



Intensification of Europium extraction through a supported liquid membrane using mixture of D2EHPA and Cyanex272 as carrier



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ABSTRACT

Solvent extraction and supported liquid membrane transport properties of Eu from nitric acid medium using mixture of D2EHPA and Cyanex272 as carrier were investigated. In the solvent extraction studies, maximum synergistic enhancement factor (R) of 2.3 was obtained for the D2EHPA/Cyanex272 mole fraction ratio of 0.5:0.5. The synergistic effect of these carriers intensified Eu extraction in the supported liquid membrane system. The effects of feed phase pH, stirring rate, carrier concentration, stripping agent concentration and membrane pore size were investigated in the supported liquid membrane experiments. Some experiments were carried out by various membrane thicknesses and the linear relationship between permeability coefficient and (membrane thickness)⁻¹ showed that the Eu transport is membrane diffusion limited. The stability of the membrane is satisfactory over 9 days.

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1. Introduction

Liquid membrane (LM) technique, as an advanced solvent extraction technique, provides an effective and simple separation method for metal ions [1]. LMs merge extraction and stripping processes in a single stage, and they have benefits of nonequilibrium mass transfer, where the solute can move from a low to high concentration solution [2]. Supported liquid membrane (SLM) and emulsion liquid membrane (ELM) are the two configurations of the membranes that are generally used for facilitated transport of the metal ions [3].

Some advantages of SLMs, such as small capital and low operating cost, considerable potential for energy saving, possible use of expensive carriers, because of tiny amounts of the membrane phase, simple operation and easy scale up, are the most important reasons for their use in experimental studies [4]. They have also received considerable attention due to high selectivity and rapid extraction capacity factors [5].

In recent years, rare earth elements (REE) have become critical and strategic materials from an industrial perspective. The largest

application of rare earth elements is found in markets such as catalysts, glassmaking, lighting, and metallurgy. These materials are also used in high-growth markets such as battery alloys, ceramics, and permanent magnets [6].

Europium (Eu), which is one of the 15 lanthanides, has unique luminescent behavior and is used as a phosphor in all TVs and computer screens to create red and blue light. Another application of this element is in fluorescent light bulbs, alloys and as an agent in fluorescent glass. Luminescence of Eu is also worthwhile in medical, biochemical and surgical applications [7].

With increasing applications of rare earth elements, various methods are used to recover such elements in high purity and large amount. LM technique provides a simple and efficient separation method for these elements [1].

Synergistic extraction, the extraction of metal ions with two different extractants, has drawn considerable attention in the last decades. In the synergistic extraction, mixtures of two different extractants will intensify the extraction of a metal as compared to the normal additive effect of these extractants separately [8]. Many authors reported the synergistic solvent extraction systems for extracting and separating rare earths using acidic, natural and solvating extractants [9–17].

In the case of liquid membrane technique, synergistic extraction of Eu from nitrate medium using trioctylamine (TOA) and

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tributyl phosphate (TBP) was studied in a contained supported liquid membrane (CSLM) system. The reported results showed better permeability in comparison with the individual extractant [18]. Joshi et al. [3] studied transport of Uranium(VI) and Europium (III) across the supported liquid membrane using D2EHPA and different organo phosphorous oxodonors (TBP, DBBP, TOPO and Cyanex923) and showed that the SLM formed by TOPO and D2EHPA has better U(VI) transport from phosphoric acid feed with a good selectivity over Eu(III). The synergistic effect of TOPO and D2EHPA also was observed for the separation of Nd from lanthanide series via hollow fiber supported liquid membrane [19]. Synergistic effect between Cyanex272 and TBP observed in the separation of yttrium ions from the mixture of rare earths by a microporous hydrophobic hollow fiber supported liquid membrane. The combination of the extractants cooperated to extract and strip yttrium from the rare earths mixture [20].

Acidic Cyanex series extractants offer high extraction power and their solubility in aqueous solutions is very low, but they are relatively expensive to be used commercially, while D2EHPA is much less expensive [10,21]. The use of mixture of Cyanex272 and D2EHPA as a carrier for rare earths in a supported liquid membrane has not been reported in the literature. In the present work, the transport of Eu from HNO₃ solution through a SLM impregnated with the mixture of D2EHPA and Cyanex272 was studied. Various parameters like feed phase pH, carrier concentration, stripping agent concentration and etc. which affect the transport of Eu, have been studied in detail. The selectivity and stability of the system also was studied.

2. Experiments

2.1. Materials

The commercial extractants di-2-ethylhexyl phosphoric acid (D2EHPA) and bis(2,4,4-trimethylpentyl) phosphinic acid (Cyanex272) were procured from Sigma–Aldrich (Germany) and used without further purification. The aqueous solutions of Europium (III) were prepared by dissolving its nitrate salt (Eu(NO₃)₃·6H₂O, Middle East Ferro Alloy Company, 99.9% purity) in deionized water. Kerosene was obtained from Alfa Aesar (Germany) and HNO₃ (65%) was purchased from Merck (Germany). All other chemicals used in this work were of A.R. grade. During the study we used flat-sheet type PTFE hydrophobic microporous membranes, procured from Millipore (USA), as membrane support. The characteristics of these membrane filters are given in Table 1. The effective area of the membrane support was determined to be 8.18 cm² that was computed from the geometrical area (which was determined to be 9.62 cm²) and the membrane porosity.

2.2. Apparatus

A Digital pH meter (Sartorius, USA), calibrated daily with standard buffer solutions, was used for measuring the pH values of the aqueous phases.

Equilibrium experiments were carried out in a temperature controlled shaking water bath (INFORS AG).

Quantitative determination of Eu was carried out using inductively coupled plasma-atomic emission spectrometry (ICP-AES, Optima 7300 DV, America). The wavelength for Eu ions is 381.967 nm. The method was proposed by Wiel [23].

2.3. Methods

2.3.1. Solvent extraction studies

In the solvent extraction experiments, appropriate volumes (10 ml) of aqueous and organic phases were contacted in reagent bottles using thermostatic shaking water adjusted to 25 °C for 30 min, enough time to reach equilibrium. Then the two phases were separated and Eu concentration in the aqueous phase before and after extraction was determined by ICP-AES after suitable dilutions. The concentration of Eu in the organic phase was determined by mass balance. The distribution coefficient (*D*) is calculated as the ratio of Eu concentration in the organic phase to that in the aqueous phase at equilibrium. The experiments were repeated two times. Average error for distribution coefficient was 5%.

2.3.2. Membrane studies

The membrane transport experiments were carried out in a permeation cell consisting of two 200 cm³ Perspex compartments separated by a microporous membrane, one for feed solution and the other for stripping solution. Initial concentration of Eu in the feed phase was 0.33 × 10⁻³ mol L⁻¹ in all the SLM studies.

The liquid membrane phase was prepared by dissolving of Cyanex272 and D2EHPA in kerosene. The PTFE support was impregnated with the carrier solution for 24 h, then removed from the solution and wiped carefully with a tissue paper to remove the excess carrier. After preparation, the membrane was put in the transport cell and the permeability coefficient was determined by monitoring Eu concentration by ICP-AES in the feed and stripping phases as a function of time. The percentage of extraction was determined as follows:

$$\% \text{Extraction} = \frac{[\text{Eu}^{3+}]_0 - [\text{Eu}^{3+}]_t}{[\text{Eu}^{3+}]_0} \times 100 \quad (1)$$

where [Eu³⁺]₀ and [Eu³⁺]_t are the concentration of Eu in the feed phase at the start of the experiment (*t*=0) and at time *t*, respectively. All experiments were carried out at ambient temperature (25 °C).

3. Results and discussion

3.1. Solvent extraction studies

The extraction behavior of Eu was studied with D2EHPA, Cyanex272 and mixtures of them by the solvent extraction method. The aqueous solutions of Eu (4.31 × 10⁻³ mol L) were used for the extraction studies. The synergistic enhancement factor (*R*) could be obtained based on the experimental data [24],

$$R = \frac{D_{AB}}{D_A + D_B} \quad (2)$$

where *D*_{AB}, *D*_A and *D*_B are the distribution coefficient when Eu is extracted by D2EHPA + Cyanex272, D2EHPA and Cyanex272, respectively. The synergistic extraction occurs when *R* > 1, whereas *R* < 1 means antagonistic extraction. Fig. 1 shows the *R* value as a function of D2EHPA mole fraction (*X*_{D2EHPA}). In these experiments, aqueous phase pH was maintained at 4 and sum of two extractants concentration was equal to 0.06 M in kerosene as organic phase. It

Table 1
Characteristics of the membrane filters used.

Membrane	Diameter (mm)	Nominal pore size (μm)	Porosity (%)	Thickness (μm)	τ = 1/ε [22]
PTFE	47	0.22	85	150	1.17

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