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Stage-efficiency of centrifugal extractor used in nuclear industry①

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Abstract: The stage-efficiency of a single-stage prototype (d70) centrifugal extractor and the cascade is tested by HNO_3-Nd^{3+} and 30% TRPO-kerosene system. The experimental results of the single-stage centrifugal extractor show that the carryover of the two phases decreases with increasing ratio of the two flow rate and rotation rate and the stage efficiency increases with not only decreasing total flow rate but also increasing rotor speed. However, the experimental results of the cascade show that the average stage efficiency of the cascade increases with not only decreasing total flow rate but also increasing rotor speed in both three-stage mode and two-stage mode.

Key words: centrifugal extractor; cascade; carryover; stage efficiency
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1 INTRODUCTION

Compared with a mixer-settler or pulsed column used in the nuclear fuel reprocessing extraction processes, a short-residence-time centrifugal extractor catches researcher's eyes because of the good phase separation and a small hold-up volume. It shows the following advantages in the nuclear industry[1]: 1) Reducing solvent degradation due to radiation; 2) Reducing inventory of aqueous and organic phases, which gives rapid start-up and shut-down; 3) Compact design that contributes to the reduction of plant cost.

Many different types of centrifugal extractors have been developed. For example, Podbielniak-extractor, where the cylindrical dual flow perforated sheets are arranged concentrically round a vertical shaft and there is no downcomer for continuous liquid, was used for penicillin extractor[2]; Robatel-extractor, which is "internal centrifugal mixer-settler" type and four stages in one extractor, has been used by the French nuclear-reprocessing industry[3].

In order to maintain conveniently the extractor, the idea that single-stage extractor instead of the former two many-stage extractors and the cascade were used in nuclear industry occurred. For small hot nuclear experiments, the miniature single-stage centrifugal extractors were developed, such as 10mm single-stage centrifugal extractor, which has been presented in our lab and the cascade is used to examine the TRPO process since 1980s[4, 5], the DIAMEX process in France[6], the DIDPA process in Japan[7], 20mm extractor cascade to test the TRUEX-SREX process[8], 12mm centrifugal extractor used in ATALANTE facility at MARCOULE[9]. However, the three parts of the miniature centrifugal extractor, i.e. the rotor, motor and mixer are fixed by mechanical connection and leads to the difficulty in maintenance. Therefore, the novel centrifugal extractor (d70), where the mixer and rotor are separated and connected by magnetic force, has been developed in our lab.

In this paper, the stage efficiency of a single-stage centrifugal extractor with the diameter of rotor 70 mm and three-extractor cascade are tested. In the stage efficiency experiment, Nd^{3+} is used to simulate Am^{3+} because the extraction properties of Nd^{3+} in the TRPO process are very similar to those of Am^{3+} [10].

2 EXPERIMENTAL

2.1 Single-stage centrifugal extractor

The diagram of the single-stage centrifugal extractor is shown in Fig. 1. The rotor diameter of the centrifugal extractor (3) is 70 mm and its hold-up volume is about 350 mL. The aqueous phase ($Nd(NO_3)_3$-1 mol/L HNO_3 solution) and organic phase (30% (volume fraction) trialkylphosphine oxide (TRPO)-kerosene solution) were fed into the centrifugal extractor (3) at the opposite sides. The rotating speed of the rotor could be adjusted by the frequency adjuster (2). The flow rates of aqueous and organic phases were determined by the rotameters (4) and (5) respectively.

The physical properties of the extraction system used in the experiment are shown in Table 1. The carryover of the single-state extractor was determined

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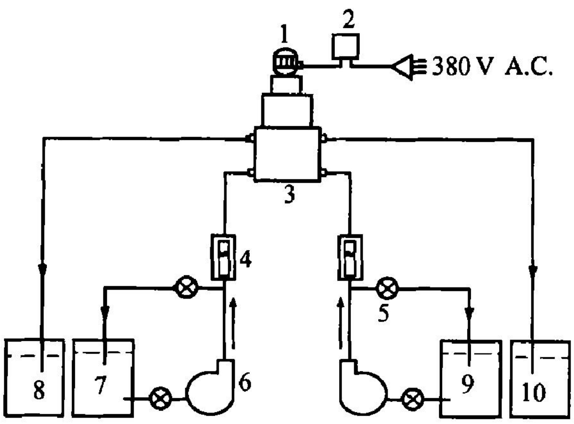

Fig. 1 Setup for single state experiment of centrifugal extractor
1—Motor; 2—Frequency adjuster;
3—Centrifugal extractor; 4—Flowmetes;
5—Valves; 6—Pumps;
7, 8—Tanks for aqueous solution;
9, 10—Tanks for organic solution

by the amount of phase carryover sampled from the outlet after the extractive systems reached steady state. The carryover was determined with a water detector (CA-05 Karl-Fisher Water Detector, USA) for the aqueous phase in the organic phase and oil detector (FF-1 Oil Detector, Xiaoshan Analytical Instrument Factor, China) for the organic phase in the aqueous phase.

Table 1 Properties of extraction system

Organic phase		Aqueous phase		Interfacial tension/
ρ_o/ (kg·m^{-3})	μ_o/ (Pa·s)	ρ_a/ (kg·m^{-3})	μ_a/ (Pa·s)	(10^{-3}N·m^{-1})
810.2	5.16×10^{-3}	1 017.9	1.12×10^{-3}	16.87

Stage efficiency was determined by extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1 mol/L HNO_3 solution and stripping Nd^{3+} with 5.5 mol/L nitric acid from 30% TRPO-kerosene solution loaded with Nd^{3+}. In aqueous phase, the concentration of Nd^{3+} is analyzed with ICP (Inductive Coupled Plasma) and the concentration of HNO_3 is measured by titration with standard NaOH solution after adding $K_2C_2O_4$ as occultation agent. However, the concentration of Nd^{3+} in organic phase should be stripped by 5.5 mol/L HNO_3 and analyzed by ICP. Equilibrium distribution of Nd^{3+} in the systems could be calculated from the mathematical model presented by Chen[7]. Therefore, the stage efficiency is described as follows[11]:

$$E_a=\frac{\rho_{a,\ in}-\rho_{a,\ out}}{\rho_{a,\ in}-\rho_{a,\ eq}}\times100\%\ \text{(for aqueous phase)}$$

$$E_o=\frac{\rho_{o,\ out}-\rho_{o,\ in}}{\rho_{o,\ eq}-\rho_{o,\ in}}\times100\%\ \text{(for organic phase)}$$

2.2 Extractor cascade

The diagram of the centrifugal extractor cascade, which consists of 3 single-stage extractors of d70 mm, is shown in Fig. 2. The cascade can run in two modes: (a) three-stages (normal operation) and (b) two-stages when one of the cascade stops working (abnormal operation), which is often met in industry.

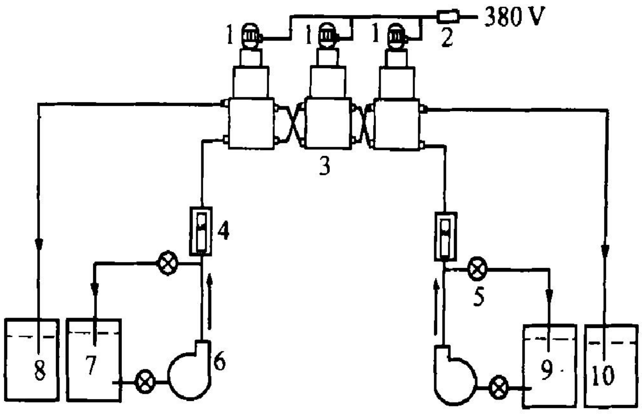

Fig. 2 Setup of centrifugal extractor cascade
1—Motor; 2—Frequency adjuster;
3—Centrifugal extractors;
4—Flowmeters; 5—Valves;
6—Pump; 7, 8—Tanks for aqueous solution;
9, 10—Tanks for organic solution

The method of measuring stage efficiency was the same as that for the single-stage extractor, but the average stage efficiency of the cascade was determined as follows: firstly, the number of theoretical equivalent stage (N_{TES}) was calculated according to the inlet and outlet concentrations of Nd^{3+} of both phases and the mathematical model of equilibrium distribution of Nd^{3+} in these systems presented by Chen[7]; then the average stage efficiency, E, was calculated by

$$E=N_{TES}/N_{st}$$

where $N_{st}=3$ for three-stage mode, and $N_{st}=2$ for two-stage mode.

3 RESULTS AND DISCUSSION

3.1 Single-stage centrifugal extractor

The carryover of the centrifugal extractor is presented in Fig. 3 and Fig. 4 when q_a: q_o= 2:1 and 1:1 respectively. From Fig. 3 (b) and Fig. 4 (b), it can be seen that the carryover of the organic phase in the aqueous phase is less than 0.1% within the experimental range. For q_a: q_o= 1:1, the carryover of the aqueous phase in the organic phase is larger than 0.5% when not only q_a> 60L/h at n= 1 764 r/min but also q_a> 80L/h at n= 2 058 r/min in Fig. 3 (a), while only when q_a> 100L/h at n= 1 764 r/min for q_a: q_o= 2:1.

During the stage efficiency experiment, the carryover must be less than 0.5%. Therefore, the measure, i. e. choosing either high rotation rate or low throughout at low rotation rate, is taken. The experimental conditions for stage efficiency are shown in Tables 2, 3, 4 and 5.

Stage efficiency of the centrifugal extractor is

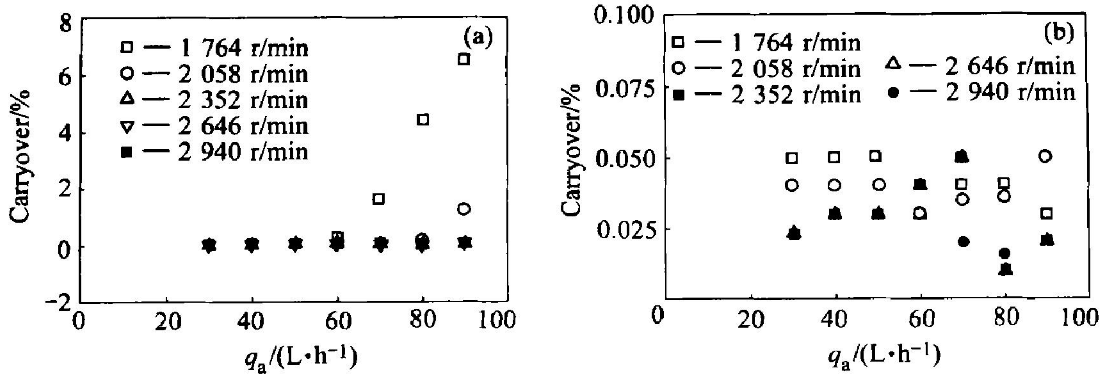

Fig. 3 Effect of rotation rate on carryover when q_a: q_o= 1: 1

(a) —Aqueous phase in organic phase; (b) —Organic phase in aqueous phase

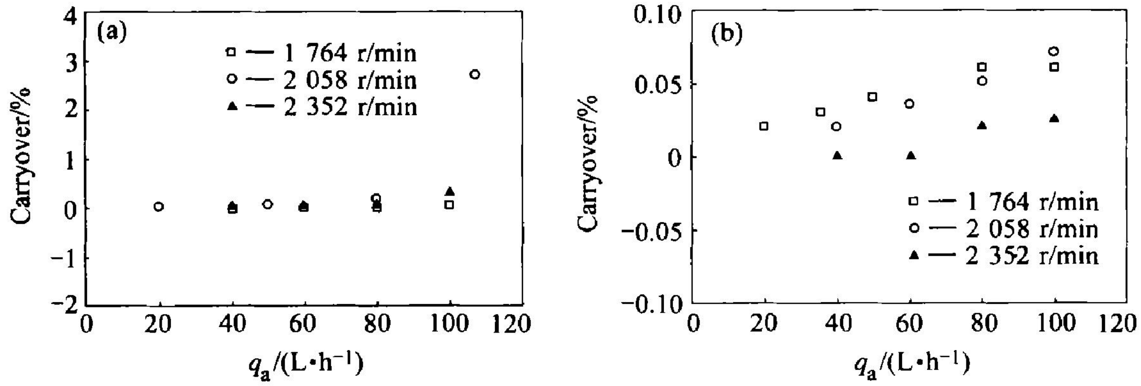

Fig. 4 Effect of rotation rate on carryover when q_a: q_o= 2: 1

(a) —Aqueous phase in organic phase; (b) —Organic phase in aqueous phase

Table 2 Stage efficiency for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution when q_a: q_o= 1: 1

n/(r·min^{-1})	q_t/(L·h^{-1})	Mass concentration/(mg·L^{-1})						E_a	E_o
		$\rho_{a,in}$	$\rho_{a,out}$	$\rho_{a,eq}$	$\rho_{o,in}$	$\rho_{o,out}$	$\rho_{o,eq}$		
1 760	37.4	506.6	122.0	103.9	543.8	1030	1 052.4	95.5	95.6
	79.2	506.6	129.9	92.1	543.8	952.8	996.8	90.9	90.3
	116.9	506.6	137.9	93.6	543.8	933.6	978.0	89.3	89.8
2 060	40.20	537.00	49.18	27.91	4.57	478.80	501.00	95.82	95.53
	82.29	537.00	63.10	26.55	4.57	456.00	494.00	92.84	92.24
	120.50	537.00	70.43	29.38	4.57	462.80	503.70	91.91	91.81
	162.89	537.00	73.06	29.62	4.57	459.20	503.00	91.44	91.21
2 350	38.38	431.00	59.09	48.85	383.40	822.00	832.00	97.32	97.77
	79.23	431.00	69.81	45.78	383.40	752.80	776.80	93.76	93.90
	118.52	431.00	74.61	47.72	383.40	764.40	794.40	92.98	92.70
	158.12	431.00	76.96	47.92	383.40	748.80	779.20	92.42	92.32
	174.79	431.00	76.78	48.10	383.40	755.20	785.60	92.51	92.44
	198.13	431.00	80.62	49.34	383.40	766.00	800.80	91.80	91.66
2 650	40.60	480.00	39.85	28.99	105.26	526.40	538.00	97.59	97.32
	79.03	480.00	51.84	31.65	105.26	544.40	564.80	95.50	95.56
	120.73	480.00	54.24	29.31	105.26	527.20	552.80	94.47	94.28
	163.33	480.00	57.03	29.26	105.26	529.60	556.80	93.84	93.98
	179.08	480.00	59.63	30.57	105.26	521.20	550.00	93.53	93.52
	201.04	480.00	58.60	32.42	105.26	546.80	575.20	94.15	93.96

Table 3 Stage efficiency for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution when q_a: q_o= 2: 1

$n/(r \cdot min^{-1})$	$q_t/(L \cdot h^{-1})$	Mass concentration/($mg \cdot L^{-1}$)						E_a	E_o
		$\rho_{a,in}$	$\rho_{a,out}$	$\rho_{a,eq}$	$\rho_{o,in}$	$\rho_{o,out}$	$\rho_{o,eq}$		
1 760	30. 00	479. 20	121. 90	107. 10	417. 20	1 077. 60	1 104. 40	96. 02	96. 10
	60. 10	479. 20	143. 00	115. 30	417. 20	1 107. 20	1 158. 40	92. 39	93. 09
	90. 89	479. 20	147. 10	115. 00	417. 20	1 092. 00	1 152. 00	91. 19	91. 83
	120. 90	479. 20	149. 50	116. 40	417. 20	1 074. 80	1 142. 80	90. 88	90. 63
	152. 48	479. 20	150. 40	115. 10	417. 20	1 072. 80	1 140. 80	90. 30	90. 60
2 060	30. 84	589. 20	113. 50	103. 20	83. 00	931. 60	951. 60	97. 88	97. 70
	60. 30	589. 20	136. 50	110. 70	83. 00	931. 60	980. 00	94. 61	94. 60
	89. 72	589. 20	148. 50	109. 60	83. 00	946. 00	1 024. 0	91. 89	91. 71
	125. 23	589. 20	146. 90	102. 30	83. 00	890. 80	971. 60	90. 84	90. 91
	153. 61	589. 20	148. 90	101. 20	83. 00	886. 00	968. 40	90. 23	90. 69
2 350	31. 57	391. 40	64. 69	56. 31	59. 50	596. 40	609. 00	97. 50	97. 71
	62. 40	391. 40	75. 20	58. 62	59. 50	604. 20	632. 40	95. 02	95. 08
	92. 60	391. 40	82. 00	62. 23	59. 50	595. 20	629. 60	93. 99	93. 97
	122. 9	391. 40	86. 28	59. 87	59. 50	589. 40	632. 20	92. 03	92. 53
	154. 7	391. 40	91. 29	58. 81	59. 50	575. 40	632. 00	90. 23	90. 11
2 650	31. 57	391. 40	62. 91	56. 05	59. 50	593. 00	606. 00	97. 95	97. 62
	62. 40	391. 40	71. 55	59. 41	59. 50	603. 40	624. 00	96. 34	96. 35
	92. 60	391. 40	76. 29	59. 20	59. 50	592. 20	624. 60	94. 67	94. 27
	122. 93	391. 40	81. 88	60. 97	59. 50	591. 60	629. 00	93. 67	93. 43
	154. 77	391. 40	85. 63	57. 26	59. 50	584. 60	629. 00	91. 51	92. 20

Table 4 Stage efficiency for stripping Nd^{3+} with 5. 5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+} when q_a: q_o= 1: 1 at n= 2 350 r/min

$q_t/(L \cdot h^{-1})$	Mass concentration/($mg \cdot L^{-1}$)						E_a	E_o
	$\rho_{a,in}$	$\rho_{a,out}$	$\rho_{a,eq}$	$\rho_{o,in}$	$\rho_{o,out}$	$\rho_{o,eq}$		
17. 75	238. 6	1 270. 00	1 304. 2	1 372. 8	159. 2	78	96. 79	93. 73
40. 39	238. 6	1 561. 20	1 673. 4	1 372. 8	189. 92	93. 08	92. 18	92. 43
85. 12	238. 6	1 526. 40	1712	1372. 8	262. 72	97. 88	87. 40	87. 07
124. 02	238. 6	1 528. 20	1 708. 8	1 372. 8	264. 28	95. 92	87. 72	86. 81
171. 53	238. 6	1 587. 80	1 810. 2	1 372. 8	277. 36	103	85. 85	86. 27
216. 57	238. 6	1 530. 6	1 765. 80	1 372. 8	278. 64	99. 4	84. 60	85. 92

Table 5 Stage efficiency for stripping Nd^{3+} with 5. 5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+} when q_a: q_o= 1: 1 at q_t= 80 L/h

$n/(r \cdot min^{-1})$	Mass concentration/($mg \cdot L^{-1}$)						E_a	E_o
	$\rho_{a,in}$	$\rho_{a,out}$	$\rho_{a,eq}$	$\rho_{o,in}$	$\rho_{o,out}$	$\rho_{o,eq}$		
1 764	238. 6	1 490. 2	1 734. 20	1 372. 8	308. 32	93. 36	83. 69	83. 20
2 058	238. 6	1 547. 8	1 760. 00	1 372. 8	268. 24	95. 52	86. 05	86. 48
2 362	238. 6	1 526. 4	1 712. 00	1 372. 8	262. 72	97. 88	87. 40	87. 07
2 646	238. 6	1 565. 4	1 748. 80	1 372. 8	230. 52	97. 92	87. 86	89. 60

shown in Tables 2 and 3 for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution when q_a: q_o = 1: 1 and q_a: q_o = 2: 1 respectively. During the extraction process, the stage efficiency is almost greater than 90%, and increases with not only decreasing the throughout of the two phases at the given rotating rate but also increasing the rotating rate at the given throughout.

Stage efficiency is also carried out for stripping Nd^{3+} with 5.5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+} when q_a: q_o = 1: 1 and the results are given in Tables 4 and 5. The conclusions in Tables 4 and 5 are in good agreement with that in Tables 2 and 3 except that the stage efficiency of stripping is less than 90%.

3.2 Three-stage centrifugal extractor cascade

The average stage efficiency of the centrifugal extractor cascade is shown in Table 6 for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution. From Table 6, it is observed that the average stage efficiency increases with not only decreasing the throughout of the two phases at the given rotating rate but alsoincreasing the rotating rate at the given throughout.

Average stage efficiency is also obtained for stripping Nd^{3+} with 5.5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+} and the experimental results are given in Table 7. The conclusion in Table 7 is in good agreement with that in Table 2 except that the stage efficiency of stripping is less than 90%.

The two stage mode of the cascade refers to the abnormal operation of the cascade, i. e. the rotor stops in one of the cascade, which often occurs in the industry. Experimental results show that the two phases can flow thoroughly even when one of the extractors stops.

The experimental results of extracting and stripping are shown in Tables 8 and 9 respectively. As expected, the trend of the extracting and stripping process in the mode is the same as that in the three-stage mode.

By comparing the data in Tables 8 and 9 with those in Tables 6 and 7, it can be seen that the average stage efficiency for two-stage mode is greater than that for three-stage mode under the same conditions. The reason is that the mixing and mass-transfer of the two phases also occur when the two phases flow through the stopped centrifugal extractor.

Table 6 Average stage efficiency for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution

N_{st}	$n/(r\cdot min^{-1})$	$q_a/(L\cdot h^{-1})$	$q_o/(L\cdot h^{-1})$	Mass concentration/$(mg\cdot L^{-1})$				E
				$\rho_{a, in}$	$\rho_{a, out}$	$\rho_{o, in}$	$\rho_{o, out}$	
3	2400	61.2	41.4	740.16	29.66	64.8	1094.4	93.6
3	1800	61.2	30	740.16	49.97	64.8	1468.8	90.6
3	2100	61.2	30	740.16	35.34	64.8	1480.3	93.5

Table 7 Average stage efficiency for stripping Nd^{3+} with 5.5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+}

N_{st}	$n/(r\cdot min^{-1})$	$q_a/(L\cdot h^{-1})$	$q_o/(L\cdot h^{-1})$	Mass concentration/$(mg\cdot L^{-1})$				E
				$\rho_{a, in}$	$\rho_{a, out}$	$\rho_{o, in}$	$\rho_{o, out}$	
3	2 100	50	57.6	69.23	1 387.50	1 292.20	123.00	60.73
3	2 400	50	50.4	69.23	1 244.00	1 292.20	51.50	84.67
3	2 400	50	38.2	69.23	1 378.50	1 292.20	50.00	88.00

Table 8 Average stage efficiency for extracting Nd^{3+} with 30% TRPO-kerosene solution from $Nd(NO_3)_3$-1mol/L HNO_3 solution

N_{st}	$n/(r\cdot min^{-1})$	$q_a/(L\cdot h^{-1})$	$q_o/(L\cdot h^{-1})$	Mass concentration/$(mg\cdot L^{-1})$				E
				$\rho_{a, in}$	$\rho_{a, out}$	$\rho_{o, in}$	$\rho_{o, out}$	
2	2 400	61.2	41.4	740.16	54.72	64.8	1 072.8	102.5
2	1 800	61.2	30	740.16	119.95	64.8	1 324.8	94.8
2	2 100	61.2	30	740.16	85.45	64.8	1 385.2	97.3

Table 9 Average stage efficiency for stripping Nd^{3+} with 5.5 mol/L HNO_3 solution from 30% TRPO-kerosene solution loaded with Nd^{3+}

N_{st}	$n/(r \cdot min^{-1})$	$q_a/(L \cdot h^{-1})$	$q_o/(L \cdot h^{-1})$	Mass concentration/$(mg \cdot L^{-1})$				E
				$\rho_{a,in}$	$\rho_{a,out}$	$\rho_{o,in}$	$\rho_{o,out}$	
2	2 400	50	38.2	4.34	69.23	1 273.00	1 292.20	113.20
2	1 800	50	38.2	4.34	69.23	1 145.00	1 292.20	192.00
2	2 400	35	35	4.3	51.85	530.00	538.11	52.89

4 CONCLUSIONS

1) The carryover of the organic phase in aqueous phase is less than 0.1% within the experimental range while the carryover of aqueous phase in the organic phase is larger than 0.5% at relatively large flow throughout and low rotation rate.

2) Both extracting and stripping experimental results show that the stage efficiency increases with not only decreasing the throughout of the two phases at the given rotating rate but also increasing the rotating rate at the given throughout.

3) The stage efficiency is larger than 90% for the extraction process, but less than 90% for the stripping process.

4) The average stage efficiency of the cascade increases with not only decreasing the total flow rate but also increasing the rotor speed in both three-stage mode and two-stage mode.

5) The average stage efficiency in two-stage mode is greater than that in three-stage mode under the same conditions due to the occurrence of the mixing and mass-transfer in the stopped centrifugal extractor.

Nomenclature

$\rho_{a,in}$ —Inlet concentration of Nd^{3+} in aqueous phase, mg/L;

$\rho_{o,in}$ —Inlet concentration of Nd^{3+} in organic phase, mg/L;

$\rho_{a,out}$ —Outlet concentration of Nd^{3+} in aqueous phase, mg/L;

$\rho_{o,out}$ —Outlet concentration of Nd^{3+} in organic phase, mg/L;

$\rho_{a,eq}$ —Equilibrium concentration of Nd^{3+} in aqueous phase, mg/L;

$\rho_{o,in}$ —Equilibrium concentration of Nd^{3+} in organic phase, mg/L;

E —Average stage efficiency, %;

E_a —Stage efficiency of aqueous phase, %;

E_o —Stage efficiency of organic phase, %;

n —Rotating rate of rotor, r/min;

N_{st} —number of stages;

q_a —Flow rate of aqueous phase, L/h;

q_o —Flow rate of organic phase, L/h;

q_t —Total flow rate of aqueous phase and organic phase, L/h;

γ —Interfacial tension, $10^{-3} N \cdot m^{-1}$;

μ_a —Viscosity of aqueous phase, Pa•m;

μ_o —Viscosity of organic phase, Pa•m;

ρ_a —Density of aqueous phase, kg/m^3;

ρ_o —Density of organic phase, kg/m^3.

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