

SELECTIVE REMOVAL OF CESIUM FROM RADIOACTIVE WASTE SOLUTIONS USING INSOLUBLE FERROCYANIDE-LOADED MORDENITES

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ABSTRACT

Potassium nickel hexacyanoferrate(II)s (KNiFC) were incorporated in the porous matrix of mordenites by successive impregnation of $\text{Ni}(\text{NO}_3)_2$ and $\text{K}_4\text{Fe}(\text{CN})_6$. The KNiFC crystals with *fcc* structure were uniformly dispersed throughout the granule, resulting in the lowering of specific surface area and pore volume. The ion-exchange equilibrium of Cs^+ in KNiFC-loaded mordenite (NMFC) was reached within 2 days. Relatively large distribution coefficients of Cs^+ , $K_{d,\text{Cs}}$, above $10^4 \text{ cm}^3/\text{g}$ were obtained even in the presence of highly concentrated 5 M NaNO_3 or 5 M HNO_3 . There was a large difference between the K_d value of ^{137}Cs and those of other nuclides; separation factor of Cs/Sr ($\alpha_{\text{Cs/Sr}} = K_{d,\text{Cs}}/K_{d,\text{Sr}}$) was estimated to be 10^4 in the presence of 2.5 M HNO_3 -0.981 M NaNO_3 . Favorable S-shaped breakthrough curves for Cs^+ were obtained by passing the simulated high-level liquid wastes containing 29 components through the column packed with NMFC exchanger, and the breakthrough capacity of Cs^+ was estimated to be 0.19 mmol/g. This exchanger proved to be effective for the selective removal of radioactive cesium from high-level liquid wastes.

INTRODUCTION

Special attention has been given to the selective removal of ^{137}Cs from radioactive waste solutions containing highly concentrated HNO_3 and NaNO_3 , in relation to the volume reduction of wastes and the partitioning of radioactive nuclides (1). Among various inorganic ion-exchangers exhibiting high selectivity to Cs^+ , insoluble hexacyanoferrates have been employed for the removal of ^{137}Cs in the treatment of nuclear waste solutions (2).

Potassium nickel hexacyanoferrate(II)s (KNiFC) can be obtained readily by precipitating $\text{Ni}(\text{NO}_3)_2$ with $\text{K}_4\text{Fe}(\text{CN})_6$ (3,4), while this products are composed of aggregates of very fine (about 30 nm in diameter) crystals and have low mechanical stability; the KNiFC precipitates tend to become colloidal in aqueous solution and seems to be unsuitable for practical applications such as column operation. In order to improve their mechanical properties, ferrocyanides exchangers have been prepared by precipitation on solid supports such as silica gels and bentonites (5). However, the preparation procedures were rather complicated and the results were often not reproducible. Mordenite, a kind of natural zeolite, with a relatively large pore volume and specific surface area is available as a carrier for the loading of microcrystalline ferrocyanides (6). This zeolite also has high resistance to acids and irradiation (7). In this study, we attempted to incorporate KNiFC crystals into the porous

support of mordenite by successive impregnation with $\text{Ni}(\text{NO}_3)_2$ and $\text{K}_4\text{Fe}(\text{CN})_6$ solutions.

The present paper deals with the procedure for the preparation of KNiFC-loaded mordenites, characterization of this exchanger, uptake properties of cesium ions and the selective removal of ^{137}Cs from simulated high-level liquid wastes.

EXPERIMENTAL

Procedure for Preparation

The unit cell content of mordenites used as a porous carrier was $\text{Na}_8[(\text{AlO}_2)_8(\text{SiO}_2)_{40}] \cdot 24\text{H}_2\text{O}$ (Linde IE-96). Three kinds of natural mordenite with different places of origin, *e.g.*, Kawarago (Miyagi pref.), Tenei (Fukushima pref.), and Itaoroshi (Miyagi pref.) were used and abbreviated as NMK, NMT, and NMI, respectively. The procedure for the preparation of KNiFC-loaded mordenite (NMFC) is as follows. A mordenite carrier (32~48 mesh) dried at 200°C was contacted with a 20 cm^3 solution of 1 M (mol/dm^3) $\text{Ni}(\text{NO}_3)_2$ under reduced pressure at 25°C for 3 hours and then washed with acetone and air-dried at 90°C for 3 hours. In a similar manner, the mordenite impregnated with $\text{Ni}(\text{NO}_3)_2$ was reacted with a 20 cm^3 solution of 0.5 M $\text{K}_4\text{Fe}(\text{CN})_6$ to form KNiFC precipitates in the pores of mordenite. The single-loaded mordenite (designated as NMFC-I) was washed with deionized water and acetone, air-dried at 90°C for 3 hours and finally stored in a sealed vessel over a saturated NH_4Cl solution at a constant vapor pressure of water (humidity: 79 % at 25°C). The double-loaded and triple-loaded samples, NMFC-II and NMFC-III, were prepared by repetition of the above procedure.

Characterization

Surface morphologies of NMFC granules were examined by scanning electron microscopy (SEM). The structure of KNiFC was determined by powder X-ray diffractometry (XRD, Rigaku RAD-B) using monochromatized $\text{Cu-K}\alpha$ radiation. The loading of KNiFC and the chemical composition of NMFC were determined by electron probe microanalysis (EPMA, Hitachi X-650S). The chemical composition of KNiFC was estimated by taking the amount of irons as unity and normalizing the nickel amount to this value. The proportion of potassium was then calculated on the basis of the iron and nickel values and assuming electrical neutrality. The specific surface areas and pore volumes of carriers and NMFC were determined by high speed specific surface area/pore size distribution measurement apparatus (Micromeritics, ASAP-2400).

Determination of Distribution Coefficient

The distribution coefficients of various radioactive nuclides were determined by the batch method. An aqueous solution (7 cm^3) containing 10 ppm metal ion labeled with a radioisotope was contacted with 0.070 g of NMFC sample at $25 \pm 1^\circ\text{C}$ for 7 days, which was found to be sufficient for attaining equilibrium. After the aqueous phase was separated by centrifugation at $10,000\text{ rpm}$ for 10 minutes, the γ -activity of the supernatant was measured with a well-type $\text{NaI}(\text{Tl})$ scintillation counter. The radioisotopes, ^{137}Cs , ^{85}Sr , ^{152}Eu , and ^{241}Am , were used as tracers. The concentration of carrier-free ^{241}Am used for the distribution experiments was $2.1 \times 10^{-9}\text{ M}$. The radioisotope ^{85}Sr was produced by the $^{86}\text{Sr}(\gamma, n)^{85}\text{Sr}$ reaction with 50 MeV bremsstrahlung from linear electron accelerator (LINAC) in the

Laboratory of Nuclear Science, Tohoku University, and was dissolved with 0.1 M HNO₃. The uptake percentage of metal ions removed from the solution, R (%), and the distribution coefficient, K_d (cm³/g), are defined as

$$R = [(A_i - A_t)/A_i] \times 100 \quad (\%), \quad (\text{Eq. 1})$$

$$K_d = [(A_i - A_f)/A_f] \times V/m \quad (\text{cm}^3/\text{g}), \quad (\text{Eq. 2})$$

where A_i , A_t , and A_f (cpm/cm³) are the counting rates at the initial stage, at time t , and at equilibrium, respectively; m (g) is the weight of NMFC sample; and V (cm³) is the volume of the aqueous phase.

Breakthrough Curve

The NMFC-III sample (32~48 mesh, 3 g) was densely packed into a glass column (8 mm ϕ ×200 mm long) with a jacket thermostated at 25°C. The column volume of NMFC-III was 5.53 cm³. A feed solution of simulated high-level liquid waste (29 components; [Cs⁺]=1.61×10⁻² M) was then passed through the column at a constant flow rate of 0.27 cm³/min [space velocity (SV)= 3.0/h]. Every 3 cm³ of the effluent was taken by a fraction collector, and then their γ -activities were measured. A breakthrough curve was obtained by plotting the breakthrough ratio (C/C_0) against the effluent volume, where C_0 and C (cpm/cm³) are the counting rates of the initial solution and the effluent, respectively.

RESULTS AND DISCUSSION

Surface Morphology and Structure

A cross section of NMFC-III granule embedded in the acrylic resin was submitted to EPMA. The constitutional elements of KNiFC (K, Fe, and Ni) were detected in the energy dispersive spectroscopic (EDS) spectrum for the inner part of this granule, indicating that the KNiFC crystals are loaded on the meso-macropores of mordenites. Here the K⁺ ion is a constituent of KNiFC as exchangeable cation. The EDS spectrum in the vicinity of the surface was almost similar to that of the inner part of the granule. The chemical composition of KNiFC in NMFC-III was determined to be K_{1.94}Ni_{0.030}[NiFe(CN)₆] by quantitative EDS analysis using the ZAF correction method (8). The KNiFC crystals were identically spherical/cubic in shape, and the crystal size as roughly estimated from SEM image was about 30 nm. As given in Table I, both surface area and pore volume were lowered after loading of KNiFC into the pores.

Table I. Specific surface areas (SSA) and pore volumes (PV) for carriers and KNiFC-loaded mordenites.

	NMK (carrier)	NMKFC-III	NMI (carrier)	NMIFC-III
SSA (m ² /g)	99	41	273	52
PV (cm ³ /g)	0.089	0.047	0.165	0.060

In order to check the homogeneity of loading of KNiFC, a loaded sample was further contacted with 0.1 M CsNO₃-5 M NaNO₃ solution, and the treated specimen was submitted

to EPMA. Here the Cs^+ ions were preferentially adsorbed on KNiFC crystals. Figure 1 shows the line analysis of Cs-L α for the cross section of a NMTFC-III granule. The Cs^+ ions are uniformly dispersed throughout the granule, indicating loading of the KNiFC crystals onto the meso-macropores.

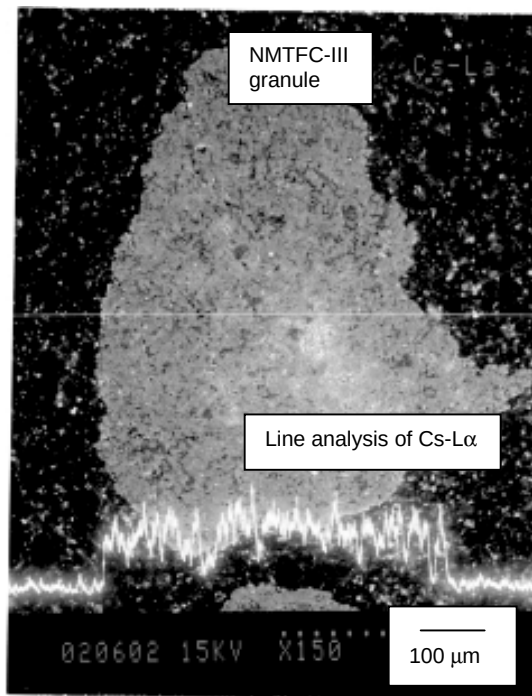


Fig. 1. Line analysis of Cs-L α of NMTFC-III treated with 0.1 M CsNO_3 -5 M NaNO_3 .

Though the XRD intensity of the KNiFC crystals loaded was rather weak, main diffraction peaks for the face-centered cubic (fcc) structure of KNiFC, *e.g.*, 200 ($2\theta = 17.44^\circ$), 220 (24.82°), 222 (30.46°), 400 (35.46°), and 420 (39.82°) were observed in the XRD pattern of NMTFC-III, together with those of mordenite. The lattice parameter of KNiFC was calculated to be 1.00846 ± 0.00264 nm (9), which is close to those prepared by precipitation methods (4). The structure of mordenite was unaltered before and after loading of KNiFC.

Preparation Conditions and $K_{d,\text{Cs}}$

The KNiFC-loaded mordenites were prepared by varying the particle size of carrier, concentration of Ni^{2+} and impregnation cycle. The $K_{d,\text{Cs}}$ values for NMTFC exchangers were summarized in Table II. Here the $K_{d,\text{Cs}}$ was determined at $V/m = 100 \text{ cm}^3/\text{g}$ in the presence of 5 M NaNO_3 . The $K_{d,\text{Cs}}$ value tends to increase markedly with decreasing particle size of carrier and increase slightly with Ni^{2+} concentration and impregnation cycle.

Table II. $K_{d,Cs}$ values for NMTFC exchangers.

	$K_{d,Cs}$ (cm ³ /g)
10~20	4.4×10^3
Mesh size 20~32	5.6×10^3
32~48	1.9×10^4
0.1 M	1.6×10^4
Ni ²⁺ concentration 0.5 M	1.8×10^4
1.0 M	1.9×10^4
Single	1.9×10^4
Impregnation cycle Double	2.0×10^4
Triple	3.2×10^4

Uptake Rate of Cs⁺

Figure 2 shows the time dependent uptake percentage, $R(\%)$, of Cs⁺ on carriers (NMT, NMK) and NMFC-III in the presence of 5 M NaNO₃. The adsorption of Cs⁺ on carriers reached equilibrium within 1 day, and relatively low R values of 31~35% were obtained. On the other hand, the adsorption of Cs⁺ on NMFC-III was fairly rapid in the initial stages, probably due to exchange on the surface, followed by a slow step controlled by mass transfer within particles (4,10). Relatively large R values, above 99%, for both NMFC-III exchangers were obtained within 2 days.

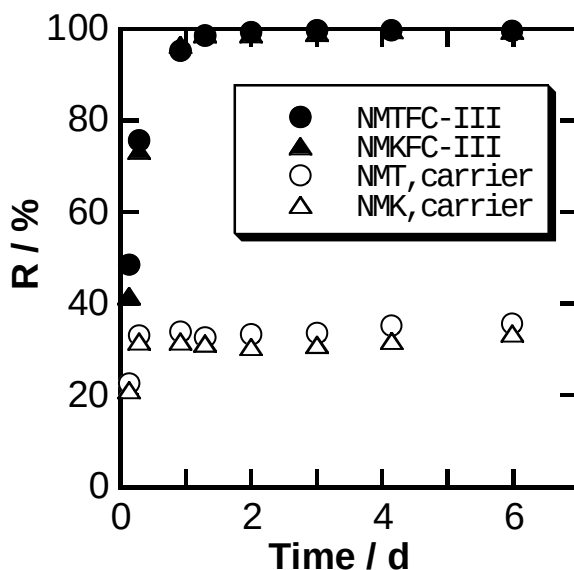


Fig. 2. Uptake rate of Cs⁺ for carriers and NMFC-III.
 V/m 100 cm³/g; 10 ppm Cs⁺-5 M NaNO₃; centrifugation 10⁴ rpm, 10 min; 25°C.

Ion-Exchange Isotherm for Cs⁺ Uptake

The ion-exchange isotherms were obtained in a wide range of initial Cs⁺ concentrations from 10⁻² to 0.1 M. The equilibrium amount of Cs⁺ adsorbed on NMFC-III approached a constant value at Cs⁺ concentrations above about 40 mmol/dm³, suggesting that the uptake of Cs⁺ follows a Langmuir-type adsorption equation (4,11). The Langmuir equation is given by

$$Q_{\text{eq}} = K Q_{\text{max}} C_{\text{eq}} / (1 + K C_{\text{eq}}) \quad (\text{mol/g}), \quad (\text{Eq. 3})$$

where C_{eq} (mol/dm^3) and Q_{eq} (mol/g) are the equilibrium concentrations of Cs^+ in the aqueous and solid phases, respectively; Q_{max} (mol/g) is the maximum amount of Cs^+ taken up; and K (dm^3/mol) is the Langmuir constant. The equation can be rewritten as follows:

$$C_{\text{eq}}/Q_{\text{eq}} = 1/K Q_{\text{max}} + (1/Q_{\text{max}})C_{\text{eq}} \quad (\text{g/dm}^3). \quad (\text{Eq. 4})$$

As seen in Fig. 3, fairly linear relations between $C_{\text{eq}}/Q_{\text{eq}}$ and C_{eq} for NMIFC-III and NMKFC-III were obtained from Langmuir plots, with correlation coefficients above 0.99. The Q_{max} values for NMIFC-III and NMKFC-III were calculated to be 1.32 and 1.37 mmol/g, respectively. The Q_{max} values for NMFC were rather low compared with those for carriers (1.46 mmol/g), suggesting that the meso-macropores of mordenites were considerably blocked by the successive loading of KNiFC crystals with relatively low Q_{max} value (1.275 mmol/g). The loading percentages of KNiFC for NMIFC-III and NMKFC-III were estimated to be 49 and 76 wt%, respectively. The Q_{max} values for NMFC-III were about 4~10 times larger than those for KNiFC-loaded silica gels (12) and CuFC-loaded resins (10).

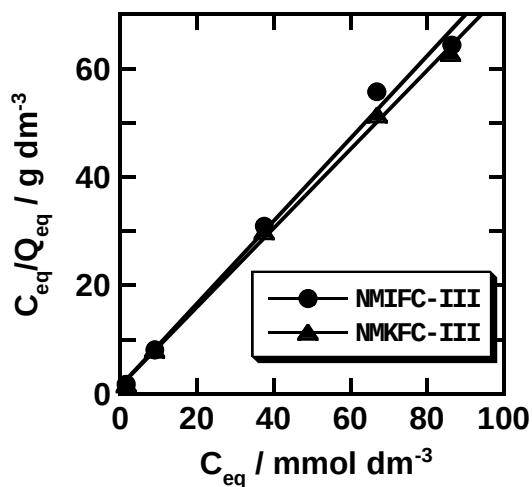


Fig. 3. Langmuir-plots for Cs^+ uptake for NMFC-III. pH_{eq} 6.45~6.63; equilibration 25°C , 7 d.

Distribution of Cs^+ in the Presence of Monovalent Cations

The effects of coexisting monovalent cations on $K_{\text{d,Cs}}$ were examined to estimate the selectivity to Cs^+ . Table III summarizes the effects of the initial concentration of competing monovalent cations (Na^+ , H^+ , K^+ , and NH_4^+) on $K_{\text{d,Cs}}$ for NMT and NMTFC-III. Even at higher Na^+ concentration of 1~5 M, the $K_{\text{d,Cs}}$ values for NMTFC-III were above $10^4 \text{ cm}^3/\text{g}$, suggesting high selectivity to Cs^+ . On the other hand, the $K_{\text{d,Cs}}$ value for NMT was considerably lower than that for NMTFC-III; the $K_{\text{d,Cs}}$ value was less than $10^2 \text{ cm}^3/\text{g}$ in the presence of 5 M NaNO_3 . The distribution of Cs^+ was considerably affected by NH_4^+ concentration. The $K_{\text{d,Cs}}$ values decreased in the order of coexisting ions, Na^+ , $\text{H}^+ > \text{K}^+ \gg \text{NH}_4^+$; this is similar to the order in the size of the hydrated cations (13).

Table III. $K_{d,Cs}$ values in the presence of monovalent cations.

	$K_{d,Cs}$ (cm ³ /g)	
	NMT(carrier)	NMTFC-III
[Na ⁺] 1 M	5.3×10^2	1.6×10^5
5 M	56	3.2×10^4
[H ⁺] 1 M	1.2×10^3	8.7×10^4
5 M	1.0×10^2	4.0×10^4
[K ⁺] 1 M	27	2.5×10^4
5 M	16	1.6×10^4
[NH ₄ ⁺] 1 M	40	4.6×10^3
5M	5.0	2.8×10^2

Distribution of Various Nuclides

The affinity of various radionuclides (¹³⁷Cs, ⁸⁵Sr, ¹⁵²Eu, and ²⁴¹Am) for NMIFC-III was examined in the presence of 2.5 M HNO₃ and 0.981 M NaNO₃. Here the concentrations of HNO₃ and NaNO₃ referred to those of simulated high-level liquid waste (SW-11) (14). Table IV summarizes the K_d values of various nuclides for NMIFC-III. There are large differences among the K_d of ¹³⁷Cs and those of other nuclides. The K_d value of ¹³⁷Cs was larger than 10^4 cm³/g, while those of other nuclides were less than 10 cm³/g; the separation factor of Cs/Sr ($\alpha_{Cs/Sr} = K_{d,Cs}/K_{d,Sr}$) was estimated to be 10^4 , suggesting that this exchanger is available for the mutual separation of these nuclides.

Table IV. K_d values of various nuclides in the presence of 2.5 M HNO₃ and 0.981 M NaNO₃.

	K_d (cm ³ /g)
¹³⁷ Cs	1.8×10^4
⁸⁵ Sr	1.8
¹⁵² Eu	4.8
²⁴¹ Am	8.2

Breakthrough Properties of Cs through NMFC Columns

To obtain information on the dynamic adsorption behavior of Cs⁺, its breakthrough property from simulated high-level liquid waste (SW-11) was examined at a constant flow rate using the column packed with NMFC-III. The chemical composition of the simulated waste (SW-11) used as the feed solution was determined according to ORIGEN-79 (14) and is given in Table V. Figure 4 illustrates the breakthrough curves for Cs⁺, which are symmetrical S-shaped profiles. This suggests no dislodgment of KNiFC from the matrices of mordenites. The break points of 5% breakthrough for NMKFC-III and NMIFC-III were estimated to be 33.6 [bed volume (BV) = 6.1] and 35.0 cm³ [BV = 6.3], respectively. The breakthrough capacity (*B.T.Cap.*) were calculated to be 0.18 and 0.19 meq./g, respectively. The column packed with NMFC was thus effective for the selective removal of ¹³⁷Cs from high-level liquid wastes.

Table V. Chemical composition of simulated high-level liquid waste (SW-11).

Element	Concn.(M)	Element	Concn.(M)
H ⁺	2.5	Ru	1.69×10^{-2}
Na	0.981	Rh	3.39×10^{-3}
P	1.27×10^{-2}	Pd	8.66×10^{-3}
Fe	7.77×10^{-2}	Ag	3.45×10^{-4}
Cr	3.95×10^{-3}	Cd	4.67×10^{-4}
Ni	9.37×10^{-3}	Sn	3.32×10^{-4}
Rb	3.64×10^{-3}	Se	5.41×10^{-4}
Cs	1.61×10^{-2}	Te	3.57×10^{-3}
Sr	8.78×10^{-3}	Y	4.78×10^{-3}
Ba	9.72×10^{-3}	La	7.92×10^{-3}
Zr	3.61×10^{-2}	Ce	5.89×10^{-2}
Mo	3.06×10^{-2}	Pr	7.46×10^{-3}
Mn	1.31×10^{-2}	Nd	2.50×10^{-2}
		Sm	5.10×10^{-3}
		Eu	7.96×10^{-4}
		Gd	3.86×10^{-4}

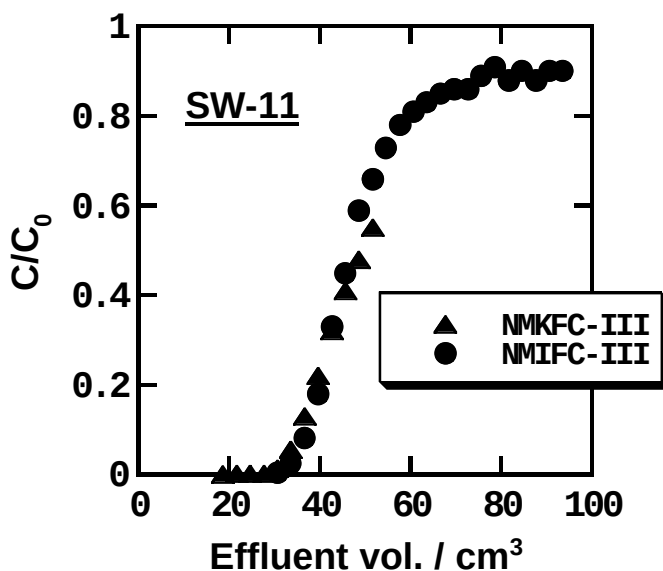


Fig. 4. Breakthrough curves for Cs⁺ through the columns packed with NMFC-III. NMFC-III 3.00 g; feed: simulated high-level liquid waste (SW-11) ; SV 3.0/h; 25 °C.

Natural mordenites used as a carrier occur abundantly in Japan (15), and the cost of using them for the purpose of separating cesium is considered to be very low. As for chemical stability, the KNiFC crystals tend to decompose with NaOH concentration above 1 M NaOH, while both mordenite and KNiFC have high resistance to acid (7,16). The NMFC exchangers are effective for the selective removal of ¹³⁷Cs from highly acidic waste solutions.

CONCLUSIONS

Potassium nickel hexacyanoferrates (KNiFC) were loaded on a porous matrix of granular natural mordenite by successive impregnation of $\text{Ni}(\text{NO}_3)_2$ and $\text{K}_4\text{Fe}(\text{CN})_6$. The loading of KNiFC on mordenites led to improvements in both mechanical stability and selectivity to Cs^+ ; the $K_{d,\text{Cs}}$ for KNiFC-loaded mordenite (NMFC) was almost independent of the coexisting NaNO_3 and HNO_3 concentrations, and relatively large $K_{d,\text{Cs}}$ values above $10^4 \text{ cm}^3/\text{g}$ were obtained even in the presence of highly concentrated 5 M NaNO_3 or 5 M HNO_3 . The NMFC products also exhibited relatively large separation factors for Cs and other nuclides (Sr, Eu, and Am). Selective removal of Cs^+ from simulated high-level liquid wastes can be achieved by using a column packed with NMFC.

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