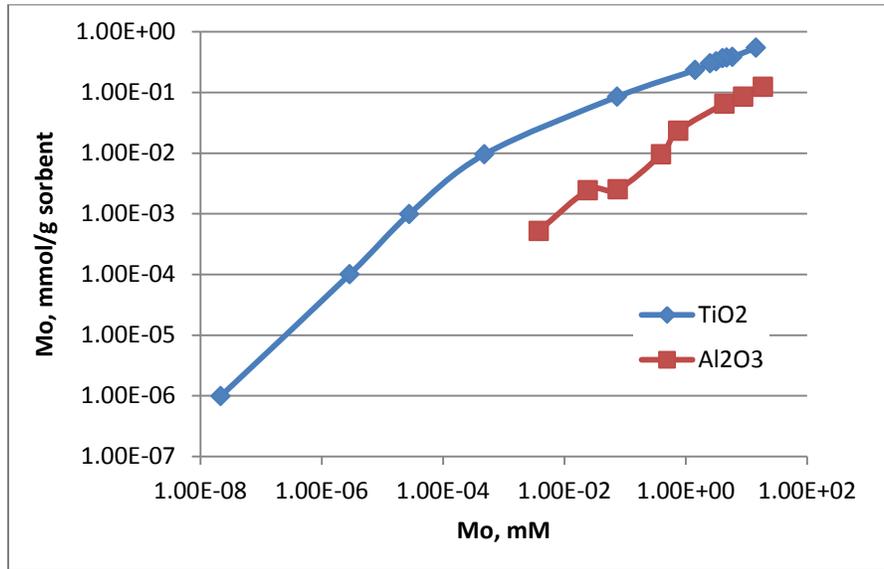


FIGURE 1 Uptake of Molybdenum on Alumina and Titania from 450-gU/L, 1-M HNO₃ Solutions

When sorption rates are higher than the controlling mass transfer rate, local equilibrium can be achieved between the solid and pore phases. Under such conditions, the solid phase concentrations are related to pore-phase concentrations by an equilibrium isotherm. The Langmuir model is tested for the sorption of Mo in this study:

$$q_i = \frac{a_i C_{p,i}}{1 + \sum_{j=1}^N b_j C_{p,j}} \quad \text{Eq. 1}$$

where q_i is the amount of species i sorbed on the column packing and equilibrated with the concentration in mobile phase, $C_{p,i}$. In this study, both q_i (meq/L) and $C_{p,i}$ (mol/L) of the Mo isotherm is described on volume basis assuming 1.3 g/mL dry packing density of TiO₂ sorbent.

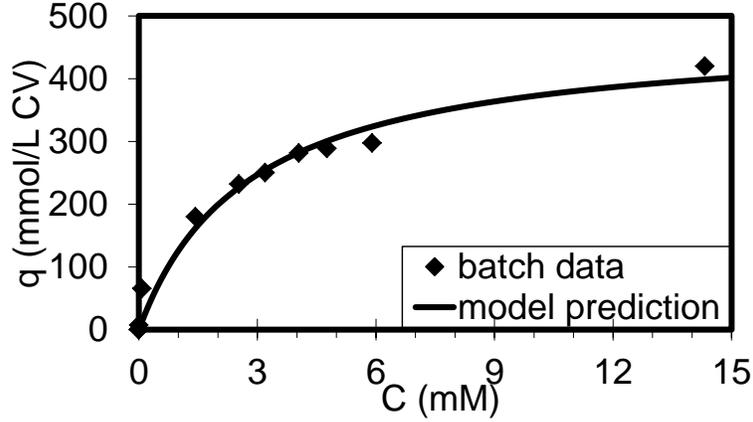


FIGURE 2 Comparison of Isotherm Data from Batch Tests and Langmuir Isotherm Model Prediction for Uptake of Mo on Titania Sorbent

The data from batch equilibrium experiments were used to estimate the effective Mo isotherm parameters for TiO_2 sorbent. All batch equilibrium data were equilibrated for 24 hours at 60°C . The linear isotherm parameter a of each sorbent was first estimated from the data in the low Mo concentration range ($[\text{Mo}] \leq 10^{-3}$ mM). The nonlinear isotherm parameter b was

estimated by minimizing the sum of the relative errors $\left[\sum \left(\frac{q - q'}{q} \right)^2 \right]$ between the experimental

(q) and the predicted (q'). The calculated Langmuir isotherm model parameter $a = 171.22$, $b = 0.36 \text{ mM}^{-1}$, and $q_{max} = 476 \text{ mmol/LCV}$ for adsorption of Mo on titania sorbent. When $C = 7 \text{ mM}$, $q_C = 340.66 \text{ mmol/LCV}$ (Figure 2).

2.2 ESTIMATION OF OTHER MASS TRANSFER PARAMETERS OF Mo

The axial dispersion coefficient, E_b , was estimated using the Chung and Wen (1968) correlation. The mass transfer coefficient, k_f , was estimated using the Wilson and Geankoplis (1966) correlation. Brownian diffusivity (D_∞) values of HMoO_4^- in aqueous solution at 25°C used in the simulations were reported to be $8.3 \text{ cm}^2/\text{min}$ (Marcus 1997). The Stokes-Einstein equation was then used to correct the D_∞ for solution viscosity and temperature effects. At 60°C , the viscosity of $450 \text{ gU/L UO}_2(\text{NO}_3)_2$ and 1 M HNO_3 was calculated to be 1.40 cp , using a correlation listed in HW-57386 (Krigens 1968). Therefore, at 60°C , $D_{\infty, \text{Mo}}$ in $450 \text{ gU/L UO}_2(\text{NO}_3)_2$ and 1-M HNO_3 solution was estimated to be $5.85 \times 10^{-4} \text{ cm}^2/\text{min}$.

2.3 ESTIMATION OF THE INTRAPARTICLE DIFFUSIVITY VALUES (D_p) OF Mo FROM BREAKTHROUGH CURVES

Two breakthrough experiments, at 7 mM Mo in 450 gU/L, 1-M HNO₃, were run to estimate D_p and Mo capacity from breakthrough curves (Figures 3a and 3b). In the first experiment, the breakthrough curve was obtained by loading 110 mL of feed solution onto a 6.6 × 32 mm column at 5 cm/min. The second breakthrough curve was obtained by loading 135 mL of feed onto a 6.6 × 42 mm column at 9 cm/min. Mo capacity from breakthrough experiments was estimated using equation (2):

$$q \text{ at } C = C \times \left(\frac{V_{br}}{CV} - \varepsilon_t - \frac{DV_{sys}}{CV} - \frac{DV_{cap}}{CV} \right) q \text{ at } C = C \times \left(\frac{V_{br}}{CV} - \varepsilon_t - \frac{DV_{sys}}{CV} - \frac{DV_{cap}}{CV} \right) \quad \text{Eq. 2}$$

where C is Mo concentration in feed solution (mM), V_{br} is the raw breakthrough volume (mL), q is the amount of Mo adsorbed per packing volume of sorbent (mmol/L CV), CV is column volume (mL), ε_t is total column void fraction (assume $\varepsilon_t = 0.77$ for TiO₂), DV is the dead volume in the system; for system (subscript “sys”) or for column caps/adjusters (subscript “cap”).

Capacity at feed concentration q_{Cf} was determined to be 326 and 336 mmol/LCV, respectively for breakthrough curves in Figures 3a and 3b. The capacity estimated from breakthrough experiments is within 5% of the q_{Cf} determined from batch experiments.

The intraparticle diffusivity was estimated by comparing the experimental Mo breakthrough curves with VERSE simulations (Figures 3a and 3b). In 450 g-U/L UO₂(NO₃)₂ and 1-M HNO₃ solution, the intraparticle diffusivities of Mo on TiO₂, assuming $D_\infty/D_p = 50$ (as in Figures 3a and 3b) is estimated to be $D_p = 1.17 \times 10^{-5}$ cm²/min. Intraparticle diffusion of Mo is assumed to be pore diffusion. As shown in a recent paper of Chung et al. (2010), in a linear isotherm range, surface diffusion effects cannot be distinguished from pore diffusion effects, and therefore an effective pore diffusion coefficient is sufficient to take into account of pore diffusion, surface diffusion, or parallel pore diffusion.

The agreement between experimental Mo breakthrough curves and model prediction indicates that (1) the adsorption of Mo in 450 g-U/L UO₂(NO₃)₂ and 1-M HNO₃ can be described by the effective Langmuir isotherm; and (2) the numerical parameters and the intrinsic model parameters (including void fractions, isotherm, and mass transfer parameters) are sufficiently accurate to predict the Mo breakthrough curves in 450 g-U/L UO₂(NO₃)₂ and 1-M HNO₃ solution.

2.4 COLUMN DESIGNS FOR Mo RECOVERY FROM 450 g-U/L $\text{UO}_2(\text{NO}_3)_2$ AND 1 M HNO_3 , 7 mM Mo

VERSE simulations were carried out to estimate MTZ at various linear velocities and loading times. Determination of MTZ lengths allowed design of separation processes at various column IDs and velocities. The Mo recovery processes aim to (1) load 500 mL of 7-mM Mo, 450 g-U/L uranyl nitrate and 1 M HNO_3 ; (2) recover (a) 99% of Mo and (b) 99.9% Mo from the feed; and (3) keep the pressure drop of the column at less than 0.8 atm. Column designs for loading 99% of Mo are listed in Table 1, and for loading 99.9% Mo are listed in Table 2.

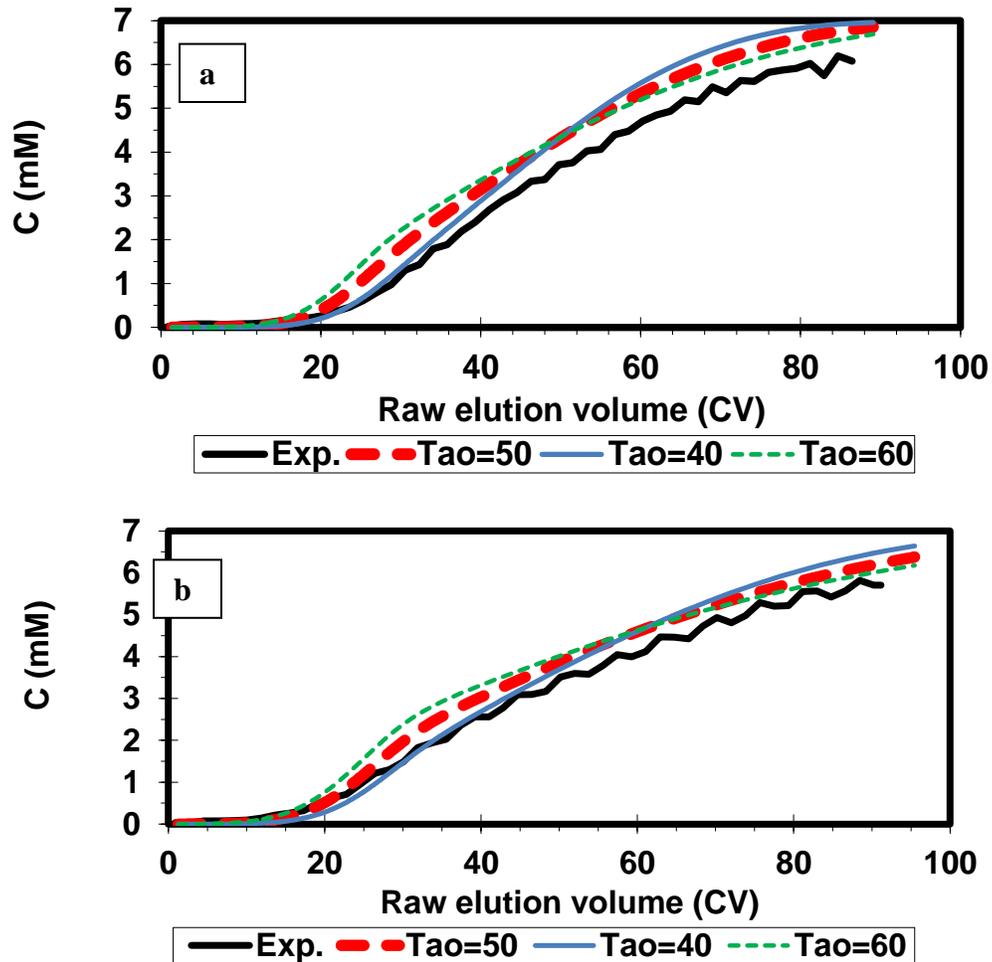


FIGURE 3 Breakthrough Experiments and D_p Estimation for Mo

TABLE 1 Column Designs for 99.0% Mo Recovery

u_s (cm/min)	ID (cm)	$t_{Loading}$ (min)	$L_{1\%Cf}$ (cm)	When $L_{min} \geq 2 L_{1\%Cf}$			
				L_{min} (cm)	CV_{min} (mL)	$W_{sorbent}$ (g)	ΔP (atm) ^a
3	4.0	13.26	2.5	5	63	82	0.1
3	3.0	23.58	3.4	7	50	64	0.1
3	2.5	33.95	4.2	9	44	57	0.2
3	2.0	53.05	5.5	11	35	45	0.2
3	1.5	94.31	8.2	16	28	37	0.3
5	3.0	14.15	3.1	6	42	55	0.2
5	2.0	31.83	4.9	10	31	41	0.3
5	1.5	56.59	7.0	14	25	32	0.5
5	1.0	127.32	12.4	25	20	26	0.8
9	3.0	7.86	6.3	12	85	110	0.7
9	2.5	11.32	7.6	15	47	61	0.9
9	2.0	17.68	9.7	20	35	46	1.2
9	1.5	31.44	13.5	27	21	28	1.6

^a Designs with $\Delta P > 0.8$ atm are in gray rows.

TABLE 2 Column Designs for 99.9% Mo Recovery

u_s (cm/min)	ID (cm)	$t_{Loading}$ (min)	$L_{0.1\%Cf}$ (cm)	When $L_{min} \geq 2 L_{1\%Cf}$			
				$2L_{min}$ (cm)	CV_{min} (mL)	$W_{sorbent}$ (g)	ΔP (atm) ^a
3	4.0	13.26	3.1	6	75	98	0.1
3	3.0	23.58	4.1	8	57	74	0.2
3	2.5	33.95	5.0	10	49	64	0.2
3	2.0	53.05	6.4	13	41	53	0.3
3	1.5	94.31	9.1	18	32	41	0.4
5	3.0	14.15	3.8	8	57	74	0.3
5	2.0	31.83	5.8	12	38	49	0.4
5	1.5	56.59	8.0	16	28	37	0.5
5	1.0	127.32	13.6	27	21	28	0.9
9	3.0	7.86	7.9	16	113	147	1.0
9	2.5	11.32	9.4	18	88	115	1.1
9	2.0	17.68	11.6	23	72	94	1.4
9	1.5	31.44	14.3	28	50	64	1.7

^a Designs with $\Delta P > 0.8$ atm are in gray rows.

Column design analysis was performed to evaluate most efficient designs in terms of time versus sorbent mass. Figures 4 and 5 show the column design analyses. The results in Figures 4 and 5 indicate that, for both 99% and 99.9% Mo recovery, the most efficient designs are at a superficial velocity of 5 cm/min. Therefore, these designs primarily will be evaluated experimentally. The column designs with loading velocities of 9 cm/min will not be evaluated due to high ΔP and/or inefficient design.

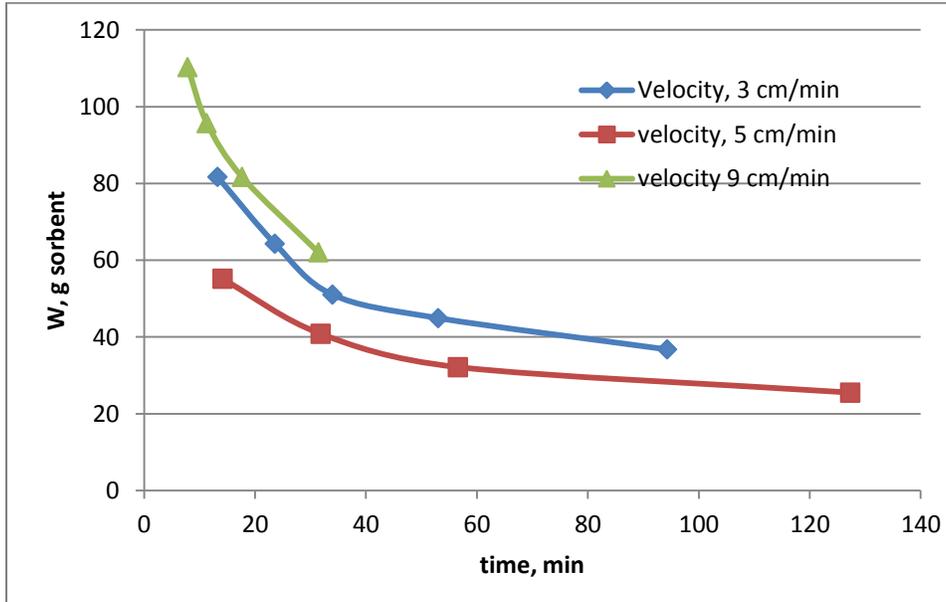


FIGURE 4 Analysis of Column Designs for 99% Mo Recovery

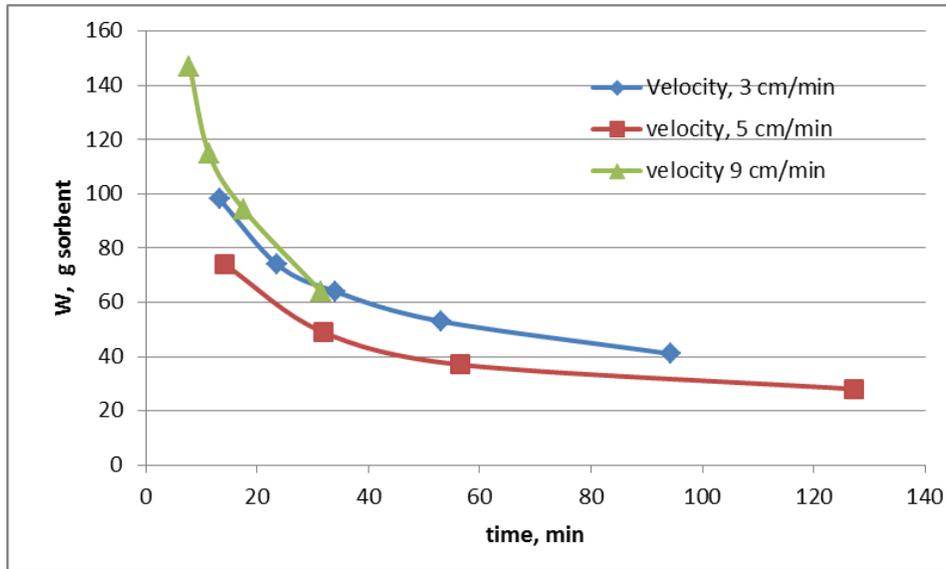


FIGURE 5 Analysis of Column Designs for 99.9% Mo Recovery

2.5 EXPERIMENTAL VERIFICATION OF LAB-SCALE Mo RECOVERY PROCESSES

Experiments were carried out to test the column designs reported in Tables 1 and 2, with the inner diameter scaled down to 6.6 mm (Table 3). The percent Mo loading are listed in Table 4. The verification of column designs for loading 99% of Mo was successful, and $99\% \pm 5\%$ loadings were achieved. The experimental verification of 99.9% Mo loading column designs clearly suffered from incorrect experimental conditions, as columns longer than those designed for 99% capture, at the same linear velocities, should have achieved equal or better %Mo uptake. Some possible experimental conditions leading to the poor results include third reuse of the 0.66×10 column, poor packing of the 0.66×12 column, or a loading temperature lower than 60°C . Experimental verification of 99.9% Mo loading column designs will be repeated. Overall, however, the results of experimental verification of column designs indicate that the NCP-MTZ method is a precise and highly efficient approach to design of column processes for Mo recovery.

TABLE 3 Scaled Down Column Designs for Experimental Verification

u_s (cm/min)	ID (cm)	L_{min} (cm)	$t_{Loading}$ (min)	V_{feed} (mL)	Target Mo loading (%)
3	0.66	7	23.58	24.2	99
5	0.66	6	14.15	24.2	99
5	0.66	10	31.83	54.4	99
5	0.66	14	56.59	96.8	99
3	0.66	10	33.95	34.8	99.9
5	0.66	12	31.83	54.4	99.9

TABLE 4 Results of Experimental Verification of Column Designs

u_s (cm/min)	ID (cm)	L_{min} (cm)	$t_{Loading}$ (min)	V_{feed} (mL)	Target Mo loading (%)	Mo loading (%)
3	0.66	7.5	23.58	28	99	99.1
5	0.66	6.2	14.15	25	99	99.0
5	0.66	10	31.83	56	99	98.7
5	0.66	10	56.59	97	99	98.8
3	0.66	10	33.95	35	99.9	86.8
5	0.66	12	31.83	55	99.9	94.1

2.6 RECOVERY OF Mo FROM TiO₂ COLUMNS

Full recovery of Mo can be achieved by counter-current stripping using 0.1-M NaOH. The Mo recovery for counter-current stripping with 0.1-M NaOH was between 88% and 105% \pm 5%, with an average of 95.3% \pm 5% (Table 5). Figure 6 shows the stripping curves for breakthrough and loading experiments performed with solution containing 7-mM Mo, 450 g-U/L and 1-M HNO₃. The resulting stripping curves demonstrate that Mo can be qualitatively stripped from titania sorbent at 3 and 5 cm/min linear velocities. The different stripping curve areas are reflected in the %Mo sorbent loading relative to sorbent capacity at feed concentration (q_{cf}) as specified in Table 5. The results indicate that Mo can be stripped within 15 bed volumes or, depending on design, 600–900 mL of 0.1-M NaOH (process scale column).

TABLE 5 Results of Column Stripping with 0.1-M NaOH

Stripping u_s (cm/min)	ID (cm)	L_{min} (cm)	Sorbent loading, % q_{cf}	Mo recovery (%)
5	0.66	6.2	16.1	93.5
3	0.66	7.5	95.2	88.4
5	0.66	10	40.3	97.4
5	0.66	12	17.7	93.9
3	0.66	10	11.7	95.0
5	0.66	3.2	78.6	104
5	0.66	4.2	79.0	105

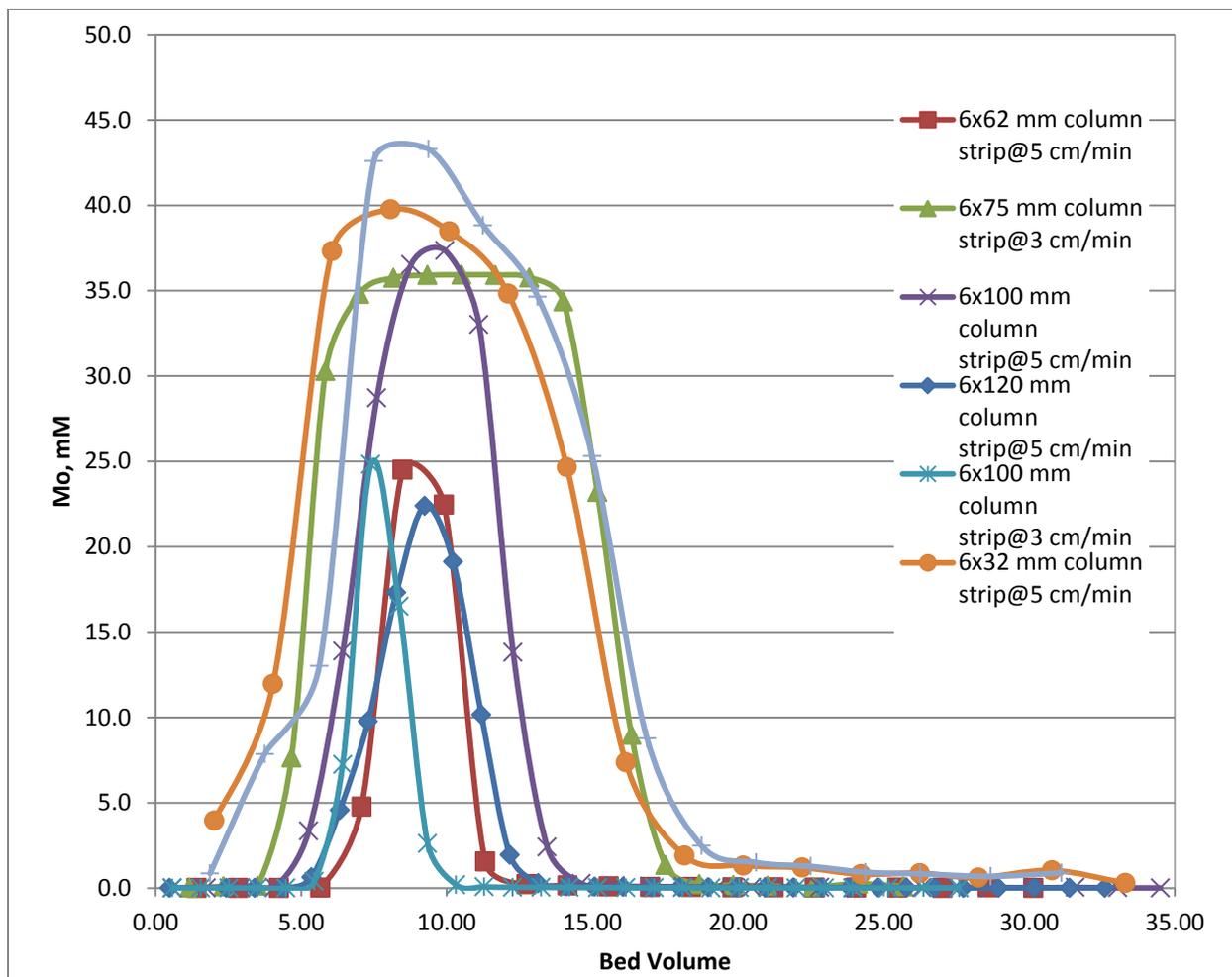


FIGURE 6 Counter-Current Stripping of Mo from Titania Columns with 0.1-M NaOH

3 EXPERIMENT

Titania sorbent (TiO_2 , 80 μm , 60 \AA) was purchased ZirChrom Separations, Inc. (Anoka, Minnesota). Alumina (Al_2O_3 , 75–150 μm , 60 \AA) was obtained from Sorbent Technologies.

Uranyl nitrate, $\text{UO}_2(\text{NO}_3)_2$, solutions were prepared by dissolving a known amount of uranium metal in 8-M HNO_3 . The resulting solution was titrated in presence of oxalate and H^+ was adjusted with concentrated HNO_3 . Molybdenum was added as 0.3-M Na_2MoO_4 , pH 0, solution.

Molybdenum-99 was obtained from a commercial $^{99\text{m}}\text{Tc}$ generator by stripping with 1-M NH_4OH , evaporating to dryness and dissolving in nitric acid.

Isotherm data were obtained by equilibrating 10 ± 1 mg of sorbent with 1 mL of 450 g-U/L containing appropriate amount of Mo for 24 h at 60°C .

The amount of activity in the aqueous samples was determined using a NaI(Tl), HPGe well detector or NaI(Tl) flow-through detector. Molybdenum-99 was quantified by measurement of its 739 keV γ -ray. The activity of ^{99}Mo in each sample was corrected for decay.

The Omnifit chromatography columns (VWR Internal, West Chester, Pennsylvania) were packed with titania sorbent and tested in the experiments. An ÅKTA Purifier unit (GE Healthcare, Piscataway, New Jersey) was used in the frontal and lab-scale Mo recovery experiments. During frontal and lab-scale Mo recovery experiments, the temperatures of the column and the mobile phases were controlled by heat tapes, heating coils, and solution heating.

4 FUTURE EXPERIMENTS

A 4-g depleted uranium (DU) target encapsulated in aluminum casing will be irradiated at the 20-MeV Argonne Linear Particle Accelerator (LINAC). Irradiation will be done using a W photoconverter to generate high-energy x-rays that will generate photofissions in the target. The target will be irradiated for 6 hours at 18 MeV and 200 microA, and the expected ⁹⁹Mo yield after a 14-h cooling time is 0.6 mCi.

The target will then be disassembled and the DU foil will be dissolved in concentrated nitric acid. The irradiated DU solution (~10 mL) will then be added to 500 mL of 450 g/L uranyl nitrate solution, 7-mM Mo, 1-M H⁺ to produce a solution representative of the process scale dissolved, irradiated LEU target solution. The solution will be passed through a 2 × 10 cm TiO₂ column at 5 cm/min. The expected loading time is ~30 min. A 0.1-M NaOH solution was chosen to strip Mo from the column in order to best reflect current chemical conditions during process target digestion or to best align with subsequent Mo purification steps. Therefore, 470 mL is expected to recover 95% ± 5% of Mo in 30 min. The eluent (waste) is expected to contain all of the actinides, lanthanides, alkali, and alkaline earth metal ions. The Mo-product stream is expected to contain a fraction of the fission products known to have chemistries somewhat similar to Mo, specifically elements known to be present as oxoions or anions in acidic solution. Therefore, the Mo product stream is expected to contain a small fraction of As, Se, Nb, Tc, Ru, Rh, Sn, Sb, and Te as well as the iodine not volatilized in the dissolution; the fraction in the Mo product is assumed to be 1%. The K_d values for these anions in 0.1-M HNO₃ tend to be quite a bit lower than that of Mo; therefore, it is likely that a significant fraction of As, Se, Nb, Tc, Ru, Rh, Sn, Sb, Te, Br, and the remaining I will be found in waste stream and not in the Mo product stream.

The timeframe for completing the column design verification experiments with the irradiated target solution is about 4 weeks.

5 CONCLUSIONS

The NCP-MTZ design method and VERSE simulations were utilized to determine intraparticle diffusivity, MTZ, and column designs. The results of scale-down indicate that Mo can be fully loaded on the proposed titania columns and then recovered by counter-current stripping with 0.1-M NaOH. By using the largest diameter column and intermediate flow rates, the Mo recovery operation should be completed in less than 1 h.

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