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Adsorption of Radionuclides from Aqueous Solutions by Inorganic Adsorbents

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ABSTRACT

Radionuclides such as ^{137}Cs and ^{85}Sr were removed from dilute aqueous solutions by means of inorganic adsorbents, MS-13X and chabazite. Adsorption data of radionuclides on the inorganic adsorbents were satisfactorily correlated by the Dubinin–Astakov (DA) equation. Gaussian-type distributions of adsorption energies were obtained from the parameters of the DA equation by applying the condensation approach. A surface diffusion model was applied to estimate the intraparticle mass transfer of radionuclides. The range of surface diffusion coefficients estimated was 3.2×10^{-12} to 3.5×10^{-11} m^2/s. The adsorption model, which describes the surface diffusion mechanism with the DA equation, satisfactorily simulated the adsorption behavior of radionuclides on zeolites in batch adsorbers.

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INTRODUCTION

Removal of long-lived radionuclides has been extensively studied in the separation and solidification of high level liquid waste (HLLW) (1, 11, 14, 15, 25) and in controlling cooling water contamination from defective spent fuels in storage pools (10, 20). Adsorption by inorganic adsorbents is a promising technique for this purpose (3). Various types of inorganic adsorbents, such as mordenite and chabazite, have been widely used because of their high resistance to radiation and high ion-exchange capacity. Furthermore, radioactive isotopes adsorbed on inorganic adsorbents can be readily immobilized in the form of a stable solid by calcination at high temperatures without additional treatment (25).

Several workers (9, 25) have studied the removal of radionuclides from aqueous solutions. However, it is still not easy to describe the adsorption process quantitatively. Quantitative analysis of adsorption in general requires accurate information on adsorption equilibrium and mass transport of adsorbates. For single species adsorptions, determining the adsorption data presents no serious problem. However, it is essential to choose the most promising isotherm type among various types of theoretical or empirical ones (5). The Langmuir equation (19) has been proven to adequately describe the adsorption of ions in many instances. However, the Langmuir equation is not generally useful in describing adsorption in heterogeneous systems because of its intrinsic assumption of homogeneous sorption sites. The Freundlich equation, usually assumed to be empirical, can be derived from an adsorption model where the adsorption energy decreases exponentially as the amount of adsorbed ions increases. However, the Freundlich equation does not satisfy the required limiting cases, such as Henry's law at concentrations near zero, and the finite adsorption capacity (5).

For this work we selected the Dubinin–Astakov (DA) equation as a single species isotherm for following reasons: 1) good fit of data over wide concentration ranges, 2) good temperature correlation, and 3) easy evaluation of the adsorption energy distribution. The DA equation can be regarded as a hybrid between the Dubinin–Radushkevich (DR) isotherm and the Freundlich isotherm (27).

The disadvantage of adsorption processes is the relative slowness of the removal rate. This drawback should be overcome if this method is applied on an industrial scale. Intraparticle diffusion within porous sorbents depends on the mechanism of adsorbate transport: pore diffusion, surface diffusion, and others. However, the mechanism of adsorbate transport cannot be completely understood because of its complicated

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characteristics. To circumvent this difficulty, a number of workers have dealt with a certain major mechanism as a matter of convenience, and subsequently the effective diffusion coefficient was determined by comparing the experimental data with those predicted by the model (13, 21).

The present work is concerned with the adsorption of radionuclides such as ^{137}Cs and ^{85}Sr onto inorganic adsorbents such as MS-13X and chabazite for the treatment of cooling water contaminated by defective spent fuel assemblies. A model for batch adsorption was formulated by incorporating the DA equation with the intraparticle diffusion equation. The major emphasis of this study is placed on the acquisition of valuable information on adsorption equilibrium and intraparticle diffusion in order to analyze and simulate the adsorption of zeolite filter in storage canisters for controlling cooling water contamination from defective spent fuels.

THEORETICAL APPROACH

Adsorption Equilibria

There are various isotherm equations for single-species adsorptions. Here, equilibrium data will be correlated by the Langmuir, the Freundlich, the Dubinin–Radushkevich (DR), and the Dubinin–Astakov (DA) equations.

$$q = \frac{q_m bc}{1 + bc}; \quad \text{Langmuir} \tag{1}$$

$$q = Kc^{1/n}; \quad \text{Freundlich} \tag{2}$$

$$q = q_m \exp\left\{-\left(\frac{kT}{E}\ln\frac{c_s}{c}\right)^2\right\}; \quad \text{DR} \tag{3}$$

$$q = q_m \exp\left\{-\left(\frac{kT}{E}\ln\frac{c_s}{c}\right)^n\right\}; \quad \text{DA} \tag{4}$$

The DA equation has an empirical basis confirmed by a large number of experiments. Its drawback from a theoretical point of view is the fact that it contains a free energy instead of a true adsorption energy. However, it is possible to link this equation to a simple theoretical model which allows a formal description of its basis and its parameters. The mean energy of adsorption (more correctly, sorption) E is the free energy of transfer of 1 mol of concentration from infinity (bulk solution) to the surface of the adsorbent (7). It has also been interpreted as the most probable adsorption energy on the heterogeneous surface of the adsorbent.

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Intraparticle Diffusion Model

The following assumptions have usually been made in developing an adsorption model (12, 13):

- The concentration of adsorbates is uniform in the batch adsorber.
- The adsorbents are spherical particles with an identical radius.
- Resistance to mass transport between the external solution phase and the particle surface as well as that to intraparticle diffusion is included.

In addition to the above assumptions, the transport mechanism of an adsorbate within the adsorbent particle is often simplified by assuming that the intraparticle transport is governed either by pore diffusion through the pore fluid or by surface diffusion on the pore surface. A more rigorous model would combine and consider the two diffusion mechanisms at the same time.

However, the choice of the type of diffusion is a matter of convenience because two diffusion models give nearly the same predictions in simulating the adsorption behavior (13). Provided that the surface diffusion is dominant, the rate of adsorption for a spherical particle can be described by the following equation:

$$\frac{\partial q}{\partial t} = D_s \left(\frac{2}{r} \frac{\partial q}{\partial r} + \frac{\partial^2 q}{\partial r^2} \right) \tag{5}$$

with the following initial and boundary conditions:

$$q(r, 0) = 0 \tag{6}$$

$$\left. \frac{\partial q}{\partial r} \right|_{r=0} = 0 \tag{7}$$

$$D_s \left. \frac{\partial q}{\partial r} \right|_{r=R_p} = k_f(c - c^*) \tag{8}$$

If the DA equation represents the single-species isotherm, the surface concentration, c^*, can be represented as

$$c^* = c_s \exp\left[\left(-\ln \frac{q|_{r=R_p}}{q_m} \right)^{1/n} \left(\frac{E}{kT} \right) \right]^{-1} \tag{9}$$

For the bulk fluid, the material balance is given by

$$V \frac{dc}{dt} = -w \frac{d\bar{q}}{dt} \tag{10}$$

with the initial condition

$$c(0) = c_i \tag{11}$$

The mean sorbate concentration inside its particle in Eq. (10) can be represented as

$$\bar{q} = \frac{3}{R_p^3}\int_0^{R_p} qr^2dr \tag{12}$$

It will not be possible to express the average accumulation of adsorbate in the solid phase as a simple function of the concentration. However, it can be calculated in terms of the adsorbate flux at the surface of the adsorbent particle. Equations (10) and (12) can be rearranged as follows:

$$\frac{dc}{dt} = -\frac{w}{V}D_s\frac{3}{R_p}\left.\frac{\partial q}{\partial r}\right|_{r=R_p} \tag{13}$$

Here, the dimensionless variables are defined as follows:

$$Q = q/q_m$$

$$X = r/R_p$$

$$C = c/c_i$$

$$C^* = c^*/c_i$$

$$\tau = \frac{D_s t}{R_p^2}$$

$$K_f = \frac{R_p k_f c_i}{q_m D_s}$$

$$Y = \frac{w q_m}{c_i V}$$

Finally, the dimensionless equations of Eqs. (5)–(13) are

$$\frac{\partial Q}{\partial \tau} = \left(\frac{2}{X}\frac{\partial Q}{\partial X} + \frac{\partial^2 Q}{\partial X^2}\right) \tag{14}$$

$$Q(X, 0) = 0 \tag{15}$$

$$\left.\frac{\partial Q}{\partial X}\right|_{x=0} = 0 \tag{16}$$

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$$\left.\frac{\partial Q}{\partial X}\right|_{x=1} = K_f(C - C^*) \tag{17}$$

$$C^* = \frac{c_s}{c_i}\exp\left[(-\ln q|_{x=1})^{1/n}\left(\frac{E}{kT}\right)\right]^{-1} \tag{18}$$

$$\frac{dC}{d\tau} = -3Y\left.\frac{\partial Q}{\partial X}\right|_{x=1} \tag{19}$$

$$C(0) = 1 \tag{20}$$

Equations (14)–(20) form a set of coupled second-order parabolic partial differential equations with boundary conditions. The method of orthogonal collocation was used to reduce these partial differential equations to a set of ordinary differential equations, and seven interior collocation points were used for the spherical particle (17).

The boundary condition at the center of the sphere is implicit in the model formulation and is not represented by the trial solution. These ordinary differential equations are solved by using DGEAR of the International Mathematics and Science Library (IMSL). DGEAR employs the variable-step size, variable-order, and the predictor-corrector technique that is suitable for stiff equations.

EXPERIMENTAL

The inorganic adsorbents used in this study were MS-13X and chabazite (AW-500) supplied by Aldrich Co. For the equilibrium isotherm experiment, the adsorbent pellets were crushed and sieved to give the fraction of 16/30 mesh particle size. The average particle diameter of the sorbent particles was calculated to be 0.50×10^{-3} m from sieve openings. All adsorbent particles were leached with deionized water, dried in an oven, and stored in a sealed vessel over a saturated NH_4Cl solution to maintain a constant equilibrium vapor pressure of water. The physical properties of these adsorbents as measured and obtained from the manufacturers' specifications are listed in Table 1. Cs and Sr were chosen as adsorbates since they are well-known radionuclides eluted from defective spent fuels in a storage pool. Stock solutions of each ion were made up by dissolving the reagent-grade CsCl and $SrCl_2$ supplied by Sigma Chemical Co. into distilled and deionized water. The concentration range of each ion was 5–30 mol/m^3. The concentrations of ions were measured by a well-type NaI scintillation counter. ^{137}Cs and ^{85}Sr were used as tracers for analysis. ^{85}Sr was particularly chosen instead of ^{90}Sr because it emits γ-rays. Since

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TABLE 1
Physical Properties of Adsorbents Used in This Study

Physical properties	Chabazite	MS-13X
Average particle size, m	0.94×10^{-3}	0.97×10^{-3}
Particle density, kg/m^3	1467	950
Bulk density, kg/m^3	670	620
Surface area, m^2/kg	2.44×10^5	3.51×10^5
Hydraulic diameter of 1/16 inch pellet, m	9.37×10^{-4}	9.69×10^{-4}

the concentrations of radionulides in the solution are in the 10^{-9}–10^{-7} mol/m^3 range, the effect of isotopes on the ionic concentration can be neglected.

Stock solutions containing tracers and 0.2 g adsorbents were poured into the centrifuge tube (Oak Ridge polycarbonate) in a constant temperature water bath and equilibrated for 7 days under shaking. One week was enough for the system to reach equilibrium. Prior to analyzing the concentration, liquid and solid phases were separated by a high-speed centrifuge with an acceleration of 15,000*g*.

A zeolite filter system in a defective spent fuel storage canister for controlling cooling water contamination is similar to a batch absorber to remove pollutants from constant sources. Adsorption experiments were conducted in a Carberry-type batch adsorber of 2×10^{-3} m^3. Adsorbent particles (1/16 inch cylinder type) were loaded into four cages made of a 80 mesh stainless-steel screen, and the cages were affixed to the rotating shaft to permit good contact with the solution. All the experiments were carried out at approximately 500 rpm so that the film mass transfer coefficient, k_f, would be nearly constant.

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RESULTS AND DISCUSSION

Adsorption Equilibrium

In this study, single-species equilibrium data were correlated by two-parameter isotherm equations such as the Langmuir, the Freundlich, and the DR equation, and by a three-parameter DA equation. In principle, one can obtain the values of the isotherm parameters by minimizing the mean percent deviation between experimental and predicted values of Q, defined as:

$$\text{dev}(\%) = \frac{100}{N} \sum_{k=1}^{N} \left\{ \frac{|Q_{\text{exp}} - Q_{\text{pre}}|}{Q_{\text{exp}}} \right\}_k \tag{21}$$

The percent deviations obtained from each isotherm are summarized in Table 2. Among four equations, the DA equation gives the best fit because it contains three adjustable parameters. However, it should be noted that the DR equation gives a better fit than the Langmuir and the Freundlich equations. Furthermore, Table 2 shows that the Langmuir equation gives the worst result. Such a result implies in principle that the adsorption systems encountered here are quite heterogeneous. It has been known that the surface heterogeneity in microporous zeolites is usually due to structural irregularities of pores. The DR equation can be derived by averaging the Langmuir local isotherms with the Gaussian-like right-hand widened adsorption energy distribution. Traditionally, the quantitative treatment of adsorption by microporous adsorbents is based on the DA equation since it has a variable heterogeneity parameter, n. Table 3 shows the parameters of the DA equations, q_m, E, and n. When $n = 1$, the DA equation becomes the Freundlich equation, which may be derived from an exponential distribution of adsorption energy and the Langmuir-type local isotherm (23). The value of n has been used as a measure of pore structure. In general, it appears that $1 < n < 2$ refers to adsorbents

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TABLE 2
Deviation between Experimental and Predicted Values for Various Isotherm Equations

		Dev(%)			
Radionuclide	Adsorbent	Langmuir	Freundlich	DR	DA
^{137}Cs	Chabazite	30.09	14.78	8.28	5.52
	MS-13X	27.86	26.68	11.59	0.67
^{85}Sr	Chabazite	11.83	3.02	1.97	1.95
	MS-13X	14.23	6.19	1.74	1.37

TABLE 3
Parameters of the DA Isotherm Equation for Radionuclides on Zeolites at 30°C

Radionuclides	Adsorbent	q_m (mol/kg)	E (kJ/mol)	n
^{137}Cs	Chabazite	2.56	23.8	2.8
	MS-13X	2.60	20.1	2.9
^{85}Sr	Chabazite	0.93	18.7	1.9
	MS-13X	2.33	25.8	2.2

with large micropores and $n > 2$ implies that the adsorbent has very fine micropores (7). However, the values of n for Cs and Sr on chabazite and MS-13X are scattered between 1.9 and 2.9, depending on the types of adsorbates and adsorbents. This implies that the surface heterogeneity is due to adsorbate–adsorbent interaction as well as the pore structure.

The DA isotherms of Cs and Sr in chabazite and MS-13X are shown in Figs. 1 and 2. Those figures show good curve fits with experimental data as linear lines. The maximum adsorption capacity and the adsorption energy may be estimated from the slope and interception of the DA isotherm curve.

The adsorption energy distributions for radionuclides on zeolites were obtained from the parameters of the DA equation. Using the Condensation Approach (CA) method (6, 16), the DA equation corresponds to the following energy distribution (8):

$$\chi(\epsilon) = \frac{n(\epsilon - \epsilon_k)^{n-1}}{E^n} \exp\left\{-\left[\frac{\epsilon - \epsilon_k}{E}\right]^n\right\} \qquad (22)$$

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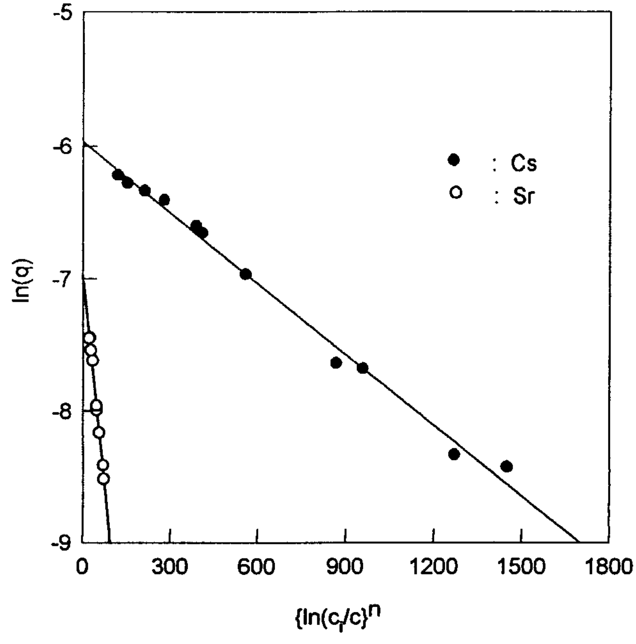

FIG. 1 DA adsorption isotherms of Cs and Sr ion on chabazite at 30°C.

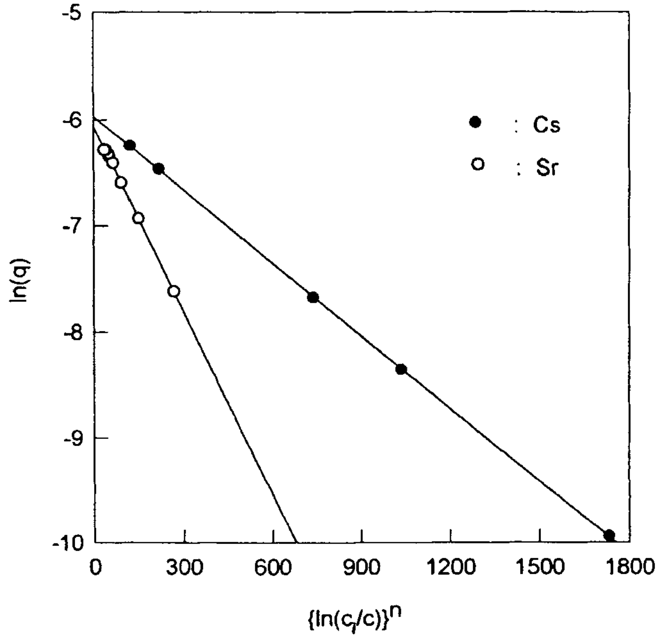

FIG. 2 DA adsorption isotherms of Cs and Sr ion on MS-13X at 30°C.

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Figures 3 and 4 represent the adsorption energy distribution curves for each radionuclides on chabazite and MS-13X, respectively. The curve for monovalent cesium has almost a normal shape of a Gaussian function, while that of bivalent strontium has a skewed adsorption energy distribution. The characteristic energy or the most probable adsorption energy, E, is in the 1.8 to 2.4 × 10^4 J/mol range, and the maximum adsorption capacities of each inorganic adsorbent from the DA equations are obtained as from 0.9 to 2.6 mol/kg.

Mass Transport and Simulation

In most adsorption processes where highly porous sorbents are used, the solution–particle mass transfer resistance can not be neglected in comparisons of intraparticle diffusion. There are some correlations for estimating the film mass transfer coefficient, k_f, in a batch system (4). In this work, k_f was estimated from the initial concentration history in which the diffusional resistance was not significant. The initial concentration history

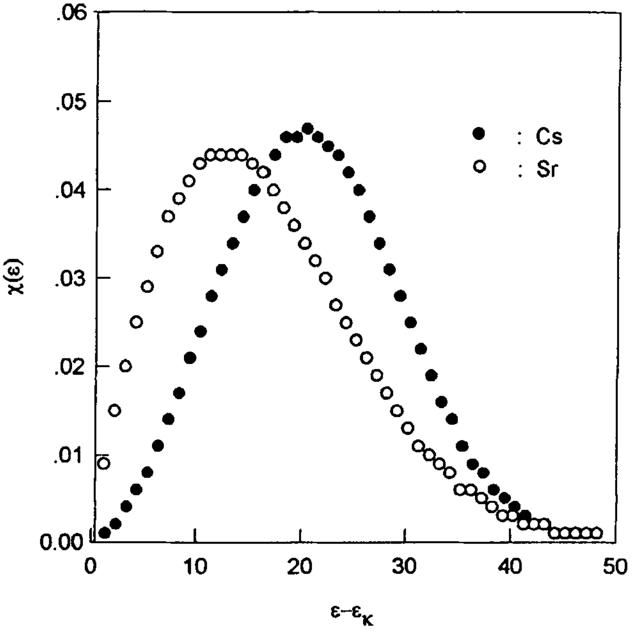

FIG. 3 Adsorption energy distribution curves of Cs and Sr ion on chabazite at 30°C.

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may be approximated by Eq. (23) when the adsorption time is less than 300 seconds (13).

$$\ln(c/c_i) = -k_f A t / V \tag{23}$$

where V is the volume of solution and A is the effective external surface area of sorbent particles, given by

$$A = 3w/\rho_p R_p \tag{24}$$

where w is the weight of sorbent particles loaded and ρ_p is the particle density. Figure 5 represents a typical plot to estimate k_f from the initial concentration history of cesium with $c_i = 5$ mol/m^3 and $w/V = 5$ kg/m^3. The value of k_f obtained from Fig. 5 is 4.78×10^{-5} m/s. For strontium, the value of k_f estimated is about 2.0×10^{-5} m/s using the same method.

Since intraparticle diffusion is the rate-controlling step in most adsorption processes, determination of the diffusion coefficients is an essential step. There are various methods presented in the literature for determining

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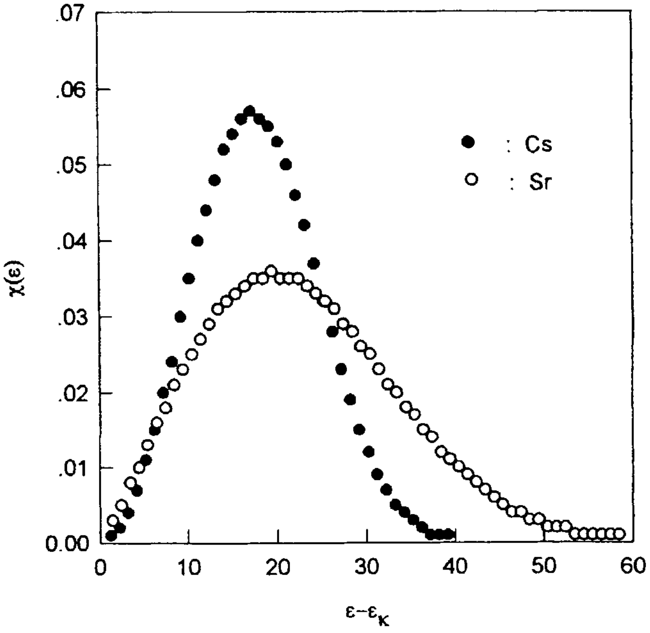

FIG. 4 Adsorption energy distribution curves of Cs and Sr ion on MS-13X at 30°C.

the diffusion coefficients. The most general method is to compare the experimental concentration history with that predicted from a specified diffusion model. Since the diffusion coefficient obtained by this method reflects all kinds of mass transfer resistances inside a particle, it has been called the effective diffusion coefficient. In this work, the surface diffusion model was employed. The effective surface diffusion coefficients obtained are listed in Table 4. The values of D_s are approximately $2.0–3.5 \times 10^{-11}$ m^2/s for the Cs–MS-13X system, $5.1–6.4 \times 10^{-12}$ m^2/s for the Cs–chabazite system, $3.2–5.2 \times 10^{-12}$ m^2/s for the Sr–MS-13X system, and $9.9 \times 10^{-12}–1.7 \times 10^{-11}$ m^2/s for the Sr–chabazite system. We cannot suggest a correlation for surface diffusion coefficients because of the scattering of values.

Figures 6–9 show the experimental and simulated concentration histories of each radionuclide in the batch adsorber. The adsorption model, which employs the surface diffusion mechanism incorporated with the DA equation, satisfactorily simulates the adsorption behavior of radionuclides on zeolites in batch adsorbers.

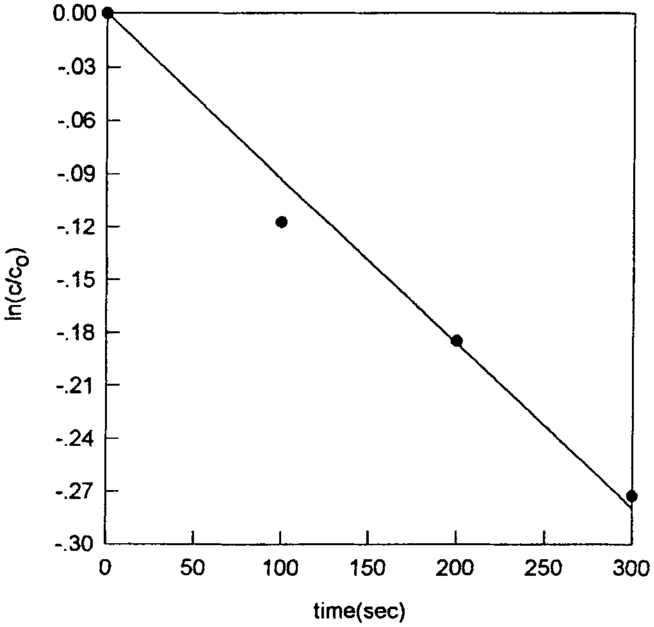

FIG. 5 Determination of k_f for Cs ion on MS-13X at 30°C.

TABLE 4
Effective Surface Diffusion Coefficients in Various Adsorption Systems

Adsorption system	c_i (mol/m^3)	D_s (m^2/s)
Cs–chabazite	5	5.3×10^{-12}
	10	5.1×10^{-12}
	30	6.4×10^{-12}
Cs–MS-13X	5	2.7×10^{-11}
	10	3.5×10^{-11}
	30	2.0×10^{-11}
Sr–chabazite	5	1.7×10^{-11}
	10	9.9×10^{-12}
Sr–MS-13X	5	3.4×10^{-12}
	10	3.2×10^{-12}
	30	5.2×10^{-12}

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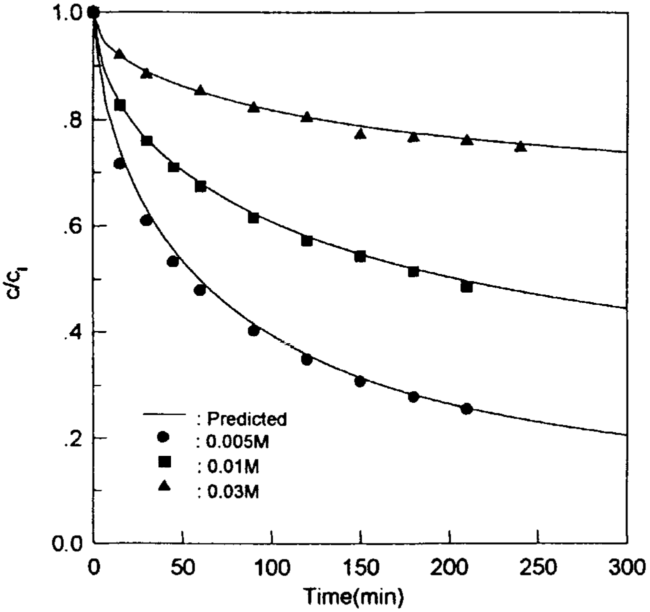

FIG. 6 Observed and predicted uptake curves of Cs ion adsorption on chabazite at 30°C.

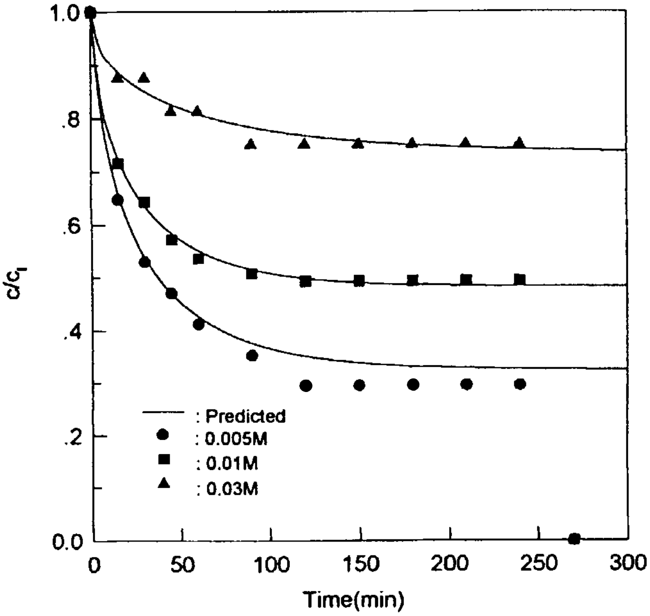

FIG. 7 Observed and predicted uptake curves of Cs ion adsorption on MS-13X at 30°C.

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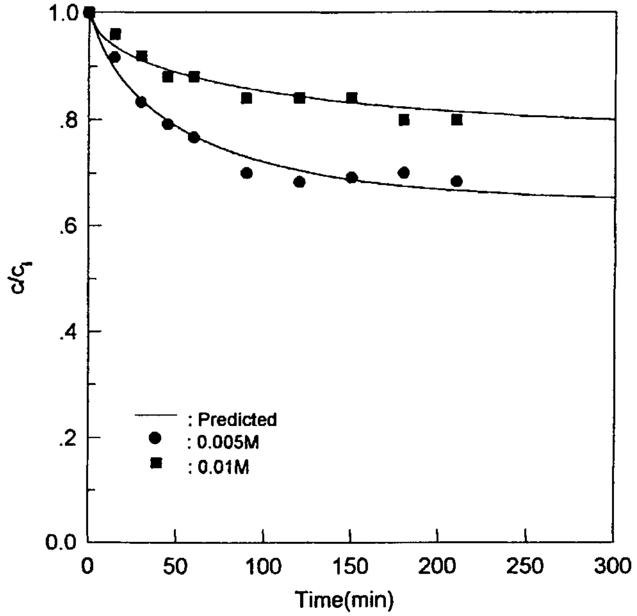

FIG. 8 Observed and predicted uptake curves of Sr ion adsorption on chabazite at 30°C.

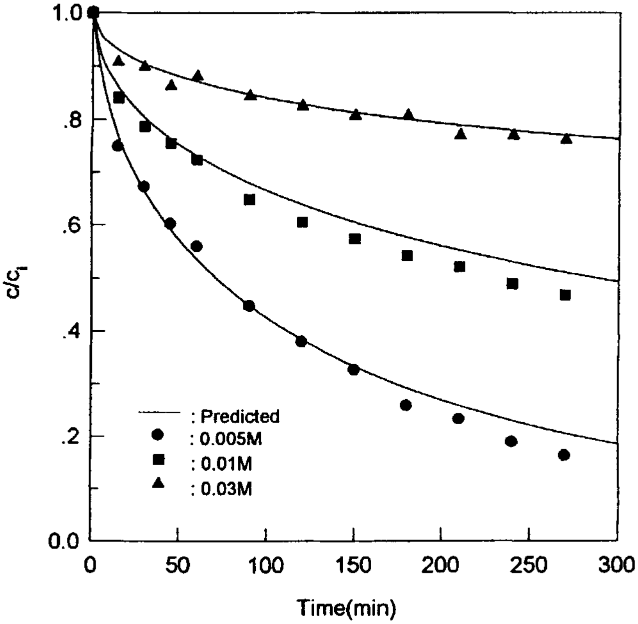

FIG. 9 Observed and predicted uptake curves of Sr ion adsorption on MS-13X at 30°C.

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CONCLUDING REMARKS

As a removal method for radionuclides dissolved in aqueous solutions, batch adsorption experiments were carried out using the inorganic adsorbents chabazite and MS-13X. Adsorption isotherms were represented by various equations, and the DA equation gave the best fit for all radionuclides on zeolites. The adsorption energy distribution was calculated from the parameters of the DA equation based on the condensation approach. The results show that the radionuclides–zeolite systems are highly heterogeneous based on the adsorbate–adsorbent interaction as well as the pore structure.

Intraparticle diffusion was assumed to be represented by the surface diffusion mechanism, and the effective surface diffusion coefficients were determined from experimental concentration histories. The adsorption model which employs the surface diffusion mechanism combined with the DA equation successfully simulates the adsorption behavior of radionuclides on zeolites in the batch adsorber. It shows that the model used here can be applied in simulating the adsorption of a zeolite filter in a defective spent fuel storage canister for controlling cooling water contamination.

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NOMENCLATURE

A	surface area of sorbent particles (m^2)
b	Langmuir constant (m^3/mol)
c	concentration in the liquid phase (mol/m^3)
c_i	initial concentration (mol/m^3)
c_s	saturated concentration in the liquid phase (mol/m^3)
C	dimensionless concentration in the liquid phase
dev(%)	percent deviation defined in Eq. (21)
D_s	effective surface diffusion coefficient (m^2/s)
E	characteristic adsorption energy (J/mol)
k_f	film mass transfer coefficient (m/s)
K_f	dimensionless variable, $R_p k_f c_i / q_m D_s$
K	Freundlich coefficients in Eq. (2)
k	Boltzman constant (J/mol·°K)
m	number of species
N	number of data points
n	exponent in Eqs. (2) and (4)
q	amount adsorbed (mol/kg)
q_m	amount adsorbed for monolayer formation (mol/kg)
Q	dimensionless concentration in the adsorbed phase
R_p	mean radius of particle (m)

r	radial distance (m)
T	temperature (°K)
t	time (s or min)
V	volume of solution (m^3)
w	weight of sorbents (kg)
X	dimensionless radial distance
Y	dimensionless variable, $wq_m/c_i V$

Greek Letters

ϵ	energy of adsorption (J/mol)
ϵ_k	critical adsorption energy (J/mol)
ρ_p	apparent particle density (kg/m^3)
τ	dimensionless variable, $D_s t/R_p^2$
$\chi(\epsilon)$	energy distribution function

Subscripts and Superscripts

0	initial value
-	average
*	equilibrium

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