

Hydrometallurgical Minor Actinide Separation in Hollow Fibre Modules

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Abstract – hollow fibre modules (HFM) were used as phase contacting devices for hydrometallurgical minor actinide separation in the Partitioning & Transmutation context. Two single-HFM setups were built, one using commercially available HFM, the other one using miniature HFM we have developed and manufactured. Several very successful DIAMEX and SANEX once-through tests were performed. The major advantage of the new miniature HFM is their size, drastically reducing chemicals consumption: Only several 10 mL of feed phases are required for a test.

INTRODUCTION

The separation of long-lived radionuclides from spent nuclear fuels is a prerequisite for their transmutation to short-lived (and finally, stable) nuclides. PARTNEW [1], a project within the EURATOM 5th framework programme, aimed at the hydrometallurgical separation of americium and curium from PUREX raffinate (HAR) or concentrate (HAC).

Part of our contribution to PARTNEW was to assess the performance of hollow fibre modules (HFM) as phase contacting devices for liquid-liquid extraction. Two HFM extraction setups were built, one using commercially available HFM, the other one using miniature HFM developed and built at our institute. With these, several very successful single-HFM, single-pass DIAMEX (*i.e.*, actinides(III)-lanthanides co-extraction) and SANEX (*i.e.*, actinides(III)-lanthanides separation) tests were performed.

HFM as Phase Contacting Devices for Liquid-liquid Extraction

Generally, continuous liquid-liquid extraction is performed using mixer-settlers, column contactors or centrifugal contactors. Alternatively, HFM can be implemented as phase contactors. They consist of a bundle of micro-porous hollow fibres in a shell tube, similarly to a shell-and-tube heat exchanger. One phase flows through the lumen of the hollow fibres, the other one counter-currently on the shell-side. Since organic and aqueous phases are macroscopically separated by the membrane, HFM offer several hydrodynamic advantages over other contactors, most notably, the absence of flooding or entrainment [2].

THE HFM AND SETUPS USED

Commercially Available HFM

A single-HFM setup, shown schematically in Fig. 1, was built and installed in a glove box. *Celgard Liqui-Cel*[®] 2.5 x 8 Extra-Flow modules [3] were used, *cf.* Fig. 2. These modules consist of 10000 *Celgard X30-240* polypropylene hollow fibres (inner diameter = 0.24 mm, outer diameter = 0.30 mm, average pore size = 0.02 μm , porosity = 40 %). Active length is 0.15 m.

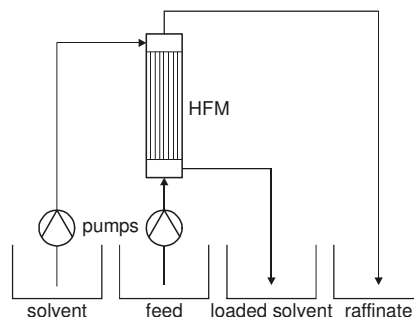


Fig. 1. Counter-current single-HFM setup (extraction mode).

Development of Miniature HFM

The large HFM setup consumes several litres of feed phases for a test. This is not convenient as the extractants used have to be synthesised in quite large quantities. Hence, we developed and built new scaled down modules. Using an epoxy resin, approx. 100 *Celgard X30-240* fibres were potted into a glass tube with two shell-side ports. Inner diameter of the shell tube is 4 mm (*cf.* Fig. 3). Active length of the modules is 0.20 m. The miniature HFM were operated in a setup analogous to the one shown in Fig. 1.

The major advantage of these miniature HFM is the reduction of feed solutions consumption: While several litres were required for a test using the large modules, as little as 20-30 mL of feed phases suffice with our new miniature HFM.



Fig. 2. *Liqui-Cel*[®] 2.5 x 8 HFM and miniature HFM compared.

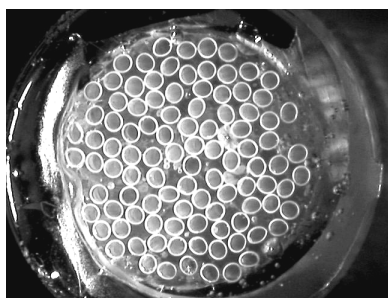


Fig. 3. View of the lumen-side entrance of a miniature HFM consisting of 100 fibres.

HFM COMPUTER CODE DEVELOPMENT

Computer codes were established to calculate transient and steady-state effluent concentrations for the HFM experiments. The codes are based on equilibrium and kinetic data, diffusion coefficients, and a hydrodynamic description of the module. Resistances to mass transfer due to diffusion (in both phases and in the membrane pores) and due to the chemical reaction (if contributing) are taken into account.

The module is divided along its length axis into an appropriate number of cells. In each of these, individual mass transfer coefficients for all species involved are calculated from hydrodynamic and diffusion data. Using these individual mass transfer coefficients, together with equilibrium

data and module and membrane geometry, changes in concentration in each cell due to mass transfer across the liquid-liquid interface are calculated. Tube and shell side cell concentrations then are passed on step-wise, and again, changes in concentration in the cells are calculated, until steady state is attained.

Several versions of the code were written for large and miniature HFM, for cation-exchanging extractants and solvating extractants, for systems with diffusional or chemical control. The calculations are described in more detail in [4] for a cation-exchanging extractant with diffusional control. A chemical reaction rate is implemented in the codes for systems where mass transfer is controlled by a chemical reaction.

DIAMEX HFM TESTS

The co-extraction of americium(III) and lanthanides from a simulated PUREX raffinate into DMDOHEMA [5] (Fig. 4) dissolved in TPH (kerosene) was studied in a miniature HFM [6].

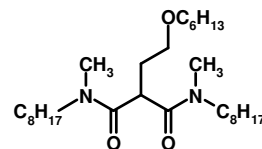


Fig. 4. *N,N'*-dimethyl-*N,N'*-dioctyl-2-(2-hexyloxy-ethyl)-malonamide, DMDOHEMA.

Computer code calculations indicated that the amount of nitric acid present in the organic phase did not influence americium back-extraction efficiency. To prove this, a back-extraction test was made, assessing the influence of nitric acid concentration in the loaded organic phase [6].

Experimental

The extraction experiment was performed in a module consisting of 100 fibres. Aqueous feed solution was a simulated inactive PUREX raffinate spiked with ²⁴¹Am. Oxalic acid and *N*-(2-hydroxyethyl)-ethylenediamine-*N,N',N'*-triacetic acid (HEDTA) were added to suppress co-extraction of zirconium, molybdenum and palladium. Table I gives the composition. It flowed inside the hollow fibres with varied flow rates, 2.0, 2.8 and 4.3 mL/h. Organic phase was a solution of 0.65 M DMDOHEMA in TPH, flowing shell-side counter-currently at 2.0 mL/h.

For the back-extraction test two organic phases containing ^{241}Am , Ce and different amounts of nitric acid were prepared. Each contained approx. 0.6 MBq/L ^{241}Am and roughly 1 g/L Ce. Their nitric acid concentrations were determined by duplicate titration to 0.11 M and 0.5 M, respectively. A module consisting of 90 fibres was used, with the organic phase flowing inside the hollow fibres at 1.0 mL/h. Aqueous phase was 0.1 M HNO_3 , flowing shell-side counter-currently at 1.8 mL/h.

TABLE I. Composition of the DIAMEX feed solution (concentrations are mg/L unless otherwise stated).

| Element | Concentration |
|-------------------|---------------|
| ^{241}Am | 20 MBq/L |
| Rb | 21 |
| Sr | 45 |
| Y | 26 |
| Zr | 360 |
| Mo | 244 |
| Ru | 111 |
| Rh | 48 |
| Pd | 208 |
| Ag | 9.6 |
| Cd | 11.6 |
| In | 1.7 |
| Sn | 2.6 |
| Sb | 2 |
| Te | 82 |
| Cs | 525 |
| Ba | 166 |
| La | 119 |
| Ce | 214 |
| Pr | 104 |
| Nd | 392 |
| Sm | 92 |
| Eu | 17 |
| Gd | 34 |
| Dy | 15 |
| oxalic acid | 0.2 M |
| HEDTA | 0.02 M |
| HNO_3 | 4 M |

Analytic

^{241}Am activities in aqueous and organic samples were determined by gamma counting. Lanthanide and non-lanthanide fission product concentrations in aqueous effluent samples and in the feed solution were analysed by ICP-MS after appropriate dilution in 2 % nitric acid (suprapur).

Organic effluent samples were prepared as follows: 25 μL of the organic sample was dissolved

in 5 mL of a solution of 10 g/L Triton X-100 in 2 % nitric acid (suprapur). Standards were prepared accordingly, *i.e.*, containing the same amount of Triton X-100 and organic phase. The samples were then analysed for non-lanthanide fission products by ICP-MS. Since lanthanides were extracted more or less quantitatively, they were not determined in the organic phase.

Results and Discussion

Extraction

With each aqueous flow rate selected in the DIAMEX extraction test, steady state was reached, as seen from the transient concentration courses (not shown). Both aqueous and organic effluents were clear, without any entrainment.

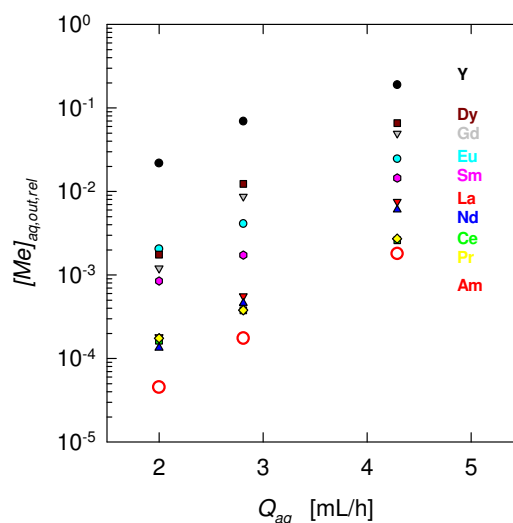


Fig. 5. DIAMEX miniature-HFM extraction test, relative aqueous effluent americium and lanthanide concentrations over aqueous flow rate.

The relative amounts of americium and lanthanides found in the raffinate, $[Me]_{aq,out,rel}$, as a function of aqueous flow rate, Q_{aq} , are shown in Fig. 5: Typically, with HFM extraction there is a strong dependency of extraction efficiency on flow rate. At $Q_{aq} = 2.0$ mL/h, americium was extracted to 99.995 %, corresponding to an americium decontamination factor of 20000. Lanthanides were co-extracted to approx. 99.9 %, yttrium to 98 %. At $Q_{aq} = 4.3$ mL/h, still 99.8 % of americium was extracted.

Non-lanthanide fission products co-extraction was quite low, so they were analysed in the loaded organic phase. Results are shown in Fig.

6 as a function of Q_{aq} : Approx. 15 % of ruthenium and molybdenum were co-extracted. Zirconium co-extraction was suppressed to approx. 1 %, due to the presence of oxalic acid in the feed phase. Palladium co-extraction was approx. 0.5 %, due to HEDTA. Other fission products were extracted to 1 % or well below.

Decontamination factors are given in Table II. It is important to keep in mind that these are results from a single-HFM extraction experiment, *i.e.*, no fission product scrubbing was made.

TABLE II. DIAMEX miniature-HFM extraction test, decontamination factors. For Am, Y and Ln, $DF = 1/[Me]_{aq, out, rel}$. For other fission products,

| Element | $DF = 1/[Me]_{org, out, rel}$ | | |
|-------------------|-------------------------------|----------|----------|
| | 2.0 mL/h | 2.8 mL/h | 4.3 mL/h |
| Rb | n/a | n/a | n/a |
| Sr | 62 | 120 | 140 |
| Y | 46 | 14.5 | 5.3 |
| Zr | 73 | 110 | 130 |
| Mo | 4.9 | 7.0 | 9.1 |
| Ru | 6.7 | 5.3 | 10 |
| Rh | 1200 | 1200 | 2200 |
| Pd | 180 | 230 | 240 |
| Ag | 79 | 120 | 120 |
| Cd | 86 | 130 | 180 |
| In | 220 | 390 | 520 |
| Sn | n/a | n/a | n/a |
| Sb | n/a | n/a | n/a |
| Te | 8400 | 27000 | 29000 |
| Cs | 1400 | 2800 | 3100 |
| Ba | 510 | 370 | 210 |
| La | 5500 | 1800 | 130 |
| Ce | 6100 | 2600 | 385 |
| Pr | 5700 | 2600 | 370 |
| Nd | 7400 | 2200 | 165 |
| Sm | 1200 | 580 | 69 |
| Eu | 490 | 240 | 41 |
| Gd | 830 | 115 | 20 |
| Dy | 570 | 80 | 15 |
| ²⁴¹ Am | 20000 | 5700 | 555 |

Back-extraction

A back-extraction test was run by contacting different organic phases with 0.1 M nitric acid. The transient americium activity in the back-extracted organic phase is shown in Fig. 7: In the first run, the organic feed phase contained 0.11 M HNO₃. Americium back-extraction efficiency approached 99 %, *i.e.*, 1 % of americium remained in the effluent organic phase. Americium mass balance was 107 %. The experiment

was re-started using the organic feed phase containing 0.5 M HNO₃. Now, 1.5 % of americium remained in the effluent organic phase. Americium mass balance was 99.7 %. Finally, the experiment was re-started, once more with the organic feed phase containing 0.11 M HNO₃. Americium back-extraction efficiency is practically unchanged, with 1.4 % of americium remaining in the organic phase. Americium mass balance was 99.2 %.

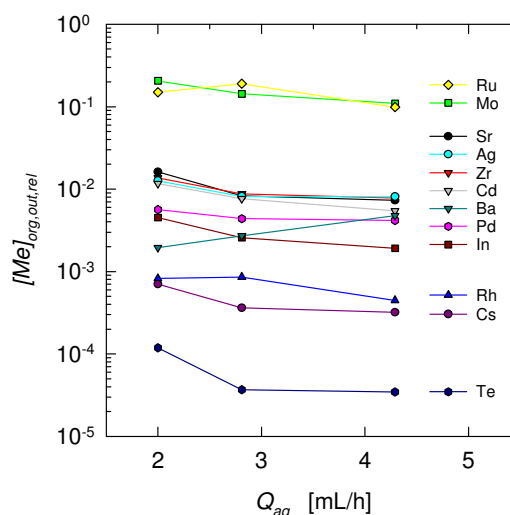


Fig. 6. DIAMEX miniature-HFM extraction test, relative organic effluent non-lanthanide fission products concentrations over aqueous flow rate.

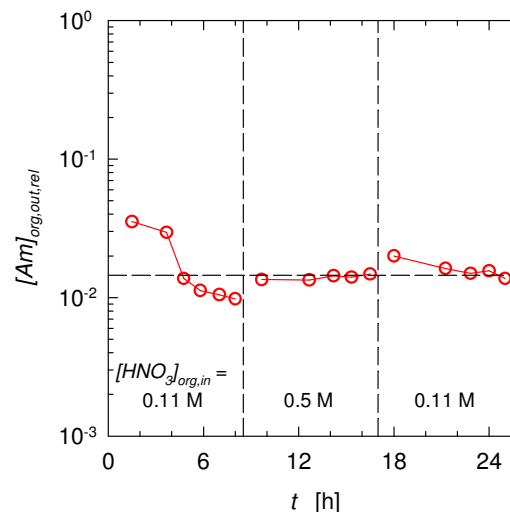


Fig. 7. DIAMEX miniature-HFM back-extraction test, transient relative americium concentration in back-extracted organic phase for varied concentration of nitric acid in loaded organic feed phase.

This means that the relatively large amount of nitric acid co-extracted in a counter-current DIAMEX process (which is in the range of 0.5 M) does not affect counter-current back-extraction performance. HFM mass transfer calculations confirm this fact. Furthermore, the calculations indicate that by reducing organic phase flow rate to approx. 0.6 mL/h, other parameters unchanged, >99.9 % back-extraction of americium is feasible.

SANEX-III TESTS

Three single-module HFM tests were performed using *n*-Pr-BTP [7] (Fig. 8) in TPH/*n*-octan-1-ol (7:3) as extractant. One test in the large setup, and two using a miniature HFM. Feed solution was a synthetic DIAMEX product, containing lanthanides at a total concentration of 2.5 g/L and ^{241}Am in trace amount in 1 M HNO_3 .

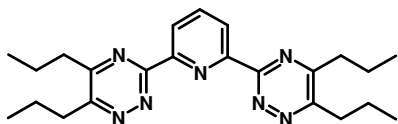


Fig. 8. 2,6-di(5,6-dipropyl-1,2,4-triazin-3-yl)pyridine, *n*-Pr-BTP.

Experimental

SANEX-III Test in the Large HFM Setup

In this test, the feed solution (*cf.* Table III, with 0.5 MBq/L ^{241}Am) flowed on the shell-side of the HFM. Organic phase was a solution of 0.04 M *n*-Pr-BTP in TPH/*n*-octan-1-ol (7:3 vol.). Aqueous flow rate was varied, $0.44 \text{ L/h} \leq Q_{aq} \leq 1.75 \text{ L/h}$, organic flow rate was $Q_{org} = 0.50 \text{ L/h}$. ^{241}Am was measured in both aqueous and organic samples by gamma counting. Lanthanides concentrations in aqueous samples were determined by ICP-OES. Organic samples were stripped into 0.01 M nitric acid before ICP-OES measurement.

SANEX-III Tests in the Miniature-HFM Setup

A SANEX-III test was performed in a miniature HFM consisting of 105 fibres. Aqueous phase (*cf.* Table III, with 3.7 MBq/L ^{241}Am) flowed through the hollow fibres. Organic phase was a solution of 0.04 M *n*-Pr-BTP in TPH/*n*-octan-1-ol (7:3 vol.). It flowed counter-currently on the shell-side of the HFM. Aqueous flow rates were varied, 0.72 mL/h, 0.95 mL/h, 1.75 mL/h, with

$Q_{org} \approx Q_{aq}$. ^{241}Am was measured in both aqueous and organic samples by gamma counting. Lanthanides concentrations in aqueous samples were determined by ICP-MS. Organic samples were stripped into 0.01 M nitric acid before ICP-MS measurement.

The test was later repeated in a HFM consisting of 100 fibres. Again, aqueous rates were varied, 0.70 mL/h and 0.92 mL/h, with $Q_{org} \approx Q_{aq}$.

TABLE III. Composition of the feed solution for the SANEX-III tests. Concentrations in mg/L unless otherwise stated.

| Element | Concentration |
|-------------------|------------------|
| HNO_3 | 1 M |
| Y | 87 |
| La | 298 |
| Ce | 559 |
| Pr | 276 |
| Nd | 1005 |
| Sm | 191 |
| Eu | 38 |
| Gd | 48 |
| ^{241}Am | 0.5 or 3.7 MBq/L |

Results and Discussion

SANEX-III Test in the Large HFM Setup

The test in the large HFM setup in fact was the first ever SANEX test performed in a HFM [8]. With an aqueous flow rate of 0.44 L/h, only 94 % of americium was removed from the aqueous phase. Increasing aqueous flow rate decreases americium extraction efficiency. Lanthanide co-extraction as determined from organic effluent samples was in the range of 1-2 % for Sm, Eu, Gd, and well below 0.5 % for the lighter lanthanides.

SANEX-III Tests in the Miniature-HFM Setup

The first experiment [9] was performed in a HFM with 105 fibres. During the complete duration of the test, both effluent phases were clear and without any entrainment. Americium mass balance was $(100 \pm 5) \%$. Mass balances for the lanthanides were 96-102 %, with the exception of europium (91-94 %).

Relative aqueous effluent concentrations over flow rate are shown in Fig. 9. With an aqueous flow rate of 0.72 mL/h, 99.95 % of Am(III) was removed from the aqueous phase, corresponding to an americium decontamination factor of 2000.

This is a significant improvement over the first SANEX-III HFM test performed in the large HFM setup (too high flow rates were used then).

Lanthanides almost completely remained in the aqueous phase. The relative amount of americium and lanthanides extracted into the organic phase is shown in Fig. 10. The heavier lanthanides are better extracted, with yttrium between neodymium and samarium. At an aqueous flow rate of 0.72 mL/h, a total of 21.5 mg/L of lanthanides was found in the organic phase. This means that, with the respective exact aqueous-to-organic flow ratio, that 22.6 mg/L were extracted from the feed phase. With a total feed concentration of 2.5 g/L lanthanides, the average lanthanide decontamination factor is 110. This is a very good value, considering it was achieved *without* lanthanide scrubbing. Mass transfer calculations are shown as lines in Fig. 9. Agreement with the experiments is good.

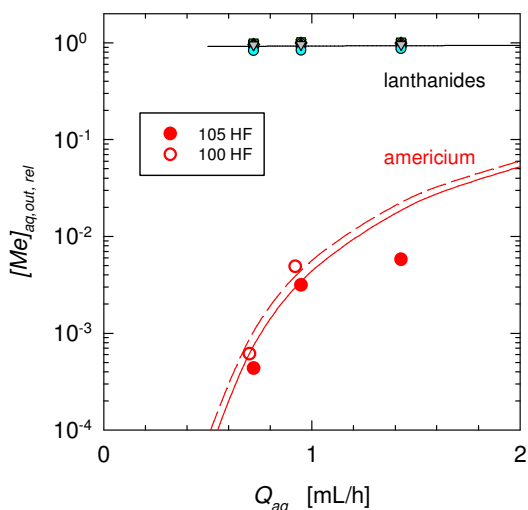


Fig. 9. 1st and 2nd SANEX-III miniature-HFM tests, relative aqueous effluent concentrations over flow rate. Symbols, experiments. Lines, calculated. Lanthanides shown for 1st test only.

The second test, performed in a HFM with 100 fibres, basically reproduced the results from the first test. As with the first test, effluents were clear and without entrainment. Mass balances were 100-110 %. The somewhat lower americium extraction efficiency can be attributed to the number of hollow fibres in the modules used, as suggested by the calculations. Experimental data for the lanthanides are omitted for clarity (they are practically identical to those from the first experiment).

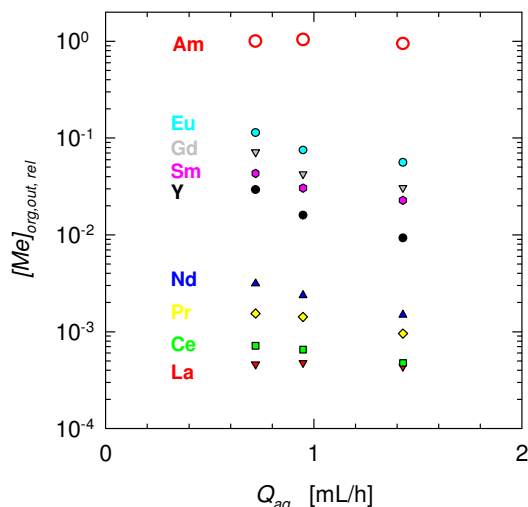


Fig. 10. 1st SANEX-III miniature-HFM test, relative organic effluent concentrations over aqueous flow rate.

SANEX-IV TESTS

Six single-module HFM tests were performed with a synergistic mixture of 0.5 M (CIPh)₂PSSH (Fig. 11) + 0.25 M TOPO (tri-*n*-octylphosphine oxide) in *tert*-butylbenzene as extractant [10]. Feed solution was a synthetic DIAMEX product, containing lanthanides at a total concentration of 6.5 g/L and ²⁴¹Am in trace amount in 0.5 M nitric acid (composition analogous to that given in Table III, however approx. 2.5-fold lanthanide concentrations, 0.5 M HNO₃ instead of 1 M).

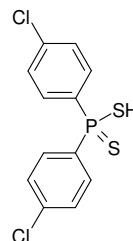


Fig. 11. Di(chlorophenyl)dithiophosphinic acid, (CIPh)₂PSSH.

Two extraction tests, one scrubbing test and two stripping tests were made in the large HFM setup (more details are given in [4]). Additionally, an extraction test was made in a miniature HFM.

SANEX-IV Tests in the Large HFM Setup

Two SANEX-IV extraction experiments were performed in the large HFM setup. The collected loaded organic phase from the first extraction

test was stripped into 1.5 M nitric acid. The collected loaded organic phase from the second extraction test was scrubbed with 1.0 M nitric acid. Then, this scrubbed organic phase was stripped into 2.0 M nitric acid.

Extraction Experiment 1

In the first extraction test, the aqueous phase was flowing in the shell side of the HFM. Equal aqueous and organic flow rates, $Q_{aq} = Q_{org}$, were varied: 400 mL/h, 800 mL/h, 1500 mL/h. Mass balances were 91-95 % (americium) and 96-103 % (lanthanides). With a flow rate of 400 mL/h, 97.5 % of americium was extracted, with approx. 30 % of the lanthanide inventory being co-extracted. Increasing flow rate decreased extraction efficiency, due to the reduced residence time of the phases in the HFM.

Extraction Experiment 2

In the second extraction test, the aqueous phase was flowing inside the hollow fibres, other than in the first extraction test. Aqueous flow rate was varied, $Q_{aq} = 300$ mL/h, 490 mL/h, 800 mL/h. Organic flow rate was kept constant at $Q_{org} = 300$ mL/h. Mass balances were 100-103 % (americium) and (100±5) % (lanthanides). The change in operating mode drastically improved the extraction efficiency: 99.99 % of americium was removed at $Q_{aq} = 300$ mL/h. Again, approx. 30 % of the lanthanides were co-extracted. Flow rate dependency of extraction efficiency is more pronounced than in the first extraction experiment. A detailed explanation for this behaviour is given in [4].

Scrubbing Experiment

An experiment on lanthanide scrubbing was performed, contacting the loaded organic phase from the second extraction test with 1.0 M nitric acid. Organic phase flowed shell-side. Aqueous and organic flow rates were equal, $Q_{aq} = Q_{org}$. They were varied, 300 mL/h, 490 mL/h, 820 mL/h. Mass balances were (100±1) % for americium, (100±5) % for yttrium, and 89-94 % for the lanthanides. Approximately 70 % of the lanthanides could be scrubbed from the organic phase at 300 mL/h. 45 % of americium was also scrubbed from the organic phase.

The calculations suggest that flowing the organic phase inside the hollow fibres would have re-

sulted in a much better lanthanide scrubbing efficiency, without affecting americium scrubbing.

Stripping Experiments

Two HFM stripping tests were performed. In the first stripping experiment, the loaded organic phase from the first extraction experiment was contacted with 1.5 M nitric acid. In the second stripping test, the organic effluent from the scrubbing experiment was stripped into 2.0 M nitric acid. In any case, organic phase flowed in the lumen of the hollow fibres. Aqueous and organic flow rates were equal. They were varied between 300 mL/h and 1500 mL/h.

Mass balances were 97-108 % for americium and 100-114 % for the lanthanides. In the first test, 80 % of americium and approx. 93 % of the lanthanides (except yttrium, 88 %) could be stripped from the organic phase at 300 mL/h. In the second test, at 320 mL/h, 88 % of americium and 93-95 % of the lanthanide inventory could be stripped from the organic phase. Regarding americium stripping, this is a slight improvement over the first stripping test, due to the increase in nitric acid concentration. Lanthanide stripping performance is not further improved since lanthanides distribute almost completely to the aqueous phase even at 1.5 M nitric acid.

To achieve more complete stripping, significantly lower flow rates would be required. The calculations indicate that to achieve e.g. 99 % stripping efficiency, stripping must be performed at an organic phase flow rate of 100 mL/h.

SANEX-IV Test in a Miniature HFM

A single-module SANEX-IV test was performed in a miniature HFM consisting of 100 fibres [11]. Aqueous phase was led inside the fibres. Aqueous flow rate was varied between 2.9 mL/h and 6.8 mL/h. Organic flow rate was 3 mL/h.

Both aqueous and organic effluents were clear throughout the test. In this test, too, americium extraction efficiency is strongly dependent on flow rate, cf. Fig. 12. At an aqueous flow rate of 2.9 mL/h, 99.996 % of americium could be removed from the organic phase, corresponding to an americium decontamination factor of 25000. A total of 4875 mg/L lanthanides was left in the raffinate at an aqueous flow rate of 2.9 mL/h. With the total lanthanides concentration being 6500 mg/L in the feed, this corresponds to a lan-

thanide decontamination factor of 4.0, without lanthanide scrubbing. The calculations are in good agreement with experimental results, cf. lines in Fig. 12.

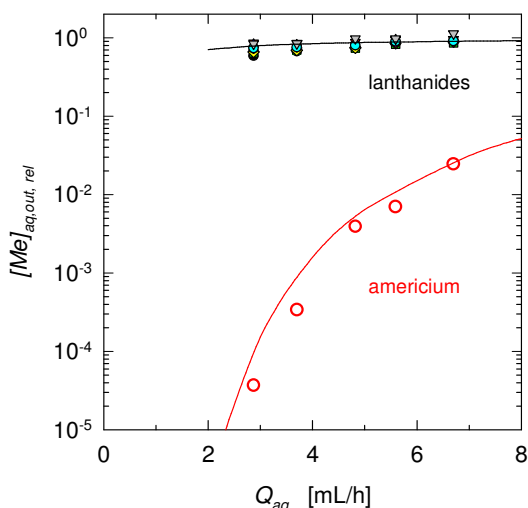


Fig. 12. SANEX-IV extraction test in a miniature HFM (100 HF), relative aqueous effluent concentrations over aqueous flow rate. Symbols, experiments. Lines, calculated.

CONCLUSIONS

HFM were tested with extractants relevant for minor actinide partitioning. It could be demonstrated that by selecting appropriate flow rates, very high decontamination factors are achievable. No entrainment which could reduce stage efficiency is detectable in the effluent phases. Due to the known interfacial area and the relatively simple hydrodynamics, mass transfer calculations yield reliable predictive results.

The development and fabrication of miniature HFM can be considered as a major improvement with respect to applicability in the laboratory and chemicals consumption.

OUTLOOK

This work is continued in the 6th framework programme project, EUROPART. Several miniature HFM are coupled in a HFM micro-plant to perform complete continuous separation processes with coupled extraction, scrubbing and stripping sections. A second version of such a HFM micro-plant is intended for hot cell use with real highly active feed solutions.

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