A Recovery Process of Strontium from Acidic Nuclear Waste Streams

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ABSTRACT

A process combining liquid–liquid extraction with ion exchange for strontium traces removal in the presence of an excess of sodium concentrations is described. In this continuous process, strontium is extracted from acidic (0.9 M) solution using a substoichiometric amount of dicyclohexano-18-crown-6 (0.1 M in $CHCl_3$). Extracted strontium is readily stripped from an organic phase with water and concentrated onto an ion exchanger. This process allows a high strontium decontamination factor (250) with a very important extraction factor (5000).

INTRODUCTION

Large volumes of liquid wastes are generated during spent nuclear fuel reprocessing. Weakly radioactive, these effluents impose a complex treatment composed of several steps of coprecipitations with various chemical reagents [$Ba(NO_3)_2$, $Fe(NO_3)_2$, . . .] before releasing, and although satis-

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factory at the level of the rejection norms presently enforced, the method presents many disadvantages. The method is not very selective; it generates important amounts of low activity solid wastes and requires a significant volume of chemical reagents before disposal can occur. Because of both the assumed high cost of waste immobilization and disposal, and the increasing concern toward protection of the environment (2), a more effective process to minimize the volume of waste is desirable. Among the radioactive elements present as traces in the liquid wastes (Table 1), ^{90}Sr, which has chemical properties close to those of calcium, is known for its harmful biological effects on bone cells. Nevertheless ^{90}Sr, because of the heat it generates, should be of value as a reliable source of thermal energy for use in radioisotopic thermal electric generators (3) and could have significant beneficial applications.

Recently, solvent extraction and ion-exchange processes for strontium isolation from alkaline and weak or strong acid waste solutions have been developed. The use of dicyclohexano-18-crown-6 (DCH18C6), a very stable crown ether (4, 5), and its dimethyl and di-t-butyl derivatives for the removal of Sr from acidic media is well documented in the literature (6–11). Many studies have focused on improvement of the efficiency of crown ethers by using different solvents and/or additives (12–15). Ion-exchange processes employing inorganic ion exchangers such as antimonic acid for sorbing ^{90}Sr from strong (0.5 to 7 M HNO_3) acid waste solutions have been extensively studied (16). On the other hand, the use of organic ion exchangers containing functional groups for the separation of metal ions has been reported (17–19) only from basic solutions. Many processes have been described for the removal of strontium from acidic or basic solutions. Without exception, however, these processes involve strontium concentrations from 70 (20) to 500 (13) times higher than these of the effluents we describe.

TABLE 1

Composition of the Effluents Produced by All the Industrial Wholes of the Spent Nuclear Fuel Reprocessing Plant of COGEMA La Hague (before treatment)

Element	Na	Zn	Mg	Fe	Ca	K	Al
Concentration (mg·L^{-1})	1870	80	50	7	5	6	3

Element	Cr	Ru	Cs	Pu	Sr	Sb	Pb
Concentration (mg·L^{-1})	0.60	0.50	0.35	0.13	6×10^{-3}	2×10^{-4}	<0.10

The aim of this paper is to report a new continuous process combining liquid–liquid extraction with ion exchange for strontium trace removal in the presence of an excess of sodium. The association of these two techniques allows a high strontium decontamination factor and a very important extraction factor (1).

The coextraction of metals present in liquid waste by solvent extraction which utilizes DCH18C6 as the Sr extractant has been studied. As reported by Horwitz et al. (21), the distributions ratios measured with 0.2 M DtBuDCH18C6 in 1-octanol were lower than 1 for all elements except strontium.

EXPERIMENTAL

Strontium Extraction Procedure

Chemicals and Reagents. All reagents were purchased commercially and used as received. The mixture of DCH18C6 isomers was supplied by Fluka, and the DCH18C6 isomers were separated according to a procedure previously reported (22). DUOLITE ARC 9652 resin (18–40 mesh, sulfonate form) was purchased from Rohm & Haas Industries. One gram of resin contains 1.2 meq of strong acid exchange sites (SO_3H). The simulated aqueous wastes were prepared by dissolving salts of sodium and strontium nitrates in nitric acid. The composition of the synthetic waste used in this study is shown in Table 2.

Liquid–Liquid Procedure. Equal volumes (3 mL) of simulated aqueous waste and organic DCH18C6 in the chosen solvent were shaken for 15 minutes in sealed tubes. The two phases were separated by centrifugation, and the strontium was then stripped from the organic phase by backextraction to an aqueous receiving phase (3 mL of distilled water).

Ion-Exchange Study

Batch Procedure. The kinetics of Sr extraction were studied by shaking 1 mL of resin with 5 mL of a 0.9 M nitric acid solution containing 0.545×10^{-3} g·L^{-1} of Sr and 4.18 g·L^{-1} of Na. After 1 to 18 hours of

TABLE 2
Chemical Composition of the Synthetic Effluents

Ion	H^+	NO_3^-	Na^+	Sr^{2+}
Concentration (mol·L^{-1})	0.9	1	0.1	2.68×10^{-6}

shaking, the solution was filtered and the concentration of Sr in the aqueous phase was measured to determine the distribution coefficient D.

Column Procedure. The desired volume of resin, 6 mL, was collected in a glass tube (h/ϕ = 12). Sr fixation was performed by passing the effluent through the column at a flow rate of 0.17 $L \cdot h^{-1}$. Strontium was then eluted with 5 M HNO_3.

Distribution Ratios

Liquid–Liquid Study. Distribution ratios of Sr and Na were determined radiometrically. The isotopes used were ^{85}Sr (65 d) and ^{22}Na (2.6 y). 514 keV γ-energy was used for ^{85}Sr determination, 511 keV γ-energy was used for ^{22}Na determination in the absence of ^{85}Sr, and 1274 keV ^{22}Na γ-energy was used for ^{22}Na determination in the presence of ^{85}Sr.

Ion-Exchange Study. Distribution ratios of Sr were measured both radiometrically using the 514 keV γ-energy of ^{85}Sr (65 d) and by atomic absorption spectrometry. Distribution ratios of Na were measured by atomic absorption spectrometry.

Analytical Section

Measurements of the Activity by γ-Ray Spectrometry. The INTERTECHNIQUE IN 1200 equipment was composed of a high-purity semiconductor Ge detector type, a preamplifier, an amplifier, a coder, an analyzer, and computer treatment. The concentrations of Sr and Na were measured with a standard deviation of ±5%.

Atomic Absorption Spectrometry Measurements. The concentrations of Na and Sr were determined by atomic absorption spectrometry using a Perkin-Elmer 5100 spectrometer (air-acetylene flame). In the case of Sr for which the amounts for analysis were extremely small, we had to use electrothermal atomization (furnace-AAS). Analyses were typically reproductible to within ±5%.

RESULTS AND DISCUSSION

Liquid–Liquid Extraction of Strontium

The nature of the solvent, the stereochemical configuration of the DCH18C6 isomer, and the extractant concentration were used to study the removal of strontium traces in the presence of excess sodium.

The distribution ratio of each metal in the liquid–liquid distribution, representing the ratio of the total analytical concentration of a metal in the extract (regardless of its chemical form) to its total analytical concentration in the other phase, was expressed as

$$D = [\bar{M}]/[M]$$

and calculated from the experimental data, where $[\bar{M}]$ and $[M]$ were the total metal concentrations in the organic and aqueous phases, respectively.

The separation factor, representing the ratio of the respective distribution ratios of the two extractible metals measured under the same conditions, was calculated as follows:

$$\alpha_{M_1,M_2} = D_{M_1}/D_{M_2}$$

Extraction Step

Choice of the Solvent. The nature of the solvent used can affect the extraction selectivity (23–25). The selection of an appropriate solvent requires that certain factors be taken into consideration: the solvent must have an acceptable density, its water solubility must be low, and its phase disengagement from mixtures with nitric acid must be rapid. The partition of DCH18C6 between water and various solvents was measured in the presence of potassium picrate by the Frensdorff's method (26), and the percentage of DCH18C6 loss was expressed by

$$p\% = \frac{[L]}{[\bar{L}]} \times 100$$

where $[L]$ and $[\bar{L}]$ indicate the concentrations of DCH18C6 in the aqueous and organic phases, respectively.

Among all the solvents tested, only those resulting in a macrocycle loss below 7% were selected. The influence of 26 solvents on Sr selectivity of extraction was studied in an attempt to correlate strontium extraction with the physicochemical properties of the solvents (dipole moment μ, dielectric constant ϵ, and Hildebrand parameter δ). Table 3 summarized the distribution ratios obtained as a function of the solvent used and of its physicochemical properties. Four classes of solvent were selected for the study: chlorinated hydrocarbons, alcohols, carboxylic acids, and aromatics.

It is interesting to note that chlorinated solvents give the highest Sr distribution ratios and the best separation factors. DCH18C6 is less effective for Sr extraction when diluted in primary or secondary alcohols, but there appears to be no correlation between the number of carbons of the aliphatic chain, the class of the alcohol, and the Sr efficiency of extraction. The same conclusions can be drawn regarding the D and α values obtained when DCH18C6 is diluted in carboxylic acids.

TABLE 3

Comparison of Strontium Distribution Ratios in Various Solvents, $T = 25°C$, DCH18C6 $= 0.1$ mol·L^{-1}, Initial HNO$_3$ = 0.9 mol·L^{-1}

Solvent	p%	DSr	DNa	$\alpha_{Sr,Na}$	$\epsilon_r{}^a$	μ^b	δ^c
Chloroform	0.05	25.00	≤0.01	≥2500	4.81	3.84	9.16
Dichloromethane	0.08	26.00	0.02	1300	9.08	5.17	9.88
1,2-Dichloroethane	0.35	14.00	0.03	467	10.65	6.20	9.86
1,1,1-Trichloroethane	1.80	0.17	≤0.01	17	7.53	5.20	8.30
1-Chloropentane	6.20	0.02	≤0.01	≥2	6.60	1.94	8.33
1-Pentanol	1.05	2.25	0.11	20	13.90	5.94	10.90
1-Heptanol	1.50	0.87	0.07	12	11.55	3.07	10.50
1-Octanol	2.00	0.6	0.05	12	10.34	5.81	10.30
1-Decanol	2.90	0.35	0.04	9	8.10	—	8.60
2-Octanol	3.50	0.34	0.03	11	8.17	5.54	7.90
2-Ethylhexanol	2.00	0.47	0.02	24	4.41	—	9.85
Cyclohexanol	1.65	2.30	0.15	15	15.00	6.20	11.40
Pentanoic acid	0.16	2.53	0.11	23	2.66	1.12	8.50
Hexanoic acid	0.22	1.08	0.03	36	2.63	1.13	8.51
Heptanoic acid	0.29	0.64	0.02	32	2.60	1.14	8.53
Octanoic acid	0.40	0.37	≤0.01	≥37	2.45	1.15	8.56
2-Ethylhexanoic acid	0.55	0.18	≤0.01	≥18	2.40	1.13	8.10
Benzene	1.30	0.21	≤0.01	≥21	2.28	0.00	9.16
Chlorobenzene	0.84	1.25	≤0.01	≥125	5.71	5.14	9.67
Bromobenzene	0.88	1.24	≤0.01	≥124	5.40	5.17	9.87
Iodobenzene	1.30	0.93	≤0.01	≥93	4.63	4.64	10.13
Toluene	0.05	0.05	≤0.01	≥5	8.93	1.43	8.93
Nitrobenzene	0.70	6.00	0.05	120	—	13.44	10.40
Benzonitrile	0.70	11.00	0.12	92	2.33	13.51	10.70
1,2-Dichlorobenzene	0.90	1.05	≤0.01	≥105	35.74	7.57	10.04
1,2,4-Trichlorobenzene	2.30	0.36	≤0.01	≥36	24.20	13.50	12.38

^{a} Electric dipole moment.
^{b} Dipole moment.
^{c} Hildebrand function (27, 28).

Aromatics are more effective solvents than alcohols and carboxylic acids, which are in turn less effective diluents than alcohols.

The solvent effect on both Sr efficiency and selectivity of extraction is not well understood and cannot be rationalized on the basis of its polarity (μ and ϵ) or its H-bonding ability (δ). In general, however, the best results are obtained with chlorinated solvents and especially with chloroform for which the required strontium distribution ratios and selectivity are observed. In order to test the potential value of our methodology, chloro-

TABLE 4

Distribution Ratios and Separation Factors as a Function of the
DCH18C6 Stereochemical Configuration, $T = 25°C$, 0.1 M DCH18C6

DCH18C6 isomer	Distribution ratio, D		Separation Factor, $\alpha_{Sr,Na} = DSr/DNa$
	DSr	DNa	
cis-syn-cis	30	0.015	2000
cis-anti-cis	15	0.008	1875
Mixture	26	0.013	2000

form was chosen as our test solvent, despite its tendency to generate corrosive HCl upon radiolysis.

Dependence of Strontium/Sodium Separation on the Stereochemical Configuration of the DCH18C6 Isomers. The distribution ratio variation as a function of the stereochemical configuration of the DCH18C6 isomers used are listed in Table 4.

A high distribution ratio and separation factor for strontium are observed with the *cis-syn-cis* isomer, and this conclusion is consistent with results previously reported by Yakshin et al. (29). Strontium and sodium distribution ratios are lowest with a mixture of the two isomers, but they lead to the same separation factor as for the *cis-syn-cis* isomer.

Dependence of Strontium/Sodium Separation on DCH18C6 Concentration. The distribution ratio variations as a function of the DCH18C6 concentration are summarized in Table 5.

Good strontium distribution ratios are observed from a DCH18C6 concentration of 0.06 mol·L^{-1}. The distribution ratio of strontium as well as

TABLE 5

Influence of the DCH18C6 Concentration on the Distribution Ratios and
Separation Factors, $T = 25°C$

DCH18C6 (mol·L^{-1})	Distribution ratio, D		Separation factor, $\alpha_{Sr,Na} = DSr/DNa$
	DSr	DNa	
0.01	2.65	0.0018	1472
0.02	5.45	0.0036	1513
0.04	10.9	0.0060	1816
0.06	15.5	0.0078	1987
0.10	24.5	0.0120	2042

the selectivity of strontium over sodium are excellent for a DCH18C6 concentration of 0.1 mol·L^{-1}.

Stripping Step

We have studied the dependence of sodium and strontium stripping on the concentration of the nitric acid solution. Indeed, it was necessary to define a range of nitric acid concentration where the extent of both strontium and sodium stripping is maximum. Stripping of sodium and strontium as a function of nitric acid concentration is represented in Fig. 1.

Strontium extraction leads to the formation of a complex with a strontium/ligand/nitrate stoichiometry of 1/1/2:

$$\bar{L} + Sr^{2+} + 2NO_3^- \rightleftharpoons \overline{LSr(NO_3)_2}$$

$$K_{ex} = \frac{[\overline{LSr(NO_3)_2}]}{[\bar{L}][Sr^{2+}][NO_3^-]^2}$$

The expression of the extraction constant (30) shows that a decrease of the nitrate concentration is responsible for decomplexation of the metals.

Water leads to the release of the major part of strontium as well as of sodium from the organic phase whereas an increase in nitric acid concentration is responsible for an increase in nitrate ions and decreases in both strontium and sodium stripping. The deciding factor for strontium stripping is the nitrate concentration.
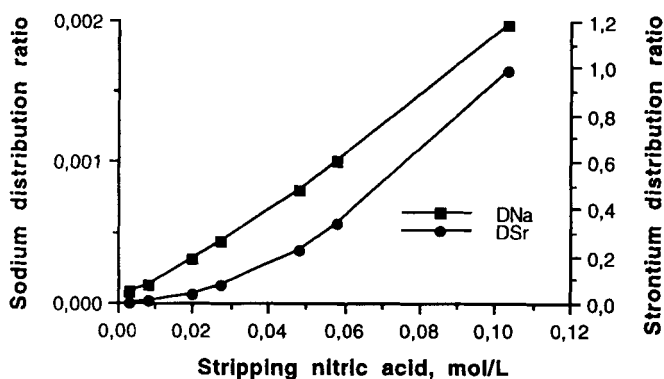
FIG. 1   Dependence of sodium and strontium stripping on nitric acid concentration. DCH18C6 = 0.1 mol·L^{-1}. T = 25°C.

Solid–Liquid Concentration of Strontium

Duolite ARC-9652 is a sulfonic acid cation-exchange macroporous resin.

Batch Concentration of Strontium

The distribution coefficients D were calculated as follows:

$$D = \frac{C_i - C_f}{C_f} \times \frac{V}{m}$$

where C_i and C_f = concentrations of strontium before and after concentration onto the resin

V = volume of solution (mL)

m = mass of resin (g)

Sorption kinetics of Sr concentration on the DUOLITE ARC-9652 from 0.9 mol·L^{-1} nitric acid containing sodium nitrate have been studied, and the results are shown in Fig. 2.

The extent of strontium concentration increases rapidly and appreciably up to 2 hours of shaking, at which time a plateau is reached. Maximum distribution ratios of 147 $\text{mL/g}_{(dry)}$ and of 5 $\text{mL/g}_{(dry)}$ are obtained after 18 hours of shaking for the strontium and the sodium, respectively.
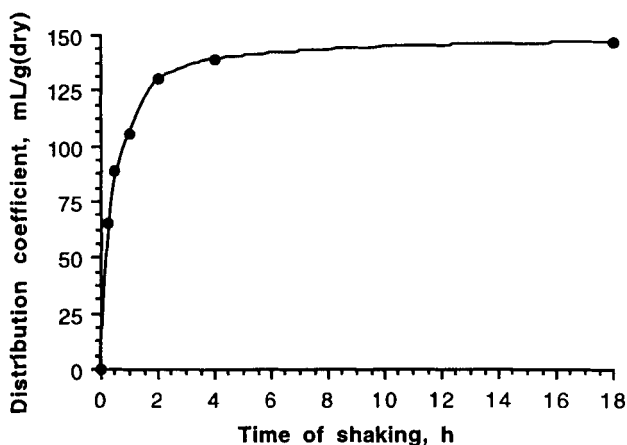

FIG. 2   Kinetic of concentration of traces of strontium onto the DUOLITE ARC-9652 in the presence of 4.18 g·L^{-1} of sodium. $HNO_3 = 0.9 \text{ mol·L}^{-1}$.

In light of these results, removal of traces of strontium from 0.9 M nitric solution containing an excess of sodium can be achieved using the DUOLITE ARC-9652 resin.

Evaluation of a Continuous Process of Extraction-Stripping-Concentration of Strontium

The number of theoretical stages of extraction was calculated using the "Kremser formula" (31a,b). In Fig. 3 a strontium stripping facility is proposed as a function of the concentrations for the nuclear waste streams studied.

The pilot installation requires a 3-stage extraction reactor and a 3-stage stripping reactor. The strontium is continuously concentrated onto an ion-exchange resin. Extraction of more than 99% of the strontium in three stages needs a DCH18C6 concentration of 0.022 M.

A pH-adjusted synthetic effluent (pH 0.46) with a flow rate of $0.17 \ L \cdot h^{-1}$ was added at the first stage and the extraction solvent was introduced at
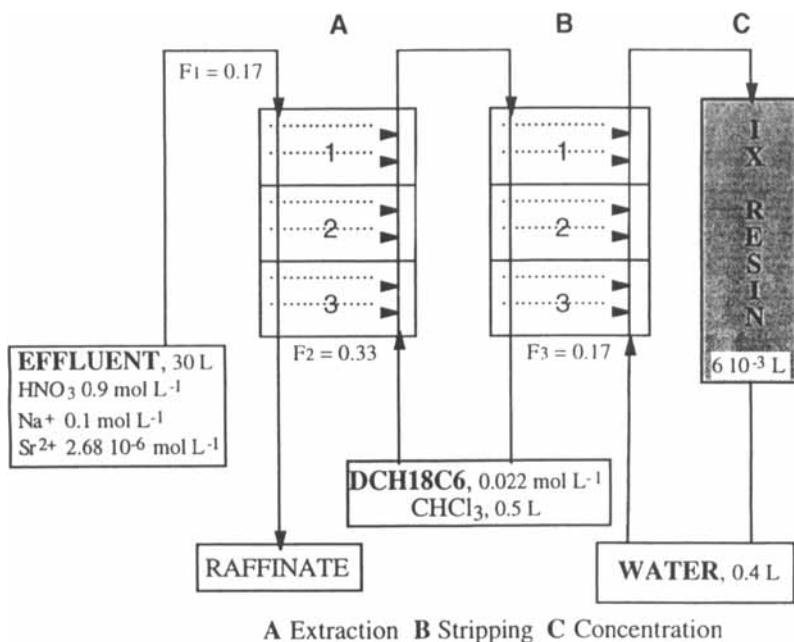
A Extraction B Stripping C Concentration

FIG. 3   Pilot installation for strontium extraction and recovery. F_1 = Effluent flow rate $(L \cdot h^{-1})$. F_2 = Organic phase flow rate $(L \cdot h^{-1})$. F_3 = Sr stripping flow rate $(L \cdot h^{-1})$.

the third stage with a flow rate of 0.33 L·h^{-1}. The majority of strontium (99.6%) was extracted in three stages. The strontium was stripped in three stages, water was introduced at the third stage at a flow rate of 0.17 L·h^{-1}. More than 90% of the strontium was stripped and continuously concentrated on to an ion-exchange resin. Traces of strontium contained in 30 L of effluent were concentrated on to 6 × 10^{-3} L of resin, leading to a calculated extraction factor of 5000; strontium was then readily eluted with 5 M HNO$_3$. Sr decontamination factors (Sr concentration in effluent/Sr concentration in raffinate) of about 250 were measured.

Leak of Strontium in the Raffinate. An effluent flow rate of 0.17 l·h^{-1} and an organic phase flow rate of 0.33 L·h^{-1} led to a strontium leak in the raffinate of less than 0.5%. An increase of the flow rate to 0.75 L·h^{-1} allowed 30 L of the effluent to be reprocessed in 40 hours of running time. At this flow rate an important strontium leak was observed in the raffinate. Figure 4 shows the concentration of strontium in the raffinate as a function of time.

One hour of running time allows a strontium leak in the raffinate of 3.7%; up to 40 hours of running time the strontium concentration in the raffinate increases by about 0.03%·h^{-1}. It is interesting to note that after 40 hours of running time with a flow rate of 0.75 L·h^{-1}, the strontium leak in the raffinate does not exceed 5%. Moreover, this leak can be avoided in using countercurrent reactors made of one or two additional stages.
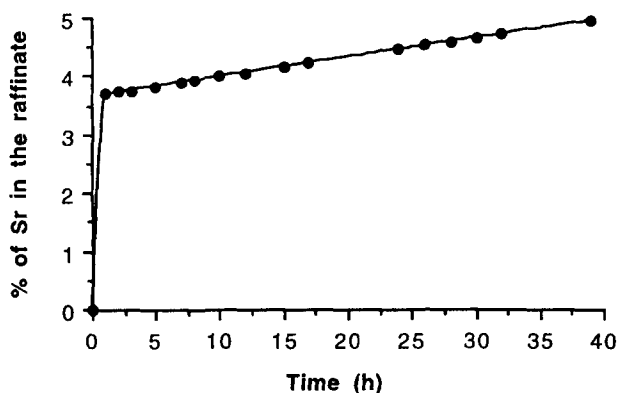

FIG. 4   Percentage of strontium in the raffinate as a function of the time. Effluent flow rate = 0.75 L·h^{-1}.

CONCLUSION

The results presented here demonstrate that a continuous process based on liquid–liquid extraction (DCH18C6 in chloroform)/solid–liquid concentration (ion exchanger) provides an effective method for the extraction and recovery of strontium traces in the presence of an excess of sodium from nitric acid media.

The liquid–liquid extraction study shows that the solvent effect on both Sr efficiency and selectivity of extraction is not well understood and cannot be rationalized on the basis of physicochemical parameters like μ, ϵ, or δ. The best results are obtained with chlorinated solvents, especially with chloroform.

This process has several advantages over other previously described strontium extraction methods. The process uses commercially available reagents and works easily. Strontium contained in a 30-L sample of radioactive effluent is selectively extracted by the DCH18C6 and then concentrated onto 6 mL of an ion-exchange resin. The overall process therefore reduces the volume of material containing Sr by a factor of 5000 and fixes this soluble specie onto an organic incinerable material.

Although we have described only one specific application in this report, the process should be suitable for use with any environmental, biological, geological, or nuclear waste sample whose preparation requires digestion or leaching with nitric acid.

ACKNOWLEDGMENT

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