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Application of 70-mm-dia Centrifugal Contactors in Pilot Plant Tests of Two Simplified TRPO Processes with Simulated High Level Waste

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Abstract: High level waste (HLW) produced from reprocessing of the spent nuclear fuel still contains actinides, ^{90}Sr, ^{137}Cs, etc., which presents a significant hazard to the environment. Two simplified TRPO processes for the partitioning of HLW were developed with N, N′-dimethyl-3-oxa-glutaramic acid (DMOGA) as the stripping agent at the Institute of Nuclear and New Energy Technology (INET), Tsinghua University, China. The cold pilot plant tests of two simplified TRPO processes were carried out with the simulated HLW respectively, where 70-mm-dia centrifugal contactors were used in the stripping sections of two simplified TRPO processes for the removal of actinides in the loaded 30% TRPO-kerosene. The test results showed that 99.9% of the Nd and 99.9% of the Zr were stripped respectively, and the organic phase was recycled after being stripped and scrubbed. During the pilot plant tests, all of the 70-mm-dia centrifugal contactors were stable, working continuously with no stage failure or interruption in operation. All of the results demonstrate that these centrifugal contactors are promising for the stripping sections of two simplified TRPO processes.

Keywords: Centrifugal contactor, high level waste, partitioning, pilot plant test, simplified TRPO process

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INTRODUCTION

The safe treatment and disposal of high level waste (HLW) that is produced from reprocessing of the spent nuclear fuel and presents a significant hazard to the environment is crucial to the sustainable development of nuclear energy. The partitioning and transmutation (P&T) of long-life nuclides from HLW is a promising method to reduce the long-term risk of HLW (1–5). Some partitioning processes, such as the TRUEX process, the DIAMEX process, the DIDPA process, and the TRPO process, were developed to separate actinides from HLW (3,6–9).

The TRPO process has been shown to remove efficiently actinides in HLW by a series of cold and hot tests at the Institute of Nuclear and New Energy Technology (INET), Tsinghua University, China (10–15). In particular, a laboratory-scale hot test of a total-partitioning process including the original TRPO process for actinides removal, the Crown Ether Strontium Extraction (CESE) process for Sr removal, and Cs removal with a small ion-exchange column loaded with potassium titanium hexacyanoferrate, was successfully carried out in 1996, where 50 stages of 10-mm-dia centrifugal contactors were used in both the TRPO process and the CESE process (12). In this hot test, the max total flow rate of two phases was about 110 mL/h. The decontamination factors (DFs) for total α activity, ^{99}Tc, ^{90}Sr, and ^{137}Cs were 588, 125, >2500, and >200 respectively. Moreover, a cold pilot plant test of the total-partitioning process was successfully carried out with simulated HLW at INET in 2005, where 24-stage, 70-mm-dia centrifugal contactors were used in the stripping section of the original TRPO process (14,15). In this pilot plant test, the total flow rate of the two phases was about 110 L/h, and the decontamination factors (DFs) for the total U, Nd, Zr, Sr, and Cs were >3000, >500, >1000, ~160, and ~94 respectively.

In the original TRPO process, actinides (An) and lanthanides (Ln) are extracted together from HLW with 30% TRPO (mixed trialkylphosphine oxide)–kerosene, then the loaded organic phase is stripped with 5.5 mol/L HNO_3 and 0.1 mol/L HNO_3 to recover An(III) and Ln(III) (section 1B), with 0.6 mol/L $H_2C_2O_4$ to recover Pu and Np (section 1C), with 5.5 mol/L HNO_3 and 0.01 mol/L HNO_3 to scrub $H_2C_2O_4$ loaded in the organic phase (sections 1CA1 and 1CA2), with 5% $(NH_4)_2CO_3$ to recover U (section 1D), and with 0.01 mol/L HNO_3 to scrub the 30% TRPO-kerosene (section 1E), namely section 1E is solvent wash section (14,15). The stripping section of the original TRPO process for actinides removal is shown in Fig. 1. However, one of the disadvantages for the process is that 5.5 mol/L HNO_3 is used for stripping Am(III) and Ln(III), which presents some problems in the subsequent separation of Am(III) and Ln(III) with bis(2,4,4 trimethylpentyl)

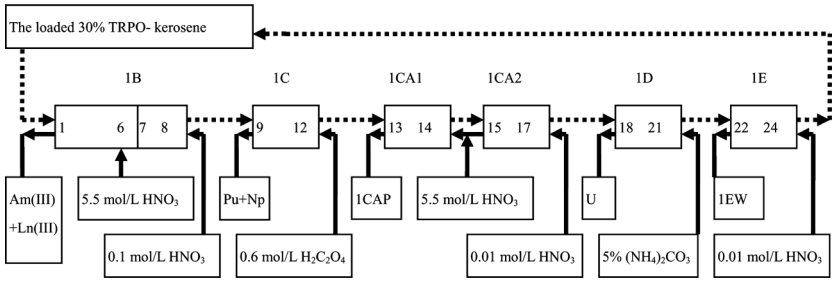

Figure 1. The stripping section of the original TRPO process.

dithiophospninic acid (Cyanex 301), which needs a dilute HNO_3 solution. The process for separation of Ln(III)/Am(III) with the purified Cyanex 301 has been developed and verified by the hot test in hot cell with 10 stages of 10-mm-dia centrifugal contactors (16). In addition, the stripping section of the original TRPO process seems complicated for practical industrial use (17).

To overcome these disadvantages of the original TRPO process, a new stripping agent, N,N′-dimethyl-3-oxa-glutaramic acid (DMOGA), was synthesized to simplify the original TRPO process (17,18). DMOGA is a bridge-quadridentate ligand and that all of its coordination donor atoms are oxygen. The coordination ratios of all the complexes are 3. DMOGA can form stable complexes with Am(III), Nd(III), Pu(IV), Np(IV), Fe(III), and Zr(IV). It can effectively strip Am(III), Nd(III), Pu(IV), Np(IV), Fe(III), and Zr(IV) in dilute nitric acid solution ($[HNO_3] < 0.8\,mol/L$) from the loaded TRPO. However, DMOGA does not form stable complex with UO_2^{2+}, therefore U in the 30% TRPO-kerosene can not be stripped by DMOGA. Because DMOGA consists of C, O, and N, it can be completely combusted without producing the secondary waste. Therefore, DMOGA is a promising stripping agent in the treatment of HLW. For these reasons, two simplified TRPO processes were developed on the basis of DMOGA as the stripping agent at INET in recent years.

Centrifugal contactors are efficient extraction devices for phase mixing and separation in extraction processes. Compared with conventional contactors such as mixer-settlers and pulsed columns, centrifugal contactors offer some advantages including low hold-up volume, short residence time (therefore less solvent degradation), excellent phase separation, high efficiency, great safety with respect to nuclear criticality, compactness (therefore low capital costs), rapid start-up and shut-down, etc., which are particularly beneficial to reprocessing of the spent nuclear fuel and partitioning of HLW (19–21).

The primary centrifugal contactor, which was the paddle type, had been successfully developed and operated for many years at Savannah River Site (SRS). In the late 1960s, the paddle type centrifugal contactor was modified to the annular type at Argonne National Laboratory (ANL). The ANL centrifugal contactor was reliable and easy to operate and maintain. In particular, it was used for the hot tests of the TRUEX process at ANL (21–24). Recently, USA, Russia, France, Germany, U.K, India, and Japan achieved new progress on the development and application of centrifugal contactors in the nuclear industry (25–32). Since the late 1970s, INET has been developing its own annular centrifugal contactors. A series of INET centrifugal contactors have been developed with rotor diameters from 10 mm to 230 mm and used successfully in some fields including hydrometallurgy, wastewater treatment, biotechnology, the pharmaceutical industry, and especially the partitioning of HLW (33–43).

In the present paper, the results of the pilot plant tests of two simplified TRPO processes with 70-mm-dia centrifugal contactors were reported. In the pilot plant tests, the total flow rate of two phases was about 110 L/h.

EXPERIMENTAL

Materials

TRPO (commercial name Cyanex 923) was purchased from American Cyanamid Company. According to the producer, the TRPO extractant comprises a mixture of four trialkylphosphine oxides, with the general formula R_3PO (14%), $R_2R'PO$ (42%), RR'_2PO (31%) and R'_3PO (8%), in which R and R′ denotes n-octyl and n-hexyl group respectively (44). TRPO was diluted with saturated kerosene to 30% TRPO-kerosene (v/v), and first purified with anion exchange resin, then scrubbed once with 1.0 mol/L HNO_3, and adjusted to neutral with deionized water, lastly scrubbed with 50 g/L $(NH_4)_2CO_3$ and readjusted to neutral with deionized water before use.

DMOGA (>99% of purity)is the synthesized product from INET. The molecular structure of DMOGA is shown in Fig. 2. Its melting point is 80–82°C. Its solubility in water is about 7 g in 100 mL H_2O at 25°C (17,18). pH is a key factor affecting the stripping efficiency. DMOGA aqueous solutions were adjusted to different pH values with ammonia.

Other reagents were all analytical reagents. Table 1 gives the composition of the simulated HLW. Based on their similar extraction behaviors

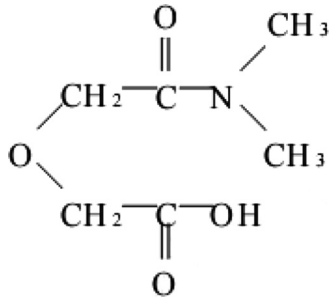

Figure 2. The molecular structure of DMOGA.

in the simplified TRPO processes, Nd and Zr were used to simulate Am and Pu respectively (13).

The 70-mm-dia Centrifugal Contactor

A diagram of the 70-mm-dia centrifugal contactor, made of stainless steel, is shown in Fig. 3. A cutaway view is shown in Fig. 4. The inside diameter of its rotor is 70 mm, and its hold-up volume is about 350 mL. Its new design characteristics and performance were shown in the previous literature (42). Two immiscible liquids are fed from the opposite sides into the annular mixing zone between the spinning rotor and the stationary housing, and are mixed by shear forces as they flow down the annular space. Four radial vanes in the bottom of the housing inhibit the rotation of the mixture and direct it through the inlet in the bottom and into the inside of the rotor. Here the emulsion breaks rapidly

Table 1. The composition of the simulated high level waste

Element	Concentration (mg/L)	Element	Concentration (mg/L)
Na	1.83×10^4	Sr	222
Al	5.68×10^3	Cs	715
Fe	5.98×10^3	Zr	78
Cr	715	Ba	27
Mo	309	U	1.63×10^3
K	160	HNO_3	1.0 mol/L
Ni	2.93×10^3	SO_4^{2-}	0.034
Nd	1.50×10^3		

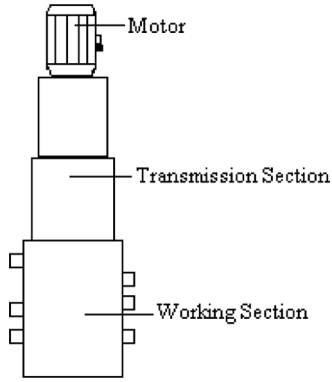

Figure 3. General view of the 70-mm-dia centrifugal contactor.

under the high centrifugal force. The separated phases flow separately through the heavy-phase weir and the light-phase weir of the rotor into their collector rings in the housing. Then each liquid leaves its collector ring through a tangential exit and flows into an adjacent stage respectively.

Pilot Plant Tests of the Simplified TRPO Processes

The pilot plant tests of two simplified TRPO processes with the simulated HLW were carried out to demonstrate feasibility and reliability of the processes. In the pilot plant tests, the 30% TRPO-kerosene was used to

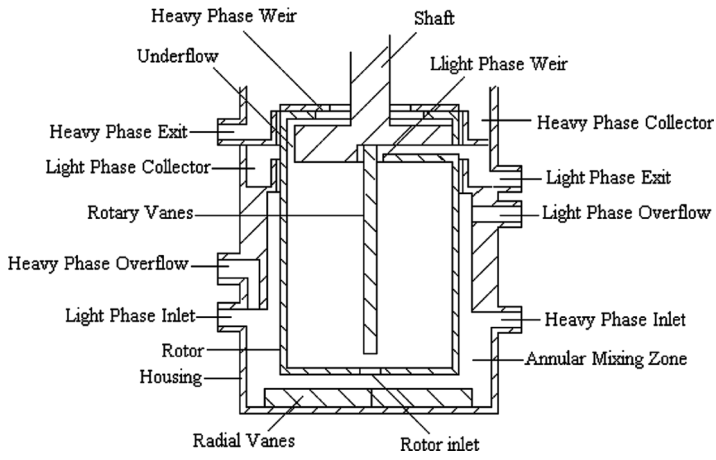

Figure 4. Cutaway view of the 70-mm-dia centrifugal contactor.

Table 2. The concentration in the loaded 30% TRPO-kerosene

Element	Concentration
Nd	2510 mg/L
Zr	131 mg/L
Fe	119 mg/L
Mo	457 mg/L
HNO_3	0.59 mol/L

extract Nd and Zr from the simulated HLW in the first pulsed column with 100 mm diameter. The loaded 30% TRPO-kerosene after scrubbing in the second pulsed column with 100 mm diameter was successively stripped with different effluents in 70-mm-dia centrifugal contactors. The main material of the pulsed columns is glass. The continuous phase in the first pulsed column was the organic phase, while the continuous phase in the second pulsed column was the aqueous phase. The concentration in the loaded 30% TRPO-kerosene after extraction in the first pulsed column and scrubbing in the second pulsed column is shown in Table 2. The 30% TRPO-kerosene after stripping was recycled. The concentrations of Nd, Zr, and other elements in the sampled solutions were determined by ICP-AES and ICP-MS.

The stripping efficiency is described as follows:

$$\rho = \frac{C_{O,in} - C_{O,out}}{C_{O,in}} \times 100\%$$

Where ρ is the stripping efficiency (%), $C_{O,in}$ and $C_{O,out}$ are the inlet and the outlet concentrations of a metal ion in the organic phase respectively (mg/L).

RESULTS AND DISCUSSION

The Test of the First Simplified TRPO Process

The stripping section of the first simplified TRPO process is shown in Fig. 5. In section 1B, 0.05 mol/L HNO_3 is used to scrub the loaded 30%TRPO-kerosene. In section C1, a 0.35 mol/L DMOGA solution with pH 5.3 is used to strip Am, Ln, Np, and Pu (only Nd and Zr in the present pilot plant test). In section C2, a 0.35 mol/L DMOGA solution with pH 3.8 is used to strip not only Am, Ln, Np, and Pu (only Nd and Zr in

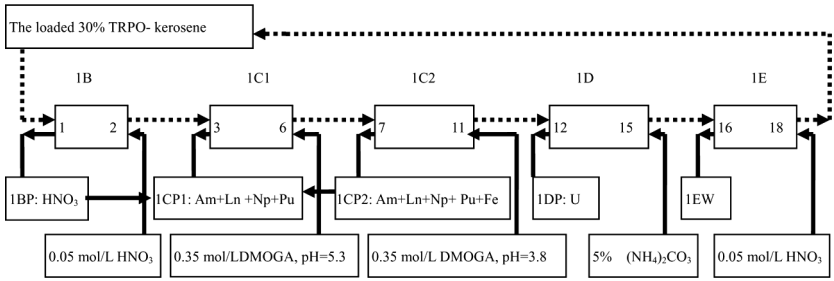

Figure 5. The stripping section of the first simplified TRPO process.

the present pilot plant test) but also Fe in the loaded organic phase in order to avoid the precipitation in section 1D, where U is stripped with 5% $(NH_4)_2CO_3$. In section 1E, 0.05 mol/L HNO_3 is used to scrub the 30% TRPO-kerosene before recycle. The required flow rate of every effluent in the test of the first simplified TRPO process is shown in Table 3.

The rotor speed (ω) of 70-mm-dia centrifugal contactors can be varied from 1500 r/min to 3000 r/min. The diameter of the heavy-phase weir (D) can be varied among 36 mm, 38 mm, and 40 mm. Both the rotor speed (ω) and the diameter of the heavy-phase weir (D) in each stage were determined under the required flow rate (see Table 3) to make all of the effluent stream entrainment in either phase below 0.5%. The operation parameters of centrifugal contactors in the test are shown in Table 4. For section 1D, there were gases produced including NH_3 and CO_2 when 5% $(NH_4)_2CO_3$ reacted with HNO_3 in the organic phase. Therefore, in order to avoid entrainment in the outlet aqueous phase, both the rotor speed and diameter of the heavy-phase weir of stage 12 test should be lower. Meanwhile, both the rotor speed and diameter of the heavy-phase weir of stages 13–15 should be larger in order to avoid entrainment in the outlet organic phase. Under these operation factors and the required flow rate, all of the end stream entrainment in either phase was below 0.5%.

Table 3. The required flow rate of every effluent in the test of the first simplified TRPO process

Effluent	The loaded 30% TRPO-kerosene	0.05 mol/L HNO_3	0.35 mol/L DMOGA (pH = 5.3)	0.35 mol/L DMOGA (pH = 3.8)	5% $(NH_4)_2CO_3$	0.05 mol/L HNO_3
Flow rate (L/h)	55	27.6	55	55	55	55

Table 4. The operational parameters of centrifugal contactors in the test of the first simplified TRPO process

Stage	ω (r/min)	D (mm)
1–11	2700–3000	38–39
12	2400	38
13–15	2700–3000	40
15–18	2700–3000	38–39

The variation of the metal concentrations with time in the aqueous phase effluent is shown in Table 5. The results showed that the stripping process could reach the steady state within 20 min because there was low hold-up volume in the centrifugal contactors. Variation of concentration of each element in the two phases of each stage is shown in Figs. 6–9 respectively. In section 1B, the scrubbing efficiency of the Nd^{3+}, Mo^{6+}, and Fe^{3+} was low, and Zr^{4+} was not stripped. After section 1C1, the total stripping efficiency of the Nd^{3+}, Zr^{4+}, Mo^{6+}, and Fe^{3+} was 99.98%, 99.6%, 26%, and 39% respectively. After section 1C2, the total stripping efficiency of the Nd^{3+}, Zr^{4+}, Mo^{6+}, and Fe^{3+} was 99.99%, 99.9%, 48%, and 61% respectively. These results imply that just section 1C1 is not enough to strip transuranic elements in the loaded 30% TRPO-kerosene. The exit aqueous phases from sections 1B, 1C1, and 1C2 were discharged into the same group effluent, because the exit aqueous phases from sections 1B and 1C2 contained a little amount of Nd or Zr. Most of Fe in the loaded organic phase needs to be removed before section 1D in order to avoid the precipitation of ferric carbonate. Fe can be easily stripped with DMOGA solution with a high pH. Only 39% of Fe was stripped from the loaded organic phase after section 1C1. Sixty-one

Table 5. The variation of the metal concentrations with time in the aqueous phase effluent of the first simplified TRPO process

Time, h	1B (mg/L)				1C1 (mg/L)				1C2 (mg/L)				1D (mg/L)		1E (mg/L)
	Nd	Mo	Fe	Zr	Nd	Mo	Fe	Zr	Nd	Mo	Fe	Zr	Mo	Fe	Mo
0.05	46	6	4	0.1	1910	18	46	122	3.7	95	27	3.3	35	3	8
0.17	46	6	3	0.1					4.2	35	33	2.1	6	3	18
0.33	48	6	3	0.1	1997	13	41	128	3.4	167	31	1.3	183	3	55
0.67	46	6	3	0.1	1957	15	42	127	4.2	171	29	0.9	183	3	63
1.17	45	6	3	0.1	1946	18	43	125	3.2	180	27	0.9	191	3	59
1.5	48	6	3	0.1	1899	26	42	122	3.7	168	26	0.7	180	3	57

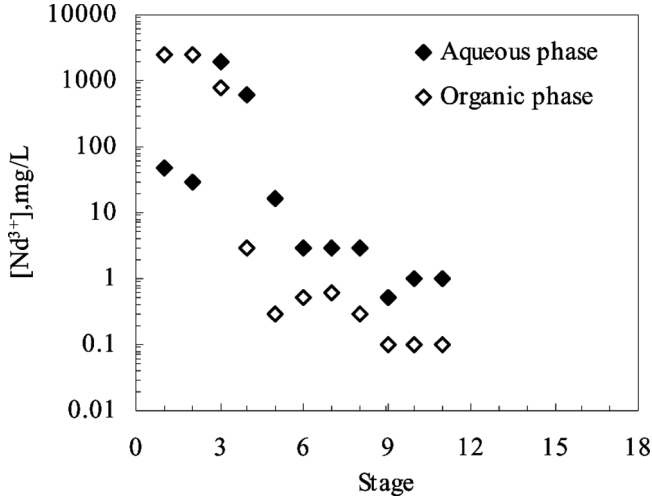

Figure 6. The effluent concentrations of Nd^{3+} in each stage.

percent of Fe was stripped from the loaded organic phase after section 1C2. After section 1D, the total stripping efficiency of the Mo^{6+} and Fe^{3+} was about 90% and above 95% respectively. Moreover, precipitation was not observed in section 1D. After section 1E, about 10% of the Mo^{6+} was still remained in the 30% TRPO-kerosene.

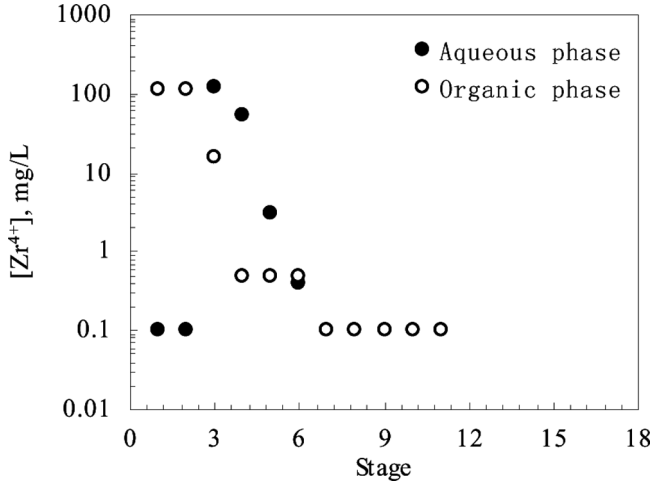

Figure 7. The effluent concentrations of Zr^{4+} in each stage.

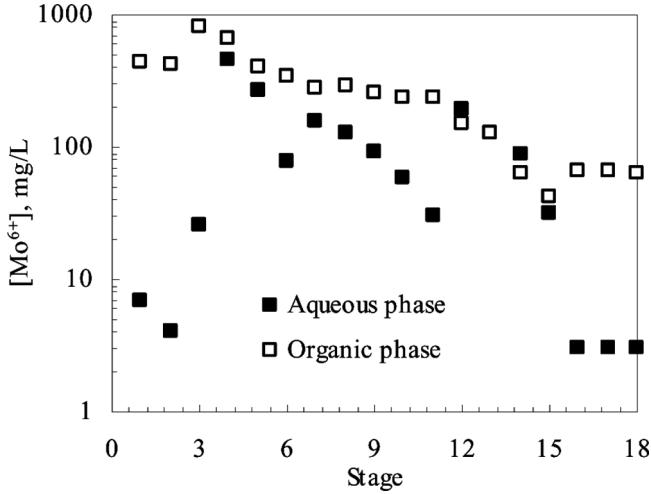

Figure 8. The effluent concentrations of Mo^{6+} in each stage.

The Test of the Second Simplified TRPO Process

The stripping section of the second simplified TRPO process is shown in Fig. 10. In section 1B, a 0.4 mol/L DMOGA solution with pH 5.7 is used to strip Am, Ln, Np, and Pu (only Nd and Zr in the present pilot plant test). In section C, 0.3 mol/L $(NH_4)_2C_2O_4$ is used to mainly strip Fe in the

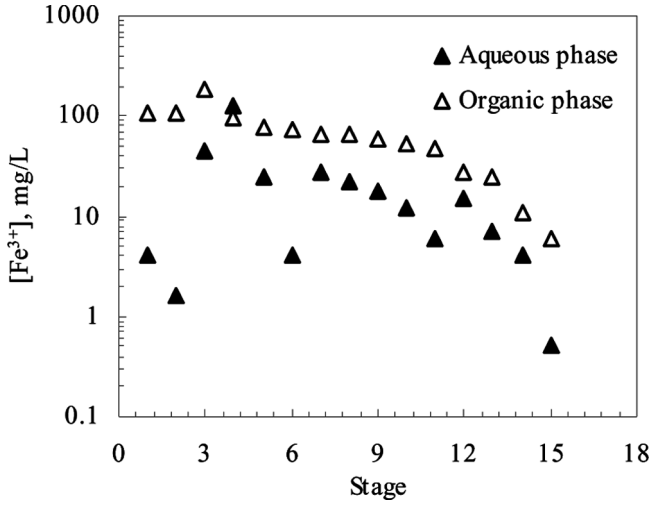

Figure 9. The effluent concentrations of Fe^{3+} in each stage.

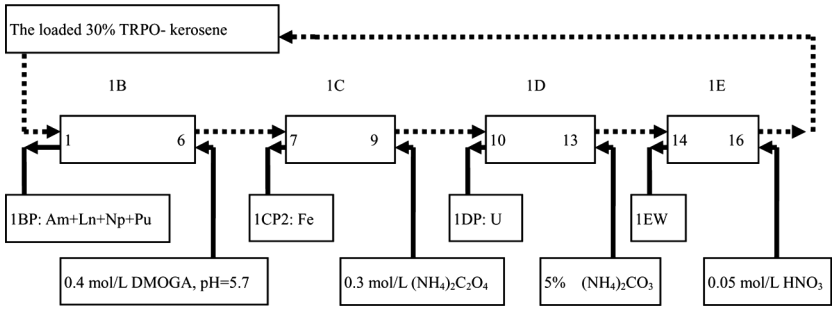

Figure 10. The stripping section of the second simplified TRPO process.

loaded organic phase in order to avoid the precipitation in section 1D, where U is stripped with 5% $(NH_4)_2CO_3$. In section 1E, 0.05 mol/L HNO_3 is used to scrub the 30% TRPO-kerosene before recycle. The required flow rate of every effluent in the second test is shown in Table 6.

The operation parameters of centrifugal contactors in the test are shown in Table 7. Similarly, for section 1D, there were gases produced including NH_3 and CO_2 when 5% $(NH_4)_2CO_3$ reacted with HNO_3 in the organic phase. Thereby, in order to avoid entrainment in the outlet aqueous phase, both the rotor speed and diameter of the heavy phase weir of stage 10 should be lower. Meanwhile, both the rotor speed and diameter of the heavy phase weir of stages 11–13 should be larger in order to avoid entrainment in the outlet organic phase. Under these operation parameters and the required flow rate, all of the end stream entrainment in either phase was below 0.5%.

The variation of the metal concentrations with time in the aqueous phase effluent is shown in Table 8. The results showed that the stripping process could also reach the steady state within 10 min.

Variation of concentration of each element in the two phases of each stage is shown in Figs. 11–14 respectively. In section 1B, the stripping efficiency of the Nd^{3+}, Zr^{4+}, Mo^{6+}, and Fe^{3+} was 99.98%, 99.9%,

Table 6. The required flow rate of every effluent in the test of the second simplified TRPO process

Effluent	The loaded 30% TRPO-kerosene	0.4 mol/L DMOGA (pH = 5.7)	0.3 mol/L $(NH_4)_2C_2O_4$	5% $(NH_4)_2CO_3$	0.05 mol/L HNO_3
Flow rate (L/h)	55	65	55	55	55

Table 7. The operation parameters of centrifugal contactors in the test ofthe second simplified TRPO process

Stage	ω (r/min)	D (mm)
1–9	2700–3000	38–39
10	2400	38
11–13	2700–3000	40
14–16	2700–3000	38–39

5.7%, and 53% respectively. This result implies that section 1B is enough to strip transuranic elements in the loaded 30% TRPO-kerosene. After section 1C, the total stripping efficiency of the Mo^{6+} and Fe^{3+} was 65% and 91% respectively, which ensured the absence of precipitation in section 1D. After section 1D, the total stripping efficiency of Mo^{6+} and Fe^{3+} was about 80% and 97% respectively. After section 1E, about 2% of the Mo^{6+} was still remained in the 30% TRPO-kerosene.

Compared with the first simplified TRPO process, the second simplified TRPO process with only one section using the DMOGA solution as the stripping agent achieved a satisfactory stripping efficiency. This mainly results from a higher concentration of DMOGA and higher initial pH of the stripping solution.

Comparison of Two Simplified TRPO Processes

The extraction sections of two simplified TRPO processes are the same as the original TRPO process, namely, actinides and lanthanides are extracted from HLW with 30% TRPO-kerosene. The different parts are their stripping sections. In the first simplified TRPO process, 0.05 mol/L

Table 8. The variation of the metal concentrations in the exit aqueous phase of the second simplified TRPO process

Time, h	1B (mg/L)				1C (mg/L)		1D (mg/L)		1E (mg/L)
	Nd	Mo	Fe	Zr	Mo	Fe	Mo	Fe	Mo
0.05	2128	10	50	109	1	1	31	5	262
0.17	2205	12	53	108	209	43	75	6	70
0.33	2209	13	51	108	224	47	88	6	85
0.67	2132	14	49	105	226	47	90	6	92
1.0	2109	14	48	106	225	48	85	6	93

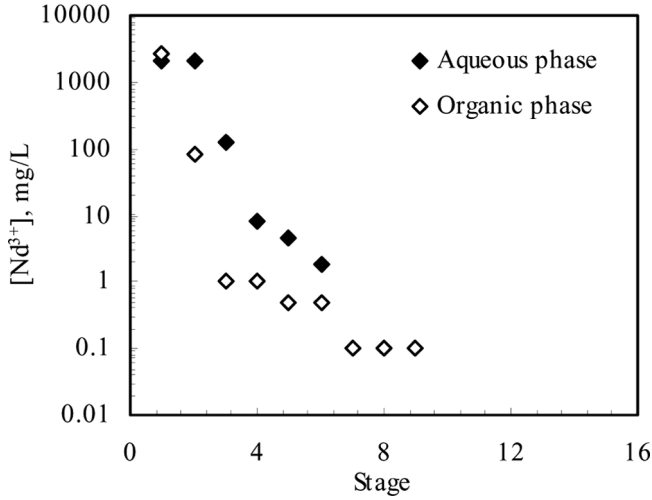

Figure 11. The effluent concentrations of Nd^{3+} in each stage.

HNO_3 is firstly used to scrub the loaded 30% TRPO-kerosene, and then 0.35 mol/L DMOGA with different acidities is used to strip Am(III), Ln(III), Np(IV), and Pu(IV) respectively. Eighteen stages of centrifugal contactors are needed for the stripping section of the first simplified TRPO process. In the second simplified TRPO process, 0.4 mol/L

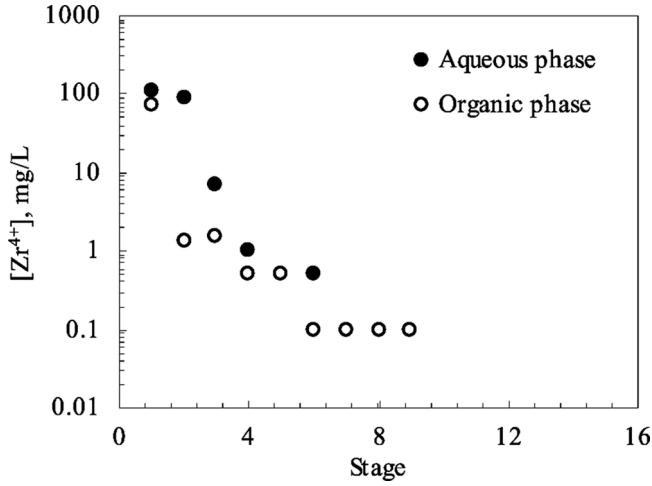

Figure 12. The effluent concentrations of Zr^{4+} in each stage.

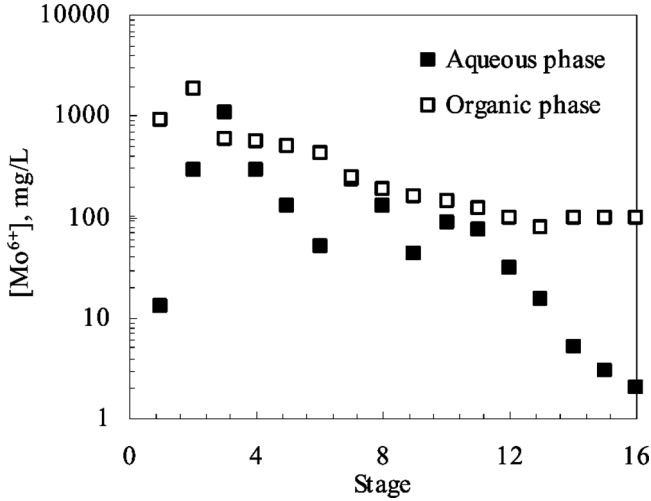

Figure 13. The effluent concentrations of Mo^{6+} in each stage.

DMOGA with pH 5.7 is firstly used to strip Am(III), Ln(III), Np(IV), and Pu(IV), and then 0.3 mol/L $(NH_4)_2C_2O_4$ is used to remove Fe in the loaded 30% TRPO-kerosene. 16 stages of centrifugal contactors are needed for the stripping section of the second simplified TRPO process. The stripping of the U (section 1D) and the scrub of the organic phase

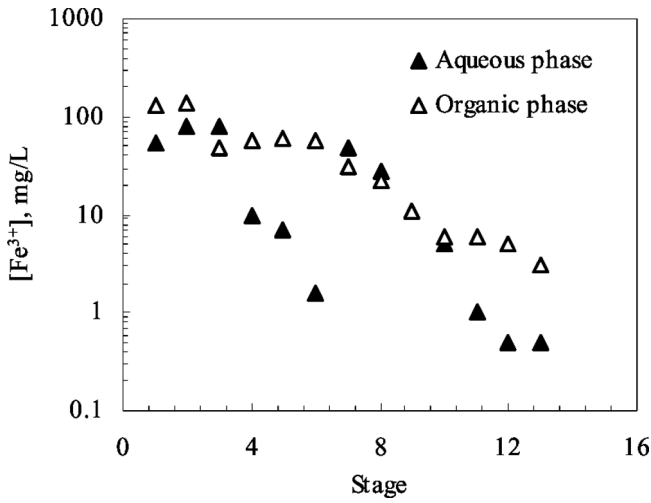

Figure 14. The effluent concentrations of Fe^{3+} in each stage.

after the stripping of the U (section 1E) are same in the two simplified TRPO processes and the original TRPO process. So the second simplified TRPO process is simpler than the first simplified TRPO process.

Moreover, the result in the pilot plant test of the first simplified TRPO process showed that about 40% of Fe was still remained in the organic phase after section 1C2, while Fe can react with $(NH_4)_2CO_3$ to produce some precipitates. So the aqueous phase effluents from sections 1B, 1C1, and 1C2 need to merge into an effluent. Thereby the volume of α waste becomes larger in the first simplified TRPO process. In the second simplified TRPO process, the exit aqueous phase from section 1C is not α waste, and the stripping efficiency of the Mo is small in section 1B, so accordingly, the volume of α waste is greatly reduced. Although, the amount of Mo remained in the organic phase after stripping in the second simplified TRPO process is larger than that in the first simplified TRPO process, the recycle of the organic phase is not affected by Mo remained in the organic phase.

CONCLUSIONS

Two simplified TRPO processes were developed by adopting DMOGA as the stripping agent to strip Am(III), Ln(III), Np(IV), and Pu(IV).

The cold pilot plant tests of two simplified TRPO processes were carried out with the simulated HLW and 70-mm-dia centrifugal contactors. The stripping rates of some elements (e.g. Nd, Zr) in the loaded 30% TRPO-kerosene from the extraction section were higher than 99.9%. Compared to the original TRPO process, two simplified TRPO processes are more compact and simpler. Although the nuclides can not be completely separated, the objects of non-α and volume-reduction of waste are achieved.

During the pilot plant tests of two simplified TRPO processes, all of the 70-mm-dia centrifugal contactors were stable, working continuously with no stage failure or interruption during operation. All of the results demonstrate that these centrifugal contactors are promising for the stripping section of the simplified TRPO process.

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