

# DESIGN OF FIXED-BED ION EXCHANGE COLUMNS FOR WASTEWATER TREATMENT

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## ABSTRACT

Oak Ridge National Laboratory plans to use chabazite zeolites for decontamination of process wastewater which contains ppb levels of Sr-90 and Cs-137. Treatability studies have indicated that chabazite zeolites have high selectivities and loadings for removal of trace amounts of Cs-137 and Sr-90 from wastewater containing high concentrations of calcium and magnesium. These studies also indicated that the efficiency of the zeolite system is dependent on the column design and operating conditions. Results from 20-mL, 566-L, and 3,760-L column tests indicated that the optimized design of full-scale columns could halve the generation rate of loaded zeolite. The corresponding annual waste disposal costs for loaded zeolite generated at the ORNL plant varied from \$80,000 to \$170,000 based on the present disposal charges of \$1400/m<sup>3</sup> indicating that design of zeolite ion exchange systems for minimization of secondary waste is imperative. This report summarizes the results of a study to model multicomponent ion-exchange columns.

## INTRODUCTION

The process waste system at Oak Ridge National Laboratory (ORNL) collects wastewater that is (or is potentially) contaminated with very low levels of radioactivity. A typical characterization of the ORNL process waste stream is shown in Tables I and II. Wastewater whose pH is typically 8.8, contains 250 mg/L total dissolved solids, 3 mg/L total suspended solids, 133 mg/L total hardness, 125 mg/L alkalinity, and 12 mg/L total organic carbon. The major chemical constituents are bicarbonates of calcium, magnesium, and sodium. Discharge limits for radionuclides are presently set at 11.1 Bq/L for Sr-90 and 740 Bq/L for Cs-137; a proposal has been made to change the limits to 37 Bq/L and 111 Bq/L, respectively. The effluent concentrations of the other radionuclides are negligible and are well below discharge requirements.

Lower discharge limits and increased costs for disposal of secondary waste have prompted ORNL to upgrade the Process Waste Treatment Plant (PWTP). The PWTP presently consists of chemical precipitation, filtration, and ion exchange processes as shown in Fig. 1. In the scavenging precipitator, calcium and magnesium, along with a fraction of the radionuclides, are precipitated in the clarifier at pH 11.4. The resulting sludge is dewatered in a filter press and disposed of as a solid low-level radioactive waste. The remaining radionuclides are removed by contact with Dowex HCR-S, a strong-acid cation resin. The resin is regenerated with nitric acid, which is then concentrated by evaporation and transferred to the liquid low-level radioactive waste system for storage and ultimate disposal. Secondary waste generation rates, shown in Fig. 1, include 85 m<sup>3</sup>/year of precipitator sludge, 2 m<sup>3</sup>/year of spent resin, and 40 m<sup>3</sup>/year of Sr-rich LLW.

From 1986 through 1988, bench-scale treatability studies were performed to determine an optimal method for upgrading the PWTP to meet more stringent discharge limits while reducing secondary waste generation. A com-

ination of 4 chemical precipitation and 16 ion exchange processes were tested for Cs-137 and Sr-90 decontamination. The ion exchange materials used included weak-acid cation resins, strong-acid cation resins, specialty resins with selective properties for cesium and strontium, titanates, and natural and synthetic zeolites. The results from scoping tests (1) indicated that chabazite zeolite, an inorganic aluminosilicate molecular sieve, has an extremely high capacity for removal of Cs-137 and is the best of the materials tested for removing Sr-90 in the presence of calcium and magnesium. Flowsheet analyses (2) indicated that a series of zeolite ion-exchange columns to remove both Sr-90 and Cs-137 would be the most economical and efficient method for long-term upgrade of the PWTP. The proposed flowsheet for the upgraded PWTP, shown in Fig. 2, consists of a series of zeolite columns. As Sr-90 begins to break through, the front column will be taken off-line and replaced with a column of fresh zeolite at the back end. The contaminated zeolite will be disposed of as solid waste because regeneration methods for the zeolite have not been developed and disposal of liquid waste is much more expensive. Estimated secondary waste generation rates for this process (based on experimental results) are approximately 50% of those for the present process.

Pilot-scale and near-full-scale zeolite column tests began at the PWTP in 1987 to demonstrate the feasibility of the process, to verify modeling results obtained from laboratory-scale tests, and to determine factors which would be important in the full-scale design of a permanent system. These tests were performed using one-tenth-plant-scale and near-full-scale demineralizer pressure vessels supplied by Chem Nuclear. Near-full-scale tests have been routinely performed since 1987 using one zeolite column to pre-process 50 to 100% of the plant feed for Cs-137 removal before the feed is sent to the ion-exchange columns. Pilot-scale tests were performed for approximately one year using two to four columns operated in the "merry-go-round" fashion. In this process, the zeolite columns are operated in series and the first column is periodically removed from operation,

**TABLE I**  
Radiochemical Composition of ORNL Process Wastewater

Radionuclide	Concentration, Bq/L	Concentration, N
Gross alpha	5	--
Gross beta	6000	--
Co-60	25	2.0E-14
Sr-90	4000	1.7E-11
Cs-137	400	9.1E-13
Ru-106	10	2.3E-15

**TABLE II**  
Chemical Composition of ORNL Process Wastewater

Component	Concentration, mg/L	Concentration, N
Ca <sup>2+</sup>	40	2.0E-03
Mg <sup>2+</sup>	8	6.6E-04
Na <sup>+</sup>	5	2.2E-04
K <sup>+</sup>	2	5.1E-05
Si <sup>3+</sup>	2	2.1E-04
Sr <sup>2+</sup>	0.1	2.3E-06
Al <sup>3+</sup>	0.1	1.1E-05
Fe <sup>2+</sup>	0.1	3.6E-06
Zn <sup>2+</sup>	0.1	3.1E-06
HCO <sub>3</sub> <sup>-</sup>	93	1.5E-03
SO <sub>4</sub> <sup>2-</sup>	23	4.8E-04
Cl <sup>-</sup>	10	2.8E-04
NO <sub>3</sub> <sup>-</sup>	11	1.8E-04
CO <sub>3</sub> <sup>2-</sup>	7	2.3E-04
F <sup>-</sup>	1	5.3E-05

**TABLE III**  
Operating Conditions for PWTP Chabazite Zeolite Columns

Column Scale	Zeolite Volume, L	L/D Ratio <sup>a</sup>	Residence Time, min	Distribution Coefficient, K <sub>d</sub>	Number of Transfer Units, N
Bench	0.020	7.1	7.0	3,100	16.7
Pilot	566	1.6	10	3,100	7.0
Near-full	3760	0.63	13	2,600	15.0

<sup>a</sup> Length-to-diameter ratio

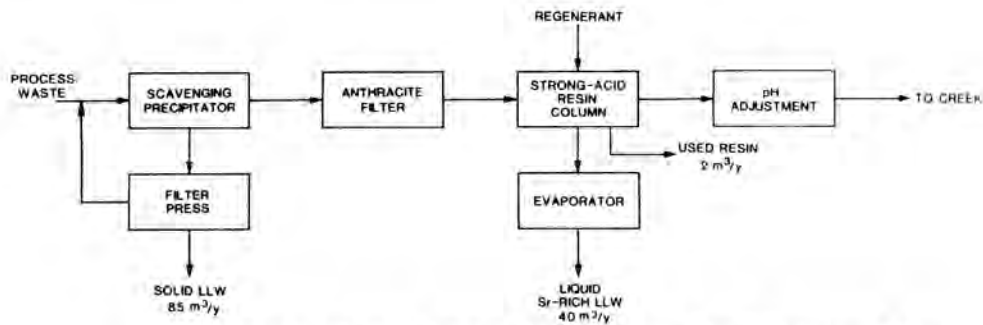


Fig. 1. Flowsheet for the Existing ORNL Process Waste Treatment Plant.

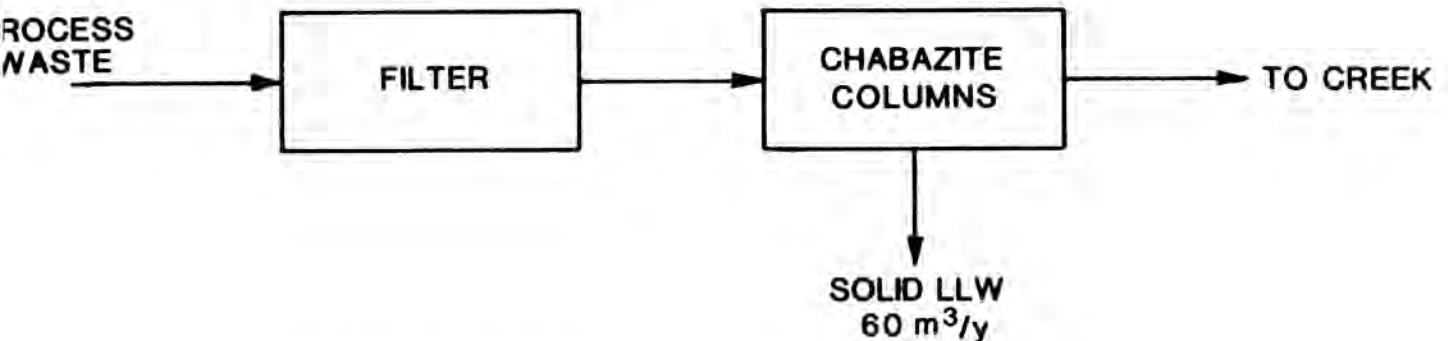


Fig. 2. Flowsheet for the Proposed ORNL Process Waste Treatment Plant.

filled with fresh material, and placed last in the train of columns.

These studies (3,4) successfully demonstrated the feasibility of zeolite systems to treat ORNL process wastewater and identified two areas where additional development was needed: (a) the zeolite columns tend to plug more easily than organic resin columns and (b) design of the columns is a major factor determining the efficiency of the zeolite system. Additional studies have shown that problems with column plugging can be eliminated by proper prefiltration and backwashing systems. Results from laboratory-scale, pilot-scale, and near-full-scale column tests indicated that full-scale columns would generate between  $2E-04$  and  $4E-04$   $m^3$  of loaded zeolite per  $m^3$  of treated wastewater. The resulting annual waste disposal costs vary between \$80,000 and \$170,000 based on the present charges of \$1400/ $m^3$ . These results indicate that design of zeolite ion exchange systems for minimization of secondary waste is the key to economic operation of the zeolite ion exchange plant for treatment of radioactive waste.

### THEORY

Although fixed-bed ion exchange has been an important unit operation for purification and component recovery for many years, the analysis of its application to this process is unexpectedly complex. Ion exchange in a fixed-bed col-

umn is characterized by a breakthrough curve which is the effluent concentration profiles as a function of throughput (i.e., the volume of liquid processed). The rate of appearance and the shape of breakthrough profiles depend on several factors, including the physical and chemical properties of the sorbate and sorbent, the equilibrium relationship between the solution and solid phases, the rate-limiting mass transfer mechanisms (film diffusion, particle diffusion, pore diffusion, axial dispersion, or reaction kinetics), the bed depth, and the fluid velocity.

The objective of fixed-bed column design is to predict the characteristics of the breakthrough curve for a particular column operating under a given set of conditions. For a given component, the convective diffusion equation must be solved as a function of time and column position. Analytical solutions for breakthrough curves can only be obtained for the simplest ion-exchange systems. If analytical solutions cannot be obtained, ion-exchange columns must be designed by empirically fitting experimental data for scale up or numerically solving the transport equations which describe the ion-exchange process. Each of these options is discussed below.

### Analytical Solutions

Analytical solutions for binary ion exchange have been obtained for isothermal plug-flow systems with linear and

irreversible equilibrium (a rectangular isotherm). Solutions have also been obtained for plug-flow systems with constant-pattern equilibrium relationships (the mass transfer front does not change shape as it moves down the column) where mass transfer is controlled by a single mechanism. The analytical solutions are summarized in standard text and reference books (5,6). Three dimensionless parameters are usually used to define the shape of these breakthrough curves: (a)  $N$ , the number of transfer units, represents the bed length, (b)  $T$ , the throughput parameter, represents operating time or the amount of feed processed; and (c)  $R$ , the separation factor. Experimental breakthrough curves can be matched to theoretically-derived graphs to determine values of  $N$ ,  $R$ , and  $T$  for the appropriate ion-exchange equation.

If more than one mass-transfer mechanism controls the ion exchange rate or if the equilibrium equations are complex, the system can sometimes be approximated as a second-order chemical reaction. A general analytical solution for a system which obeys the second-order reaction rate law with nonlinear equilibrium (Langmuir isotherm) has been solved by Thomas (6). The three dimensionless parameters  $N$ ,  $R$ , and  $T$  described above are also used in the solution to this equation. Experimental breakthrough curves can be matched to theoretically-derived graphs to determine values of  $N$ ,  $R$ , and  $T$  for the Thomas equation.

Guidelines are available in the Chemical Engineers' Handbook (6) for empirical scaleup of ion exchange systems using  $N$ ,  $R$ , and  $T$ . To predict scaleup, one must determine how  $N$  and the slope of the breakthrough curve change with column size and operating conditions. The number of transfer units,  $N$ , is a function of several parameters:

$$N \propto vS^{-a} F^{a-1} d^{a-2} D^{1-a} \quad (\text{Eq. 1})$$

where  $v$  is the volume of sorbent,  $S$  is the cross sectional area of the column,  $F$  is the volumetric flow rate of the fluid phase,  $d$  is the particle diameter, and  $D$  is the diffusion coefficient. The exponent,  $a$ , has a value of zero for particle or pore diffusion, 0.5 for film diffusion, 1.0 for axial dispersion, and 2.0 for molecular diffusion. If the controlling mass transfer mechanism is known in terms of ratios (Eq. 1) and not changing,  $N$  can be calculated, based on any reference  $N$ , for each set of conditions. If the flow rate or particle size change during scaleup, the following proportionality must be applied to determine the change in the slope of the breakthrough curve:

$$\left( \frac{N_{\text{exp}}}{N_{\text{pred}}} \right)^{\beta} = \frac{(T-1)_{\text{pred}}}{(T-1)_{\text{exp}}} \quad (\text{Eq. 2})$$

where the exponent  $\beta$  depends on both  $R$  and  $N$ . Applicability of these equations depends on the stoichiometry,

equilibrium, and the controlling mass transfer mechanisms remaining constant.

### Mathematical Modeling

Analytical solutions are not available for systems with complex mass transfer relations, complex equilibrium relations, and/or irregular boundary conditions. Simultaneous integration of the following equations is required: (a) fluid phase and solid phase mass balances, (b) equations for each mechanism affecting mass transfer, and (c) the equilibrium equation. The resulting partial differential equations are often highly nonlinear, and numerical methods are generally required to obtain their solution.

Models of multicomponent liquid ion-exchange systems were virtually nonexistent prior to the 1980s. Several multicomponent models have been developed in the last decade, but models which are based purely on theory are generally restricted to gaseous or non-interactive systems. Although a considerable effort has been made in this area, multicomponent models have not been developed to the point where they can be used in general industrial applications without using laboratory- or pilot-scale data to predict the equilibrium and mass transfer relationships.

### Pilot Plant Approach

Ion exchange systems can be designed using pilot-plant data only, but this method has several disadvantages. The pilot plant design approach acknowledges the system specificity for particular applications, but it tends to be very time consuming and expensive. The approach does not lend itself easily to predicting system responses to variables other than those specifically tested. In addition, there is always a degree of uncertainty associated with scaleup of pilot-scale data.

Although most industrial multicomponent ion-exchange columns are designed using these scale-up techniques, the systems cannot be optimized without the use of more accurate models (7). The optimization of ion exchange columns becomes increasingly important in applications requiring high efficiency to meet low discharge requirements where the disposal costs for the secondary waste are extremely high. The logistics, time, expense, and uncertainty associated with pilot-plant-based designs make the use of mathematical models necessary for such systems.

### SCALEUP OF EXPERIMENTAL DATA USING THE THOMAS MODEL

Bench-scale, pilot-scale, and near-full-scale zeolite ion-exchange column tests (3) performed at the PWTP using columns as shown in Figs. 3, 4, and 5. The operating conditions for these tests are summarized in Table III. The resulting breakthrough curves, shown in Figs. 6, 7, and 8, were fitted to the theoretically derived curves for the

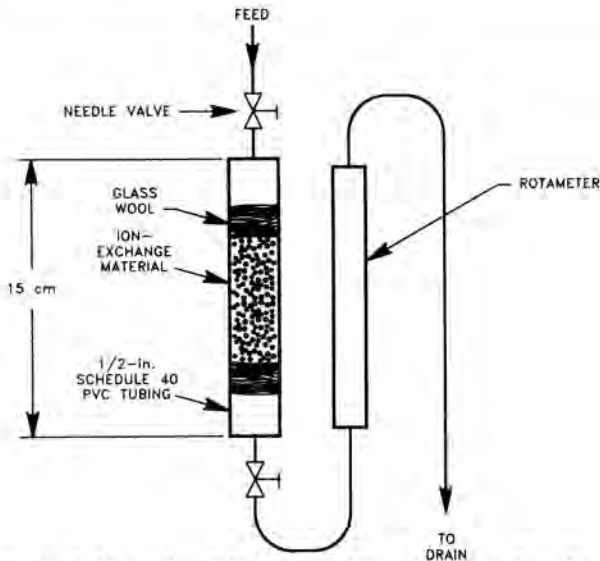


Fig. 3. Schematic of the Ion Exchange Column Used in Bench-Scale PWTP Tests.

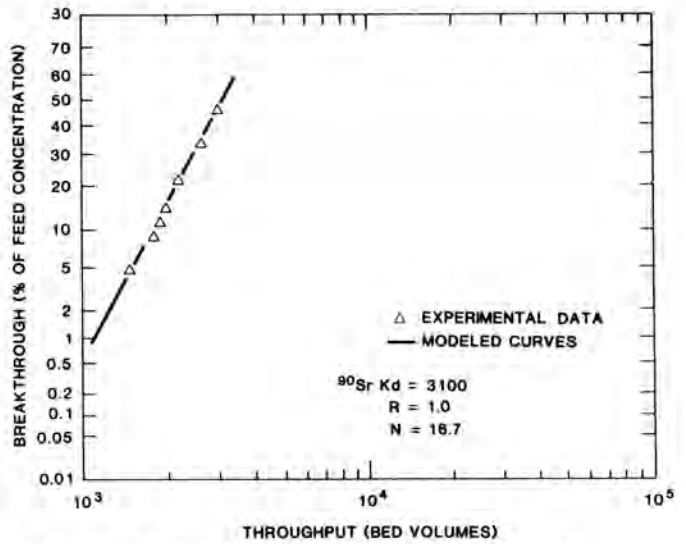


Fig. 6. Sr-90 Breakthrough Curve for Bench-Scale Zeolite Column.

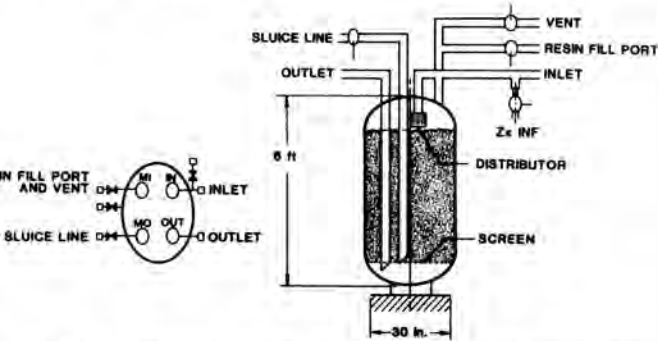


Fig. 4. Schematic of the Ion Exchange Column Used in Pilot-Scale PWTP Tests.

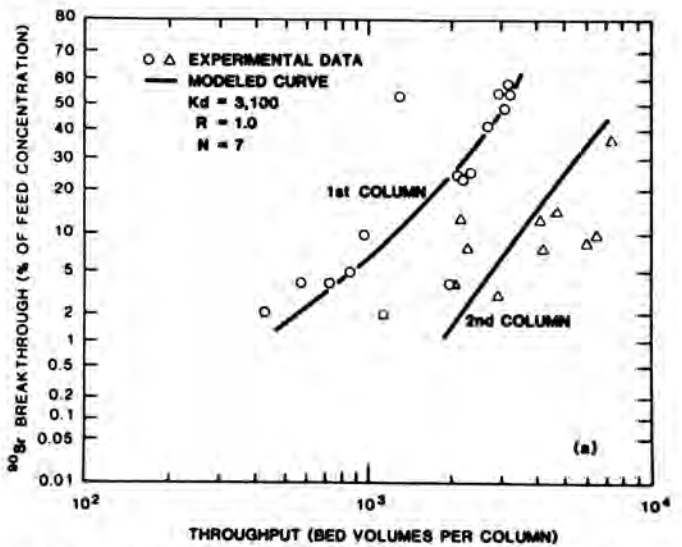


Fig. 7. Sr-90 Breakthrough Curve for Pilot-Scale Zeolite Column.

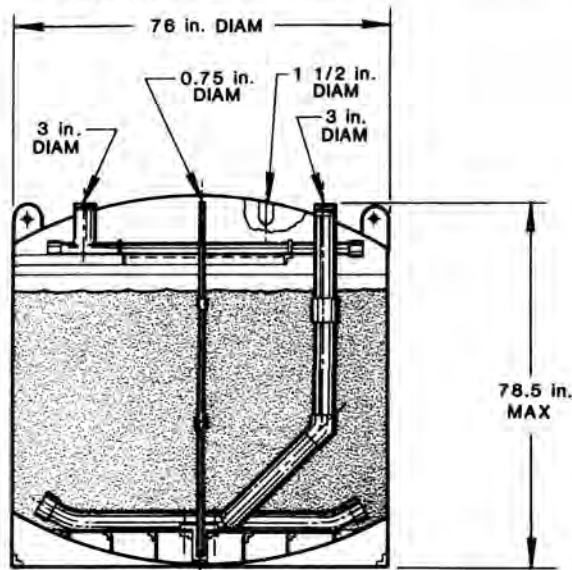


Fig. 5. Schematic of the Ion Exchange Column Used in Near-Full-Scale PWTP Tests.

Thomas model to determine the values of  $N$  and  $T$ , assuming  $R$  was equal to one (which is a reasonable assumption for dilute systems). The resulting values of  $N$  and the volume-based distribution coefficient,  $K_d$ , are also given in Table III and Figs. 6, 7, and 8. Equations 1 and 2 were then used to predict scaleup from the 20-mL column data. The resulting values of  $N$  for various controlling mass-transfer mechanisms are listed in Table IV. The predicted values for  $N$  by the Thomas model were much higher than those obtained experimentally, indicating that the Thomas model did not adequately address nonidealities associated with the interactive multicomponent liquid system and therefore cannot be used to design columns for the PWTP.

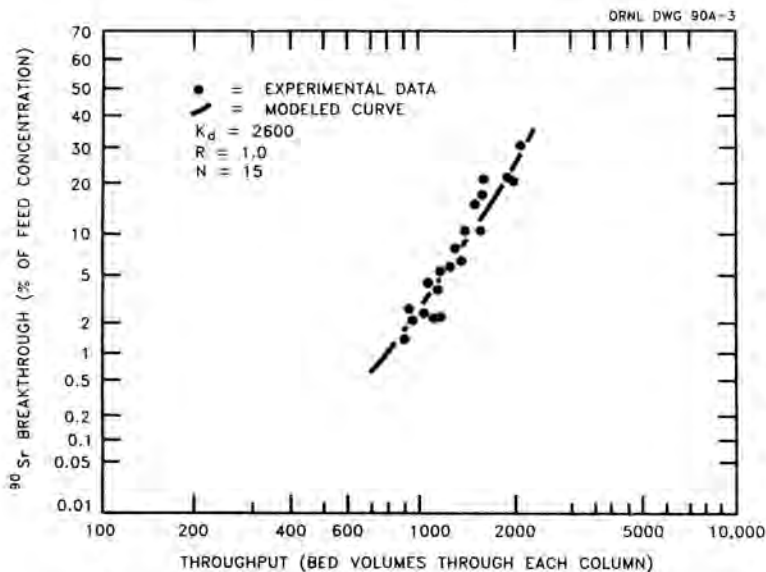


Fig. 8. Sr-90 Breakthrough Curve for Near-Full-Scale Zeolite Column.

#### MATHEMATICAL MODELING OF ZEOLITE COLUMNS FOR WASTEWATER TREATMENT

A predictive mathematical model is being developed for the design optimization of ion-exchange columns containing chabazite zeolites. This project consists of three phases: (a) equilibrium studies, (b) mass transfer studies, and (c) mathematical modeling. The first phase includes the determination of equilibrium isotherms for single and multicomponent cations in an aqueous solution. In the second phase, the controlling mass transfer mechanisms will be determined for these solutions under various conditions. Finally, a mathematical model will be developed and compared with data taken on various sizes of experimental columns. To date, only the equilibrium studies have been performed, and they are summarized below.

Ionsiv IE-96, a synthetic commercially available chabazite manufactured by Union Carbide, was used as the sorbent in these studies. It was obtained from the manufacturer in the hydrated sodium form as 20 to 50 mesh (840 to 297  $\mu\text{m}$  irregularly shaped particles).

The experimental tests in this study were performed using simulated wastewater solutions to avoid variability in feed composition. Simulated wastewater was prepared by dissolving various amounts of  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CsCl}$ ,  $\text{CaCl}_2$ , and  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  in demineralized water. The solutions containing strontium and cesium were also spiked with Sr-85 and Cs-137 tracers, respectively. The composition of the simulated wastewaters was held as near to the concentrations listed in Tables I and II as possible, but the strontium and cesium concentrations were increased to  $2.0\text{E-}3$  and  $1.0\text{E-}3 \text{ N}$ , respectively, which could be accurately measured by the atomic adsorption or gamma counting analytical equipment. The concentration of cations in the zeolite were calculated from mass balances using the initial and final solution concentrations. All tests were performed in triplicate at room temperature ( $25^\circ \text{C}$ ).

The total ion-exchange capacity of Ionsiv IE-96 was determined for  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{CsCl}$ ,  $\text{SrCl}_2$ , and a  $\text{CsCl-SrCl}_2$  mixture. The zeolite was contacted with 20 mL of 2 N NaCl per gram of zeolite overnight to assure that it was in the sodium form, washed by mixing with 20 mL of deionized water for 30 min, and dried at  $110^\circ \text{C}$ . The zeolite was then contacted with 20 mL of 0.2 N of the exchange solution overnight, then weighed, centrifuged for 30 minutes at 5000 relative centrifugal force, and the supernate was analyzed by atomic adsorption for sodium. The results indicated that Ionsiv IE-96 has a cation exchange capacity of 2.24 meq/g when the solution phase does not contain cesium and 2.60 meq/g when cesium is present.

Equilibrium isotherms were obtained using a batch method where the volume in the solution phase was held constant at 10 mL and the solids dosage was varied between 0.002 and 0.4 g. The solution and solids were contacted for

TABLE IV  
Number of Transfer Units for Column Scale-Up Using the Thomas Model

Column Scale	Experimentally Determined Values	Predicted Values for Various Controlling Mass Transfer Mechanisms			
		Particle Diffusion	Film Diffusion	Axial Dispersion	Molecular Diffusion
Bench	16.7	-- <sup>a</sup>	--	--	--
Pilot	7.0	24	68	19	150
Near-full	15.0	46	306	215	9,940

<sup>a</sup>Base Case

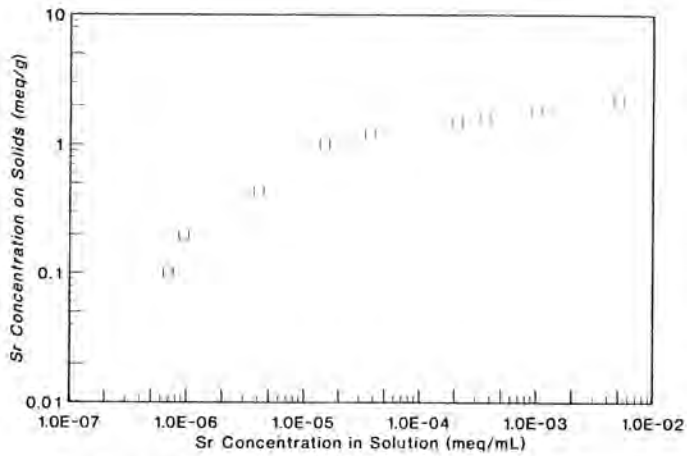


Fig. 9a. Isotherms for Binary Ion Exchange. (a) Sr-Na; (b) Cs-Na; (c) Ca-Na; (d) Mg-Na.

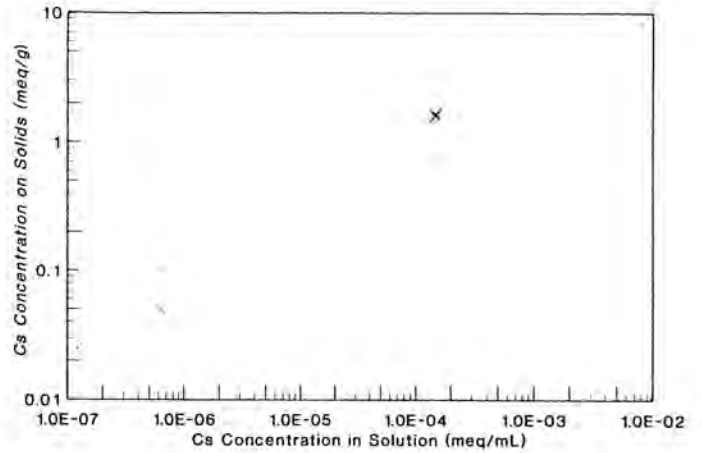


Fig. 9b. Isotherm for Sr-Cs-Ca-Mg-Na Ion Exchange.

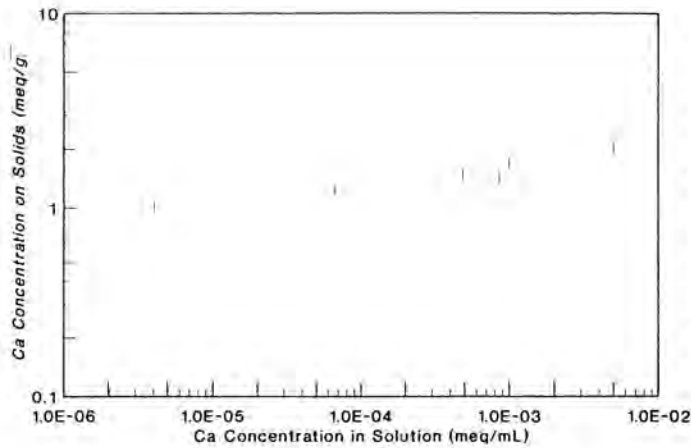


Fig. 9c. Ca Concentration in Solution (meq/mL).

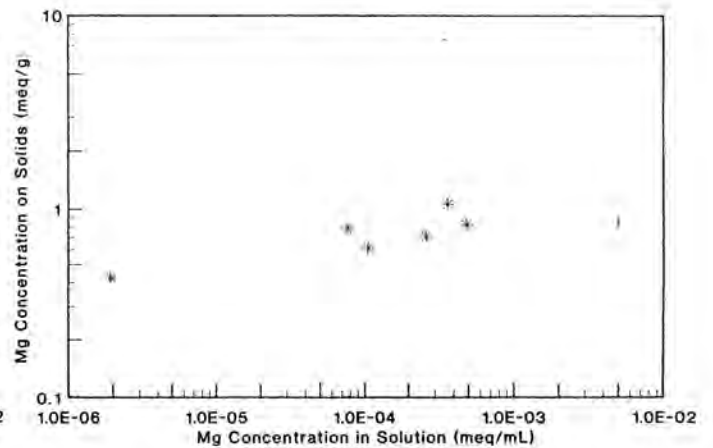


Fig. 9d. Mg Concentration in Solution (meq/mL).

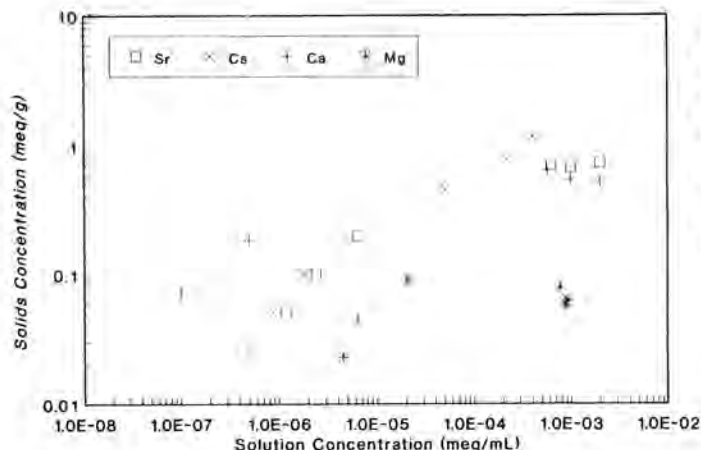


Fig. 10. Isotherm for Sr-Cs-Ca-Mg-Na Ion Exchange.

24 to obtain each isotherm point. The initial concentrations of the exchanging cations in the solution phase were 2.0E-03 N Sr, 1.0E-03 N Cs, 2.0E-03 N Ca, and/or 8.0E-04 N Mg. The binary isotherms are given in Fig. 9, and multicomponent isotherms are shown in Fig. 10.

This isotherm data indicate that Ionsiv IE-96 has the following selectivity for the cations tested: Cs > Sr > Ca > Mg. The data in Fig. 9 indicate that the binary isotherms are selective for the entering cations over the entire range of zeolite composition. This type isotherm has been classified as a Type I isotherm and is typical for zeolites

(5). Type I isotherms are said to be "favorable" for the uptake of the solute.

The shape of the isotherm for cesium, the cation with the highest selectivity, in the multicomponent system (Fig. 10) was similar to that of the corresponding binary isotherm. However, in the multicomponent system, cation interactions affected the isotherms for the species with lower selectivities. The cations with lower selectivities (Ca and Mg) were displaced when the solution concentration increased relative to the available exchange sites. This phenomenon was more pronounced as the selectivity decreased and resulted in a magnesium isotherm with a convex shape.

Standard isotherm equations were used to model the data shown in Figs. 9 and 10. The Langmuir model,

$$\frac{q_i}{q_{ai}} = \frac{b_i c_i}{1 + b_1 c_1 + b_2 c_2 + \dots} \quad (\text{Eq. 3})$$

the Freundlich model,

$$\frac{q_i}{q_{ai}} = \frac{b_i c_i^{n_i}}{b_1 c_1^{n_1} + b_2 c_2^{n_2} + \dots} \quad (\text{Eq. 4})$$

and the Dubinin-Polyani model,

$$q = \exp \left\{ b_0 + b_1 \times \ln(c) + b_2 \times [\ln(c)]^2 \right\} \quad (\text{Eq. 5})$$

each fit the binary data. In these equations,  $q$  and  $c$  are the equilibrium concentrations in the solid and liquid phases, respectively,  $q_a$  is the saturation concentration in the solid, and the  $b$ 's and  $n$ 's are coefficients fitted to the experimental data. The Dubinin-Polyani model was the best equation for predicting the data for all four systems. The coefficients for each cation are given in Table V. The multi-

TABLE V  
Parameters for Isotherm Equation

Isotherm	$b_0$	Coefficients			$R^2$
		$b_1$	$b_2$		
Binary Systems					
Sr Isotherm	-1.635	-0.6835	-0.05013		0.97
Cs Isotherm	-2.165	-0.8850	-0.06261		0.95
Ca Isotherm	1.827	0.2641	-0.00954		0.96
Mg Isotherm	-0.465	-0.1279	-0.01202		0.74
Multicomponent System					
Sr Isotherm	-4.742	-1.157	-0.07507		0.99
Cs Isotherm	-1.532	-0.5890	-0.05002		0.99
Ca Isotherm	-4.212	-0.8560	-0.04703		1.00
Mg Isotherm	-17.28	-3.345	-0.1818		0.93



component isotherms were not accurately predicted using the standard multicomponent Langmuir, Freundlich, Langmuir-Freundlich,

$$\frac{q_i}{q_{ai}} = \frac{b_i c_i^{n_i}}{1 + b_1 c_1^{n_1} + b_2 c_2^{n_2} + \dots} \quad (\text{Eq. 6})$$

and the Ideal Adsorbed Solution Theory Model (IAST). The IAST is based on the Gibbs adsorption equation and only requires single-solute data to predict multicomponent equilibrium. The Dubinin-Polyani equation accurately modelled each of the multicomponent isotherms. These coefficients (shown in Table V) will be used in the ion-exchange column model.

### SUMMARY

The ORNL PWTP will be upgraded using a series of chabazite zeolite columns to remove Sr-90 and Cs-137 from wastewater. Bench-scale, pilot-scale, and near-full-scale tests have indicated that standard scaleup techniques cannot be used to optimize the zeolite column design. Since the optimization of ion-exchange columns is the key to economic operation of this system, a predictive mathematical model is being developed for this purpose. This model will incorporate the effects of mass transfer mechanisms and equilibria on the column dynamics for multicomponent liquid ion exchange with zeolites. Once verified by pilot tests, this model can be used to examine design parameters other than those directly measured and project column response and sensitivity to various variables. Because the model is based on the transport and equilibrium parameters of the system, it should also greatly reduce the need for

future pilot-scale tests and reduce the uncertainty involved in the design of full-scale zeolite columns.

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