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Partitioning of actinides from high-level liquid waste employing supported liquid membrane technique using TOPO in *n*-dodecane as carrier

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ABSTRACT

This paper describes the studies on the partitioning of actinides from high-level liquid waste of PUREX origin employing supported liquid membrane technique. The process uses solution of tri-n-octyl phosphine oxide in *n*-dodecane as a carrier with polytetrafluoroethylene support and a mixture of citric acid, formic acid and hydrazine hydrate as a receiving phase. Transport of ^{241}Am has been studied as a function of various parameters such as acidity of feed, concentration of carrier, effect of uranium concentration, effect of trivalent ions and salt concentration in the feed in order to optimize efficient transport conditions. The studies indicated good transport of actinides like uranium, neptunium, plutonium and americium across the membrane from nitric acid medium. Under the optimized conditions, the transport of ^{241}Am is also studied from a uranium-depleted synthetic pressurized heavy water reactor-high-level liquid waste (HLLW). Also the technique has been used for the partitioning of alpha emitters from research reactor-HLLW.

Keywords: TOPO; ^{241}Am; Supported liquid membrane; Partitioning of actinides

1. Introduction

The alpha-emitting long-lived actinides present in the HLLW originating from the reprocessing of the spent nuclear fuel by PUREX process are of great environmental concern. Actinide partitioning is emerging as the strategy for the removal of minor actinides from HLW prior to the burial of latter in the form of vitrified blocks [1,2]. In the last two decades, considerable work has been carried out for the partitioning of these nuclides from HLLW employing many solvents in several modes such as solvent extraction, extraction chromatography (EC) and supported liquid membrane (SLM) [3–7]. Among these, EC and membrane-based techniques are relatively newer and gaining importance because of various advantages. Earlier, with the object of partitioning of actinide from HLW, octyl (phenyl)-*N*,*N*-di-isobutyl carbamoyl methyl phosphine oxide (CMPO) was studied in our laboratory in all the three modes and results were found to be promising [4–6,8]. In similar context, the present study deals with the partitioning of actinides from HLLW using tri-n-octyl phosphine oxide (TOPO) as carrier in SLM mode. In our earlier studies using solvent extraction, citric acid in the presence of formic acid and hydrazine hydrate was used for stripping actinides from acid bearing loaded Truex solvent (0.2 M CMPO + 1.2 M

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TBP in *n*-dodecane) [9,10]. The same mixture is used in the present studies as a receiving phase.

The transport of ^{241}Am was studied under different experimental conditions like variation in the acidity of the feed, effect on the concentration of the carrier, the transport of other radionuclides *viz.*^{233}U, $^{237+238}$Np and ^{239}Pu from nitric acid solution. The study has been extended to simulated HLLW originated from the reprocessing of spent fuel from pressurized heavy water reactor (PHWR)-HLLW as well as actual HLLW solution originating from the reprocessing of spent fuel from research reactor to test the applicability of this technique for the separation of actinides.

2. Experimental

2.1. Reagent

TOPO used was a product from Fluka, Switzerland and used as such without further purification. Tri-n-butyl phosphate (TBP) was procured from M/s. Bharat Vijay Chemicals, India and purified by contacting it with a 2% (w/v) solution of sodium carbonate for the removal of the acidic impurities. It was then washed with distilled water and dried over anhydrous sodium sulphate. Dodecane (~ 93% C-12, Sp. gr. = 0.751, refractive index = 1.42) was procured from M/s. Transware Chemia Handelsgeselschaft Hamburg, Germany. Formic acid (assay - 90%, M.W. = 46.03) used was of analytical grade reagent. Citric acid (assay - 99.5%, M.W. = 210.14) used was laboratory-grade reagent.

Hydrazine hydrate (assay- 80%, M.W. = 50.06) used was laboratory-grade reagent. All other chemicals used were of analytical reagent grade.

2.2. Radiotracers

Transport studies were carried out from feed solutions containing known concentration of nitric acid spiked with tracer activities of individual radiotracers. Radiotracers used in the transport studies were ^{233}U, ^{239}Pu, ^{144}Ce, ^{241}Am and $^{237+238}$Np. The tracer ^{144}Ce were separated and purified from various streams of the PUREX process using the methods reported in the literature[11,12].^{233}U was purified by the cation-exchange procedure[13] before its use. Plutonium (mainly ^{239}Pu) was purified by the anion-exchange method [14,15]. The valency of the plutonium was maintained at IV by adding small amounts of $NaNO_2$ and NH_4VO_3 and was checked by HTTA extraction [16] prior to the actual experiments. The final stock solution was stored in ~1.0 M HNO_3. ^{241}Am used was from the stock and purified by the method reported elsewhere [11,12,17]. $^{237+238}$Np was obtained by irradiating ^{237}Np in research reactor and purified by the method reported elsewhere [17–19]. Oxidation state of neptunium was adjusted to either (IV) or (VI) using 0.02 M ferrous and 0.01 M dichromate ions, respectively.

2.3. Analysis

In all the experiments, samples of 20 μL were taken from feed and receiving compartments periodically and analysed radiometrically. Gross α-activity in the actual sample solutions, ^{233}U and ^{239}Pu were analysed using 2π geometry argon flow alpha proportional counter, standardized against standard ^{239}Pu. Single-channel gamma analyser with NaI(Tl) detector was used for the analysis of ^{238}Np, ^{241}Am and ^{144}Ce when transport studies were carried out from pure nitric acid medium or from simulated waste solution. Uranium analysis in the waste and U-depleted HLW was carried out using a spectrophotometric method employing 2-(5-bromo-2-pyridylazo)-5-diethyl aminophenol as chromogenic reagent [20,21].

2.4. Membrane cell

Commercially available polytetrafluoroethylene (PTFE) membranes were procured from Millipore (India) Pvt. Ltd., Mumbai. Membrane had an average pore diameter and thickness of 0.45 and 160 μm, respectively. The porosity of the membrane was ~70%. The membrane was impregnated with TOPO by immersing it in the organic phase of required concentration for at least 12 h before use. After the impregnation, membranes were given a mild wash with water and used for the transport studies.

2.5. Feed solutions and strippant

Single-stage permeation measurements across SLM were carried out with a two compartment permeation cell consisting of feed chamber and receiver chambers, with a volume of 4.5 mL each, separated by a SLM having an effective membrane area of 1.13 cm^2 (Fig. 1). The source and receiving phases were mechanically stirred to minimize the thickness of the aqueous diffusion layer and to avoid concentration polarization. Transport studies were carried out from feed solutions containing nitric acid of known molarity spiked with required radiotracer and a mixture of citric acid (0.1 M), formic acid (0.4 M), and hydrazine hydrate (0.4 M) was used as the receiving phase (strippant) throughout the studies [22].

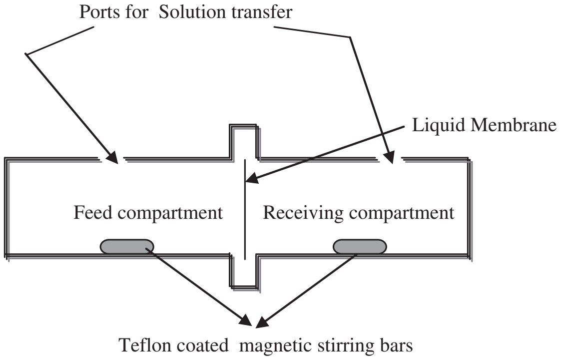

Fig. 1. Sketch of SLM cell used for the transport of radionuclide.

During the study on simulated waste solutions, uranium-depleted waste spiked with ^{241}Am was used as the feed. The composition of the simulated HLW used in the present studies was based on fission product inventory of the spent uranium fuel from pressurized heavy water reactor with a burn up of 6,500 MWd/Te of UO_2 and 3 years of cooling. Inert constituents, such as sodium and corrosion products like iron, chromium and nickel, were added in quantities anticipated in the HLW. The composition of simulated HLW used in the studies is shown in Table 1 [8,22–24]. The composition of simulated HLW with respect to uranium in this study lowered to ~10 ppm, by contacting it thrice with equal volume of 30% TBP in *n*-dodecane and the resultant raffinate was used as a feed for the experiments carried out with simulated waste solution. Similar procedure was adopted for the removal of uranium from actual HLW generated during the reprocessing of spent fuel from research reactor.

2.6. Permeation of radionuclides and calculation of permeability coefficients

The permeation of radionuclides *viz.* ^{144}Ce, $^{152+154}$Eu, U, ^{237}Np, ^{239}Pu, and ^{241}Am from feed compartment containing radionuclides at tracer level across the carrier supported liquid membrane was studied as a function of time at required acidity and the strip solution in the receiving compartment. Samples (generally 20 μL) from feed as well as receiving compartments were drawn periodically and assayed. Percentage transport of the radionuclide at any time interval was calculated using Eq. (1).

$$\%\ \text{Transport} = [A_t/A_i] \times 100 \tag{1}$$

where A_t is the activity of the radionuclide at any time t in the receiver phase and A_i the initial activity of the radionuclide in the feed compartment.

Various experimental parameters studied include the concentration of TOPO in *n*-dodecane in the membrane phase, concentration of nitric acid, uranium, effect of trivalent metal ions and salt ($NaNO_3$) in the feed. Under optimized conditions, studies were carried out for the transport of americium from uranium-depleted simulated HLLW of PHWR origin,

Table 1
Composition of simulated high level waste solution (Acidity = 3.0 M)

Constituent	Concentration (g/L)	Constituent	Concentration (g/L)
Selenium	0.0123	Barium	0.3088
Rubidium	0.0745	Lanthanum	0.2638
Strontium	0.1863	Cerium	0.5325
Yttrium	0.0990	Praseodymium	0.2438
Zirconium	0.7713	Neodymium	0.8625
Molybdenum	0.7313	Promethium*	0.0283
Technetium*	0.1813	Samarium	0.1638
Ruthenium	0.4638	Europium	0.0226
Rhodium*	0.1275	Gadolinium	0.0165
Palladium	0.2675	Terbium	0.0005
Silver	0.0186	Dysprosium	0.0002
Cadmium	0.0159	Uranium	18.325
Tin	0.0151	Sodium	3.0
Antimony	0.0047	Iron	0.5
Tellurium	0.1028	Chromium	0.1
Cesium	0.5438	Nickel	0.1

*Lanthanum was added for promethium; molybdenum for technetium and cobalt for rhodium.

and finally, partitioning of alpha emitters was studied from an actual uranium-depleted HLLW originated from the reprocessing of spent fuel from research reactor.

The permeability coefficients for the metal ions were calculated using Eq. (2).

$$\ln(M_t/M_o) = -(A\varepsilon/V)P \times t \tag{2}$$

where M_o is concentration of the radionuclide in the feed at time $t = 0$ and M_t in feed at t in s, A is the geometric area of the membrane in m^2, ε is porosity, V is the volume of feed in m^3, P is the permeability coefficient in m/s and t is the lapsed time in s. The plot of ln (M_t/M_o) vs. time for different radionuclides gave straight line, and the permeability coefficients were calculated from the slopes of the straight lines.

Percentage transport of the radionuclide was calculated using the original feed activity and activity of solution in the receiver phase compartment at any time interval. Transport data were also used to calculate permeability coefficients for actinides using the Danesi equation [24]. The flux is defined as the rate of mass transport of the solute through the SLM, and it is the criterion used to evaluate the performance of SLM. According to Fick's first law of diffusion, the rate of diffusion (dc/dt) of a solute across an area (A), known as flux (J) [25] is calculated using Eq. (3) (considering the initial concentration in receiving phase is zero)

$$J = (\mathrm{d}C_{AM,\mathrm{receiving}}/\mathrm{d}t)_0(V/A\varepsilon) \tag{3}$$

where $C_{\mathrm{Am,\ receiving}}$ = Am concentration in the receiving phase, moles/m^3, V = volume of receiving phase, in m^3, A = geometric area of the membrane, m^2; t = time elapsed, s.

3. Results and discussion

Fig. 2 shows the results of transport studies carried out from an americium spiked feed solution containing 0.02 M nitric acid. The results indicate that the transport increases with time as well as with increasing concentration of TOPO. It increases from 32 to ~94% in 6 h, while the concentration of TOPO (in *n*-dodecane) was increased from 0.1 to 0.7 M. Further increase in carrier concentration from 0.7 to 0.9 M did not change the transport rates significantly (it remained almost constant ~96% in 6 h). Because of the solubility problems of TOPO in *n*-dodecane the higher concentrations were not used for further studies. The abnormal behaviour of lower two curves (0.1 and 0.2 M), that is, slow transport is due to the insufficient carrier concentration. Leaching of carrier is one of the major drawbacks in supported liquid membrane based techniques, and hence, a higher concentration of TOPO (0.9 M in *n*-dodecane) was chosen in subsequent studies.

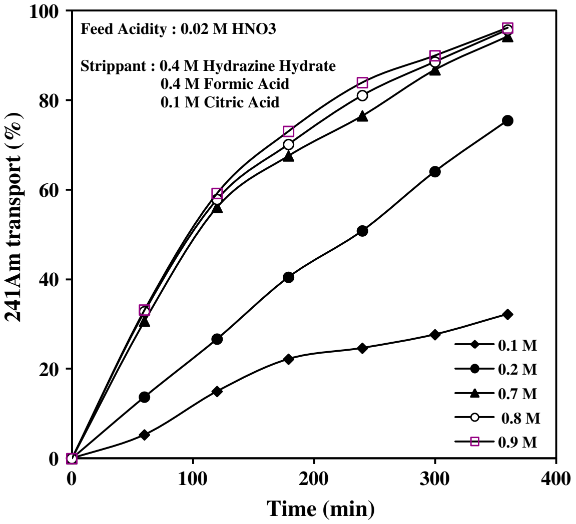

Fig. 2. Transport studies of ^{241}Am using varying concentration of TOPO in PTFE membrane phase.

Fig. 3 shows the transport of ^{241}Am studied as a function of time as well as with varying concentration of the nitric acid in feed from 0.01 to 2.0 M, while maintaining the concentration of TOPO impregnated in the membrane phase at 0.9 M in dodecane. The transport was followed up to 6 h. The results indicate that as the transport decreases with increases in acidity of feed solution. More than 95% transport of ^{241}Am was observed in 6 h, at the acidity up to 1 M, and was only about 27% at 2 M.

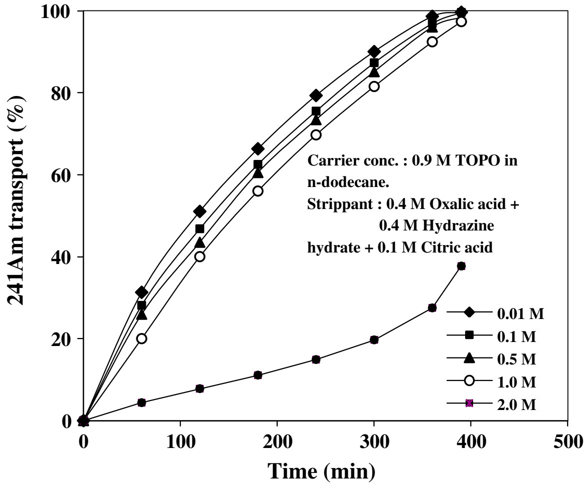

Fig. 3. Transport behaviour of ^{241}Am as a function of varying concentration of HNO$_3$ in the feed compartment.

The transport increases from 27.5 to 98.6%, as the acidity of the feed decreases from 2.0 to 0.01 M in 6 h. These observations suggest that lower concentrations of nitric acid favors the transport of americium into the product compartment.

The transport of various radionuclides relevant to nuclear waste management *viz.* actinides ^{233}U,^{238}Np, ^{239}Pu, ^{241}Am and fission product $^{152+154}$Eu were carried out using 0.9 M TOPO in *n*-dodecane as the carrier at 0.5 M acidity. This is the approximate feed acidity that is expected after the uranium depletion step by TBP in HLW. It can be seen that >95% transport within 6 h was found for all the actinides under study (Fig. 4). In these experiments, the citrate-containing buffer was used as the receiver phase. The radiotracers of elements were spiked individually. The transport of Np was studied in its tetravalent as well as hexavalent state. Further, it can be seen that the transport behaviours of actinides and lanthanides are of similar nature, and thus, the partitioning of actinides can be achieved from the feed acidity of 0.5 M.

Using the transport data linear graphs were plotted for all the radionuclides between $\ln(M_t/M_o)$ and time at 0.5 M. Using the slopes of these straight lines, permeability coefficients were calculated for each radionuclide using the Danesi equation and corresponding fluxes calculated by the equation given in the initial part of paper and are shown in Table 3.

The use of 0.04 M nitric acid as the receiver phase (strippant) resulted in the cotransport of nitric acid from the feed to the receiver phase, and hence, did not allow the complete transport of the americium. In spite of nearly 95% transport of americium being transported to the receiving chamber in the initial stages of the experiment, subsequent migration of americium back to the feed chamber was observed later. Reversal in the direction of the movement of americium was found to be due to increase in the nitric acid content of the receiving chamber resulting from co-transport of nitric acid. Therefore, a mixture of 0.1 M citric acid, 0.4 M formic acid and 0.4 M hydrazine hydrate were used as receiving phase which gives the buffering action and not allows the back migration.

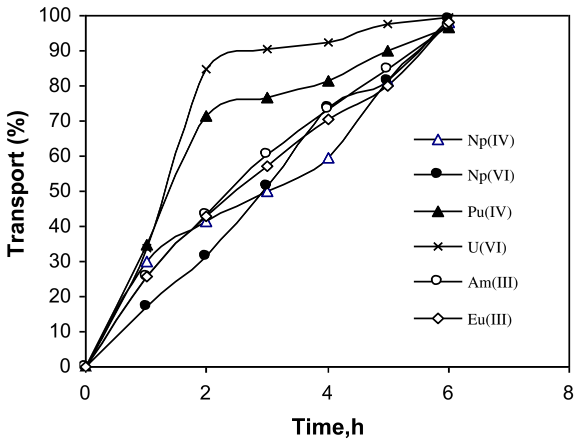

Fig. 4. Transport of different radionuclides from 0.5 M nitric acid.

The transport of ^{241}Am was found to be reduced substantially in the presence of uranium in the feed due to its cotransport. In these studies, uranium concentration in the feed was increased up to 1.5 g/L at 0.5 M HNO_3. These studies clearly indicate the need to deplete the uranium content from the HLLW. The transport decreases to ~90% in the presence of 0.5 g/L of uranium as compared to >97.2% in the absence of uranium. It decreased further to 76.7, 58.4 and 28.8% when the uranium concentration was increased to 0.75, 1.0, 1.5 g/L, respectively (Fig. 5).

The transport of ^{241}Am in the presence of increasing concentration of Nd(III) showed that the presence of macroconcentration of Nd(III) also reduce the rate of transport of ^{241}Am because of its cotransport. The results for ^{241}Am, in such studies, carried out from feed solutions at 0.5 M HNO_3 with increasing Nd(III) up to 1.0 g/L showed that at 0.1 g/L of Nd in the feed reduced transport to only about 63.4% against 98.2% in its absence. On further increasing the concentration of Nd to 0.5 and 1.0 g/L, the transport of ^{241}Am decreased to 58.2 and 36.5% respectively (Fig. 6).

^{241}Am transport carried out in the presence of increasing concentration of salt ($NaNO_3$) up to 1.0 M, showed improvement in the transport rates of ^{241}Am, due to the salting out effect [26] and its quantitative transport was obtained in 4 h, as compared to 6 h required in the absence of $NaNO_3$ in a feed containing 0.5 M HNO_3 (Table 2). This shows that the presence of salt enhance the transport kinetic. In another important study, highly radioactive solutions were left in the experimental set-up undisturbed for 3 more days, to study the effect of radiation on the performance of membrane. After the removal of solutions, the membrane was reused to study the transport of americium from uranium lean simulated HLW using receiving phase containing citric acid, formic acid and hydrazine hydrate. No change in permeation characteristics was observed indicating the stability of the membrane under above experimental conditions.

In transport studies using simulated waste solutions also, uranium was first depleted by solvent extraction using repeated batch contacts of 30% TBP in *n*-dodecane (three contacts). The resultant raffinate was used as feed after spiking ^{241}Am. More than 98% transport of ^{241}Am was observed in 12 h (Fig. 7). In

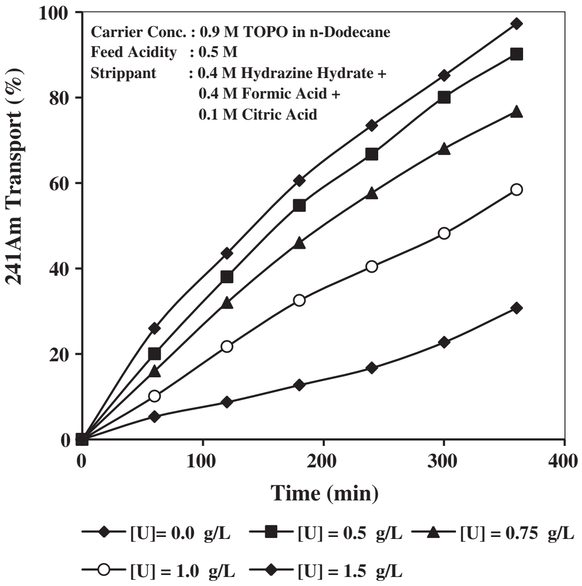

Fig. 5. Transport studies of ^{241}Am from 3.0 M nitric acid in presence of Uranium.

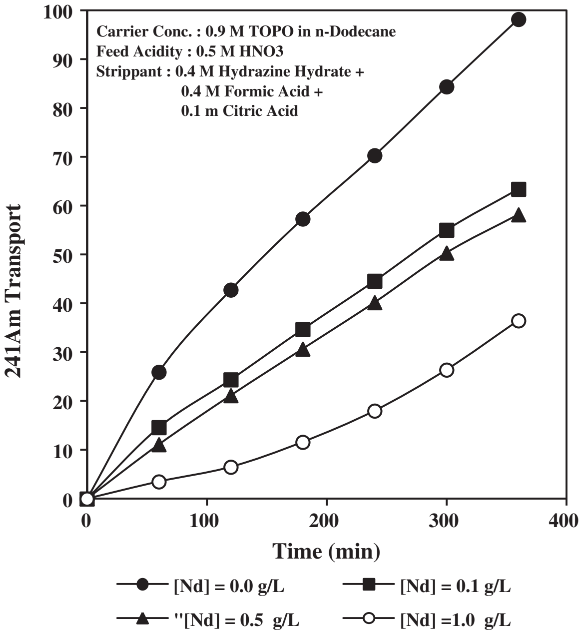

Fig. 6. Transport studies of ^{241}Am from 3.0 M nitric acid in presence of Neodymium.

the final experiment, actual HLLW from reprocessing of the spent fuel of research reactor origin after depleting uranium to less than 10 mg/L was used as the feed. Gross α analysis indicated transport of >98% in 10 h (Fig. 7). This shows that this technique can be used for the partitioning of alpha emitters from HLLW prior to the burial of the nuclear waste in deep geological surveillance facilities.

Table 2
Effect of $NaNO_3$ concentration on the transport of ^{241}Am, Feed: 0.1762 µg /mL, Acidity of Feed = 0.5 M, Strippant: 0.4 M Formic acid + 0.4 M hydrazine hydrate + 0.1 M citric acid, Volume of feed and receiver compartment: 5.0 mL, Concentration of carrier: 0.9 M TOPO in *n*-dodecane

Time (min)	Transport of ^{241}Am in varying conc. of salt ($NaNO_3$), M		
	0.0	0.5	1.0
60	25.95	29.55	32.79
120	42.78	49.45	55.58
180	57.33	62.78	69.28
240	70.31	94.67	98.81

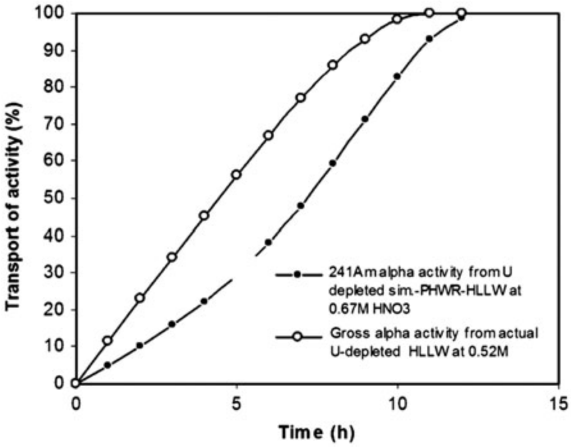

Fig. 7. Partitioning of α-emitters from HLLW.

Table 3
Permeability coefficients and flux for different actinide ions

Species	Permeability coefficients (m/s) × 10^6	Flux (moles/m^2 s) × 10^{12}
U(VI)	7.3	7.55
Np(IV)	3.5	2.25
Np(VI)	5.3	5.24
Pu(IV)	5.0	1.20
Am(III)	2.2	1.50

4. Conclusion

TOPO containing supported liquid membranes can be employed for the partitioning of actinides from high-level waste solution. Fast separation of neptunium, uranium, plutonium and americium can be accomplished using a solution of TOPO in *n*-dodecane at a concentration of 0.9 M as carrier, while using a mixture of 0.1 M citric acid, 0.4 M formic acid and 0.4 M hydrazine hydrate as receiving phase. Partitioning of these actinides will avoid the radiological risk due to these minor actinides during the storage of nuclear waste. If these actinides are not separated, it may leach into the environment during their long-term storage also cost of their surveillance gets reduced. The proposed receiving phase can be used for the feed with nitric acid content between 0.01 and 2.0 M. Since the transport rates are found to be very low in the presence of high concentration of uranium, the present method can give best results only after depleting the uranium content of the waste solution. Lower acid concentration of the feed (<1 M) exhibited faster transport of actinides under these conditions.

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