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ABSTRACT

A standard pilot plant resin column was fitted with a teflon expansion bellows. Resin bed compression was thus maintained under various chemical conditions. Hydraulic and experimental data were reported for these conditions at various flow rates through the resin bed.

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BELLOWS TYPE RESIN COLUMN EVALUATION ORNL METAL RECOVERY PLANT

Part Two

SUMMARY

A 6" I. D. by 14" bed height resin column, fitted with a teflon bellows to provide a variable bed volume, was evaluated. Pressure drop and expansion data were obtained using different ionic solutions and for an additional compacting force on the bed. Flow rates ranged from 35 ml/min to 750 ml/min with corresponding pressure drops of 1.5 inches H_2O and 30 inches H_2O . Flooding flow rates for different head tank heights above the column were obtained by extrapolation. Gassing did not occur in elution runs with 5.7 M HNO_3 and 0.3 M NH_4SO_3H . Solutions of sulfamic acid, nitric acid, and hydroxylamine sulfate were used in the tests.

INTRODUCTION

Processes for the concentration and decontamination of fissionable material employ both anion and cation exchange. At the ORNL Metal Recovery Plant uranium and plutonium are sorbed on Dowex-50, a sulfonated styrene-divinylbenzene cation exchange resin. Selective elution of the uranium and plutonium and reconditioning of the resin bed involve contacting the resin with several different aqueous solutions. The following characteristics observed during the cyclic operations were found to greatly influence column design:

1. The resin bed expanded when contacted with water and contracted upon contact with concentrated nitric acid.
2. Pressure drop variations resulted when the bed volume changed.
3. Gas was evolved when the resin was contacted with 5.7 M nitric acid.
4. Channeling occurred when the resin bed was not held compact.

To permit a variable bed volume and still maintain a compact bed, a spring loaded disc was used to apply a force on the top of the bed. Binding of the disc against the wall presented a serious problem so a new design was devised. This paper is concerned with the experimental evaluation of the bellows type resin column shown in Fig. 1.

Fig. 1. Bellows type resin column

- Note (1) The liquid levels in these lines were measured
(2) The jack-leg height was held constant during runs

Fig. 2. Experimental Set-up

Column Design

A teflon bellows, Fig. 1, with expansion characteristics described by Fig. 3 and Table 1A, allowed a variable volume bed and applied a compacting

TABLE 1A

Bellows Force Characteristics

<u>Bellows Length</u>	<u>Force Exerted by Bellows</u>
cm	kilograms-force
13.80	4.49
13.95	6.39
15.76	16.79
15.85	17.79
16.00	18.79
16.10	19.79
16.50	26.27
16.74	28.19

force. The total force on the resin bed is given by the equations below:

Case 1 - No additional weight on the bellows

$$\text{Force on bed} = \text{force due to bellows} + 16.79$$

Case 2 - 22.6 kg-f weight on the top flange

$$\text{Force on bed} = \text{force due to bellows} + 39.39$$

where: forces are measured in kilogram-force

16.79 = weight of the top flange and collar
expressed in kilograms-force

Experimental data were not sufficient to justify writing for the total force exerted on the bed and are given in Table 1.

Criteria determining the most desirable force to be applied on the bed are pressure drop and channeling. Possibilities of channeling decrease and the pressure drop increases as a larger force is applied. Pressure drop data are given in the expansion and pressure drop section of the paper.

Bellows expansion data are given in Table 1. For solutions other than concentrated nitric acid, the bellows expansion was the same for different flow rates. The behavior using nitric acid may be attributed to the decreased pressure exerted on the bed in the contracted state. There was also a lack of firmness in the bed at high nitric acid flow rates.

TABLE 1

EXPANSION DATA - NO ADDITIONAL WEIGHT ON FLANGE

Solution	Flow Rate ml/min.	Bellows Height cm	Total Force on Bed kg _f	Column Height cm	Volume (approx) liters	% Change in Volume over Water-Saturated Volume
Water (Downflow)	0	14.96	29	35.60	6.49	0
	37.3	14.96		35.60	6.49	0
	343	14.96		35.60	6.49	0
0.1 M NH ₂ OH • 1/2 H ₂ SO ₄ 0.1 M HNO ₃ (Upflow)	0	15.04	30	36.67	6.51	+0.20
	35	15.04		35.67	6.51	+0.20
	410	15.03		35.66	6.51	+0.20
	666	15.03		35.66	6.51	+0.20
5.7 M HNO ₃ 0.3 M NH ₃ SO ₃ H (Upflow)	0	13.00	14	33.64	6.14	-5.5
	35	13.00		33.64	6.14	-5.5
	193	13.20		33.84	6.17	-4.9
	195	13.48		34.12	6.22	-4.2
	270	13.41		34.05	6.21	-4.4
	270	13.51		34.15	6.23	-4.2
	431	13.89		34.53	6.29	-3.0
	755	14.11		34.75	6.34	-2.4

EXPANSION DATA - 22.6 kg_f WEIGHT ON FLANGE

Solution	Flow Rate ml/min.	Bellows Height cm	Total Force on Bed kg _f	Column Height cm	Volume cm ³	% Change in Volume Over Water-Saturated Resin
Water (Downflow)	0	14.59	49	35.23	6.43	-1.0
	192	14.58		35.22	6.43	-1.0
	430	14.58		35.22	6.43	-1.0
	720	14.65		35.29	6.44	-1.0
5.7 M HNO ₃ 0.3 M NH ₃ SO ₃ H	0	12.50	35	33.14	6.04	-6.9
	35.6	12.56		33.20	6.06	-6.7
	192	12.56		33.20	6.06	-6.7
	430	12.56		33.20	6.06	-6.7

Since this might have been an indication of partial fluidization of the bed, 22.6 kg-f of weight were placed on the top flange and further studies made. When the additional weight was used the bellows expanded and contracted uniformly on all sides of the bellows. Frequently, there was an 8% variation in expansion heights on opposite sides of the column when no additional weight was used on the flange. Since the pressure drop increased only slightly, the use of additional weight on the top flange is recommended. A further improvement in column design would be the use of posts to guide the movement of the top flange.

The column operation was satisfactory except for leaking around the teflon bellows-collar seal. The leaks appeared to be due to warping of the collars and could presumably be stopped by using a heavier flange.

Bellows Expansion and Flow Data

Initially, runs were made using no additional weight on the bellows. In a second group of runs, 22.6 kg-f of weight were placed on the top flange of the column. In each case pressure drop, bellows expansion, and feed line head height data were obtained. Both downflow and upflow runs were made; however, the pressure drop due to the bed resistance should be essentially the same in either case. The jack-leg height above the bottom of the resin bed was 53.5 cm for downflow runs and 67.6 cm for upflow runs. Refer to Figs. 1 to 3.

Pressure drop due to the resistance of the resin bed are plotted vs. flow velocity in Figs. 4 to 7. (Flow Velocity = $\frac{\text{flow rate, ml/min}}{\text{cross-sectional area, cm}^2}$). Differences in pressure between the top and bottom streams may be calculated as shown.

$$\text{Downflow } \Delta P = P_{\text{bottom}} - P_{\text{top}} = (\text{column height}) - (\text{resistive pressure drop})$$

$$\text{Upflow } \Delta P = P_{\text{bottom}} - P_{\text{top}} = (\text{column height}) + (\text{resistive pressure drop})$$

Solution heights in the feed line to the column are plotted vs. flow rates in Figs. 8 and 9. Overflow rates for different head tank heights may be read from the graph.

Notes supplementing the graphical information are given below.

Figure 4: Runs were made with resin that had been in water for several months. Further runs were made after reconditioning the resin with 0.1 M HNO_3 and 0.1 M $\text{NH}_2\text{OH } 1/2 \cdot \text{H}_2\text{SO}_4$.

Figure 7: The elution was upflow when no additional weight was added. Downflow elution was used when 22.6 kg-f of weight were added.

Figure 8: Head height data for the no weight and additional weight cases were nearly the same.

Gassing

An experiment was conducted to see if gas were evolved from the resin bed during contact with concentrated nitric acid. The resin was first reconditioned with sulfamic acid or hydroxylamine sulfate then was contacted with nitric acid. Results given in Table 2 indicate that gassing does not appreciably occur when sulfamic acid is present in the elutriant solution.

TABLE 2

<u>Reconditioning Solution</u>	<u>Elutriant Solution</u>	<u>Flow Rate</u>	<u>Gas Evolution</u>
0.1 M $\text{NH}_2\text{OH} \cdot 1/2 \text{H}_2\text{SO}_4$ 0.1 M HNO_3	5.7 M HNO_3	200 ml/min	> 5 liters STP
0.1 M $\text{NH}_2\text{OH} \cdot 1/2 \text{H}_2\text{SO}_4$ 0.1 M HNO_3	5.7 M HNO_3 0.3 M $\text{NH}_3\text{SO}_3\text{H}$	200 ml/min	~ 0.1 liter, STP
$\text{NH}_3\text{SO}_3\text{H}$	5.7 M HNO_3 0.3 M $\text{NH}_3\text{SO}_3\text{H}$	200 ml/min	None

Volume Determination

The volume of solution needed to fill the resin packed column was determined for the expanded and contracted states. After draining the column for 15 minutes, solution was added from the bottom until the column was full. Several trials were made. Average volumes are given in Table 3. No additional weight was added to the flange.

TABLE 3

	<u>Bellows Height</u>	<u>Volume Solution Required To Fill Column</u>	<u>% Solution in Total Volume</u>
5.7 M HNO_3 0.3 M $\text{NH}_3\text{SO}_3\text{H}$	12.99 cm	590 ml	9.6%
Water	14.86	510 ml	7.9%

Column Loading

The following procedure is recommended for loading the column with the same amount of resin used in the experiments. The only measurement necessary is the determination of 7600 ml of settled, water saturated resin. It is not necessary to carefully measure the bellows expansion.

Procedure*

1. Soak about 9 liters of wet resin in water for one day.
2. Decant most of the water leaving enough to make a resin slurry that can be easily poured.
3. Pour the resin slurry into a 10 liter graduated cylinder until the resin forms about an 8.5 liter bed. This may be observed about 15 minutes after pouring. During the transfer it will probably be necessary to decant more water.
4. Let the resin settle for 2 hours and note the settled volume.
5. Remove or add resin until the settled volume is 7600 ml.
6. Let the resin settle for one hour to make certain the settled volume is 7600 ml.
7. Expand the bellows to 7 inches and open the drain on the column.
8. Transfer all resin into the column.
9. Bolt on the top flange before allowing the bellows to contract.
10. Fill the column with water.

*The accuracy of the volume measurements determines the nearness to experimental behavior. If a 10 liter cylinder is not available, the settled resin may be added in two parts. The single requirement is that 7600 ml of settled, water saturated resin be added.

Fig. 3. Bellows Force Characteristics

Fig. 4. Data for Water

Fig. 5. Data for Reconditioning Solution

Fig. 6. Data for Water

Fig. 7. Data for Nitric Acid

Fig. 8. Data for Water

Fig. 9. Data for Nitric Acid

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