Separation and Purification Technology 164 (2016) 12–18

Contents lists available at ScienceDirect

Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

Separation of indium from iron in a rotating packed bed contactor using Di-2-ethylhexylphosphoric acid

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ARTICLE INFO

Article history:
Received 27 September 2015
Received in revised form 7 March 2016
Accepted 10 March 2016
Available online 11 March 2016

Keywords:
Separation
Impinging Stream–Rotating Packed Bed
Indium
D2EHPA

ABSTRACT

In order to separate In(III) and Fe(III) from the sulfate leaching solution, solvent extraction with Di-2-ethylhexylphosphoric acid (D2EHPA) was investigated using a Impinging Stream–Rotating Packed Bed (IS–RPB) contactor in laboratory scale. The effects of operating parameters, such as feed solution pH, flow rate ratio, the high gravity factor, the concentration of D2EHPA, initial iron concentration, stripping solution concentration were examined. The experimental results showed that the IS–RPB was effective in the separation of In(III) and Fe(III)under favorable operating conditions. The extraction and stripping efficiency as high as 99% could be achieved, at a separation factor in excess of 3000. Compared with other mixing devices, IS–RPB contactor has better micromixing characteristics, which has a good application prospect.

1. Introduction

As a scarce metal, indium plays a major role in high-tech industry, as it finds application in liquid crystal displays (LCDs), solder alloys, semiconductor devices, photovoltaic cell, highspeed sensor, and so on [1–3]. Indium is most commonly found in zinc-bearing materials such as solution of sphalerite or marmatite and hence is usually a by-product of zinc metallurgy [4]. Rotary kiln reduction is popularly adopted in many zinc plants to enrich indium from zinc leach residue through oxidation at high temperature. The zinc oxide flue dust (ZOFD) containing 0.1–0.3% (mass fraction), serves as a potential source of indium [5]. In recent years, due to the extensive demand of indium, the recovery of indium from byproduct of zinc hydrometallurgical process is becoming more and more significant. Therefore, indium-bearing zinc oxide dust has become a new source raw material.

The leached solution of indium-containing zinc oxide dust include impurities like iron and zinc ion. Hence, it is necessary to further enrich or purify indium from leachate. The separation methods such as deposition, ion exchange, solvent extraction and so on are reported in open literature [6]. Solvent extraction is a most widely used method for the purification of indium in process metallurgy [7], where indium is usually recovered from sulfate solution [8]. Bis (2-ethylhexyl) phosphate (CAS-number 298-07-7), usually known as Di-2-ethylhexylphosphoric acid (D2EHPA), is by far the most popular extractant and it is almost always used in practical applications since it has a high loading capacity as well as good selectivity for indium over many other metal ions such as Zn(II), Fe(II), Cd(II), As(III) and Cu(II) [9]. However, Fe(III) can be extracted inevitably from sulfate solution by D2EHPA. A significant reduction in the emulsification and aging of organic phase was reported at concentration of Fe(III) higher than 100 mg/L. Additionally at concentrations of Fe(III) higher than 2 g/L, extraction using 20% D2EHPA–kerosene, resulted in 15% co-extraction of Fe (III), thus contributing to a significant reduction in the quantum of indium extracted, due to accumulation of Fe(III) in the organic phase [10]. Thus it establishes the fact that it is difficult to separate indium and iron based on equilibrium separation concept as it is limited by the thermodynamic equilibrium.

Although equilibrium conditions are unfavorable, it is well known that the rate of diffusion of one ion could be significantly different from the other, which can be harnessed to effect the

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http://dx.doi.org/10.1016/j.seppur.2016.03.017
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separation [11]. The utilization of difference in extraction rates of metal ions, by controlling the contact time of two phases, is called non-equilibrium solvent extraction. There were reports about the separation of metal ions using non-equilibrium solvent extraction, such as the separation of cobalt and nickel, indium and iron, neodymium and praseodymium, yttrium and erbium, yttrium and thulium [12–16].

Among the various liquid–liquid contactors such as mixer-settlers, extraction columns, centrifugal contactors, and impinging stream–rotating packed bed reactor, only centrifugal contactors and impinging stream–rotating packed bed reactor offer enhanced rates of separation. Annular centrifugal contactors (ACCs) use centrifugal force to separate the mixture of two immiscible liquids with different densities [17], but at a higher capital and operating cost in addition to serious phase entrainment loss. Compared with other contactors such as mixer-settlers, extraction columns and ACCs, IS–RPB reactor offer the following advantages: (i) micromixing time in the RPB can be less than 1 s [18], which is about three orders of magnitude smaller than in conventional packed beds, (ii) small degree of back mixing and solvent inventory demonstrate wide prospect of engineering application. In IS–RPB, two stainless capillary nozzles are located in-line with the rotational shaft. The two different liquids flow along the same axis in the opposite directions and collide, which causes a narrow zone in which a high turbulence intensity is created which leads to high micromixing, and excellent mass transfer efficiency [19,20].

In the last decades, liquid–liquid extraction using IS–RPB has received reasonable attention, Liu et al. [21] have studied the mass transfer characteristics in IS–RPB with tributyl phosphate (in kerosene)-water and phenol as the solute. The experimental results showed that the mass transfer efficiency was more than 97%. Jiao et al. [20] reported IS–RPB with tributyl phosphate solvent for extraction of acetic acid highlighting better separation efficiency compared to conventional systems [22]. The same authors have utilized LIX984 N as solvent to extract copper using IS–RPB and have reported the results covering the process variables in the following range: 1:1 phase ratio, 80 L/h flow rate, 5 vol%. LIX984 N and high gravity factor was 135, extraction ratio of copper reached 98.8% [23].

The literature clearly indicates the advantages of IS–RPB and hence the present work attempts to investigate the separation of indium and iron with D2EHPA–kerosene from sulfuric acid solution by non-equilibrium solvent extraction using a IS–RPB reactor. The effect of process parameters were assesses the extent of selective In(III) recovery and an effective In(III)–Fe(III) separation.

2. Experimental

2.1. Materials

The aqueous phase feed used in the experiments was supplied by a factory in Mengzi, Yunnan, China, which was H_2SO_4 leachate from zinc oxide flue dust. The chemical analysis results are listed in Table 1.

As seen in Table 1, in addition to indium, there are a great deal of metallic elements such as zinc, arsenic and iron in the solution. Organic reagents D2EHPA, was employed as solvent for extraction without further purification and commercial grade kerosene was used as diluent for organic extractant. The sulfuric acid and hydrochloric acid were used to adjust solution pH and to strip the organic phase, respectively.

2.2. Apparatus

The main structure of IS–RPB reactor is shown in Fig. 1. The two titanium capillary nozzles were located at the each side of the chamber with an inner diameter of 1.5 mm. Two different liquid flow along the same axis in the opposite direction are allowed to collide. As a result of such collision, a relatively narrow zone, called the impingement zone of high turbulence intensity, is created which offers excellent conditions for intensifying micromixing and improving mass-transfer efficiency. Secondly, the collided liquids are redistributed through the rotating packing, which is fully dispersed and flows through the rotor under an imposed collision. Finally, the liquid is expelled from the bottom of the IS–RPB.

Table 2 summarizes the characteristics of the IS–RPB used in this investigation. The type of packing is made of corrosion resistance teflon mesh with a diameter of 0.3–0.4 mm and a porosity of 0.95. The axial height of the bed is 5 cm. The inner and outer diameters of the rotor packing bed are 4 cm and 10 cm, respectively. The acceleration of packed bed leads to the formation of thin liquid films and tiny liquid droplets at the surface of dense packing channeling. the mass-transfer interfacial area between organic phase and aqueous solution in a RPB could be significantly increased and thus result in a high extraction rate within a short period. Moreover, fast and homogeneous mixing of the two phases can avoid the flooding characteristics in a centrifugal field.

2.3. Experimental section

Fig. 2 shows the schematic of the experimental setup. The feed solution and solvent stored in tank 14 and 1, were pumped through a rotameter 4 and 11 respectively, into the IS–RPB, by opening the valves 3 and 12. Both aqueous phase and organic phase streams contacted counter-currently in the RPB. The mixed phase moved outwardly and leave from the outer edge of the RPB via a centrifugal force into the tank 6. After demixing, the organic phase and

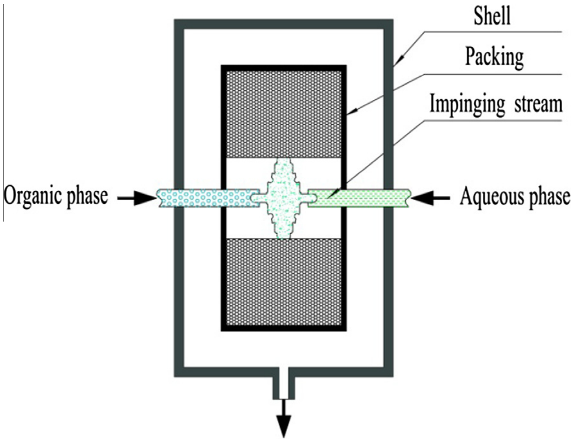

Fig. 1. Schematic of the rotating packed bed unit.

Table 1
Chemical composition of the indium-rich solution.

Element	Zn	As	In	Al	Fe	Sn	Mg	Ca	Bi	Cu
Conc. (g/L)	16.46	9.62	2.92	2.33	2.04	0.50	0.44	0.12	0.01	0.01