

An Evaluation of Lithium Sorption Isotherms and Their Application to Ground-Water Transport

by Brent D. Newman^a, Hector R. Fuentes^b, and Wilfred L. Polzer^c

Abstract

Batch adsorption and desorption equilibrium experiments were used to evaluate lithium sorption on crushed tuff in contact with ground water. The laboratory studies were conducted in support of saturated zone characterization field tests to be performed near Yucca Mountain, Nevada. These studies included equilibrium experiments conducted at 38°C (expected subsurface temperature) for a 1- to 2,000-mg/l range of initial lithium solution concentrations. Data from the equilibrium experiments were used in the evaluation of Linear, Langmuir, Freundlich, and Modified Freundlich isotherm expressions.

Comparisons of adsorption and desorption isotherms showed that lithium adsorption was reversible for solution concentrations up to 150 mg/l. This conclusion was based on F-test comparisons of predicted adsorption and desorption data from Langmuir isotherms.

Lithium sorption was best represented by the nonlinear isotherms where the Langmuir gave the best fit followed by the Modified Freundlich and Freundlich. When these isotherms were used in a one-dimensional, column adsorption transport code, differences occurred in the predicted breakthrough curves. The transport results showed differences of as much as a factor of five in the time of arrival of lithium for the various isotherms depending on the influent concentration chosen. The differences in the simulation breakthrough curves indicate the importance of isotherm selection. Statistical discrimination and column experiments are suggested as better evaluations of isotherms that are to be incorporated into field-scale transport simulations.

Introduction

Yucca Mountain, Nevada, is currently the only candidate site for the first high-level nuclear waste repository in the United States (Figure 1). Extensive site characterization is required to establish that the site will be an adequate host for long-term storage of these wastes (Nuclear Waste Policy Act, 1982).

The stratigraphy of Yucca Mountain that includes the proposed repository location contains a series of Upper Miocene tuff units (Figure 2). The oldest unit is the Crater Flat Tuff Formation which is approximately 13.5 million years (m.y.) in age and is composed of the Tram, Bullfrog, and Prow Pass Members. The three members comprise a

sequence of calcalkaline rhyolitic ash flow tuffs (Dockery et al., 1985).

The Crater Flat Tuff Formation is overlain by the tuffaceous beds of the Calico Hills (13 to 13.5 m.y.) that are characterized by a variable tuff lithology. The Calico Hills beds consist of nonwelded ash-flow, air-fall, and reworked tuffs and are heavily zeolitized. This unit is considered to be an important potential near-site sorptive and low-permeability barrier to radionuclide transport (Dockery et al., 1985).

The Topopah Spring Member of the Paintbrush Tuff Formation (12 to 13 m.y.) lies above the Calico Hills tuffs and is a multiple-flow compound cooling unit. The welded, devitrified portion of the Topopah Spring is the proposed host rock for the candidate repository. This unit is the thickest and most extensive member of the Paintbrush Tuff Formation and was selected as a potential host rock because of its lateral continuity, dense welding, and stratigraphic setting above the low-permeability Calico Hills tuffs (U.S. Department of Energy, 1988b).

A bedded tuff layer overlays the Topopah Spring Member, and it is overlain by the Tiva Canyon Member of the Paintbrush Tuff. The Tiva Canyon is a densely welded multiple-flow compound cooling unit and caps most of the surface of Yucca Mountain (U.S. Department of Energy, 1988b).

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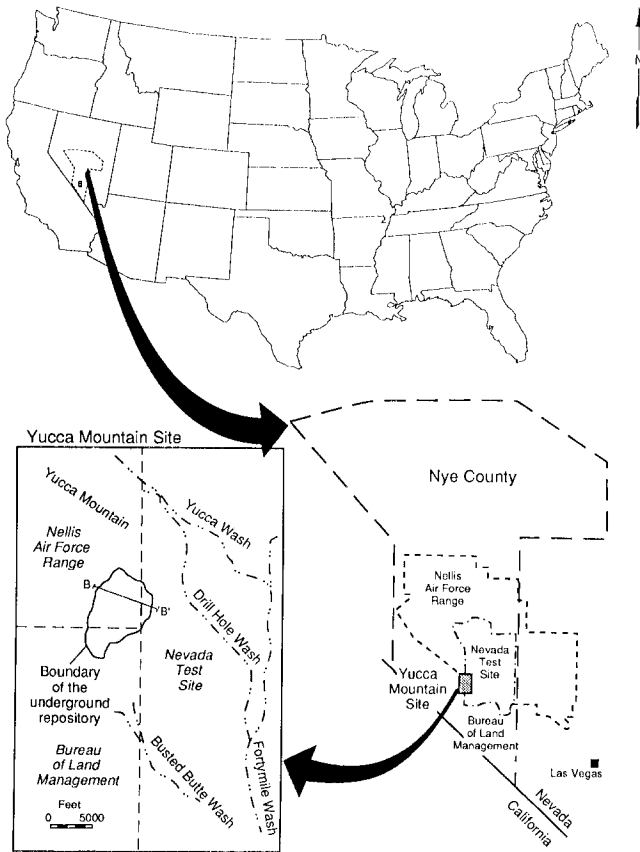


Fig. 1. Location of Yucca Mountain site. The line labeled B-B' marks the location of the cross section shown in Figure 2 (from U.S. Department of Energy, 1988a).

As part of the saturated zone characterization at Yucca Mountain, in situ field tests will be performed at the C-Well complex near the southeastern edge of the mountain (Springer, 1989). Reactive (nonconservative) and nonreactive (conservative) ground-water tracers will be used to evaluate the retardation potential of the Bullfrog and Prow Pass Members of the Crater Flat Tuff Formation (Figure 2). Work to date has focused on lithium as a reactive tracer. Bromide will be used as the conservative tracer.

Objectives of this paper include the evaluation of (1) the reversibility of lithium adsorption; (2) the effectiveness of four isotherm models to represent lithium adsorption; and (3) the potential impact of the estimated isotherm parameters on transport simulations.

Lithium (Li^+) as lithium bromide (LiBr) was evaluated in conventional batch reactors containing Prow Pass tuff suspended in samples of the regional ground water. Lithium was selected because of its expected physical mode of sorption, a relatively weak adsorption that should result in a reasonable field recovery time, high solubility, minor environmental impact, and low cost (Fuentes et al., 1988).

Techniques to study retardation include various batch and column approaches. The batch mode was used here because it provided a rapid and economic way to get information on the effect of different variables on adsorption. Certainly, column experiments are more useful in measuring retardation under flow conditions, and are planned for future work.

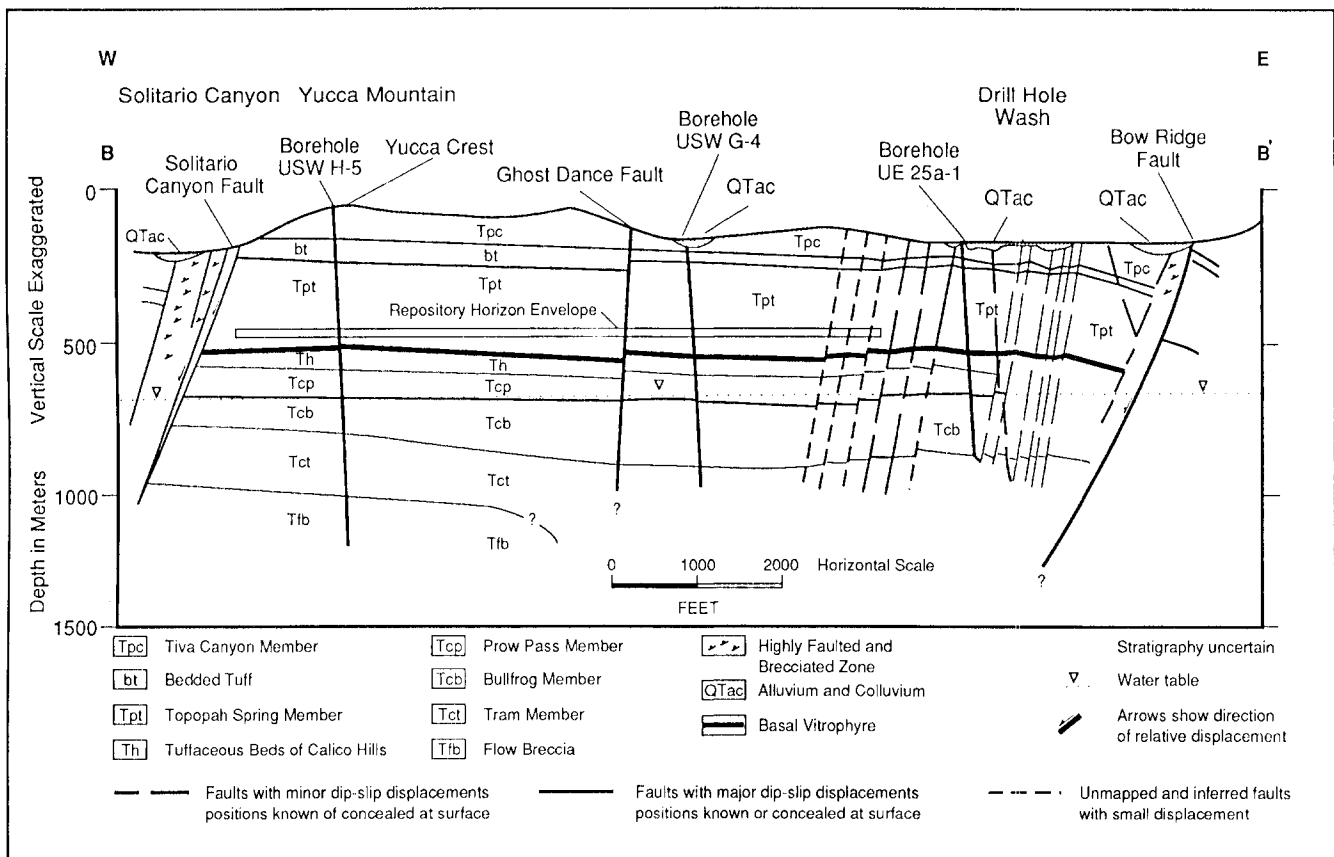


Fig. 2. Geologic cross section of Yucca Mountain (from U.S. Department of Energy, 1988a).

Linear, Langmuir, Freundlich, and Modified Freundlich isotherms were used to relate the lithium surface concentration to the lithium concentration in solution at equilibrium. Isotherms were developed from the experimental data using best-fit statistics in order to select the best model(s) for use in laboratory (column) and field-scale transport codes.

Desorption isotherms were developed to evaluate the reversibility of lithium adsorption. F-test comparisons of adsorption and desorption isotherms were made to determine if there was a significant difference between the two. Reversibility is indicated by no significant difference between the adsorption and desorption isotherms. A significant difference indicates no or limited reversibility.

Emphasis has been given to the representation of adsorption by isotherms because of their widespread application in transport modeling. However, it should be noted that there is a need to study the fundamental mechanisms controlling the results presented here. Such a study is not within the scope of this paper.

Methods and Materials

Reagent grade lithium bromide from EM Science was used as the Li^+ source in the batch experiments. Ground water was collected from Well J-13 on the Nevada Test Site for use in these experiments. J-13 water has been used extensively as a reference water in adsorption site characterization experiments for the Yucca Mountain Project (Thomas, 1987). The J-13 water was characterized using inductively coupled plasma atomic emission spectroscopy (ICPAES), inductively coupled plasma mass spectroscopy (ICPMS), and ion chromatography. Tuff cuttings from the Prow Pass Member were obtained from Well UE-25P #1 (located near the C-Well complex). The cuttings were crushed, and the fraction that passed through a 500- μm sieve was collected for use in the experiments and for x-ray diffraction analysis. The cation exchange capacity of the <500- μm fraction was determined using a sodium acetate procedure (U.S. Salinity Laboratory, 1954).

Ten batch adsorption kinetics experiments were conducted in glass vessels with initial lithium concentrations of 15, 75, 100, 250, and 2,000 mg/l in order to establish minimum equilibration times for the equilibrium experiments. These experiments were conducted at 38°C in a temperature-controlled chamber because 38°C is the expected downhole temperature for the field experiments. Samples were taken over time to characterize concentration changes from 1 to 12 hours. Sampling time intervals varied between 20 seconds after the experiment start-up and 2 hours by the end of the experiment. Lithium desorption kinetics was evaluated using the same experimental configuration as the adsorption experiments. J-13 water was added to lithium-treated tuff, and then desorption was monitored at 3, 6, 9, 24, and 96 hours.

Equilibrium batch experiments were conducted at 38°C and 50-ml polyallomer centrifuge tubes were used as contact vessels. Solid/liquid ratios of either 1:10 or 1:20 were used in the adsorption experiments. Initial lithium concentrations for adsorption were 0, 1, 5, 10, 25, 50, 100,

250, 500, 750, 1,000, and 2,000 mg/l, and each experiment was performed in duplicate. Vessel contents were allowed to equilibrate on a rotating tumbler for 24 hours before sampling. Equilibrium desorption experiments were conducted after the adsorption step using the same vessels and contents. The initial lithium solution was removed by centrifuging and decanting the supernatant. J-13 water was added to retain the same solid/liquid ratio as in the adsorption stage. The vessels were weighed to determine the amount of remaining solution after decanting. Because the concentration of the remaining solution was known from the adsorption step, the lithium concentration at the start of the desorption step could be calculated from the amount of J-13 water added to desorb the lithium. Thus, the desorption results reflect the small amount of nonsorbed lithium present at the start of the desorption experiments. Vessel contents were then allowed to re-equilibrate on a rotating tumbler for 24 hours before sampling.

Sampling in both the kinetics and equilibrium experiments involved withdrawal of solution (e.g., 10-20 ml) through a 0.45- μm filter using a plastic syringe. Samples were then acidified to 1% nitric acid. Sample splits for anion analysis were not acidified. Analyses for the major groundwater cations and anions were performed using ICPAES, ICPMS, and ion chromatography.

The amount of lithium adsorbed on Prow Pass tuff at equilibrium was determined from the following mass balance:

$$S = [(C_0 - C)V]/M \quad (1)$$

where S = amount of lithium adsorbed ($\mu\text{g/g}$); C_0 = initial aqueous lithium concentration ($\mu\text{g/ml}$); C = aqueous lithium concentration at equilibrium ($\mu\text{g/ml}$); V = solution volume (ml); and M = mass of the reactor solids (g). Parameters for the Linear (Travis and Etnier, 1981), Langmuir (Langmuir, 1918), Freundlich (Sposito, 1980) and Modified Freundlich (Murali and Aylmore, 1981) isotherms were determined using the following equations (Polzer et al., 1985):

Linear Isotherm:

$$S = K_d C \quad (2)$$

where S = amount of solute adsorbed ($\mu\text{g/g}$); C = concentration of solute in solution ($\mu\text{g/ml}$); and K_d = distribution coefficient (ml/g).

Langmuir Isotherm:

$$S = kbC/(1 + kC) \quad (3)$$

where k = Langmuir adsorption constant (ml/ μg); and b = maximum amount of sorbed solute ($\mu\text{g/g}$).

Freundlich Isotherm:

$$S = KC^N \quad (4)$$

where N = empirical constant describing the spread of distribution coefficients about an average distribution coefficient; and K = empirical constant related to an average distribution coefficient (ml/ μg adsorbate) ^{N} ($\mu\text{g/g}$).

Modified Freundlich Isotherm:

$$S = (S_{\max} K_D^\beta C^\beta) / (1 + K_D^\beta C^\beta) \quad (5)$$

where β = empirical constant describing the spread of distribution coefficients about an average distribution coefficient (K_D); K_D = average distribution coefficient (ml/mg); and S_{\max} = maximum amount of solute that can be sorbed ($\mu\text{g/g}$).

The Linear isotherm was evaluated using the linear regression GLM method in SAS (Statistical Analysis Systems, 1990). Values for the various constants used in the three nonlinear isotherms were determined using the nonlinear DUD ("doesn't use derivatives") method in SAS.

Transport simulations using influent concentrations of 1, 100, and 1,000 mg/l were performed to determine the effects of the four isotherm expressions on transport predictions. A computer code that simulates solute transport and solves the advective-dispersive equation [equation (6)] was used to predict breakthrough curves. Adsorption is represented in the equation by either the Linear, Langmuir, Freundlich, or Modified Freundlich isotherms (Robinson, 1990).

$$\frac{\partial C^*}{\partial t^*} + \frac{\rho_b}{\phi} \frac{\partial S^*}{\partial t^*} = D_e \frac{\partial^2 C^*}{\partial x^{*2}} - U \frac{\partial C^*}{\partial x^*} \quad (6)$$

where C^* = Li concentration (Kg/m^3); t^* = time (s); ρ_b = bulk density (Kg/m^3); ϕ = porosity (dimensionless); S^* = amount of Li sorbed, expressed as a form of the Modified Freundlich isotherm (Kg tracer/Kg rock); D_e = effective axial dispersion coefficient (m^2/s); U = fluid velocity (m/s); and x^* = distance along the flow path (m). Isotherm parameters were used as input along with influent concentrations of 1, 100, and 1,000 mg/l and an assumed pecllet number (Pe) of 40. The Pe number is used in the code to establish a length scale/dispersivity ratio for a simulation. A Pe value of 40 was selected because it was assumed to be representative of Pe values for a typical packed column experiment. The derivation of the length scale/dispersivity ratio from the Pe equation is given below:

$$\text{Pe} = UL/De \quad (7)$$

where L = column length (m).

$$De = U\alpha \text{ (assuming negligible molecular diffusion)} \quad (8)$$

Table 1. Chemical Components of J-13 Water Used in the Lithium Batch Experiments

Element or species	Concentration ($\mu\text{g/l}$)	Element or species	Concentration ($\mu\text{g/l}$)
Al	<0.03*	Mg	<0.01*
B	0.13	Na	44
Ba	<0.001*	Si	30
Ca	11.3	Sr	0.04
Fe	0.02	Cl	7
K	5	PO_4^{3-}	<2.5*
Li	0.04	SO_4^{2-}	19
Mn	0.01	HCO_3^-	124

*limit of detection.

Table 2. Mineralogic Composition of UE-25P #1 Crushed (<500 μm) Prow Pass Tuff as Determined by X-Ray Diffraction

Mineral or mineral group	Fractional percent and error
Feldspars	60 \pm 7%
Quartz	22 \pm 2%
Cristobalite	15 \pm 1%
Hematite	1 \pm 1%
Smectites	1 \pm 1%

where α = dispersivity (m). Replacing De in equation (10) with $U\alpha$ yields:

$$\text{Pe} = UL/U\alpha = L/\alpha \quad (9)$$

The computer code is part of developmental work for the C-well tracer tests to be conducted by Los Alamos for the Yucca Mountain Project (Springer, 1989). The code is in the final stages of documentation, and information is available from Robinson (1990).

Results and Discussion

The elemental composition of J-13 water and X-ray diffraction data for Prow Pass tuff are shown in Tables 1 and 2. The results presented in Table 2 show the fractional percentage of mineral phases present in one sample of the crushed, sieved, and mixed Prow Pass Tuff. The \pm values for the fractional mineral percentages are a result of the propagation of various errors including the 1σ error that is based on the number of reflections measured for a particular phase, the error associated with the purity of the standard, and error associated with compositional variations. A discussion of these quantitative X-ray diffraction considerations is given in Bish and Chipera (1988). The measured cation exchange capacity was 16 meq/100 g of tuff. This value was used as S_{\max} in the Modified Freundlich Isotherm, equation (7).

Results of the adsorption kinetics experiments indicated that equilibrium was reached in about one hour. Data from the desorption kinetics experiments showed that equilibrium was reached in about six hours. Equilibrium was established when the solution concentration remained constant over time. A 24-hour equilibration time was selected for the equilibrium experiments based on these results.

Slower rates of desorption compared to adsorption are commonly observed from sorption batch experiments. One possible reason for lithium behaving in this manner is that when the adsorption step is being performed, there is a relatively large mass of lithium (or higher ionic strength) in solution resulting in a "large driving force" enhancing adsorption kinetics. During desorption there is a relatively small amount of lithium adsorbed so the "driving force" back into solution is relatively small resulting in a slower rate of transfer.

Analyses of adsorbed lithium in equilibrium experiments as a function of concentration showed a high degree of variability between experimental replicates at high solution concentrations. The variability can be attributed largely

to the method of determining adsorbed lithium, as shown in equation (1). At high concentrations, adsorbed lithium represents a small difference between two large concentrations, and the resulting difference carries the relatively large errors from the two concentration values.

An alternative method was used to overcome the variability of lithium data at high concentrations. The method assumes that the summation of calcium, magnesium, potassium, and sodium (meq) displaced by lithium approximates the amount of sorbed lithium. However, the error associated with each cation as low concentrations resulted in a relatively large total error. Therefore, isotherm data sets were formed using lithium data for initial tracer concentrations less than 25 mg/l and summation of cation data for initial concentrations greater than 25 mg/l.

The nonlinear determinations of isotherm parameters were evaluated to assure that the DUD routine did not converge on a local minimum. This was accomplished by varying the initial guess of the parameter values and by contour plotting the two unknown parameters versus the absolute value of the SSE at each iteration. Once acceptable fits were generated the absolute value of the residuals for a given isotherm were plotted versus the independent variable, concentration. The residual plots were used to evaluate the need for weighting during the nonlinear fitting routine. In all cases, no discernible trends were observed in the residual plots, so no weighting was performed.

Regression results and curve-fitting parameters of Langmuir isotherms are shown in Table 3 for adsorption and desorption data. An F-test comparison of the regressions of adsorption and desorption was used to evaluate the reversibility of lithium adsorption. If the F-test comparison between regressions shows no significant difference, reversibility is assumed. If the regressions are significantly different, then adsorption is assumed not to be reversible, or reversibility is assumed to be limited. The F-test comparison of the regressions shown in Figure 3 indicates that there is no significant difference between adsorption and desorption at a 95% confidence level. Thus, lithium adsorption is apparently reversible.

Regression results and curve-fitting parameters for a 1- to 2,000-mg/l range of initial lithium concentrations from batch experiments at 38°C are given in Tables 4 and 5. The

Table 3. Statistical and Parameter Estimates of Langmuir Isotherms for Lithium Adsorption and Desorption in a Prow Pass Tuff Suspension in J-13 Well Water for an Initial Concentration Range of 1 to 150 µg/ml and a Solid/Liquid Ratio of 1:10 at 38°C

Estimate	Adsorption	Desorption
Sum of squares of residuals	421.5	241.6
Mean square of residuals	91.8	48.3
b	0.606	0.679
Asymptotic std. error on b	0.095	0.221
k	0.0002	0.0005
Asymptotic std. error on k	0.0001	0.0008

Units: b = µg Li/g solid; k = ml H₂O/µg Li.

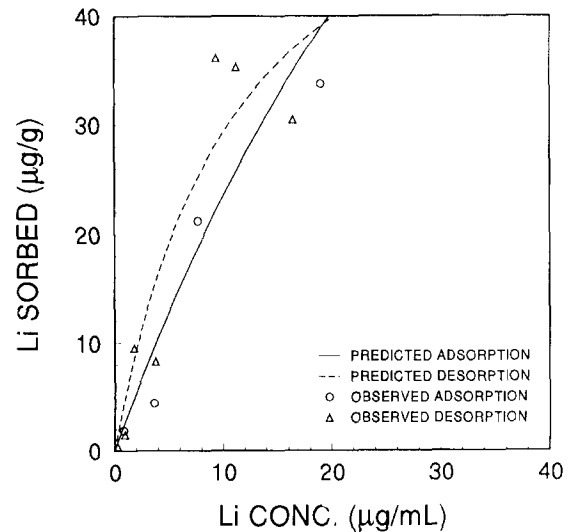


Fig. 3. Observed and predicted adsorption and desorption of lithium on a Prow Pass tuff suspension in J-13 Well water at 38°C for an initial concentration range of 1 to 150 mg/l and a solid/liquid ratio of 1:10. Predicted lithium is based on the Langmuir isotherm.

four isotherms modeled the data reasonably well based on statistical measures of curve fitting. However, as Figure 4 shows, the Linear isotherm fit was much poorer than the nonlinear isotherms. The Langmuir isotherm provided the best fit followed closely by the Modified Freundlich isotherm. One reason the fits of the Langmuir and Modified Freundlich isotherms were better than the others is that they do not allow the amount of adsorption to exceed a maximum. The Linear and Freundlich formulations do not contain constraints on how much adsorbate can be loaded on a surface.

In order to evaluate travel time differences and breakthrough patterns among the Linear, Langmuir, Freundlich, and Modified Freundlich isotherms, the one-dimensional transport code by Robinson (1990) was used. Simulations were run for each isotherm at 1, 100, and 1,000 mg/l influent concentrations. Results for 100-mg/l influent simulations

Table 4. Statistical Measures of Fitting for the Linear, Langmuir, Freundlich, and Modified Freundlich Isotherms for the Adsorption of Lithium on a Prow Pass Tuff Suspension in J-13 Well Water for an Initial Concentration Range of 1 to 2,000 mg/l and a Solid/Liquid Ratio of 1:20 at 38°C

Estimate	Linear	Langmuir	Freundlich	Modified Freundlich
R ²	0.905	NA	NA	NA
CV	45	NA	NA	NA
Square root of MSE	100.8	NA	NA	NA
Sum of squares of residuals	NA	4,446.7	13,211.0	5,767.0
Mean square of residuals	NA	555.8	1,651.4	720.9

R² = index of determination; CV = coefficient of variation; and MSE = mean squared error.

Table 5. Parameter Estimates for the Linear, Langmuir, Freundlich, and Modified Freundlich Isotherms for the Adsorption of Lithium on a Prow Pass Tuff Suspension in J-13 Well Water for an Initial Concentration Range of 1 to 2,000 mg/l and a Solid/Liquid Ratio of 1:20 at 38°C

Parameters	Value	Units
Linear:		
K_d	0.425	ml H ₂ O/g solid
Std. error on K_d	0.046	
Langmuir:		
b	860.8	$\mu\text{g Li/g solid}$
Asymptotic std. error on b	65.05	
k	0.001	ml H ₂ O/ $\mu\text{g Li}$
Asymptotic std. error on K	2×10^{-4}	
Freundlich:		
N	0.543	dimensionless
Asymptotic std. error on N	0.055	
K	11.13	(ml/ $\mu\text{g Li}$) ^N ($\mu\text{g Li/g solid}$)
Asymptotic std. error on K	4.22	
Modified Freundlich:		
β	0.850	dimensionless
Asymptotic std. error on β	0.062	
K_D	8×10^{-4}	ml H ₂ O/ $\mu\text{g Li}$
Asymptotic std. error on K_D	5.6×10^{-5}	

are shown in Figure 5. The breakthrough curves show that arrival times for the nonlinear isotherms are over three times longer than the Linear isotherm prediction. Differences in arrival times and breakthrough development also exist among the Langmuir, Freundlich, and Modified Freundlich isotherms. The 1- and 1,000 mg/l influent simulations produced results similar to the above 100-mg/l simulations with decreasing differences observed among predictions by the isotherms using the 1,000-mg/l influent concentration. Even at 1,000 mg/l, the differences between the predicted breakthrough of lithium for the different isotherms would be important.

Figure 5 shows that the different isotherm formulations can result in significantly different transport predictions. Use of an inappropriate isotherm may cause either over- or underestimation of solute travel times. Selection of inappropriate isotherms also has been discussed by Kinniburgh (1986). His research demonstrated that selection of even "well-fitting" isotherms can result in highly biased estimates. The potential bias in isotherm estimates and the apparent sensitivity of transport simulations to isotherm formulation shown here indicate additional means are needed to select representative isotherm expressions. Column experiments and statistical discrimination of the isotherms coupled to transport codes (Knopman and Voss, 1988) should help to establish more definitively the best isotherm for use in field-scale transport simulations.

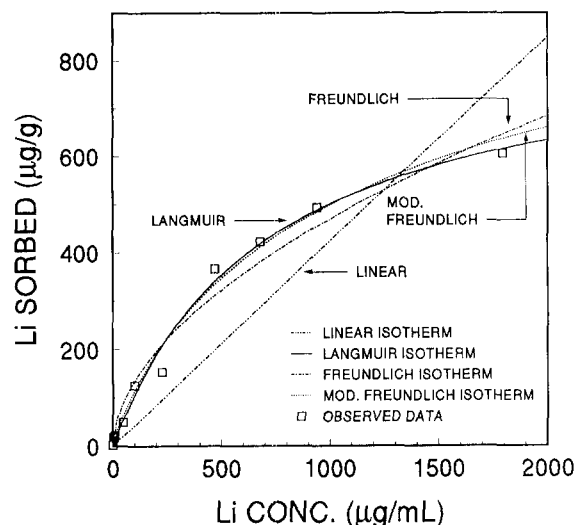


Fig. 4. Observed and Linear, Langmuir, Freundlich, and Modified Freundlich based predicted adsorption of lithium on a Prow Pass tuff suspension in J-13 Well water at 38°C for an initial concentration range of 0 to 2,000 mg/l and a solid/liquid ratio of 1:20.

Summary

Batch adsorption and desorption experiments were performed to evaluate lithium as a reactive (nonconservative) ground-water tracer in saturated zone field tests at the C-Well complex near Yucca Mountain, Nevada. Experimental and modeling activities have established that the sorption of lithium is relatively weak and reversible which is a positive characteristic for field use. These sorptive properties indicate that field recovery should be reasonably fast (i.e., days to months).

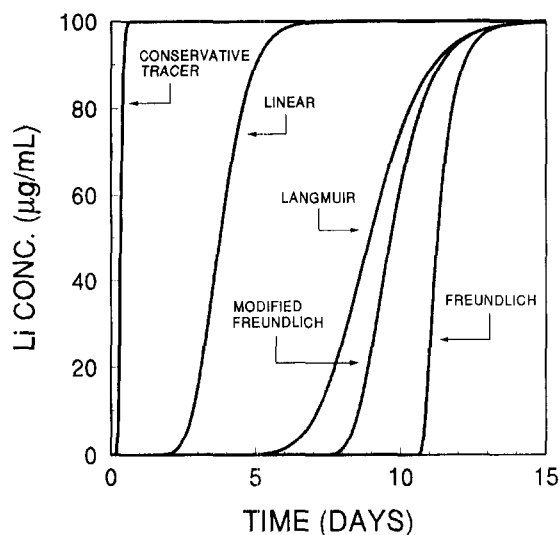


Fig. 5. Breakthrough curves based on transport simulations using a solution to the advective-dispersive equation that represents adsorption equilibrium by either the Linear, Langmuir, Freundlich, and Modified Freundlich isotherms. Influent concentration of the applied step input was 100 mg/l.

The R^2 values from the Linear isotherm fits indicate that this isotherm is effective in modeling lithium adsorption on Prow Pass tuff. However, when compared to the observed data and the nonlinear isotherms, the Linear fits are quite poor. The Langmuir and Modified Freundlich isotherms represent the observed data best, and this is probably related to their formulations which express a maximum adsorption capacity. One-dimensional transport modeling demonstrates that the four isotherms would predict different degrees of retardation. This result shows that isotherm selection is not necessarily straightforward. Therefore, additional means are needed to select appropriate isotherms.

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