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Efficient extraction and enrichment of rare earth from diluent aqueous solution by organic hollow sphere

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ARTICLE INFO

Article history:

Received 14 March 2017

Received in revised form 1 October 2017

Accepted 2 January 2018

Available online 10 January 2018

Keywords:

Rare earths

Low concentration

Enrichment

Solvent extraction

Organic hollow sphere

ABSTRACT

Extraction of rare earths from leach aqueous solution by conventional chemical precipitation brings severe amino-nitrogen pollution. In this work, a novel method was developed by organic hollow sphere to extract and enrich Er(III), a model middle-heavy rare earth ion, from diluent aqueous solution. The organic hollow sphere is a core-shell structure, in which the core is composed of gas phase and the shell is a thin layer of organic extractant. The effects of flow rates of gas phase and/or organic extractant on Er(III) extraction rate were investigated. The results demonstrated that the flow rate of gas phase played more important role than that of organic extractant in the extraction rate of Er(III). Accompanied by the achievement of a large volume ratio (1000:1) of aqueous solution to organic extractant, the enrichment ratio (100) of the rare-earth ions could reach a significant level, which is much higher than that by conventional methods. The lower flow rate of organic extractant is more beneficial to Er(III) enrichment. It demonstrates that the newly developed extraction technique in this work is promising, and as a result the development of organic hollow sphere extraction process for the recovery of REs is straightforwardly envisaged.

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1. Introduction

In recent years, the demand for middle-heavy rare earths (MHREs) has been sharply increased due to their wide application in a variety of high technologies. MHREs are mainly absorbed on the surface of weathered crust elution-deposited rare-earth ore in form of cation ions with the content in the range of 0.05 wt%–0.5 wt% (Chi and Jun, 2011; Jun et al., 2010). Conventional beneficiation methods, for example, flotation, gravity, cannot be used to obtain concentrate of MHREs. As a result, MHREs are often obtained from weathered crust elution-deposited rare-earth ore by a separation process consisting of leaching with ammonium sulfate solution and two-step precipitation with ammonium carbonate. However, in this process, the concentration of MHREs in leach solution is very low (less than 500 mg/L) (Xiao et al., 2015; Huang

et al., 2015). To concentrate MHREs from leach solution, a two-step precipitation process with ammonium bicarbonate is employed (Xiao et al., 2015). Unfortunately, the amount of ammonium bicarbonate consumed in these step is quite huge, for example, production of 1 t of rare-earth bicarbonates consumed more than 4 t of ammonium bicarbonate (Xiao et al., 2015; Huang et al., 2015) and ammonium bicarbonate cannot be recycled. Consequently, the slather of ammonium salt poses a great threat to environment.

Liquid-liquid extraction is the core technology in the hydrometallurgical industry (Samdavid et al., 2016; Rout and Binnemans, 2014; Na et al., 2013). However, in conventional extraction process, the volume ratio of aqueous solution to organic extractant was low. In addition, the concentration of MHREs leach solution is much lower than those of other separation systems. Therefore, MHREs cannot be effectively extracted and enriched from leach solution by conventional extraction

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<https://doi.org/10.1016/j.cherd.2018.01.001>

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Nomenclature

a	Total interfacial area between aqueous solution and organic extractant in the plexiglass column (m^2)
a_{single}	Surface area of one organic hollow sphere (m^2)
c	Concentration of Er(III) in aqueous solution (mol/m^3)
c_e	Concentration of Er(III) in aqueous solution when the extraction experiment ends (mol/m^3)
c_i	Concentration of Er(III) in aqueous solution at initial moment (mol/m^3)
c_{org}	Concentration of Er(III) in loaded organic extractant (mol/m^3)
c^*	Equilibrium concentration of Er(III) in aqueous solution between aqueous solution and organic extractant at time t (mol/m^3)
d_B	Diameter of the core of organic hollow sphere (m)
d_h	Outer diameter of outer tube (m)
d_o	External diameter of organic hollow sphere (m)
E	The enrichment ratio of Er(III)
g	Gravitational acceleration (N/kg)
h	The depth of aqueous solution in the plexiglass column (m)
k	Mass transfer coefficient of Er(III) (m/S)
n	Formation rate of organic hollow sphere
$Q_{\text{gas},\text{in}}$	Flow rate of gas phase (m^3/S)
$Q_{\text{org},\text{in}}$	Flow rate of organic extractant (m^3/S)
t	Extraction time (S)
u_b	Mean rising velocity of organic hollow sphere (m/S)
V_a	Volume of aqueous solution loaded in the plexiglass column (m^3)
V_B	Volume of the core of organic hollow sphere (m^3)
V_o	Volume of the shell of organic hollow sphere (m^3)
V_r	Volume ratio
Greek letters	
ρ	Density of aqueous solution (kg/m^3)
θ	Extraction rate (%)
τ	Retention time of any one organic hollow sphere in the plexiglass column (S)
σ	Interfacial tension (N/m)

technology. In conventional extraction process, the extractors used are often mixer-settler (Hadjieva et al., 1992; Khakpay et al., 2009; Pinto et al., 2009; Javanshir et al., 2012), mixer-settler column (Napeida et al., 2010; Torab-Mostaedi et al., 2009; Abdeltawab et al., 2002), and others (van Delden et al., 2006; Angelov and Gourdon, 2012; Torab-Mostaedi et al., 2012; Attarakh et al., 2013; Hemmatia et al., 2015; Wang et al., 2016). These extractors generate organic full sphere scattering in aqueous solution. Even MHREs could be completely transferred into the organic extractant from leach solution, the enrichment ratio of MHREs is too small to implement economically. Only when both enough interfacial area between aqueous solution and organic extractant and large volume ratio of aqueous solution to organic extractant could be realized simultaneously, liquid-liquid extraction technology is promising to be used to extract and enrich MHREs. To achieve these goals, one can generate organic hollow sphere by new extractor to take the place of the organic full sphere by conventional extractor. Recently, large volume

ratio of aqueous solution to organic extractant was realized through hollow sphere in microfluidic device (Wang et al., 2015; Fu et al., 2009; Nichols et al., 2011). However, it is costly to scale microfluidic device up to commercial scale and it is difficulty to clean contaminated microchannels.

In our previous work (Kun et al., 2014), a distributor was proposed to generate organic hollow sphere (Figs. 1 and 2). The organic hollow sphere is core-shell structure. The core of organic hollow sphere is composed of gas phase, correspondingly, the shell of organic hollow sphere is composed of organic extractant. However, operational factors influencing extraction efficiency and organic extractant have not clarified during extraction. Obviously, clarification of these factors will contribute to generalize the application of such a technique.

The aim of the work was to clarify the operational parameters which influencing extraction efficient and organic extractant during extraction of organic hollow sphere. In present work, organic hollow sphere was applied to extract Er(III), a model MHREs, from diluent solution. The key factors influencing the interfacial area between aqueous solution and organic extractant during extraction of organic hollow sphere was systematically investigated.

2. Experimental

2.1. Reagents and chemicals

2-Ethylhexyl phosphoric acid-2-ethylhexyl ester (EHEHPA) was purchased from Shanghai Rare-earth Chemical Co. Ltd, and used as the extractant. Kerosene was purchased from Xinhuan Cheng (Beijing) Co. Ltd. and used as solvent for EHEHPA. Both EHEHPA and kerosene were used as received, without any further purification or modification. The concentration of EHEHPA in kerosene was 1.5 mol/L. The Er_2O_3 of AR grade was supplied by Alfa Aesar (China). The solution of Er(III) with concentration 50.00 g/L was prepared by dissolving Er_2O_3 into 3.00 mol/L hydrochloric acid solution and used as stock solution. Industrial nitrogen (95%) was employed as gas phase. The other reagents used were of AR grade.

2.2. Experimental equipment

The solvent extraction equipment used in experiment is shown in Fig. 1. The equipment consists of a plexiglass column (1), distributor (2) and subsidiary equipment. The inner diameter and the height of plexiglass column are 90 mm and 2000 mm, respectively. There are four holes in the plexiglass column for the inlet and the outlet of aqueous solution, the outlet of organic extractant, and sampling. The inlet (6) and outlet (5) of aqueous solution are located at the 1300 mm and 20 mm upon the bottom of plexiglass column, respectively. The outlet of organic extractant (7) is located at the 1500 mm upon the bottom of plexiglass column. The hole of sampling (8) is located at the 1000 mm up the bottom of plexiglass column and used to get samples from aqueous solution during extraction experiment. The distributor (2) is vertically mounted under the bottom end of the plexiglass column. The inlet of organic extractant (4) is located at the side wall of distributor. Subsidiary equipment is composed of the tank of aqueous solution (11), the tank of raffinate (12), the tank of organic extractant (10), the tank of loaded organic extractant (9), pump for organic extractant (15) and pump for aqueous solution (14).

Aqueous solution is pumped into the plexiglass column through the inlet of aqueous solution from the tank of aqueous solution. Raffinate flows into the tank of raffinate through the outlet of aqueous solution. Organic extractant is pumped

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