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Oxidation and extraction of Am(VI) using a monoamidic extractant in 3D printed centrifugal contactors

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Abstract

A series of tests were performed using centrifugal contactors to evaluate the separation of Am(VI) from cerium, as well as to determine the efficiency of the contactors. All experiments were performed using 2-cm, acrylic 3D-printed centrifugal contactors. Solvent extraction tests were performed using 1 M N,N-di(2-ethylhexyl)butyramide (DEHBA)/dodecane and a nitric acid feed solution spiked with ^{243}Am and ^{139}Ce and oxidized with 60 mg mL^{-1} sodium bismuthate. Approximately 72 % of the Am was extracted with a single contactor stage. Co-stripping of Am and Ce was demonstrated but attempts at selective stripping were not successful. Successful recycle of the used organic phase was demonstrated. Contactor efficiencies of 95 % to 100 % were obtained.

Keywords

Americium (VI), Centrifugal contactors, Minor actinides, Sodium bismuthate, Solvent extraction, 3D printing

Introduction

Advanced used nuclear fuel recycle research internationally includes the separation of minor actinides, in addition to the historical separation of uranium and plutonium. Minor actinide separation is primarily focused on the separation of americium from the lanthanides. This allows for the recycle of the americium along with the uranium, plutonium, and neptunium as fast reactor fuel or for the preparation and transmutation of americium targets. Partitioning and transmutation of the americium is desirable to minimize the long-term heat load of material interred in a future high-level waste repository. Separation of curium from americium is also desirable to reduce heat generation during minor actinide fuel fabrication [1].

Separation of the minor actinides from the lanthanides is challenging given there are only subtle differences between the ions of the trivalent actinide and lanthanide series. Of a particular challenge is the development of a partitioning scheme amenable to process scale-up. One approach to this problem is to oxidize Am(III) to Am(VI) and extract the americium from the lanthanides [2,3,4]. Of several oxidants that have been investigated, only sodium bismuthate has been used successfully for solvent extraction experiments [5]. Several ligands have been evaluated for Am(VI) extraction including conventional tributylphosphate (TBP) [6], its more basic analogues dibutylbutylphosphonate (DBBP) [5] and diamylamylphosphonate (DAAP) [7]. Prior development efforts have resulted in the demonstration of the extraction of Am(VI) in engineering-scale centrifugal contactors using DAAP [8].

This work focuses on the use of N,N-di(2-ethylhexyl)butyramide (DEHBA) as an extractant with the goal of improved Am(VI) separation. Monoamides such as DEHBA have been proposed as replacement compounds for TBP in some fuel cycle proposals because they are incinerable for waste disposal purposes and their higher radiolytic stability [9]. Preliminary batch contact work showed that Am distribution ratios using this extractant are about 20 % higher than for the DAAP used in previous tests, and that the reduction of Am(VI) was also somewhat slower than for DAAP [9]. Therefore DEHBA was chosen for the contactor test reported here. Additionally, smaller-scale, 3D printed acrylic 2-cm centrifugal contactors were used and evaluated for testing.

Theory

Americium is present in dissolved used nuclear fuel as Am(III). As Am(III), it is not extractable with conventional extractants utilized for the separation of uranium and plutonium from used nuclear fuel. Rather, the Am(III) will partition with Ln(III) and Cm(III) due to the chemical similarities caused by their like valence states and similar ionic radii, resulting in similar charge densities. One potential method to extract Am is to oxidize the Am(III) to Am(VI). Since most lanthanides and Cm are not oxidized, the hexavalent Am can then be separated in analogy with U(VI) and/or Pu(IV)/Pu(VI) using the same extractants developed for U/Pu recovery [10]. The standard potential for the Am(III)/Am(VI) redox couple under acidic conditions is about 1.7 V, and thus strong oxidizing agents are required [1,11]. Several oxidants have been explored including peroxydisulfate [12,13], copper(III) periodate [14,15], electrochemical techniques [16], and sodium bismuthate [5-8,17]. Once oxidized the Am(VI) exists as the americyl AmO_2^{2+} ion and behaves similarly to the other hexavalent actinyl cations. However, Am(VI) is easily reduced, even by its own radiolysis products, to Am(V) [18]. Additionally, the presence of reducing agents, including the organic extractant, will facilitate more rapid reduction to a mixture of Am(V) and Am(III). Thus, short solvent extraction contact times are necessary to effect the Am(VI) separation to mitigate its reduction and back-extraction to the aqueous phase.

Ce (III) is the only lanthanide that is also oxidized, to Ce(IV), which is also extracted in analogy with Pu(IV). The standard potential for the Ce(III)/Ce(IV) redox couple in HNO_3 is about 1.6 V [18]. This poses two issues: 1) the Ce(IV) requires selective reduction and stripping of the loaded solvent if a separation of Am from Ce is desired, and 2) the Ce(IV) is very corrosive to many metals, including stainless steel, which requires the use of alternative equipment materials of construction not only for longevity of the equipment but also to prevent corrosion products from forming and acting as a reductant to the Am(VI).

Experimental

All chemicals were reagent grade or higher. Radiotracers (^{243}Am, ^{139}Ce) and depleted uranium were obtained from laboratory stocks. Organic solutions consisted of 1 M diamylamylphosphonate (DAAP) (Eichrome, Darien, IL, USA) or 1 M DEHBA (Technocomm, UK) in dodecane (Aldrich Chemical, St. Louis, MO, USA).

Oxidation of the feed solutions was performed by subjecting the radiotracer spiked feed simulant to a 2 h oxidation with 60 mg/mL sodium bismuthate in a stirred, 1-L, glass beaker which served as an oxidation and feed tank. This feed was then pumped through a 1 mm pore size ZenPure PureFlow Capsule PTFE Teflon filter contained in a polypropylene filter housing into the extraction contactor. For the Am(VI) separation testing, gravity settling was utilized instead of filtration as described below. Two sets of testing were performed: efficiency testing of the centrifugal contactors and oxidation/extract/stripping testing of Am(VI) from a nitric acid feed solution using 1M DEHBA.

Efficiency testing

Testing was performed using 2-cm rotor diameter, acrylic, centrifugal contactors fabricated by Argonne National Laboratory (ANL) using 3D printer technology. The contactor housings and internal rotors were printed with acrylic material and 3600 rpm motors provided. The contactors were assembled and supplied as a four-pack as shown in Fig. 1. The four contactors can be used individually or integrated as a group of two to four stages.

Reduction of the Am(VI) in the presence of the acrylic materials used for the contactors was evaluated prior to fabrication of the contactors. Samples of four acrylic plastic materials used for contactor printing were received from ANL to be evaluated for their reducing reactivity toward oxidized Am. This was examined qualitatively by performing a sodium bismuthate Am oxidation in 6.5 M HNO_3, in the presence of a 100 ± 50 mg specimen of each material, and this aqueous phase was used in a batch extraction with 2 mL of bismuthate pre-treated 1 M DAAP/dodecane. The results are shown in Table 1. Also shown for comparison is the mean D_{Am} for duplicate contacts with 6.5 M HNO_3 containing no plastic specimen.

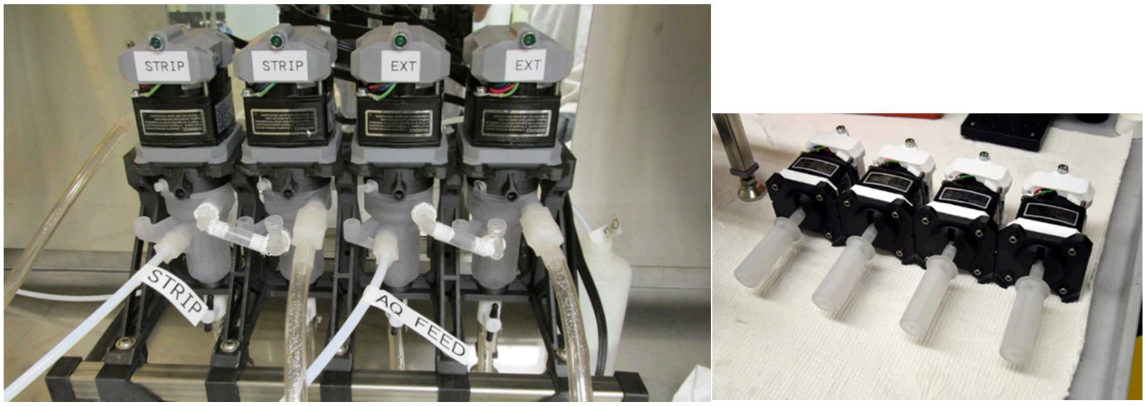

Fig. 1 2-cm 3D printed centrifugal contactors used for Am(VI) solvent extraction testing

Table 1 Effect of materials of construction on D_{Am} for batch contact measurements

Specimen type	mean D_{Am}
Grey plastic	3.35 ± 0.35
Clear plastic	3.17 ± 0.55
Black plastic	4.54 ± 0.24
Polyethylene terephthalate	2.93 ± 0.57
6.5 M HNO_3 only	5.16 ± 0.03

It can be seen in Table 1 that contact with the acrylics did decrease the extraction efficiency compared to specimen-free solution, probably via reduction of Am(VI). However, given the contact times of two hours and the surface areas in this qualitative test it was concluded that these materials would be adequate for short contacts during testing.

To measure contactor efficiency it was necessary to select a well-characterized extraction system that would eliminate any confounding variables such as incomplete oxidation or reduction during the contacts. However, it was also desirable to use a solvent system actually under consideration for Am(VI) oxidation and separation research. The organic solvent selected was 1 M DAAP/dodecane. Prior to the test, the solvent was pre-equilibrated for 1 h with 3 M HNO_3. The aqueous feed for the efficiency test was 3 M HNO_3, spiked to approximately 3 ppm with depleted U (3.75 uL of 200 mg mL^{-1} ^{238}U stock solution diluted to 250 mL). A single centrifugal contactor stage in a HEPA-filtered radiological fume hood was utilized for the efficiency test.

Aqueous and organic solutions were fed to the contactor stage and both phases were sampled while exiting the contactor at three different times into the contactor test (3, 4, and 5 minutes). These multiple samples were obtained to estimate the time necessary for equilibrium to be achieved. This entire process was completed at two different contactor total throughputs (aqueous flowrate + organic flowrate), 30 mL min^{-1} and 40 mL min^{-1}, maintaining an organic to aqueous phase ratio of 1. All tests were performed under ambient temperature conditions and hood temperature was recorded.

In addition to measuring the equilibrium contactor distribution ratios ($[U_{Org}]/[U_{Aq}]$), aliquots of the phases of these samples were vortex mixed under batch conditions using 1 minute contact times, to generate true equilibrium distribution ratios. The contactor efficiency was then calculated as the ratio of the contactor test D_U, versus the equilibrium batch contact D_U as shown in Eq.(1).

$$Efficiency\ (\%) = \frac{D}{Dequil}\ x\ 100 \qquad (1)$$

Uranium concentrations in both phases were measured using a Thermo X series 2 ICP-MS with a Teflon sample introduction system and platinum cones. During analysis of organic solutions, 0.2 L min^{-1} of 20 % oxygen in argon was added to the spray chamber to aid in combustion of the organic material and to reduce build up on the cones. Organic solutions were emulsified with Triton TX100 into the normal 1 % nitric acid solution used to dilute aqueous samples prior to analysis, and stirred for 20 sec. The same emulsification was applied to the aqueous samples for consistency.

Am(VI) separation and strip testing using centrifugal contactors

For the Am oxidation, extraction and stripping testing, the contactors were configured for one stage of extraction, followed by two stages of stripping as shown if Fig. 2. A 6.5 M HNO_3 aqueous feed containing 700 Bq/mL ^{243}Am + ^{139}Ce and a 1 M DEHBA/dodecane organic solution, recycled from prior flowsheet tests, were used. The organic feed was scrubbed with an equal volume of 6.5 M HNO_3 + 9 g L^{-1} sodium bismuthate for 1 hour to remove any trace reducing agents in the organic. Sodium bismuthate was also added to the aqueous feed (60 mg mL^{-1}) and mixed for 2 hours to oxidize the Am. After this oxidation, the mixer was shut off and the bismuthate solids allowed to settle for 10 minutes prior to sampling and starting the feed to the extraction stage.

Filtration was not used for this test due to issues with the filter media reducing the Am(VI). Prior testing with DAAP as an extractant utilized a 1 μm ZenPure PureFlow Capsule PTFE Teflon filter with no issues. For these tests, the same type of filter was initially utilized (different lot) and results were poor, showing reduction of the Am in the feed after flow through the filter. Tests were performed in which a filter from this lot was dismantled so that its materials of construction could be evaluated for their reducing tendency using batch contact oxidations and extractions. The results of single batch contacts indicated that the presence of 0.025 g of filter medium during a typical batch contact decreased the D_{Am} using 1 M DAAP/dodecane from its control value of 4.6 to 1.5, while 0.047 g of filter medium decreased the D_{Am} to 0.9. A similar batch contact using 0.2 g of polyethylene filter housing material resulted in $D_{\mathrm{Am}} = 3.5$. Thus, although the polyethylene definitely has a reducing tendency toward Am(VI), very small amounts of the filter medium reduce Am(VI).

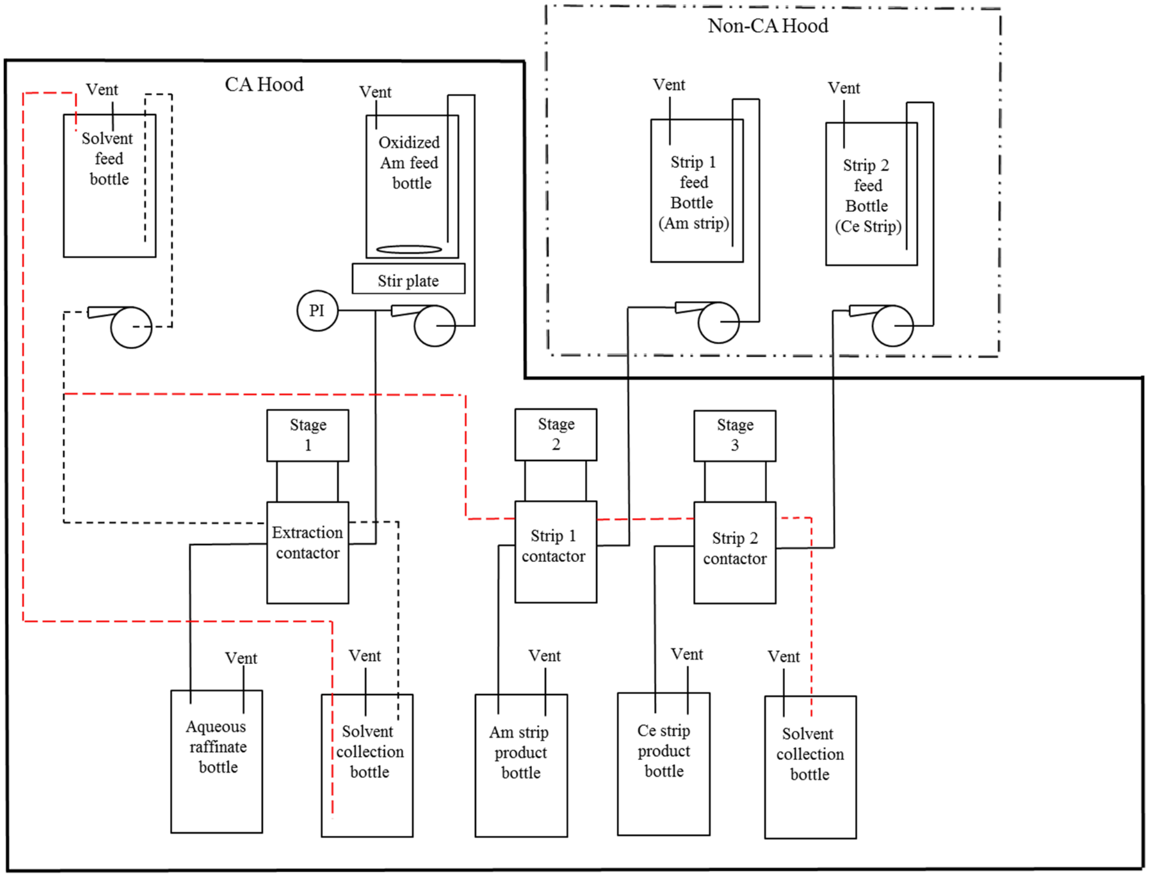

Fig. 2 Schematic representation of test bed for contactor testing

To move forward with flow sheet development in the absence of filters, a batch contact study was conducted to examine the effect of bismuthate settling time in lieu of filtration. When stirring of the suspension is stopped, the bismuthate solids settle, suggesting the possibility that a clean feed may be pumped to the contactors without filtration. Since the bismuthate acts as a holding oxidant, any carryover is not expected to adversly impact extraction results. Filtration or settling is necessary to prevent excessive buildup of the bismuthate solids in the centrifugal contactor equipment. Therefore, an Am-spiked solution of 6.5 M HNO_3 was bismuthate-oxidized for 2 h, and then the solid was allowed to settle for varying amounts of time prior to a 1 M DAAP/dodecane batch contacts. The D_{Am} decreased with increasing settling time, from 3.5 initially to about 2 after 10 minutes of settling and to about 1.5 after 20 minutes. A target of 10 minutes was chosen for testing as visible settling was complete and still achieving D_{Am} of 2.

The feed solution entered the extraction contactor at a flowrate of 20 mL min^{-1} where it contacted the organic phase at an O/A of 1. As the loaded organic phase exitied the contactor, it was collected in a feed vessel and held for 5 minutes prior to feed through the first strip contact where the organic was contacted with 6.5 M HNO_3 at an O/A of 1. As the organic exited the first strip stage it entered a second strip stage where it was contacted with 0.1M HNO_3, 0.5% H_2O_2 at a O/A of 1. Effluent streams were sampled each minute during operation. The activity of the radiotracers present in the aqueous and organic phases was determined using gamma spectroscopy.

The goal of the hold-up time for the loaded organic phase exiting the extraction contact was to enable Am reduction. Based upon prior research in which D_{Am} dropped below 1 after 2 minutes [10], a 5 minute holdup time was chosen to ensure complete Am reduction. Also, based upon prior stability testing where Ce remained extractable after 2 hours in the organic phase (1M DAAP/dodecane), it was expected that the Cm (IV) would not reduce during this time [10].

Results and discussion

Efficiency testing

The contactor efficiency test operation performed according to expectations with no hydrodynamic or mechanical issues impacting the testing. The distribution ratios for uranium based on the final samples taken (5 minutes for the 30 mL min^{-1} total throughput and 3.75 minutes for the 40 mL min^{-1} total throughput) are shown in Table 2. Additionally, the distribution coefficients resulting from re-equilibration of the aqueous and organic effluent samples using vortex mixing, which represents 100 % efficiency, are shown in Table 2.

The data collected in table 2 may be used to calculate the contactor efficiency. This was done by calculating the ratio of the distribution ratios measured during the contactor test, with the equilibrium, post-contactor batch contacts on the same samples. These results are shown in Table 3, resulting in a contactor efficiency of 95 % - 100 %. As expected, the efficiency is lower at the higher total flowrate due to the reduced contact time of the two phases. Note that the slightly lower distribution ratios found at the higher flow rate were measured at a higher ambient hood temperature. This temporary increase in hood ambient temperature associated with cycling of heaters in the laboratory likely will not have resulted in significant heating of the solution in the contactors.

Table 2 1 M DAAP/dodecane uranium distribution ratios and temperatures (^{o}C)

Sampling time	Temperature (C)	D_U
30 mL/min Experiment		
3 min-1	21.9	48.6
3 min-2	21.9	48.8
3 min average		48.7 ± 0.1
4 min-1	22.0	48.8
5 min-1	21.9	52.6
5 min-2	21.9	51.2
5 min-3	21.9	52.7
5 min average		52.2 ± 0.8
40 mL/min Experiment		
2.5 min-1	26.1	47.1
3 min-1	26.3	47.6
3 min-2	26.3	45.7
3 min average		46.7 ± 1.3
3.75 min-1	26.2	48.1
3.75 min-2	26.2	47.0
3.75 min-3	26.2	46.6
3.75min average		47.2 ± 0.8
Post-run Batch Equilibrium Contacts		
30 mL/min 5 min-1	21.4	50.5
30 mL/min 5 min-2	21.4	50.3
30 mL/min 5 min-3	21.4	51.9
average		50.9 ± 0.9
40 mL/min 3.75 min-1	21.4	48.6
40 mL/min 3.75 min-2	21.4	50.0
40 mL/min 3.75 min-3	21.4	50.9
Average		49.8 ± 1.2

Table 3 Contactor efficiency calculations

Flow rate	Ratio	Efficiency (%)
30 mL/min	52.2/50.9	103 ± 2.4
40 mL/min	47.2/49.8	94.8 ± 2.8

Am(VI) separation testing using centrifugal contactors

Following the oxidation of the aqueous feed, a total of 13 min settling time elapsed prior to the initiation of the testing. A batch contact using this prepared feed resulted in D_{Am} = 3.71 ± 0.10 and D_{Ce} = 9.34 ± 0.22. After initiation of the extraction contactor test, both effluent phases were sampled at 2, 4 and 5 min. The resulting distribution ratios for Am and Ce are shown in Fig. 3, where it can be seen that the distribution ratios have decreased slightly during the test. The mean values for the composite distribution ratios were 2.51 ± 0.21 (72 % extracted), and 4.69 ± 0.69 (82 % extracted) for D_{Am} and D_{Ce}, respectively. Although the settling time allowed for the delivery of an aqueous feed to the contactor without visible solids, there was evidence of bismuthate on the contactor rotor. Thus, filtration of the sodium bismuthate solids prior to the feed solution entering the centrifugal contactor equipment is preferred for long-term operation as compared to allowing the solids to settle. A non-reducing material of construction for the filtration would be required.

The first-stage strip consisted of 6.5 M HNO_3, conducted after a delay time of 5 min 40 sec. The hold-up time was intended to allow the loaded organic phase to reduce the Am(VI) to Am(V)/Am(III) thus allowing it to be stripped by the acidic aqueous strip solution. It was anticipated that Ce(IV) would be more stable and therefore not be stripped as Ce(III). Strip distribution ratios were calculated based on the measured activity of Am and Ce in the organic phase feed and the measured activity in the aqueous effluent, with the assumption of mass balance. These strip distribution ratios are also shown in Fig. 3. It can be seen that, contrary to expectations, both Am and Ce were stripped in the first strip stage. Thus, the Ce was unexpectedly reduced during the hold time. Future testing should reduce or possibly eliminate this hold time to better effect the desired selective strip.

A second strip stage consisting of 0.5 % H_2O_2 in 0.1 M HNO_3, designed to separately strip the remaining Ce, further stripped both metals, although the distribution ratios are unreliable due to the low amounts of activity that remained after the first strip. The composite distribution ratios for the second strip were 0.008 ± 0.003 and 0.005 ± 0.001 for D_{Am} and D_{Ce}, respectively. The behavior of nominally Am(VI) and Ce(IV) were nearly identical in this strip contact.

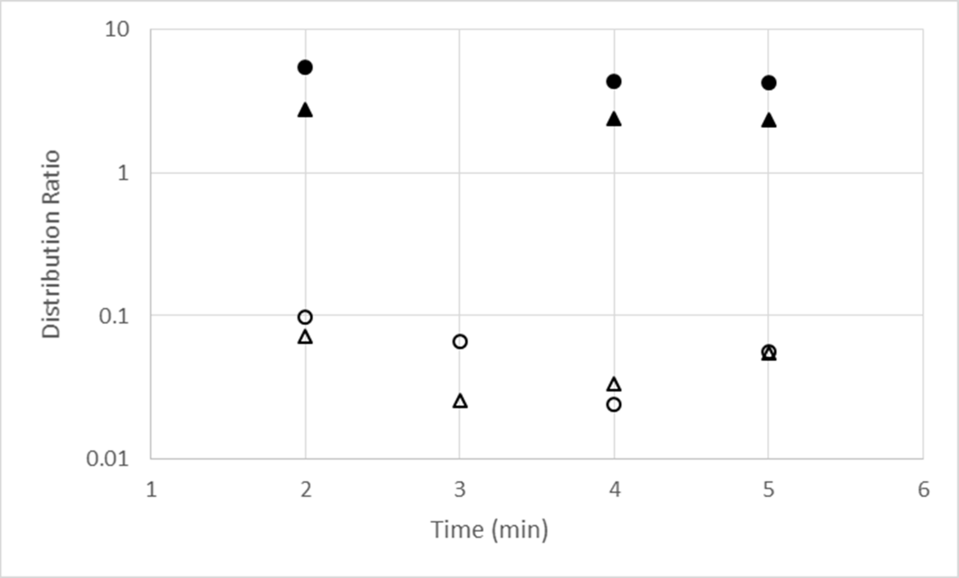

Fig. 3 Extraction distribution ratios (filled symbols) and strip distribution ratios (open symbols) for Ce (circle) and Am (triangle)

Conclusions

The contact efficiency of the 2-cm acrylic centrifugal contactors, fabricated by ANL using 3D printer technology, was measured using uranium extraction from 3 M HNO_3 by

1 M DAAP/dodecane. The efficiency was determined to be 95 % to 100 %, by comparison to 1-min batch contacts using the same solutions.

As expected, both Am and Ce were readily oxidized by sodium bismuthate. Their solvent extraction distribution ratios using DEHBA indicate that Am was oxidized predominantly to Am(VI), and that Ce was oxidized predominantly to Ce(IV). Overall extraction efficiency (72 % for Am and 82 % for Ce) was somewhat lower for both than previously reported using batch contacts. This is consistent with reduction of both species during somewhat longer organic –phase contact times, and contact with larger surface areas of potential reducing agents in the materials of construction of the solvent extraction system. Cerium (IV) was rapidly reduced by organic phase contact to an extent not anticipated. Thus design of a selective stripping separation will require additional work. Possibilities include elimination of the organic-phase hold-up time and/or substitution of an immediate Am(VI) strip using a dilute reducing agent that more rapidly reduces Am(VI) over Ce(IV).

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