EXTRACTION AND SEPARATION STUDIES OF Ce (III) FROM ACIDIC NITRATE MEDIUM WITH BINARY MIXTURE OF ALIQUAT 336 AND CYANEX 921 IN KEROSENE

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ABSTRACT

In the present study, the extraction of Ce(III) from acidic nitrate medium with mixture of Aliquat 336 and neutral organophosphorus extractant (Cyanex 921) in kerosene has been investigated. Synergistic effects were observed for all the diluents and at all temperature except at 328K. According to the various extraction effects, the separation possibilities of Nd(III) and Ce(III) were found to be maximum using the binary mixture. The extraction mechanisms have been studied with the methods of slope analysis in the synergistic systems. The thermodynamic functions have also been determined from the extracted equilibrium constants.

Keywords: extraction, cerium(III), Aliquat 336, Cyanex 921, separation.

INTRODUCTION

Liquid-liquid extraction has been widely used as a simple, versatile technique for the recovery and separation of rare earths [1 - 4]. Rare earths are specific and important due to their applications in various fields [5 - 6]. Solvent extraction has been widely used for obtaining pure metals under different extraction conditions [7 - 10]. Synergistic extraction systems have been employed for the separation studies of rare earths using various extractants [11 - 13]. Replacing a single extractant by a mixture of extractants may give rise to enhancement in the extraction and better selectivity of extraction and separation studies [14]. This phenomenon “synergism” refers to an increase in the lipophilicity of the extracted metal complexes arising from the replacement of residual water in the inner coordination sphere or to occupy open coordination sites by neutral electron donors, called synergists. This may be attributed to the replacement of residual water molecules from the coordinately unsaturated metal complex or expansion of coordination sphere to accommodate the synergist [15]. Furthermore, synergistic extraction of metal ions has various advantages over ordinary solvent extraction system, such as enhancement of extractability, widening the optimum extraction pH range, stabilization of extracted species by forming an adduct. Extraction of some lanthanides with ternary and quaternary mixture of extractants has been studied [16]. Atanassova et al. have reported that the separation factors between adjacent rare earths are small in the synergistic extraction using binary mixture of the nonyltrifluoroacetone and quaternary ammonium salts in chloride and perchlorate media [17]. Synergism was observed in the extraction of Nd (III) from chloride solution with mixture of Alamine 308 (triiso-octylamine) and Cyanex 302 (bis-2,4,4-trimethylpentyl)monothiophilosphinic acid in kerosene [18]. Growing attention is being paid to the development of new synergistic systems to improve the extractability and selectivity of metal ions. The addition of neutral organophosphorus extractants to amine extractants has great potential for this purpose. Therefore, it will be of great interest to develop new mixing systems based on these two kinds of extractants [19]. The synergic extrac-
tion of Sm and Gd using a mixture of tri-butyl phosphate and Aliquat336 has been reported. The separation factor of Sm and Gd was found to be larger than 3 [20]. Ionic liquids have attracted attention as green solvents due to their unique properties such as high thermal stability, negligible vapour pressure and non-flammability [21-22]. Banda et al. have reported the separation of Ce as a precipitate using NaClO₄ and separation of La from Pr and Nd in chloride medium with cationic extractants (PC 88A, D2EHPA, Cyanex 272, LIX 63) and anionic extractants (Alamine 336 and Aliquat 336) [23]. The extraction of cerium (IV) from sulfuric acid medium using bifunctional ionic liquid extractants [A336][P507], [A336][P204] and [A336][C272] in n-heptane have been investigated by Zhang et al. [24]. The extraction efficiency order followed as: Ce(IV) > Th(IV) > Ce(III). Ionic liquid, 1-butyl-3-methylimidazolium hexafluorophosphate ([C₄mim][PF₆]) and tri-n-octylphosphine oxide (TOPO) as extractant in the extraction of Eu(III) and Th(IV) ions from nitrate media was investigated [25].

In the present study, trivalent cerium extraction from acidic nitrate medium has been investigated in detail using mixture of Aliquat 336 and Cyanex 921 in kerosene. The influence of different experimental parameters such as aqueous phase acid concentration, extractant molarity, temperature, diluents, phase volume ratio and nature of stripping agents on the extraction and stripping behaviour of Ce(III) using binary mixture has been studied. Synergistic aspect of extraction has been explained with special reference to temperature and nature of diluent. The separation of Ce (III) and La/Pr/Nd from acidic nitrate medium has also been studied with binary mixture of extractants.

EXPERIMENTAL

Materials

Cerium nitrate (Merck) was used to prepare the metal stock solution. Cyanex 921 (Cytec, Canada) and Aliquat 336 (Sigma Aldrich) were used as received. As the metal nitrate [Aliquat 336] [NO₃] was prepared by equilibrating a solution of Aliquat 336 in kerosene with an equal volume of aqueous KNO₃ solution (3M) for one hour followed by separation of the organic phase. Then it was used for extraction studies. Commercially available kerosene (SD-Fine Chemicals Ltd.) was used as organic phase diluent. All the chemicals used in this study are of analytical reagent grade.

Methods

A feed solution of Ce(III) (0.01M) was prepared by dissolving a required amount of Ce (NO₃)₃ in double distilled water. A small amount of concentrated HNO₃ was added to suppress hydrolysis of Ce(III). The organic phase extractant solution was prepared in kerosene. For extraction experiments an equal volume of aqueous (required concentration) and organic phases (10 ml) have been shaken for five minutes using a mechanical shaker at room temperature. After separation of the two phases, the concentration of cerium in the aqueous phase before and after the extraction was measured spectrophotometrically using Arsenazo (III) by an UV-Vis spectrophotometer (ELICO). The cerium content in the organic phase was calculated by material balance. The distribution ratio (D) was determined from the ratio of Ce (III) concentration in the organic phase to that in the aqueous phase after extraction. The percentage of extraction was calculated as % E = [100D/(D + V₀aq/V₀org)] where V₀aq = volume of the aqueous phase and V₀org = volume of the organic phase. Stripping experiments were carried out by mixing equal volumes of loaded organic phase and the stripping agent. The extent of synergism is expressed in terms of ∆D [Dₘᵢₓ - (D₁₊D₂)] and synergistic coefficient (S.C) = log (Dₘᵢₓ/D₁ + D₂), where Dₘᵢₓ, D₁ and D₂ are the distribution ratios of Ce(III) using bynary mixture, Aliquat 336 and Cyanex 921, respectively. For separation studies, the metal ion concentrations in the aqueous phase before and after the extraction were determined using ICP-AES (ULTIMA HORIBAJOBIN YVON).

RESULTS AND DISCUSSION

Effect of shaking time

To determine the effect of shaking time on the extraction of 0.001 M Ce(III) from 0.01 M HNO₃ and 1.0 M KNO₃, a binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene was used. Extraction tests were performed by varying the shaking time from one minute to forty minutes.

Under the above experimental condition, it was observed that the extraction percentage of Ce (III) increased from 35.6 % to 50.7 % when time increased from one minute to five minutes and then it became constant with further increase in time. Therefore, for all experiments the shaking time was maintained equal to five minutes.
Effect of aqueous phase acid concentration

To know the influence of aqueous phase H\(^+\) ions concentration on the extraction of Ce(III), experiments were carried out using 0.001 M Ce(III) from 1.0 M KNO\(_3\) at various HNO\(_3\) concentrations in the range of 0.001 - 1.0 M with binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene. The extraction percentage was decreased from 54.9 % to 5.7 % when the nitric acid concentration increased from 0.001 M to 1.0 M (Fig. 1). The increase in nitric acid concentration leads to a competition between the acid and the metal ion for the extraction by Cyanex 921 present in the mixture which might be responsible for less Ce(III) extraction. At higher HNO\(_3\) concentration the extraction of acid seems to be favoured compared to metal extraction.

Effect of nitrate ion concentration

Extractions were performed using Ce(III) at various concentrations of KNO\(_3\) from 0.5 M to 2.0 M with a constant HNO\(_3\) concentration (0.01 M) using binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene. The experimental results showed that the extraction percentage increased from 37.5 % to 79.6 % when the concentration of nitrate ions increased from 0.5 M to 2.0 M. The percentage of extraction increases with increase in the concentration of nitrate ions due to a salting out effect which ensures the removal of water of hydration and make the metal species more lipophilic [25]. Plotting of distribution ratio (D) against the nitrate ions concentration as a log-log relation (Fig. 2), gives a straight line with slope of 2.77 ≈ 3.0 indicating the participation of three nitrate ions in the extraction reaction.

Effect of extractant concentration

The extraction of 0.001 M Ce(III) from 0.01 M HNO\(_3\) and 1.0 M KNO\(_3\) was studied by varying Aliquat 336 concentration in the range from 0.01 M to 0.6 M, keeping the concentration of Cyanex 921 constant (0.04 M) in the organic phase. The extraction of Ce(III) was increased from 22.5 % to 59.7 % when the Aliquat 336 concentration increased from 0.01 M to 0.1 M and then decreased to 10.7 % with further increase in Aliquat 336 concentration. Initially extraction increases due

![Fig. 1. Effect of nitric acid concentration on the extraction of 0.001 M Ce(III) from 1.0 M KNO\(_3\) using binary mixtures of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene.](image1)

![Fig. 2. Plot of log D\(_{\text{mix}}\) versus log [NO\(_3\)]\(^-\), for the extraction of 0.001 M Ce(III) from 0.01 M HNO\(_3\) using binary mixtures of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene.](image2)

![Fig. 3. Plot of log D\(_{\text{mix}}\) versus log [Aliquat 336] for the extraction of 0.001 M Ce(III) from 0.01 M HNO\(_3\) and 1.0 M KNO\(_3\) using binary mixture of Aliquat 336 and Cyanex 921 in kerosene.](image3)
to the extraction of mixed complex of cerium with the binary mixture but at higher Aliquat 336 concentration an antagonistic effect is observed due to the competition between the quarternary ammonium salt and neutral donor for the metal ion at higher Aliquat 336 molarity. The log-log plot of $D_{\text{mix}}$ versus concentration of Aliquat 336 (Fig. 3) produced a straight-line with a slope of 0.735 $\approx$ 1.0 indicating first power dependence on Aliquat 336 molarity. The effect of Cyanex 921 concentration was examined by varying it in the range 0.04 - 0.4 M for the extraction of Ce(III) keeping the Aliquat 336 molarity (0.05 M) in kerosene. It was observed that the extraction percentage was increased from 50.7 % to 99.3 % when the concentration of Cyanex 921 increased from 0.04 M to 0.4 M. The plot of log $D_{\text{mix}}$ versus log [Cyanex 921] (Fig. 4) yielding a slope of 2.28 $\approx$ 2.3 indicates the presence of two molecules of Cyanex 921 in the extracted metal complex. Synergistic effect (enhancement) was observed when Cyanex 921 concentration increased from 0.08 M to 0.4 M which may be attributed to the solvation of Ce-Aliquat 336 complex. Antagonistic effect was observed with 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene. The plot of synergistic coefficient (S.C.) versus [Aliquat 336/Cyanex 921] (Fig. 5) indicates that increase in the extraction was observed using binary mixture of 0.05 M Aliquat 336 and varied Cyanex 921 molarity in kerosene as extractant for Ce(III).

**Extraction equilibrium**

From the above slope analysis results, the cerium extraction mechanism from acidic nitrate solution using binary mixture of [Aliquat 336] [NO$_3$], (R$_3$R$'$N$^+$ NO$_3^-$) and Cyanex 921 at higher Cyanex 921 concentration in kerosene, can be presented as

$$\text{Ce}^{3+} (\text{aq}) + 3 \text{NO}_3^- (\text{aq}) \rightarrow \text{R}_3\text{R}'\text{N}^+\text{Ce}(\text{NO}_3)_4^- + 2\text{Cyanex 921}_{(\text{org})}$$

(1)

The distribution ratio ($D$) is given as

$$D_{\text{mix}} = \frac{[\text{R}_3\text{R}'\text{N}^+\text{Ce}(\text{NO}_3)_4^-; 2\text{Cyanex 921}_{(\text{org})}]}{\text{[Ce}^{3+}]_{(\text{aq})}}$$

(2)

The extraction equilibrium constant is expressed as

$$K_{\text{eq}} = \frac{D_{\text{mix}}[\text{NO}_3^-]^3}{[\text{R}_3\text{R}'\text{N}^+\text{Ce}(\text{NO}_3)_4^-][\text{Cyanex 921}]^2_{(\text{org})}}$$

(3)

Fig. 5. Plot of S.C versus [Cyanex 921/Aliquat 336], M for the extraction of 0.001 M Ce(III) from 0.01 M HNO$_3$ and 1.0 M KNO$_3$ using binary mixture of Aliquat 336 and Cyanex 921 in kerosene.
Effect of metal ion concentration

The metal ion concentration was varied from 0.0005 to 0.008 M, keeping other parameters constant, i.e 0.01 M HNO₃ and 1.0 M KNO₃ in aqueous phase solution using binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene. The extraction percentage was found to increase from 38.6 % to 63.8 % (0.0005 - 0.002 M), and then decreased from 63.8 % to 54.3 % when metal ion concentration was increased from 0.002 M to 0.008 M (Fig. 6). The decrease in extraction of metal at its higher molarity may be because of the saturation of extractant with metal thus reaching its maximum loading capacity [26].

Effect of temperature

The extraction process depends greatly on temperature as it usually involves diffusion and mass transfer mechanism. The effect of temperature on extraction of 0.001 M Ce(III) from 0.01 M HNO₃ and 1.0 M KNO₃ using Aliquat 336, Cyanex 921 and their binary mixture in kerosene, was studied in the range 25ºC to 55ºC (298 K - 328 K). It was observed that the percentage of the extraction increased from 0.9 % to 11.5 % when 0.05 M Aliquat 336 is used as an extractant. The percentage of the extraction of Ce(III) was decreased from 50.3 % to 23.7 % when 0.04 M Cyanex 921 is used as an extractant. But in case of binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921, the percentage of the extraction of Ce(III) was decreased from 50.7 % to 27.0 % when temperature increased from 298 K to 328 K. Synergistic effect was observed when the temperature increased from 298 K to 328 K (Table 1). Antagonistic effect was marked at 328 K. The Vant Hoff equation was used to determine thermodynamic properties (∆H° and ∆S°) of the above studied extraction system.

\[ \log K_{eq} = -\Delta H^\circ /2.303RT + \Delta S^\circ / 2.303R \]  

(4)

From Fig. 7, ∆H° and ∆S° were calculated to be −145.3 kJ mol⁻¹ and −330.7 J K⁻¹ mol⁻¹ for the system under study. The standard enthalpy and entropy changes

<table>
<thead>
<tr>
<th>Temperature, Kelvin</th>
<th>D₁</th>
<th>D₂</th>
<th>Dₘixa</th>
<th>ΔD</th>
<th>S.C</th>
</tr>
</thead>
<tbody>
<tr>
<td>298</td>
<td>0.01</td>
<td>1.01</td>
<td>1.03</td>
<td>0.01</td>
<td>0.004</td>
</tr>
<tr>
<td>308</td>
<td>0.02</td>
<td>0.74</td>
<td>0.85</td>
<td>0.09</td>
<td>0.049</td>
</tr>
<tr>
<td>313</td>
<td>0.06</td>
<td>0.57</td>
<td>0.67</td>
<td>0.04</td>
<td>0.025</td>
</tr>
<tr>
<td>318</td>
<td>0.08</td>
<td>0.42</td>
<td>0.58</td>
<td>0.08</td>
<td>0.064</td>
</tr>
<tr>
<td>323</td>
<td>0.09</td>
<td>0.34</td>
<td>0.49</td>
<td>0.06</td>
<td>0.056</td>
</tr>
<tr>
<td>328</td>
<td>0.13</td>
<td>0.31</td>
<td>0.37</td>
<td>-0.07</td>
<td>-0.075</td>
</tr>
</tbody>
</table>

Table 1. Effect of the temperature on the extraction of 0.001M Ce(III) from 1.0 M KNO₃ and 0.01 M HNO₃ using 0.05 M Aliquat 336 (D₁) and 0.04 M Cyanex 921(D₂) and their binary mixture(Dₘixa) in kerosene.
were negative due to the evolution of heat in the extrac-
tion process and formation of a stable complex which
restricts the randomness of the extraction system.

**Effect of O/A phase volume ratio**
The tendency of increasing extraction of 0.001 M
Ce(III) from 0.01 M HNO₃ and 1.0 M KNO₃
by binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene
was studied by extraction experiments using 0.05 M Aliquat 336, 0.04 M Cyanex 921 and their binary mixture in different organic solvents. Various organic solvents such as benzene, toluene, kerosene, xylene, chloroform, carbon tetrachloride were used as diluents. It was observed that the extraction of Ce(III) was maximum when xylene was used as diluent in both 0.05 Aliquat 336 and binary mixture of 0.05 M Aliquat 336 and 0.04 M Cyanex 921. But in case of 0.04 M Cyanex 921, the extraction was maximum when kerosene was used as diluent. This concludes that the extraction process of cerium is affected by the nature of diluent. However, kerosene was used as a diluent for the above extractants throughout the extraction studies of cerium for low cost and ready availability. Synergism was observed in case of all diluents. Maximum S.C. was estimated for CCl₄ (Table 2).

**Effect of salts**
The extraction of 0.001 M Ce(III) from 0.01 M HNO₃ using binary mixture of 0.05 M Aliquat 336 and

Table 2. Effect of diluents on the extraction of 0.001 M Ce(III) from 1.0 M KNO₃ and 0.01 M HNO₃
using 0.05 M Aliquat 336 (D₁) and 0.04 M Cyanex 921(D₂) and their binary mixture (D_mix) in kerosene.

<table>
<thead>
<tr>
<th>Diluents</th>
<th>D₁</th>
<th>D₂</th>
<th>D_mix</th>
<th>ΔD</th>
<th>S.C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kerosene</td>
<td>0.01</td>
<td>1.01</td>
<td>1.03</td>
<td>0.01</td>
<td>0.004</td>
</tr>
<tr>
<td>Benzene</td>
<td>0.0</td>
<td>0.75</td>
<td>0.84</td>
<td>0.09</td>
<td>0.049</td>
</tr>
<tr>
<td>Xylene</td>
<td>0.02</td>
<td>0.69</td>
<td>1.18</td>
<td>0.47</td>
<td>0.22</td>
</tr>
<tr>
<td>Toluene</td>
<td>0.04</td>
<td>0.63</td>
<td>1.24</td>
<td>0.57</td>
<td>0.27</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>0.0</td>
<td>0.16</td>
<td>0.29</td>
<td>0.13</td>
<td>0.26</td>
</tr>
<tr>
<td>Chloroform</td>
<td>0.0</td>
<td>0.07</td>
<td>0.12</td>
<td>0.05</td>
<td>0.21</td>
</tr>
</tbody>
</table>
0.04 M Cyanex 921 in kerosene was studied by taking different nitrate salt such as KNO₃, NH₄NO₃ and NaNO₃ in the aqueous phase with varying concentration (0.5 - 2.0 M). The percentage of extraction of Ce(III) was increased from 41.8 % to 80.8 %, in the case of sodium nitrate, from 39.0 % to 79.9 % for ammonium nitrate and from 37.5 % to 79.6 %, in case of potassium nitrate. The increasing order of extraction in various salts follows the order KNO₃ < NH₄NO₃ < NaNO₃. The extraction was increased in each case when the nitrate salt concentrations increase from 0.5 M to 2.0 M (Fig. 8). This is due to salting out effect.

**Separation of Cerium(III) from binary mixtures**

The separation of lanthanoids using binary mixture of Aliquat 336 and Cyanex 921 can be assessed by the separation factors calculated as a ratio of the distribution

![Fig. 8. Plot of % E versus [NO₃⁻], M on the extraction of 0.001 M Ce(III) from 0.01M HNO₃ and 1.0 M KNO₃ using binary mixtures of 0.05 M Aliquat 336 and 0.04 M Cyanex 921 in kerosene.](image)

Table 3. Distribution ratios (D) and Separation factors (β) of light rare earths (Ce, La, Pr and Nd) using binary mixture 0.05 mol/L Aliquat 336 and of 0.4 mol/L Cyanex 921 in kerosene.

| Rare earth metal(III) added to Ce(III) | Concentration, M | D<sub>Ce</sub> | D<sub>La</sub> | β (D<sub>Ce</sub>/D<sub>La</sub>)
--- | --- | --- | --- | --- |
| La | 0.0005 | 119.0 | 28.28 | 4.20 |
| | 0.0010 | 84.34 | 31.68 | 2.66 |
| | 0.0015 | 66.91 | 34.26 | 1.95 |
| | 0.0020 | 49.77 | 39.15 | 1.27 |
| Nd | 0.0005 | D<sub>Ce</sub> | D<sub>Nd</sub> | β (D<sub>Nd</sub>/D<sub>Ce</sub>) |
| | 0.0010 | 58.15 | 92.1 | 1.58 |
| | 0.0015 | 45.16 | 122.3 | 2.71 |
| | 0.0020 | 38.41 | 145.8 | 3.82 |
| | 0.0020 | 29.67 | 195.6 | 6.63 |
| Pr | 0.0005 | D<sub>Ce</sub> | D<sub>Pr</sub> | β (D<sub>Pr</sub>/D<sub>Ce</sub>) |
| | 0.0010 | 87.67 | 144.2 | 1.65 |
| | 0.0015 | 71.22 | 147.3 | 2.15 |
| | 0.0020 | 52.6 | 179.2 | 3.43 |
| | 0.0020 | 39.0 | 196.9 | 5.14 |
coefficients of two adjacent lanthanoids. The separation of Ce(III) from La(III)/Pr(III)/Nd(III) was carried out by using the mixture containing Ce(III)/La(III), Ce(III)/Pr(III), Ce(III)/Nd(III) in 0.01 M HNO₃ and 1.0 M KNO₃ with binary mixture of 0.05 M Aliquat 336 and Cyanex 921 in kerosene. Thus, it was observed that the extraction of Ce(III) decreased when the La(III) concentration increased from 0.005 M to 0.002 M. Data shown in Table 3 reveals that separation factors for Ce-La decrease with the increase in concentration of La whereas for Nd-Ce and Pr-Ce they increase with increase in Nd/Pr concentration. Highest separation factor for Nd-Ce was found to be 6.63.

**Stripping**

Reuse of the solvent can only be done after the back extraction of the metal from the loaded organic phase. Hence, the loaded organic phase containing 138 mg/L of cerium in a mixture of 0.05 M Aliquat 336 and 0.4 M Cyanex 921 was stripped off the metal using various concentrations of Na₂CO₃, NaOH, HCl and H₂SO₄ in equal phase ratio (1:1) at room temperature (298K) to find out a suitable stripping agent. Then aqueous phase was analyzed for the Ce(III) concentration. With Na₂CO₃ and NaOH, the turbidity appeared inhibiting separation of phases. Stripping was 100 % when 0.2 M HCl, 0.8 M H₂SO₄ were used as stripping agents (Fig. 9).

**CONCLUSIONS**

The distribution data for the extraction of 0.001 M Ce(III) from 0.01 M HNO₃ and 1.0 M KNO₃ showed that extraction was quantitative (97.6 %) with 0.05 M Aliquat 336 and 0.4 M Cyanex 921. From slope analysis results, the composition of the extracted species in synergistic extraction has been proposed to be R₂R'N⁺Ce(NO₃)₄·2 Cyanex 921. The negative value standard enthalpy change indicated that the extraction process was exothermic. The negative value standard entropy change confirmed the formation of a stable complex. It was found that HCl and H₂SO₄ are effective stripping agents for the recovery of 100 % metal from the loaded organic phase. The separation factor for Nd/Ce was the highest.

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