



Extraction of iron (III) from chloride leaching liquor with high acidity using tri-n-butyl phosphate and synergistic extraction combined with methyl isobutyl ketone



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ARTICLE INFO

Article history:

Received 27 April 2015

Received in revised form 11 June 2015

Accepted 1 July 2015

Available online 3 July 2015

Keywords:

Synergistic extraction

Iron (III)

TBP

MIBK

Chloride leaching liquor with high acidity

ABSTRACT

Separation of iron (III) from simulated chloride leaching liquor with high acidity by tri-n-butyl phosphate (TBP) and methyl isobutyl ketone (MIBK) extraction was investigated. Synergistic effect in iron extraction was found using TBP and MIBK extractant and evaluated by synergistic enhancement coefficient. Based on the physical properties and extraction ability of organic phase, the suitable composition was 70 vol.% TBP and 30 vol.% MIBK. Iron extraction increased with an increase in HCl concentration from 4.22 mol/L to 7.74 mol/L. Under optimal extraction conditions, that is, TBP volume fraction 70%, contact time 2 min, temperature of 25 °C, and phase ratio (O/A) 1:1, the iron extraction in one stage was above 91.0% with initial HCl concentration 5.1–5.5 mol/L and iron concentration 36–39 g/L, consistent with the practical chloride leaching liquor. Results showed that negligible metal ions Cr, V, Ca, Mg, Al, Mn, Ti and Si were co-extracted. The loaded organic phase can be effectively stripped by 0.1 mol/L ammonia compared with other stripping reagents. Under the optimal stripping conditions: contact time 2 min, reaction temperature 25 °C, and phase ratio (A/O) 2:1, more than 89.0% of iron was stripped in one stage so the organic phase could be regenerated. The Fe₂O₃ product with high purity was prepared from the stripping solution by precipitation and calcination processes. The mechanism of iron (III) extraction using synergistic system was discussed according to FT-IR spectra results.

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1. Introduction

As the anticorrosive technology of equipment has developed [1–3], considerable attention has been paid to the chloride hydrometallurgical method because of its efficient leaching of valuable metals from ores and possibility of HCl regeneration [4–6]. When iron enters into leaching liquor in most cases, it is required to be removed or recovered. There have been more concerns on the recovery of iron to prepare high added value materials rather than discarding it as waste [7–9]. The Hongge ores (titanomagnetite reserves 3.55 billion tons) in China could not be effectively utilized by traditional pyrometallurgical routes such as blast furnace and direct reduction. A novel hydrometallurgical

route for the treatment of high-chromium vanadium-bearing titanomagnetite concentrates by hydrochloric acid leaching of the titanium-bearing tailing was developed in our laboratory [10]. The existence of iron in leaching liquor hindered subsequent recovery of vanadium and chromium. Solvent extraction was used to separate and recover high concentration iron from the leaching liquor that contained 36–39 g/L Fe, approximately 200 g/L hydrochloric acid and small amount of Cr, V, Ca, Mg, Al, Mn, Ti and Si metal ions. Extraction was chosen because of its convenient operation, mild reaction condition, good selectivity, high recovery rate and product of high purity [11,12].

The solvent extraction of iron has been studied using various extractants, such as organo-phosphorus extractants e.g. D2EHPA [7,13,14], PC88A [15], carboxylic acid extractants e.g. Versatic 10 acid [16,17], amine extractants e.g. primary amine N-1923 [18], tertiary amine Alamine 336 [15,19], quaternary amine salt Aliquat 336 [20], neutral extractants e.g. MIBK [9,13–15], TBP [8,21,22], Cyanex 921 [22], Cyanex 923 [22,23], amides [24,25],

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chelating extractants e.g. LIX 860 [26], 3-phenyl-4-benzoyl-5-isoxazolone [27]. The extraction mechanism of organo-phosphorus is usually cation exchange, therefore, other metal ions are extracted along with iron. Stripping iron from loaded organic phase is also difficult even using strong acid [28,29]. Most importantly appropriate extraction condition for phosphates is not strong acid system [13,30]. The aqueous solubility of carboxylic acid is high leading to serious loss of extractant. The third phase in amine extraction system is easy to form [18]. Researches on chelating extractant for separation of iron are few. Neutral extractants have been reported to have higher extraction efficiency for iron extraction from acidic solution while complete stripping could be achieved.

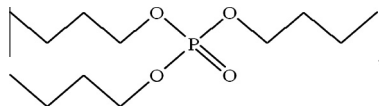
Both TBP and MIBK as neutral extractants have been investigated for iron extraction from chloride solution [9,14,21,22]. Negligible TBP is dissolved in aqueous phase (0.039 mass%) and a little water (4.67 mass%) is co-extracted by TBP [31]. However, the viscosity and density of TBP are relatively high, thereby hindering phase disengagement. Considering the low viscosity and density of MIBK and its quick kinetics for extraction [9], mixing TBP with MIBK has been studied for different extraction systems. Caproic acid was recovered using TBP in both MIBK and xylene systems [31]. TBP in the MIBK system was found to be better than TBP in the xylene system based on extraction efficiency. The extraction of phosphoric acid was performed with 45% TBP and 55% MIBK mixtures [32], and the H_3PO_4 -water-MIBK and TBP phase diagram was determined. Research on the extraction of iron from titania waste liquor was conducted with the TBP and MIBK mixed system [33]. The results showed that complete iron extraction was obtained in two-stage cross current operation at A/O ratio of 1:2. Studies also showed that with the increase of acid concentration, phase separation time decreased when MIBK extracted iron [13]. Thus, combining TBP with MIBK could reduce phase separation time, which is suitable for iron removal in this strong acidic system. However previous researches paid attention to low acid concentration condition, the solvent extraction of iron using TBP and MIBK mixed extractant from chloride leachate with high concentration of hydrochloric acid (about 200 g/L) has not been reported.

In the present study, extractant composed of TBP and MIBK was used to separate iron (III) from the simulated chloride leaching liquor with high-concentration HCl. The effects of different extraction and stripping conditions on iron separation were investigated. Synergism was observed, and the mechanism of the mixed system extracting iron was studied. This research may provide related data and guidance for effectively separating high concentration iron from strong acidic leaching liquor to produce high purity iron oxide.

2. Experimental

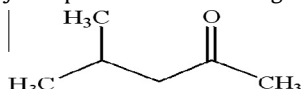
2.1. Chemicals and reagents

The extractant tri-n-butyl phosphate was obtained from Xilong Chemical Co., Ltd. with chemical structure



, and methyl isobutyl ketone

was supplied by Sinopharm Chemical Reagent Co., Ltd. with chemical structure



. All the extractants were used without purification. Based on previous study, feed chloride liquor that contained ferric iron approximately 38 g/L was prepared by dissolving $FeCl_3 \cdot 6H_2O$ ($\geq 99.0\%$, Xilong Chemical

Co., Ltd.) and HCl in distilled water, which was used to recover iron by extraction and stripping. Ammonia (25–28 wt.%, Xilong Chemical Co., Ltd.) dissolved in deionized water was used as stripping agent. All other reagents were of analytical reagent grade. The metal concentrations in solution were measured with inductively coupled plasma optical emission spectroscopy (ICP-OES, Optima 5300DV, Perkin-Elmer). The hydrochloric acid concentrations of solutions were analyzed by acid-base titration. The density and viscosity of organic phase were measured with vibrating densitometer-automated microviscometer (DMA 5000-AMVn, Anton Paar). The structure of organic phase was evaluated by FT-IR spectra (Spectrum GX, Perkin-Elmer). The iron oxide product was characterized using X-ray diffraction (XRD, SmartLab, Rigaku Corporation).

2.2. Method for extraction and stripping

Extraction experiments were performed in conical flasks by mechanically shaking with frequency of 150 r/min, and then phase disengagement was conducted in separating funnels. After phase disengagement within 2 min, the concentrations of metals in raffinate were determined by ICP-OES after dilution and the wavelength used for determining iron was 238.204 nm. The concentrations of metals in organic phase were obtained from mass balance.

The loaded organic phase and stripping reagent were mixed in conical flasks by mechanically shaking with frequency of 150 r/min, and then phase disengagement was conducted in separating funnels. After phase disengagement within 2 min, the concentrations of metals in stripping solution were determined by ICP-OES after dilution and the concentrations of metals in loaded organic phase were obtained from extraction experiments.

2.3. Data treatment and related formula

Extraction (E), distribution coefficient (D), synergistic enhancement coefficient (R), and stripping (S) were provided as Eqs. 1–4, respectively.

$$E = \frac{V_{Org} \cdot C_{Org}}{V_F \cdot C_F} \times 100\% = \frac{D}{D + \frac{V_{Aq}}{V_{Org}}} \times 100\% \quad (1)$$

$$D = \frac{C_{Org}}{C_R} \quad (2)$$

$$R = \frac{D_{T+M}}{D_T + D_M} \quad (3)$$

$$S = \frac{V'_{Aq} \cdot C'_{Aq}}{V_{Org} \cdot C_{Org}} \times 100\% \quad (4)$$

In the mentioned equations, C_F is the concentration of metal in feed solution; C_{Org} or C_R is the concentration of metal in organic phase or raffinate when reaction reaches equilibrium; V_F , V_{Org} , and V_{Aq} represent the volume of feed solution, organic phase, and aqueous phase, respectively; D_{T+M} is the distribution coefficient of TBP and MIBK mixture extraction; D_T or D_M represents the distribution coefficient of single extraction using TBP or MIBK, respectively; V'_{Aq} is the volume of stripping aqueous phase; C'_{Aq} is the metal concentration in stripping solution.

3. Results and discussion

3.1. Solvent extraction

3.1.1. Effect of extractant composition on the extraction of iron

A series of experiments were performed to determine extractant components under the following condition: initial HCl

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