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DIAMEX Counter-Current Extraction Process for Recovery of Trivalent Actinides from Simulated High Active Concentrate

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Abstract: The partitioning of trivalent actinides was demonstrated with a new version of the French DIAMEX (DIAMide EXtraction) process. A continuous counter-current experiment using a 16-stage centrifugal extractor battery was tested using 1 mol/L N,N′-dimethyl-N,N′-dioctyl-hexylethoxy-malonamide (DMDOHEMA) in TPH as the extractant. A high active concentrate (HAC), obtained after concentration and denitration of a high active raffinate (HAR) with a concentration factor of 10, was used as a feed. Based on results from cold and hot batch extraction experiments and computer code calculations, a flowsheet was developed and a full test was carried out using a simulated HAC solution spiked with radionuclides (^{241}Am, ^{244}Cm, ^{152}Eu, and ^{134}Cs). In the DIAMEX process, five extraction stages were sufficient to obtain Am and Cm (feed/raffinate) greater than 5000 and for the coextracted lanthanides decontamination factors between 1100 and 4500. Co-extraction of zirconium, molybdenum, and palladium was prevented by using oxalic acid and HEDTA. The back extraction comprising 4 stages was also efficient and the recoveries of actinides were greater

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than 99.8%, which can be further improved by a minor process flowsheet optimisation. The experimental steady-state concentration profiles of important solutes were determined and compared with model calculations and good agreement was generally obtained.

Keywords: Solvent extraction, DIAMEX process, DMDOHEMA, minor actinides, partitioning

INTRODUCTION

Nuclear power constantly produces a mass of spent fuel which contains, apart from the short-lived fission products, a significant amount of the higher actinides neptunium (Np), plutonium (Pu), americium (Am), and curium (Cm) which are responsible for long-term radiotoxicity. Selective partitioning followed by transmutation (P&T strategy) to short-lived or stable nuclides by physical means is an attractive option for reducing the radiotoxicity of the high-level waste by a factor proportional to the separation yield (1, 2). The current reprocessing technique according to the PUREX process, as commercially operated in France and the UK, only separates U and Pu as valuable materials from the spent fuel elements to reuse them as nuclear fuel (e.g. U/Pu-MOX). Laboratory studies have shown that the PUREX process can be adapted to also include complete Np separation (3, 4).

However, partitioning of the remaining minor actinides (MA), Am, and Cm, is not possible with the present reprocessing technique, and so together with the fission products, they are channelled into the high active raffinate (HAR) of the PUREX process. The partitioning of americium and curium is a much more difficult task since their chemical properties are very similar to those of the lanthanides (Ln), which are up to 20 times more concentrated in the HAR of the PUREX process. The separation of Am and Cm additionally requires more complex partitioning steps and is the subject matter of our investigations. The following flowchart (Fig. 1) gives an overview of advanced reprocessing of spent fuel based on hydrochemical processes. One of the most promising options for the removal of MA is the DIAMEX-SANEX concept (5, 6). In the first step, An(III) and Ln(III) are coextracted directly from HAR, e.g. by malonamide within the DIAMEX process, and separated from the remaining 2/3 of the fission products, while in the second step, trivalent actinides are separated from Ln(III) by a SANEX (Selective ActiNide EXtraction) process. The advantages of these processes are the high purity of the An(III) product and the reduced volume of the secondary waste.

The French DIAMEX process, further developed in European collaborations (5, 6) uses a diamide, a solvating extractant in an aliphatic diluent as the solvent. Besides the good extractability of trivalent actinides from HAR, the diamide derivatives are completely incinerable and produce no further radioactive waste (7–10).

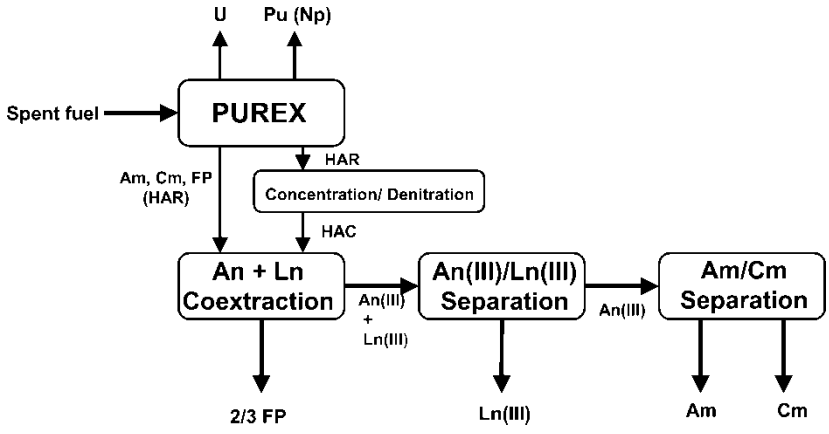

Figure 1. European partitioning strategy for the separation of all actinides from spent fuel.

Recently, N,N′-dimethyl-N,N′-dioctyl-hexylethoxy-malonamide (DMDO HEMA) was selected as the new reference molecule for the DIAMEX process instead of the previously used DMDBTDMA (N,N′-dimethyl-N,N′-dibutyl-tetradecyl-malonamide). DMDOHEMA (see Fig. 2) is sufficiently robust against hydrolysis and radiolysis and, due to the increased lipophilicity, the third phase formation is surpressed.

Within the PARTNEW project (6) of the European Union's 5th Framework Programme, several partners (ITU, CEA and FZJ) worked together to define a DIAMEX process for treating high active concentrates (HACs), the product after the concentration and denitration of HAR. The expected advantage when processing HACs instead of HARs is to be found in the low volumes of feed to be processed and hence the compactness of the experimental facilities required. Due to the high metal ion concentrations (up to 15 times greater than HAR) the question arises as to how to prevent the co-extraction of the second row transition elements (fission products such as Zr, Mo, Ru, Rh, and Pd) with trivalent actinides and thus avoid third-phase formation.

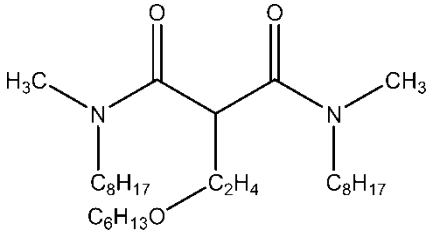

Figure 2. Structure of DMDOHEMA, the new malonamide for the DIAMEX process.

A successful concentration and denitration of a genuine raffinate has been reported by the ITU team (11). In this hot test, a high active concentrate was generated with a concentration factor of approx. 11 and an acidity of 4 mol/L HNO_3. The experimental conditions for a hot DIAMEX-HAC test using 16 centrifugal extractors were established by cold and hot batch extraction studies followed by computer code calculations. However, before that, a final spiked test needs to be carried out in order to reduce possible sources of malfunction, such as poor phase separation or precipitation inside the centrifuges. The continuous counter-current process using a spiked HAC was carried out at the Research Centre Jülich with four batteries of centrifugal contactors and the main results obtained are presented in this paper.

EXPERIMENTAL

Reagents

N,N′-dimethyl-N,N′-dioctyl-hexylethoxy-malonamide (DMDOHEMA), purity 97.3%, was used as provided by Panchim, Lisses, France. The industrial diluent TPH (hydrogenated tetrapropylene) used in COGEMA's reprocessing plants was employed as the diluent. Nitric acid solutions were prepared by diluting concentrated nitric acid (Merck KGaA, Darmstadt, Germany) with ultrapure water. The acidity of the initial aqueous solutions is determined by potentiometric titration, using a METROHM 798 MPT Titrino device and a [NaOH] = 0.1 mol/L or 0.01 mol/L solution. All reagents and chemicals were of analytical reagent grade. The radiotracers ^{241}Am, ^{244}Cm, ^{152}Eu, and ^{134}Cs were supplied by Isotopendienst M. Blaseg GmbH, Waldburg, Germany.

The feed solution used for the batch extraction studies as well for the spiked tests was a simulated high active concentrate (HAC), obtained by concentration and subsequent denitration of a simulated PUREX-HAR solution. Details of the production of the HAC can be found in (11). The composition of the synthetic HAC is shown in Table 1. The feed was spiked with ^{241}Am, ^{244}Cm, ^{152}Eu, and ^{134}Cs giving a final volume of 200 mL and an activity of around 6.25 MBq/L for each nuclide. Oxalic acid (for Zr and Mo complexation) and N-(2)-(hydroxyethyl)ethylenediaminetriacetic acid (HEDTA) (for Pd complexation) were also added.

Analysis

Activity measurements of the γ-ray emitters ^{241}Am, ^{152}Eu, and ^{134}Cs were performed with a γ-ray spectrometer provided with a high-purity germanium detector from EG & Ortec, Munich, Germany and equipped with the Gamma Vision software. The γ-lines at 59.5 keV, 121.8 keV, and

Table 1. Composition of the synthetic HAC feed solution used for the spiked DIAMEX-HAC test

Element	mg/L
Rb	142
Sr	154
Y	169
Zr	217
Mo	552
Ru	903
Rh	352
Ag	61
Cd	78
Sn	28
Te	1777
Cs	2947
Ba	177
La	894
Ce	1730
Pr	702
Nd	2684
Sm	824
Eu	159
Gd	275
Nuclide	MBq/L
^{241}Am	6.25
^{152}Eu	6.25
^{244}Cm	6.25
^{134}Cs	6.25
Complexant	mol/L
$[H_2C_2O_4]$	0.1
[HEDTA]	0.05
$[H^+]$	3.96

604 keV were examined for ^{241}Am,^{152}Eu, and ^{134}Cs, respectively. The nuclides ^{244}Cm and ^{241}Am were measured by means of alpha spectrometry with an α-Analyst from Canberra-Packard GmbH, Dreieich, Germany. Stable elements were determined by ICP-MS on an Elan 6100 DRC from Perkin Elmer Sciex, Roggau-Jügesheim, Germany. The aqueous phase was measured directly after adequate dilution. The organic phase was co-analysed after having been digested by means of microwave digestion. A standard Meinhard nebulizer and a cyclone spray chamber were used for sample introduction. The ICP-MS calibration solutions have been purchased from CPI International, Amsterdam, The Netherlands.

Equipment and Procedure

A DIAMEX process with spiked simulated HAC was carried out using miniature centrifugal extractors in a counter-current mode. The contactor setup consisted of 4 batteries of four stages each, reagent feed and receiving vessels, and pumps with associated controllers. The contactors were designed and fabricated at INET, Tsinghua University, Beijing, China. Their rotor diameter was 10 mm and hold-up volume was 3–4 mL in each stage.

The organic extractant used was 1 mol/L DMDOHEMA dissolved in TPH. On the basis of the results obtained from batch tests and from computer code simulations with the aid of the PAREX code (12), an optimized flowsheet for a 16-stage counter-current process was designed by the CEA, see Fig. 3. The design of the flowsheet was based on the earlier HAR flowsheet which used 0.65 mol/L DMDOHEMA in TPH as the solvent modified with regard to the batch experiment data obtained with 1 mol/L DMDOHEMA (10).

The process comprises 5 stages for extraction, 5 stages for scrubbing, 2 stages for acid scrubbing of the organic phase and 4 stages for back extraction. A/O flow rate ratios were set to about 1 for both the extraction and the back extraction. The acid scrubbing section was included in order to reduce the general acidity in the back-extraction section and to thus improve the stripping effficiency.

In order to reach equilibrium faster, the continuous test was carried out as follows. First of all, pre-adjusted flows of the aqueous solutions were fed into the running centrifugal extractors. A cold solution of the same composition (without radionuclides) was first used as the feed solution. The extractant was added after the raffinate had emerged from stage 1. The active feed solution was added after the organic phase had left the outlet of the organic phase at stage 16.

The test was performed for three and a half hours. During the first hour and a half, the stripping solution was 0.1 mol/L HNO_3. However, an emulsion formed in the outgoing organic solution (water content approx. 20–30%). To solve this problem the acid content was increased to 0.3 mol/L. Although a great improvement was achieved, some drops (less than 1%) of

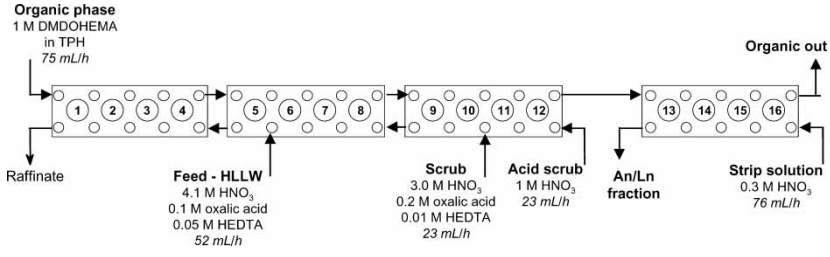

Figure 3. Schematic of the DIAMEX-HAC process flowsheet.

water were still found in the outgoing organic solution. The new stripping solution was in contact with the organic phase during the last two hours of the experiment, which was sufficient time to reach equilibrium. Attainment of the steady state was checked by gamma spectrometric measurements of ^{241}Am, ^{152}Eu, and ^{134}Cs at the outlets of stage 1 [raffinate] and stage 13 [An/Ln fraction].

At the end of the experiment, the motors and pumps were switched off and the individual stages were drained. Subsequently, the organic phase was separated from the aqueous phase by centrifuging and aliquots from both phases were used for the analyses.

The following analyses were carried out from all stages and from the samples collected (raffinate, An(III)/Ln(III) product, spent solvent): γ spectroscopy for ^{241}Am, ^{152}Eu, and ^{134}Cs, α spectroscopy for ^{241}Am, ^{244}Cm, and ICP-MS for all inactive elements. The acidity profile was determined for the aqueous phase by titration with NaOH.

RESULTS AND DISCUSSION

Optimization Studies before DIAMEX

In the DIAMEX process, oxalic acid has to be added to the DIAMEX feed in order to prevent third-phase formation due to the co-extraction of Zr and Mo. However, it has been demonstrated that with a high concentration of oxalic acid (>0.15 mol/L) in the HAC (depending on the concentration factor, CF) a precipitate is formed which, in addition to Zr and Mo as the main constituents, also contains considerable amounts of lanthanides (up to 20% of the initial concentration), and thus will also lead to a loss of trivalent actinides (13). For cold HAC solution with a concentration factor of up to CF = 10 no oxalate precipitation was observed after adding 0.2 mol/L oxalic acid. During batch extraction experiments with 0.65 and 1.0 mol/L DMDOHEMA in TPH no third phase formation was observed either. The results in Table 2 show good extraction for Am(III) and the lanthanides with 1.0 mol/L DMDOHEMA in TPH and distribution ratios between 4 and 9 at an acidity of 4.2 mol/L HNO_3 were obtained. As expected, the addition of oxalic acid prevented co-extraction of Zr and Mo, and the addition of HEDTA prevented the extraction of Pd.

DIAMEX Test

The aim of this test was to separate the trivalent actinides and the lanthanides from the bulk of the fission products by extracting and back-extracting both element groups more than 99%. Furthermore, high decontamination factors were to be achieved for most fission products.

Table 2. Extraction of Am(III), Eu(III) and HAR elements with DMDOHEMA from synthetic HAC (CF = 10, similar composition as in Table 1, (nitric acid) = 4.2 mol/L). The organic solution is composed of 1.0 or 0.65 mol/L DMDOHEMA in TPH

Element	Distribution ratios D	
Rb	0.001	0.001
Sr	0.012	0.006
Y	1.74	0.90
Zr	0.03	0.03
Mo	0.26	0.22
Ru	0.40	0.35
Rh	0.014	0.010
Pd	0.02	0.01
Cd	0.013	0.008
Te	0.001	0.001
Cs	0.001	0.001
Ba	0.028	0.024
La	5.8	2.8
Ce	7.1	3.5
Pr	7.0	3.4
Nd	5.9	2.9
Sm	4.7	2.4
Eu	4.0	2.0
Gd	3.6	1.9
^{152}Eu	4.7	2.0
^{241}Am	8.8	3.7
[DMDOHEMA]	1 mol/L	0.65 mol/L
[$H_2C_2O_4$]	0.2 mol/L	0.2 mol/L
[HEDTA]	0.05 mol/L	0.05 mol/L

The process decontamination factor $DF_{feed/raff}$ was calculated acccording to the following Equation 1, where V is the flow rate in mL/h and C is the concentration of the element in mg/L:

$$DF_{feed/raff} = \frac{V_{feed} \cdot C_{feed}}{V_{raff} \cdot C_{raff}} \quad (1)$$

The results in Table 3 show that the goals set were achieved. Good balances were achieved in the material flows (raffinate, An(III)/Ln(III) product, spent solvent) for the lanthanides(III), actinides(III) and all fission products. As can be seen from Table 3, hardly any lanthanides (<0.1%) and actinides(III) (<0.01%) are found in the raffinate. The addition of oxalic acid and HEDTA efficiently prevented the co-extraction of Zr and Pd, which are normally strongly extracted in the DIAMEX process (8). Most fission products were not extracted and have DFs of around 1. However, 7.4% Mo, 20% Ru, and 2.2% Sr were also coextracted. After back extraction with

Table 3. Recovery and process decontamination factors obtained in the spiked DIAMEX-HAC test

Element	% in raffinate	% in An/Ln product	% in solvent (stage16)	Total balance raff/prod/solv	$DF_{feed/raff}$
Rb	104.8	0.08	0.1	105.0	0.95
Sr	119.7	0.83	1.35	121.9	0.84
Y	0.67	99.8	0.05	100.5	150
Zr	100.1	0.29	0.84	101.2	1
Mo	92.3	4.4	3.01	99.7	1.08
Ru	78.8	1.03	19.01	98.8	1.27
Rh	102.7	0.01	0.04	102.8	0.97
Pd	101.8	0.11	0.27	102.2	0.98
Cd	103.1	0.05	0.09	103.2	0.97
Te	101.1	0.03	0.15	101.2	0.99
Cs	109.4	d.l.	d.l.	109.4	0.91
^{134}Cs	100.7	d.l.	d.l.	100.6	0.99
Ba	104.6	0.09	0.15	104.8	0.96
La	0.09	99.8	0.08	99.9	1109
Ce	0.07	99.5	0.17	99.7	1513
Pr	0.05	99.4	0.15	99.6	1972
Nd	0.02	100.2	0.09	100.3	4146
Sm	0.02	101.1	0.03	101.1	4389
Eu	0.06	101.7	0.03	101.8	1624
Gd	0.06	101.9	0.03	101.9	1732
^{152}Eu	0.04	98.8	0.03	98.9	2468
^{241}Am	0.02	103.9	0.13	104.1	4770
^{244}Cm	0.01	103.9	0.04	103.9	8461

d.l. = below detection limit.

0.3 mol/L HNO_3, the An/Ln product stream contains more than 99.8% of the actinides(III) and lanthanides(III) and is only contaminated by 0.1% Pd, 0.3% Zr, 4.4% Mo, 1% Ru, and 0.8% of Sr. The decontamination factors could be improved by increasing the number of scrubbing stages, whereas a lower scrub acidity must only be considered with care to avoid lanthanide oxalate precipitation, since the aqueous lanthanide concentrations are high in the scrubbing section (see Fig. 4).

The analysis of the spent solvent shows that four stages with 0.3 mol/L HNO_3 are sufficient to almost completely strip the actinides and lanthanides. Only 0.13% of the Am(III) and less than 0.05% of the lanthanides(III) are found in the extractant. Even better results can easily be achieved by increasing the number of stages in the stripping section or by increasing the flow rate from 75 ml/L to 90 ml/L HNO_3. Ruthenium shows an unusual behavior. Although the distribution ratios are relatively low under the extraction conditions ($D_{Ru} = 0.4$, see Table 2), approx. 19% is irreversibly coextracted and left in the spent solvent. The experimental aqueous concentration

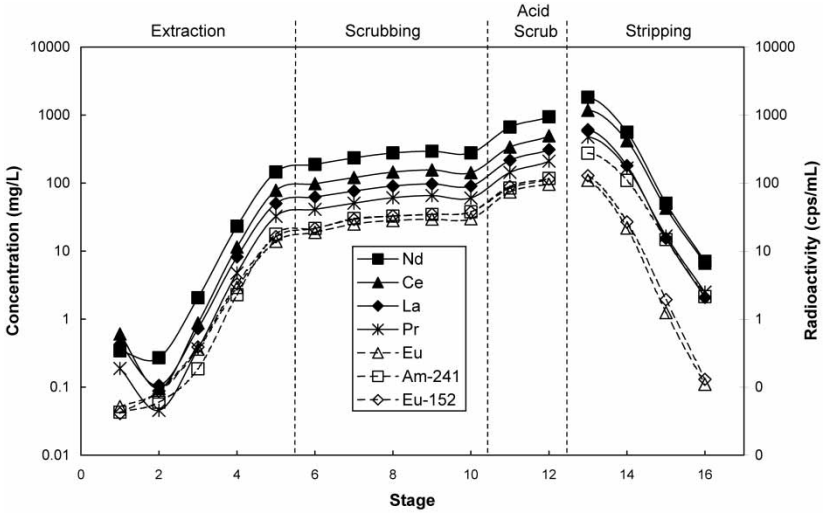

Figure 4. Aqueous concentration profiles of some relevant An and Ln during the spiked DIAMEX-HAC process.

profiles of some relevant fission products including Ru are shown in Fig. 5. The Ru profile does not indicate that the stripping of Ru can be improved by more stages, since the Ru concentration does not decrease from stage 13 to stage 16 and a plateau is formed. The behavior of Ru in the solvent

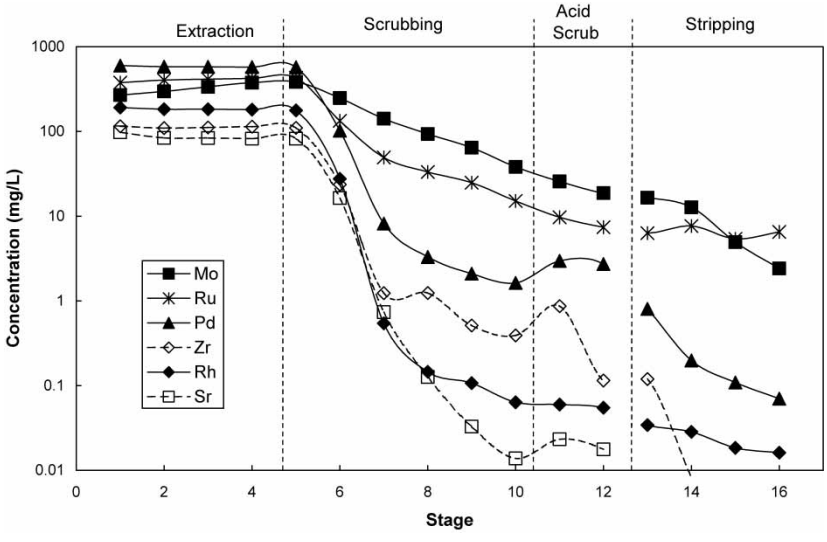

Figure 5. Aqueous concentration profiles of some relevant fission products during the spiked DIAMEX-HAC process.

clean-up operation step should therefore be studied. Problems are also encountered with the Mo, 3% of which is left in the solvent. The behavior of Mo and Ru must therefore be investigated more closely in the DIAMEX process treating a high active concentrate. The results obtained during a hot demonstration of the DIAMEX process using DMDOHEMA and a genuine HAR showed similar behaviour for the trivalent actinides and lanthanides. With the nearly same flowsheet (with the exception of stripping with 0.1 mol/L HNO_3) used here, the coextraction of Mo, Zr, and Pd was prevented, thanks to oxalic acid and HEDTA scrubbing, respectively. Only 4% of Ru was extracted and 3% was left in the spent solvent (10).

The experimental aqueous acidity profile and the comparison with the model during the DIAMEX HAC test is shown in Fig. 6. The experimental acidity was estimated considering that the first endpoint corresponds to nitric acid + the two $H_2C_2C_4$ acidities + the two first HEDTA acidities. The nitric acid extraction explicitly takes into account the solvation of nitric acid with DMDOHEMA. A co-extraction of acid is observed in the extraction and scrubbing sections, yielding a maximum acidity in stage 5. Although 1 mol/L HNO_3 is used for the acid scrubbing of the organic phase, the acidity in the first back-extraction stage is always as high as 0.7 mol/L. This means that a subsequent process for separation of actinides from lanthanides needs either a feed adjustment by means of denitration or a solvent capable of extracting directly from 0.7 mol/L HNO_3 concentration. A large number of SANEX extractants have been developed in European research projects (5, 6). Unfortunately, most of them were only able to selectively extract trivalent actinides from solutions of very low acidity (>pH 2),

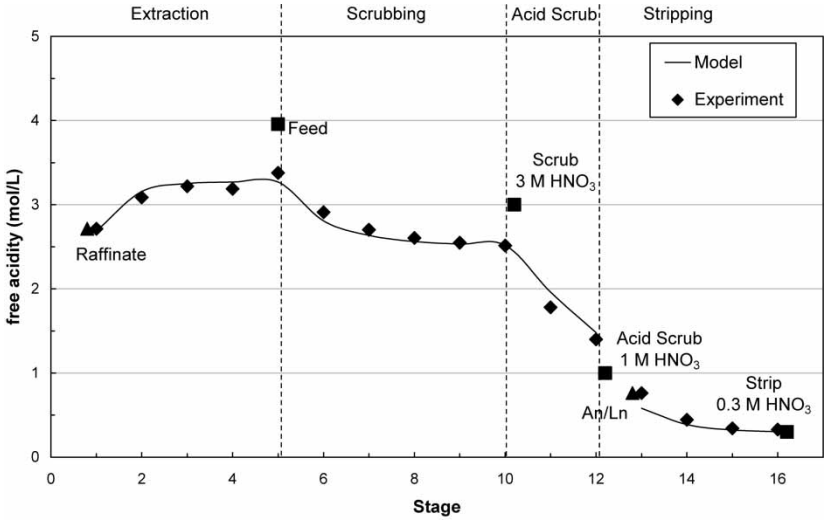

Figure 6. Experimental and calculated acidity profile.

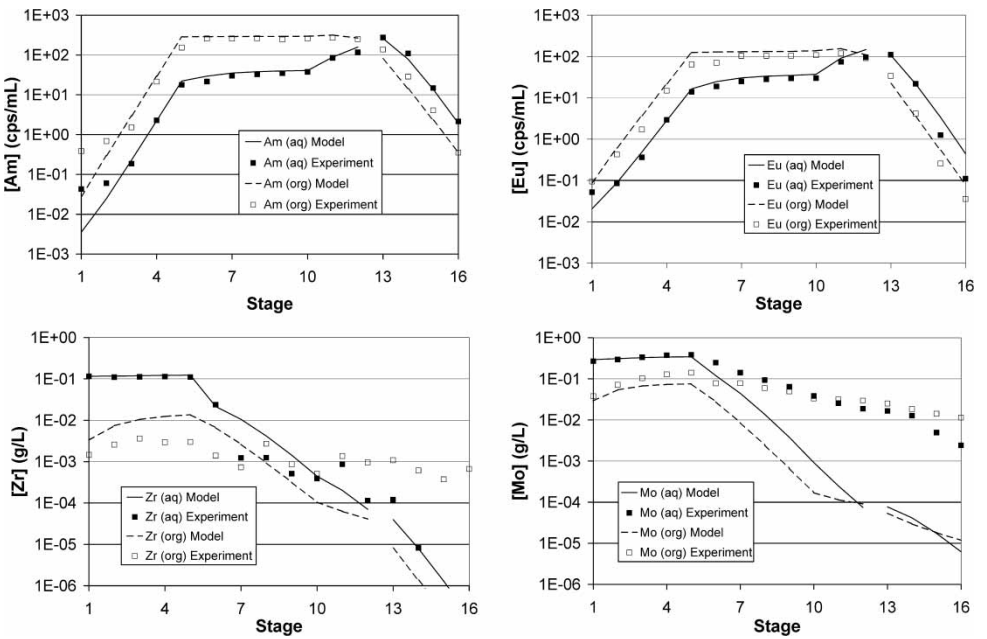

Figure 7. Comparison between the experimental and simulated concentration profiles for Am, Eu, Zr, and Mo (both aqueous and organic).

rendering them useless for further process development. Extractants from the family of aromatic dithiophosphinic acids (14) or the heterocyclic N-donor extractants, such as the BTBPs (bistriazinylbipyridenes) (15, 16) were recently found to be able to perform the difficult separation from feed solutions of rather high acidities (0.5–1.0 mol/L HNO_3). Process development studies are under way in the current European project, EUROPART.

In Fig. 7 the aqueous and organic concentration profiles of Am, Eu, Zr, and Mo are shown and compared with results from computer code simulations (PAREX). The experimental concentration profiles are fairly well simulated for trivalent actinides (represented by Am) and lanthanides (represented by Eu).

At contrast, simulation of Zr and Mo in the scrubbing and stripping section are not well simulated. The main hypothesis for explaining the retention of these elements in the solvent is slow kinetics for back extraction, since a thermodynamic equilibrium was not reached during the test. The simulation of the Zr and Mo concentrations can be improved by taking into account a lower transfer kinetic. Such calculations were not performed in the present study, since we need to know the exact volume of each phase in each well of the extractors to simulate the equilibrium state reached after sample collection.

CONCLUSIONS

The spiked DIAMEX-HAC test showed that this process could treat a feed solution containing high concentrations of fission products. The extraction performance goals were achieved: actinide(III) and lanthanide(III) extraction and stripping were quantitative.

Zirconium and palladium backextraction was very good thanks to oxalic acid and HEDTA.

Molybdenum was coextrated to some extent (7.4%) and distributed to the An(III)/Ln(III) product (4.4%) and to the spent solvent (3%). In summary, it may be stated that the good performance of the spiked test and the subsequent analytical evaluation of the experiment furnished very positive results, so that a successful hot DIAMEX test with a genuine HAC could performed after a minor modification of the flowsheet.

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