Communication

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Extraction of Rare Earth Elements from chloride Solutions Using Mixtures of P507 and Cyanex 272

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Abstract: In this study, the extraction of rare earth elements (REEs) from chloride solutions containing mixtures of these metals with solutions of the mixtures of P507 and Cyanex 272 (1:1) of various concentrations was experimentally studied. It was shown that the distribution coefficients of all REEs decrease with increasing concentration of these metals in the initial solution, which is associated with the loading of the organic phase. The most significant improvement in the extraction is observed for heavy group of rare earth elements. The extractability of REEs enhances with increasing atomic number of the element, as is typical for the extraction of these metals with acidic organophosphorus extractants. The data obtained show that separation factors of adjacent rare earth elements mainly decrease with increasing concentration of metals in the initial aqueous solution. Increasing the concentration of the extractant mixture does not have a significant effect on the values of the adjacent REE separation factors. Analysis of the experimental results shows that two commercial products can be obtained from a concentrate of the medium-heavy group. In the first cascade, yttrium can be separated, in the second, a concentrate of dysprosium and terbium can be obtained.

Keywords: rare earth elements; chloride solutions; mixtures of P507 and Cyanex 272; extraction; separation

1. Introduction

Rare earth elements (REEs) including the lanthanides, scandium and yttrium are widely used both in traditional industries and in modern production, since they have a variety of useful and unique properties (for example, electronic, luminescent, magnetic, catalytic etc.). New applications in electronics and other industries have led to an increase in demand for REEs, as well as high purity REE materials. Owing to the similarity in the chemical and physical properties, separating REEs is a complicated and urgent problem. Recently, the number of studies on the recovery and separation of REEs by various methods, especially with the use of solvent extraction, has been growing. This is due to the fact that solvent extraction is presently one of the major techniques on industrial scale for recovery, separation and concentration of metals from aqueous solutions. Particularly, the extraction of rare earth elements with acidic organophosphorus extractants of various kinds is widely used. Among these extractants, bis(2,4,4-trimethylpentyl)phosphinic acid (Cyanex 272), di(2-ethylhexyl)phosphoric acid and 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester (P507) are commonly applied for the recovery and separation of REEs [1-7]. Sufficiently high separation factors between adjacent elements are observed in P507 extraction systems [5–9], but the separation factors for some lanthanide pairs (such as Nd/Pr, Gd/Eu, Lu/Yb) are very low. Cyanex 272 also exhibits high separation efficiency between adjacent rare earth elements; however, the extraction power of Cyanex 272 towards REEs is low owing to its high pKa value [10]. Moreover, the use of Cyanex 272 is restricted due to the poor physical characteristics of this extractant. To overcome the low extractability of rare earth elements in systems with Cyanex 272, synergistic mixtures with other extractants have been suggested [11-18]. Recently, a binary mixture of P507 and Cyanex 272 was proposed as a synergistic system for the extraction of REEs. It has been found that the separation of heavy lanthanides(III) was higher than those with using P507 [19]. Besides, stripping processes in systems involving mixtures of P507 and Cyanex 272 proceeded more easily than with P507 alone. Later, Cytec Industries developed a new extractant called Cyanex 572, which is a mixture of phosphinic (Cyanex 272) and phosphonic (P507) acids (Cytec Industries, 2015) [20]. Cyanex 572 provides efficient extraction of heavy REEs simultaneously, with the stripping of these metals from the loaded organic phases using mineral acid solutions with a lower concentration than in the P507 system [21-25]. The ability of Cyanex 572 to form complexes with metal ions is between phosphonic and phosphinic organic acids [23].

The aim of this work was to study the use of mixtures of P507 and Cyanex 272 (1:1) for the extraction and separation of rare earth elements (Ce, Pr, Nd, Sm, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu and Y).

2. Materials and Methods

The chemicals and reagents used in this study were of analytical grade and they were used without any purification.

All the extraction studies were carried out in triplicate at 298 K unless otherwise mentioned and the results indicate the average of three independent measurements.

The initial solution of REE chlorides was obtained by dissolving a concentrate of REE carbonates (Apatit Cherepovets, Vologda region) in a 10 M HCl solution. The carbonates were added to 10 M HCl until the aqueous solution was neutralized. The resulting orange precipitate was filtered off, and HCl solution was added to pH 1.5. The resulting solution of REE chlorides had the following composition (%): Ce – 1.2, Pr – 0.2, Nd – 3.4, Sm – 19.7, Eu – 4.5, Gd – 14.5, Tb – 1.5, Dy – 7.5, Ho – 1.1, Er – 2.6, Tm - 0.2, Yb - 1.1, Lu – 0.1, Y – 41.4.

Solutions of REE chlorides with concentrations from 0.05 to 0.90 mol/L were prepared from the initial solution.

A mixture of P507 (China, Luoyang Zhongda Chemical Co) and Cyanex 272 (Canada, Cytec Solvay Group) in a molar ratio of 1:1 was used as an extractant. Isopar L (fraction C_{11}–C_{13}) was used as a solvent with the addition of 15 vol. % isooctanol. For the extraction, 0.3, 0.6, 0.9 and 1.2 M solutions of extractant in isopar L were prepared. To improve the extraction ability of the mixture of P507 and Cyanex 272, a saponified extractant was used (the degree of saponification of the extractants did not exceed 40%).

The phase contact time on the orbital shaker was 30 minutes, which was sufficient to establish constant REE distribution coefficients.

After phase separation, the aqueous phase concentrations of REEs were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) using a Shimadzu ICPE-9000 with an analytical error < 5%. The REE concentrations in the organic phase were determined as follows. An aliquot of the organic phase was burned in a muffle furnace at 650°C, the resulting residue was dissolved in a 6 M nitric acid solution on heating, and then the solution (after neutralization) was transferred into a volumetric flask and the REEs were analyzed by ICP-OES.

The total concentration of REE chlorides in the initial solutions and aqueous phases after extraction was determined via titration with a standard solution of EDTA at pH 5.6, using xylenol orange as an indicator.

The distribution ratio (D) was determined as the ratio of the concentration of REE in the organic phase to that in the aqueous phase, and was calculated according to the following equation:

$$D = C_o/C_{aq}, \qquad (1)$$

where C_o and C_{aq} are the equilibrium concentrations of REE in the organic and aqueous phase, respectively. The separation factor (β) characterizes the separation degree between two rare earth elements (E^1 and E^2) in the extraction system and was calculated according to the following equation:

$$\beta = D(E^1)/D(E^2) \qquad (2)$$

3. Results and Discussion

The extraction of rare earth elements from chloride solutions containing mixtures of these metals, the concentration of which varied in the range of 0.05 – 0.9 mol/L, with solutions of a mixture of P507 and Cyanex 272 (1:1) of various concentrations was studied. The calculated distribution coefficients for each metal are given in Tables 1-4. From the data obtained it follows that the distribution coefficients of all REEs decrease with increasing concentration of these metals in the initial solution, which is associated with the loading of the organic phase and a decrease in the free concentration of the extractant mixture. An increase in the initial concentration of the extractant mixture leads to an increase in the distribution coefficients of REEs. The most significant improvement in the extraction is observed for heavy group of rare earth elements (Ho, Er, Tm, Yb, Lu). It should be noted that in a wide range of initial concentrations of REE chlorides, the distribution coefficient of yttrium exceeds the distribution coefficient of dysprosium by 2-4 times, depending on the equilibrium concentration in the aqueous phase. The extractability of rare earth elements enhances with increasing atomic number of the element, as is typical for the extraction of these metals with acidic organophosphorus extractants.

Table 1. Distribution coefficients of REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.3 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$.	Distribution coefficients											
mol/L	Ce	Nd	Sm	Gd	Tb	Dy	Ho	Er	Tm	Yb	Y	∑REE
0.050	-	-	0.160	-	-	0.180	0.460	0.770	-	-	0.470	0.340
0.100	-	0.120	-	-	-	0.081	0.220	0.360	1.44	4.21	0.220	0.130
0.160	-	-	-	-	-	0.048	0.130	0.220	0.930	2.50	0.130	0.083
0.210	0.089	0.035	-	-	-	0.040	0.100	0.160	0.630	1.93	0.100	0.068
0.330	0.068	0.078	0.0095	-	0.235	0.024	0.063	0.100	0.410	1.11	0.062	0.046
0.410	-	-	0.0016	-	-	0.019	0.049	0.081	0.300	0.860	0.048	0.032
0.520	0.020	0.015	0.0028	-	-	0.017	0.041	0.065	0.230	0.620	0.036	0.026
0.630	-	-	0.0018	0.0026	-	0.015	0.035	0.054	0.190	0.580	0.036	0.024
0.990	0.047	0.012	0.0025	0.0024	-	0.015	0.027	0.043	0.130	0.350	0.026	0.020

Table 2. Distribution coefficients of REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.6 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$	Distribution coefficients											
. mol/L	Ce	Nd	Sm	Gd	Tb	Dy	Ho	Er	Tm	Yb	Y	∑REE
0.050	0.190	0.160	0.048	0.150	0.790	2.24	-	12.6	-	-	7.82	1.29
0.110	0.074	-	0.008	0.025	0.130	0.320	0.590	1.55	-	-	1.01	0.430
0.120	0.370	-	0.008	0.025	0.130	0.300	0.530	1.54	-	-	0.990	0.430
0.220	0.120	-	0.003	0.0090	0.044	0.110	0.190	0.530	1.27	6.03	0.340	0.190
0.340	0.088	-	0.002	0.0058	0.026	0.060	0.100	0.280	0.760	3.14	0.190	0.110
0.430	-	-	0.003	0.0059	0.023	0.047	0.082	0.220	0.570	2.41	0.140	0.085

0.550	-	-	0.003	0.0061	0.020	0.041	0.067	0.170	0.450	1.78	0.110	0.069
0.640	-	0.007	0.004	0.0063	0.020	0.036	0.055	0.140	0.360	1.44	0.093	0.060
0.960	-	-	0.005	0.0080	0.023	0.036	0.052	0.110	0.260	0.920	0.074	0.049

Table 3. Distribution coefficients of REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.9 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$. mol/L	Distribution coefficients													
	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Y	∑REE
0.050	0.990	0.180	1.31	3.33	5.10	-	83.6	-	-	-	-	-	-	6.04
0.090	0.560	0.020	0.021	0.070	0.110	0.540	1.65	2.97	9.36	19.4	98.5	-	5.18	1.14
0.130	0.880	0.022	0.017	-	0.040	0.160	0.470	0.790	2.52	6.88	24.2	-	1.52	0.590
0.180	0.520	0.017	0.011	-	0.023	0.100	0.300	0.530	1.51	4.31	15.5	9.90	0.950	0.430
0.270	0.330	-	0.008	-	0.013	0.040	0.150	0.250	0.700	2.39	8.87	6.63	0.450	0.240
0.370	0.220	-	0.006	-	0.011	0.040	0.110	0.160	0.490	1.44	4.78	4.50	0.310	0.180
0.450	0.230	0.010	0.006	-	0.010	0.030	0.080	0.120	0.370	1.06	3.49	3.50	0.250	0.140
0.530	0.190	0.006	0.008	-	0.011	0.030	0.080	0.110	0.310	0.900	2.95	3.19	0.200	0.120
0.810	0.140	0.006	0.009	0.010	0.013	0.040	0.070	0.080	0.210	0.490	1.61	1.92	0.130	0.090

Table 4. Distribution coefficients of REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 1.2 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$. mol/L	Distribution coefficients													
	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Y	∑REE
0.090	1.83	0.075	0.340	0.800	1.21	8.31	24.5	-	-	-	-	-	86.7	-
0.140	0.920	0.027	0.029	0.085	0.130	0.600	1.79	3.15	9.60	19.2	93.6	-	5.74	2.99
0.170	0.830	0.023	0.021	0.033	0.051	0.220	0.660	1.20	3.42	9.90	33.4	-	2.14	1.23

0.260	0.410	0.015	0.011	-	0.024	0.120	0.290	0.480	1.40	3.97	13.4	8.80	0.920	0.740
0.340	0.260	0.010	0.009	-	0.017	0.063	0.170	0.260	0.770	2.20	7.54	5.97	0.540	0.420
0.450	0.260	0.008	0.010	0.013	0.017	0.063	0.150	0.220	0.600	1.79	5.71	4.99	0.430	0.270
0.520	0.190	0.039	0.010	0.013	0.016	0.051	0.110	0.160	0.450	1.20	3.96	3.83	0.300	0.240
0.800	0.120	0.011	0.011	0.016	0.019	0.047	0.094	0.120	0.270	0.690	2.16	2.39	0.190	-

The obtained REE distribution coefficients (Tables 1-4) were used to calculate the separation factors of adjacent REEs depending on the initial concentration of these elements in the initial aqueous solutions in the extraction with solutions of the mixtures of P507 and Cyanex 272 of various concentrations. The results obtained are shown in Figure 1 and in Tables 5-8.

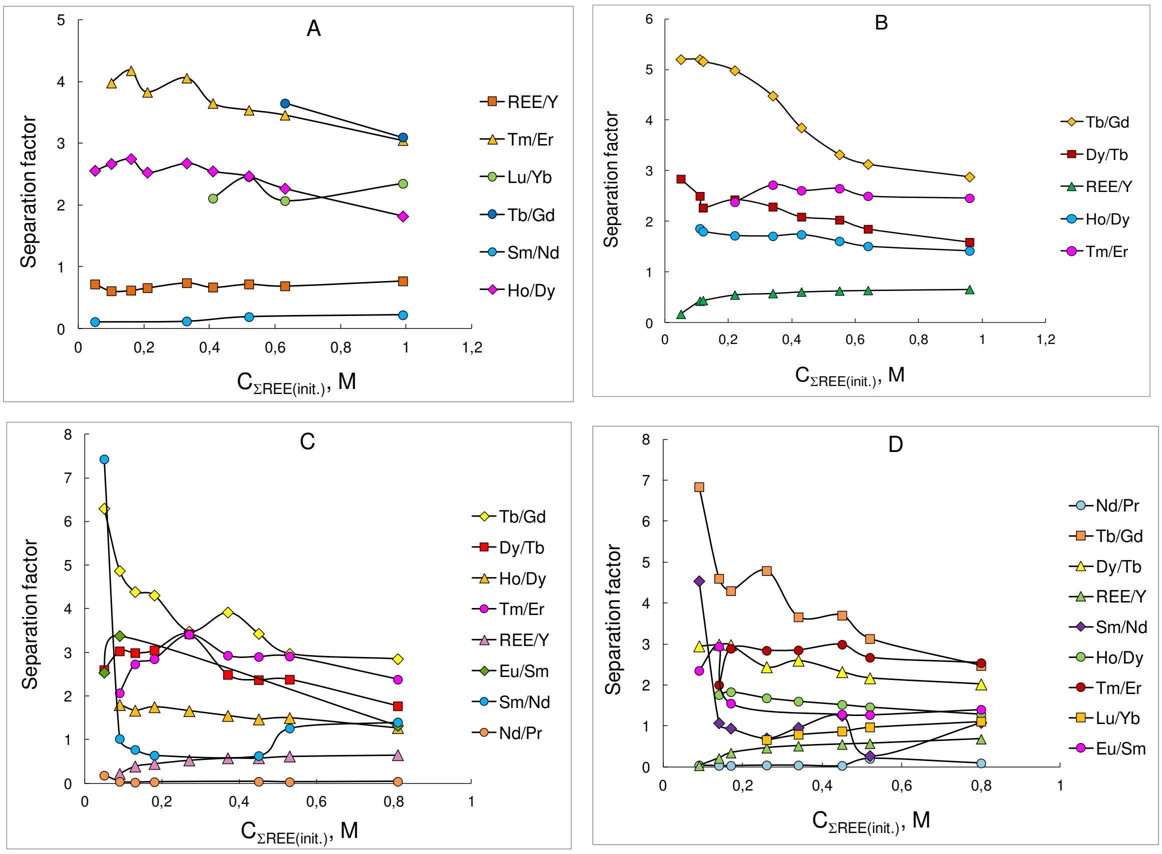

Figure 1. Dependency of separation factor of REE pairs on the initial REE concentration in aqueous solution at extractant concentrations, mol/L: (A) 0.3; (B) 0.6; (C) 0.9; (D) 1.2.

Table 5. Separation factors of adjacent REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.3 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$, mol/L	Separation factor						
	Sm/Nd	Tb/Gd	Dy/Tb	Ho/Dy	Tm/Er	Lu/Yb	∑REE/Y
0.050	0.110	-	1.58	2.56	-	-	-

0.100	-	-	-	2.67	3.98	-	0.610
0.160	-	-	-	2.75	4.18	-	0.620
0.210	-	-	-	2.53	3.83	-	0.660
0.330	0.120	-	-	2.68	4.06	-	0.740
0.410	-	-	-	2.55	3.65	2.11	0.670
0.520	0.190	-	-	2.47	3.54	2.46	0.720
0.630	-	3.65	1.64	2.27	3.46	2.07	0.690
0.990	0.220	3.10	2.00	1.82	3.05	2.35	0.770

Table 6. Separation factors of adjacent REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.6 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$.	Separation factor					
mol/L	Sm/Nd	Tb/Gd	Dy/Tb	Ho/Dy	Tm/Er	∑REE/Y
0.050	0.300	5.20	2.84	-	-	0.170
0.110	-	5.20	2.50	1.86	-	0.430
0.120	-	5.16	2.27	1.80	-	0.440
0.220	-	4.98	2.43	1.72	2.38	0.550
0.340	-	4.48	2.29	1.71	2.72	0.580
0.430	-	3.85	2.09	1.74	2.61	0.610
0.550	-	3.32	2.03	1.61	2.65	0.630
0.640	0.530	3.13	1.85	1.51	2.50	0.640
0.960	-	2.88	1.59	1.42	2.46	0.660

Table 7. Separation factors of adjacent REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 0.9 M solution of a mixture of P507 and Cyanex 272.

$C_{REE(init.)}$.	Separation factor					
mol/L	Sm/Nd	Tb/Gd	Dy/Tb	Ho/Dy	Tm/Er	∑REE/Y
0.050	7.43	6.30	2.60	-	-	-
0.090	1.02	4.87	3.03	1.80	2.07	0.220
0.130	0.770	4.39	2.99	1.67	2.73	0.390
0.180	0.640	4.31	3.05	1.75	2.85	0.450
0.270	-	3.48	3.42	1.66	3.41	0.530
0.370	-	3.92	2.49	1.55	2.93	0.580
0.450	0.630	3.43	2.37	1.47	2.90	0.580
0.530	1.26	2.97	2.38	1.50	2.91	0.620
0.810	1.44	2.85	1.77	1.27	2.38	0.650

Table 8. Separation factors of adjacent REEs depending on the initial concentration of rare earth elements in aqueous solutions in the extraction with a 1.2 M solution of a mixture of P507 and Cyanex 272.

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$C_{REE(init.)}$,	Separation factor							
mol/L	Sm/Nd	Eu/Sm	Tb/Gd	Dy/Tb	Ho/Dy	Tm/Er	Lu/Yb	∑REE/Y
0.040	-	-	-	-	-	-	-	-
0.090	4.54	2.35	6.84	2.95	-	-	-	-
0.140	1.07	2.94	4.60	2.99	1.76	2.00	-	0.210
0.170	0.940	1.55	4.30	2.98	1.83	2.89	-	0.340
0.260	0.700	-	4.79	2.44	1.68	2.84	0.660	0.460
0.340	0.960	-	3.66	2.60	1.60	2.85	0.790	0.510
0.450	1.25	1.28	3.70	2.32	1.52	2.99	0.870	0.550
0.520	0.270	1.27	3.13	2.17	1.46	2.67	0.970	0.570
0.800	1.08	1.40	2.47	2.02	1.29	2.54	1.11	0.680

As can be seen from the data obtained the separation factors of adjacent rare earth elements mainly decrease with increasing concentration of metals in the initial aqueous solution. However, the separation factor of the sum of REEs to yttrium increases with increasing concentration of metals in the initial aqueous solution. The most significant separation factors are observed for pairs of medium-heavy (Tb/Gd) and heavy group of metals (Tm/Er, Dy/Tb). When using a 0.9 M solution of a mixture of P507 and Cyanex 272, the dysprosium/terbium separation coefficient reaches a value of 3.42, and the thulium/erbium separation coefficient reaches 3.41. Increasing the concentration of the extractant mixture does not have a significant effect on the values of the adjacent REEs separation factors as well as the separation factors of the sum of REEs to yttrium.

Analysis of distribution coefficients and separation factors of rare earth elements (Tables 1-8) shows that two commercial products can be obtained from a concentrate of the medium-heavy group. In the first cascade, yttrium can be separated, in the second, a concentrate of dysprosium and terbium can be obtained. The optimal extractant concentration for the extraction and separation of rare earth elements using a mixture of extractants is 0.6 mol/L. At lower concentrations of the extractant mixture, the REE distribution coefficients are not high enough, and at higher concentrations, precipitation may occur, which is unacceptable when using technological processes.

5. Conclusions

In this study, the data on the extraction of rare earth elements from chloride solutions with solutions of a mixture of P507 and Cyanex 272 (1:1) were obtained. It has been shown that the distribution coefficients of all REEs decrease with increasing concentration of these metals in the initial solution, while with increasing the initial concentration of the extractant mixture, the distribution coefficients of REEs increase. The most significant improvement in the extraction is observed for heavy group of rare earth elements. The separation factors of adjacent rare earth elements mainly decrease with increasing concentration of metals in the initial aqueous solution. Based on the experimental results obtained, it was concluded that two commercial products can be obtained from a concentrate of the medium-heavy group. Experimental data can be used in mathematical modeling and optimization of extraction cascades to obtain individual rare earth elements using a mixture of P507 and Cyanex 272.

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