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APPLICATION OF MINIATURE CENTRIFUGAL CONTACTORS FOR TREATING HIGH-LEVEL LIQUID WASTE

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ABSTRACT

The hydraulic performance of the H_2O–30% trialkyl phosphine oxide (TRPO)–kerosene system and the mass transfer properties of HNO_3 and Nd^{3+} were studied in a single-stage miniature centrifugal contactor with 10 mm diameter. A cascade cold test for partitioning Nd^{3+} from the simulated high-level liquid waste (HLLW) using the TRPO process was conducted on the miniature contactors. Based on results from the cold tests, a hot test for partitioning Am^{3+} from the genuine HLLW using the TRPO process was conducted on miniature contactors. The results showed that approximately 200 mL waste feed will allow the partitioning process with 24-stage miniature contactors of 10 mm diameter to reach steady state. The test results also showed that the miniature centrifugal contactors have good hydraulic performance and high stage efficiency. The cascade cold and hot test results agreed well with calculations from extraction models.

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INTRODUCTION

The final disposal of high-level liquid (radioactive) waste (HLLW) is a subject of great public concern. Partition and transmutation (P-T) is a candidate method for pretreatment of HLLW (1). Under the P-T process, the actinides, ^{99}Tc, and others are first removed from the HLLW, and then some actinides, such as Am and Cm, are transmuted. Others, such as U and Pu, can be recycled. Therefore, the partitioning can be implemented with subsequent transmutation to reduce the long-term risk of nuclear waste. The partitioning method can also be used to reduce α waste and high-level radioactive waste volumes.

Various extraction processes have been developed for partitioning actinides from HLLW solutions, such as the DIAMEX process in France (2), the DIDPA process in Japan (3), the TRUEX process in the USA (4), and the H_2O–30% trialkyl phosphine oxide–kerosene system process (TRPO) in China (5). These processes have been evaluated previously (6,7). The Chinese HLLW is stored in underground stainless steel tanks for future treatment. In recent years, the TRPO process has been studied for removing actinides from Chinese HLLW. Miniature centrifugal contactors were developed and used to evaluate the TRPO process.

Several researchers have developed and used miniature centrifugal contactors (8). For example, researchers at Argonne Laboratory developed 40-mm contactors for laboratory-scale testing. They also developed and used 20-mm contactors to test the TRUEX-SREX process (9). The miniature contactor proved to be a valuable tool in the development of solvent extraction processes, especially for HLLW. We have been developing miniature centrifugal contactors in our laboratory since the 1980s (10). A series of contactors has been developed from miniature to large, such as the 230-mm contactors for partitioning rare earth elements (11) and the 10- and 20-mm contactors for partitioning actinides from HLLW (12). In this paper we discuss the application of the 10-mm contactors to the TRPO process for partitioning americium from HLLW.

EXPERIMENTAL

Reagent and Apparatus

TRPO was obtained from the Jinan Phosphorous Factory, P.R. China. TRPO was diluted with saturated kerosene to 30% TRPO: kerosene (vol/vol) and scrubbed once by 1.0 mol/L HNO_3, by 5% Na_2CO_3 twice, and then by H_2O to reestablish neutralility before use. The other reagents were analytical reagents.

The main composition of the genuine Chinese HLLW is given in (13). The main composition of the simulated HLLW is given in Table 1. The genuine Chinese HLLW was the raffinate from the PUREX process of a reprocessing plant.

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Table 1. The Main Composition (g/L) of HLLW

Element	Concentration	Element	Concentration
Na	51.2	Zr	0.01
Al	16.5	Ru	0.62
Fe	17.4	Re	4.6
Ni	8.2	HNO_3 (mol/L)	2.6
Cr	2.0		
Sr	0.62		
Cs	1.76		

The miniature centrifugal extractors were developed and manufactured by our laboratory. The rotor diameter is 10 mm and its hold-up volume is approximately 4 mL in each stage.

Analytical Methods

The metallic ion concentrations were determined with a 180–80 Polarized Zeeman Absorption Spectra-Photometer (Hitachi, Japan) and an ICP-AES (Leeman 950, USA). The acid concentration in the aqueous sample was determined by caustic titration with oxalic acid used as a masking agent. The metallic ions and acidity in the organic solvent were stripped into the aqueous phase and then measured.

The carryover was determined with a water detector (CA-05 Karl-Fisher Water Detector, USA) for the aqueous phase in the organic phase and an oil detector (FF-1 Oil Detector, Xiaoshan Analytical Instrument Factor, China) for the organic phase in the aqueous phase.

^{241}Am was separated from the samples and determined by a Au-Si surface barrier detector (Fh445, $\phi = 20$ mm, Beijing Nuclear Instrument Factory, China).

PROCEDURES

Hydraulic Performance of Single-Stage Contactor

The diagram of the single-stage centrifugal contactor is shown in Fig. 1. The aqueous and organic phases were deionized water and 30% TRPO–kerosene (vol/vol), respectively. The hydraulic performance of the single-stage contactor was determined by the amount of phase carryover found from the outlets after the test reached steady state. The solubility of the aqueous phase in the organic phase and the solubility of the organic phase in the aqueous phase were measured through the following process: 1 mL water and 1 mL organic phase were magnet-

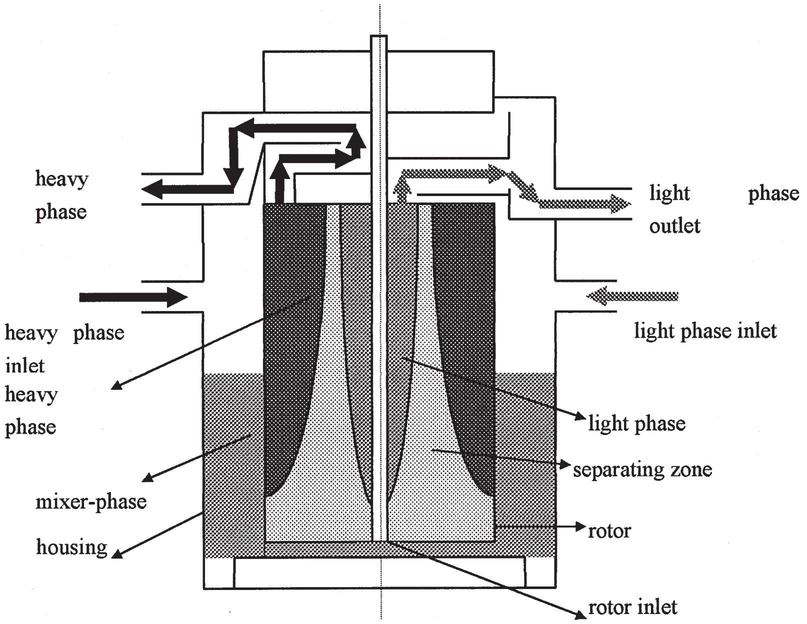

Figure 1. Schematic diagram of the single-stage contactor.

ically stirred for 30 minutes in a test tube, and then the 2 phases were separated by a supercentrifuge and were analyzed.

Mass Transfer Properties of HNO_3 and Nd^{3+}

In these tests, Nd^{3+} was used to simulate Am^{3+} because the extraction properties of Nd^{3+} in the TRPO process are very similar to those of Am^{3+}. The extraction efficiencies of HNO_3 and Nd^{3+} were studied in a single-stage 10-mm centrifugal contractor with the aqueous phase consisting of HNO_3 and Nd^{3+} and the organic phase consisting of 30% TRPO–kerosene at different conditions. The elements in the 2 outlet phases were determined when the extraction process reached steady state. The extraction stage efficiency was described as follows (14):

$$E_A = \left(\frac{X_i - X_o}{X_i - X_{eq}}\right) \times 100$$

for the aqueous stage and

$$E_o = \left(\frac{Y_o - Y_i}{Y_{eq} - Y_i}\right) \times 100$$

for the organic phase. X_i and X_o are the aqueous inlet and outlet concentrations of element i, respectively. Y_i and Y_o are the organic inlet and outlet concentrations of element i, respectively. X_{eq} and Y_{eq} are the aqueous and organic equilibrium concentrations of element i, respectively.

Extraction Distribution Ratio Model and The Simulation

The extraction-distribution ratio models of Am^{3+}, Nd^{3+}, and H^+ were developed using the experimental homogeneous-design method as presented in (15) and (16).

$$\begin{aligned} D(\mathrm{Am}^{3+} \text{ or } \mathrm{Nd}^{3+}) = {} & 19.936 - 14.284[\mathrm{H}^+] - 0.471[\mathrm{Fe}^{3+}] - 2.057[\mathrm{Nd}^{3+}] \\ & + 3.195[\mathrm{H}^+]^2 + 0.617[\mathrm{H}^+][\mathrm{Nd}^{3+}] + 0.0283[\mathrm{Fe}^{3+}]^2 \\ & + 0.117[\mathrm{Nd}^{3+}] - 0.211[\mathrm{Al}^{3+}] - 0.024[\mathrm{Na}^+] \end{aligned}$$

$$\begin{aligned} D(\mathrm{H}^+) = {} & 0.924 - 0.575[\mathrm{H}^+] + 0.053[\mathrm{Fe}^{3+}] \\ & - 0.0804[\mathrm{Nd}^{3+}] + 0.161[\mathrm{H}^+]^2 - 0.0414[\mathrm{H}^+][\mathrm{Fe}^{3+}] \\ & + 0.0452[\mathrm{H}^+][\mathrm{Nd}^{3+}] + 0.0028[\mathrm{Al}^{3+}] + 0.000239[\mathrm{Na}^+] \end{aligned}$$

H^+ was measured in mol/L for concentrations between 0.5 and 2.5 mol/L, while the other components were measured by g/L. Their concentrations were 0 to 25 g/L for Na^+, 0 to 8 g/L for Al^{3+}, 0 to 8.6 g/L for Fe^{3+}, and 0 to 5 g/L for Nd^{3+} (Am^{3+}).

A set of equations can be obtained using the material balance for each stage. If the extraction stage number N_1, the scrubbing stage number N_2, and the entrance conditions are known, the concentration profile of each component can be determined by solving the equations using the extraction-distribution ratio models. The decontamination factor (D.F.) of Am was calculated by

$$\mathrm{D.F.} = \frac{\text{Am Content in the Feed}}{\text{Am Content in the Raffinate}}$$

The analysis was used to determine the best conditions for satisfying the D.F value for Am.

Cold and Hot Tests of the TRPO Process in Miniature Centrifugal Contactors

The cold test for partitioning Nd^{3+}, used to simulate Am $^{3+}$, from simulated HLLW through the TRPO process was studied with a 24-stage contactor set. The hot test of the TRPO process for partitioning actinides from HLLW was completed in a hot cell. The flow sheet for the cold and hot tests is presented in Fig. 2.

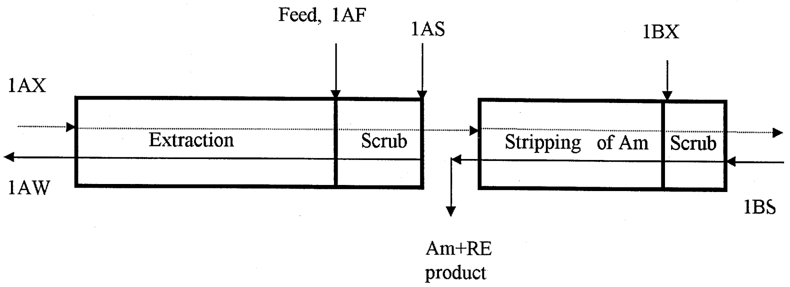

Figure 2. The TRPO process flow sheet.

The process was characterized by 12 extraction stages, 4 scrubbing stages in the extraction, 6 stripping stages, and 2 scrubbing stages for Am back-extraction. The TRPO process parameters are presented in Tables 2 and 3. The parameters selected for the computer simulation were based on the extraction models.

The miniature, centrifugal, extractor set was first filled with scrubbing solution, then the process solutions were added to the set with a gear pump for the 30% TRPO–kerosene solution and peristaltic pumps for the stripping and scrubbing solutions. After the organic solvent had flowed out of the set, the feed solution was pumped into the centrifugal contactor set, and the test was initiated. The cold test reached steady state when acidity concentrations at the outlets no longer changed. The equilibration time for the hot test was estimated from the results of the cold test. All pumps and centrifugal extractors were stopped after the test reached equilibrium. The 2 phases in each stage were put into centrifugal test tubes for phase separation by centrifugation and were then analyzed.

Table 2. The TRPO Process Stage Numbers

	Extraction Section		Am Stripping	
	Extraction	Scrub	Stripping	Scrub
Stage	12	4	6	2

Table 3. The TRPO Process Parameters

	Compositions		Flow (mL/h)	
Process	Cold Test	Hot Test	Cold Test	Hot Test
1AF	3.0 times diluted simulated HLLW, HNO_3: 1.08 mol/L	2.7 times diluted HLLW, HNO_3: 1.0 mol/L	80	77
1AX	30% TRPO–Kerosene	30% TRPO–Kerosene	40	40
1AS	1.2 mol/L HNO_3	1.2 mol/L HNO_3	8	8
1BX	5.5 mol/L HNO_3	5.5 mol/L HNO_3	32	32
1BS	0.1 mol/L HNO_3	0.1 mol/L HNO_3	8	8

RESULTS AND DISCUSSION

Hydraulic Performance

The hydraulic performances of the single, miniature, centrifugal contactor are presented in Figs. 3, 4, and 5. The results showed that the carryover of the 2 phases was very low, approaching the solubility of the 2 phases when the rotation rate was changed from 3700 to 5300 rpm, the aqueous-organic (A-O) phase ratio was 1:1, and the throughput was 300 mL/h (Fig. 3). The results were the same for the following conditions: The throughput (2 phases) was changed from 100 to 800 ml/h; the phase ratio was 1:1; and the rotation rate was maintained at 4500 rpm (Fig. 4). The carryover results when the phase ratio was changed from 1:10 to 10:1, the throughput was 300 mL/h, and rotation rate was 4500 rpm (Fig. 5) were similar to those of Figs. 3 and 4. However, the data in Fig. 3 also show that the organic phase carryover in the aqueous phase increased and the aqueous phase carryover in the organic phase did not change when the rotation rate was more than 5300 rpm. The aqueous phase carryover in the organic phase increased and the organic phase carryover in the aqueous phase did not change when the rotation rate was less than 3700 rpm. The centrifugal force in the heavy phase (aqueous phase) is stronger than that of the light phase (organic phase) in the rotor. The heavy phase settles more easily than does the light phase. Therefore, at lower rotation rates, an interface exists between the heavy phase and the mixed phase, but no interface exists between the light phase and the mixed phase. At high rotation rates no interface existed between the heavy phase and the mixed phase, so the 2 phases were mixed together. At low rotation rates, lack of interface between the light phase and the mixed phase means that the aqueous phase carryover in the organic

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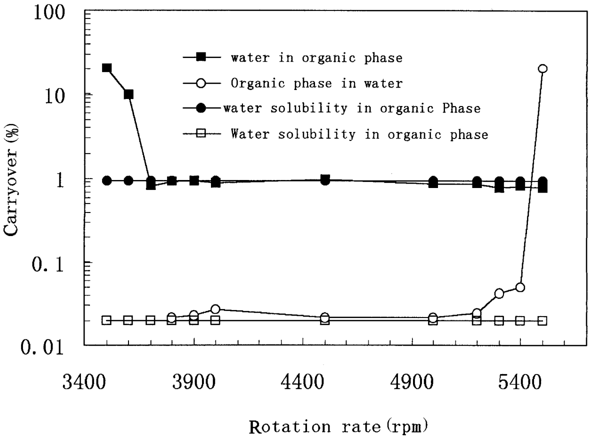

Figure 3. The dependence of carryover on rotation rate (2 phases): 300 mL/h; A-O: 1:1.

phase was increased. The data in Fig. 4 show that the carryover of water into the organic phase was more than 10% when the throughput was more than 800 mL/h. However, the carryover of organic phase into water did not increase significantly. The serious carryover conditions indicate incorrect operation of the centrifugal contactor. Carryover of the 2 phases did not occur when the phase ratio was changed from 1:10 to 10:1 at a rotation rate of 4500 rpm and throughput of 300 mL/h (Fig. 5).

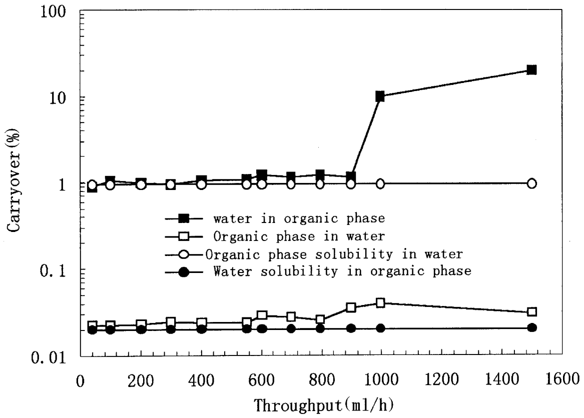

Figure 4. The dependence of carryover on throughput. Rotation rate: 4500 rpm; A-O: 1:1.

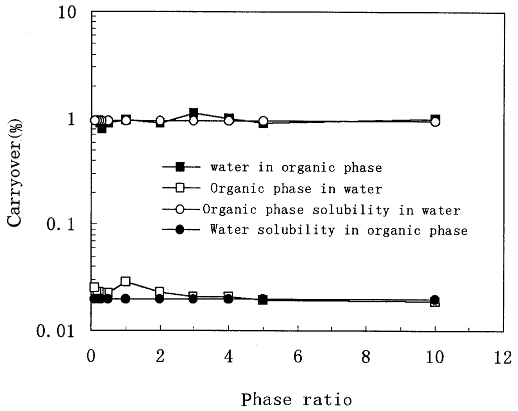

Figure 5. The dependence of carryover on phase ratio. Rotation rate: 4500 rpm; throughput: 300 mL/h.

Mass Transfer Performance

Figures 6, 7, and 8 show the results for the extraction efficiency for Nd^{3+} and H^+ at different conditions. The extraction efficiencies of Nd^{3+} and H^+ were up to 90% when the rotation rate was between 3700 and 5300 rpm, the phase ratio A-O was 1:1, and the throughput was 300 mL/h (Fig. 6). The results were the same when the throughput (2 phases) was changed from 100 to 550 mL/h, the phase ratio was 1:1, and the rotation rate was 4500 rpm (Fig. 7). The results were also the same when the phase ratio was changed from 1:10 to 10:1, the throughput was 300 mL/h, and rotation rate was at 4500 rpm (Fig. 8).

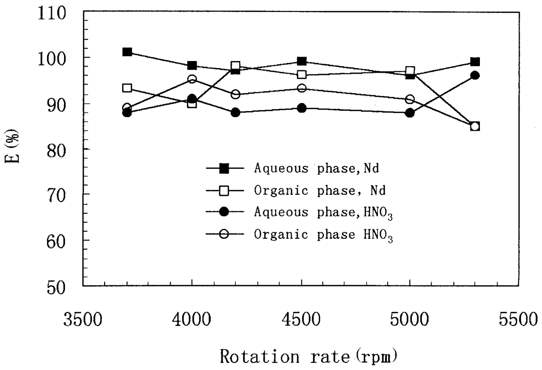

Figure 6. The dependence of efficiency (E) on rotation rate. Phase ratio: 1:1; throughput: 300 mL/h.

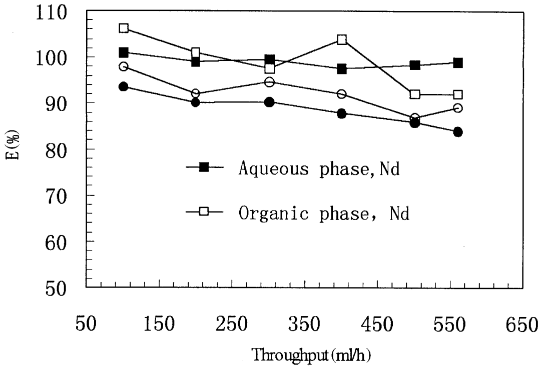

Figure 7. The dependence of efficiency (E) on the throughput. Rotation rate: 4000 rpm; phase ratio: 1:1.

Increasing the rotation rate increased the mixing of the 2 phases, which increases the extraction efficiency. However, increasing the rotation rate results in the reduction of the hold-up volume and shortening of the hold-up time, which results in decreased extraction efficiency. Figure 6 indicates that the extraction efficiency changed with rotation rate.

Increasing of the throughput also shortens the hold-up time, which reduces the extraction efficiency. The data in Fig. 7 show the results.

Changing the phase ratio affects the physical character of the extraction system, which changes the extraction efficiency. The data in Fig. 8 show the small changes of the extraction efficiency that occurred with changes of the phase ratio.

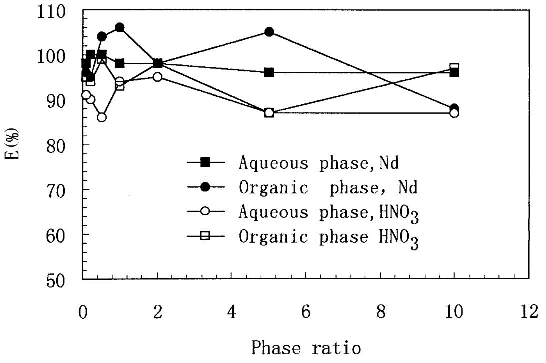

Figure 8. The dependence of efficiency (E) on phase ratio. Rotation rate: 4000 rpm; throughput: 300 mL/h.

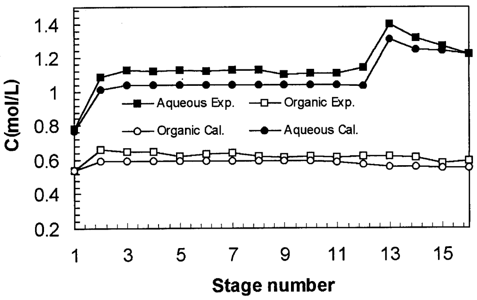

Figure 9. The HNO_3 profile in the cold test. For the model, the H^+ extraction efficiency was assumed to be 100% in the extraction section.

Cold and Hot Tests

The TRPO process consisted of 12 extraction stages, 4 scrubbing stages during the extractions, and 6 stripping stages with 2 scrubbing stages. The flow rate of the 2 phases was 125 mL/h during the extractions and 80 mL/h in the stripping section. The residence time for the 16 extraction stages (total hold-up volume was approximately 80 mL) was about 40 minutes at the flow rate of 125 mL/h. Residence time for the 8 stripping stages (total hold-up volume was approximately 40 mL) was approximately 20 minutes at a flow rate of 80 mL/h. The test reached steady state after 3 to 4 residence times (120 minutes). Acidity of the outlets in the TRPO process did not change after the test was run for 120 minutes. The test was run for 180 minutes to ensure that all experimental sections completely reached steady state.

The experimental results from the cold test are compared with the calculated results from extraction models for the TRPO process in Figs. 9 and 10. The pro-

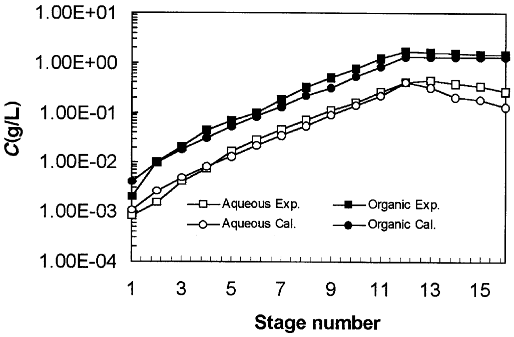

Figure 10. The profile of Nd^{3+} in cold test. For the calculated model, the Nd^{3+} extraction efficiency was assumed to be 90% in the extraction section.

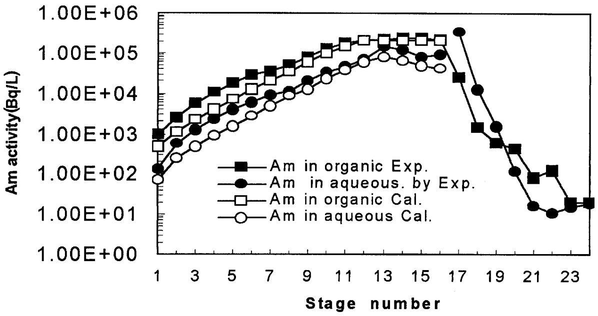

Figure 11. The Am^{3+} profiles in the hot test. For the model, the Am^{3+} extraction efficiency was assumed to be 85% in the extraction section.

files were calculated according to the experimental descriptions, and the stage efficiencies were taken into account in the calculation process. The HNO_3 profiles were calculated based on 100% stage efficiencies. The Nd^{3+} profiles were calculated based on 90% stage efficiencies. The calculations agree well with the experimental results.

These results show that the model can predict the experimental results, that the miniature centrifugal contactor worked well, and that the extraction efficiency in it was high.

The hot test was run for 180 minutes in accordancewith the results of the cold test. The Am^{3+} profiles in the hot test and the calculated Am^{3+} profile based on 85% stage efficiency are shown in Fig. 11. The Am^{3+} extraction efficiency was lower than that of Nd^{3+} in the cold test because of differences between the simulated and genuine HLLW. Another reason for the differences relates to the model setup under the simulated HLLW because it may be modified under the genuine HLLW. The D.F. for Am^{3+} was 666, which is enough for the HLLW to be recategorized into non-α waste (17). The calculated Am^{3+} profile in the stripping process cannot be calculated by extraction models because the extraction models are not suitable for acidity higher than 2.5 mol/L. The results for the other radioactive nuclides were discussed by Wang and Song (18).

CONCLUSION

A 10-mm diameter centrifugal contactor was developed and used at the Institute of Nuclear Energy Technology, Tsinghua University, P.R. China for a laboratory-scale test of HLLW treatment. The hydraulic performance and the mass transfer properties of HNO_3 and Nd^{3+} in the miniature centrifugal contactor were measured in the tests. The results of the cascade cold test for partitioning Nd^{3+} from simulated HLLW by the TRPO process showed that only about 200 mL of simulated feed was needed to reach steady state for a 24-stage miniature contactor set. The test results of the hot test for partitioning Am^{3+} from genuine HLLW using the TRPO process in the miniature contactors agreed well with the calculated results that relied on extraction models with 85% stage efficiency. These results also show that the contactor is a valuable tool for verifying effectiveness of the extraction process, which is difficult to evaluate in large-scale tests.

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