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Optimization of Re solvent extraction from molybdenite roasting dust leaching solution and the performance evaluation of extraction in a multi-stage mixer-settler



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ABSTRACT

Separation of Re from roasting dust leaching solution of molybdenite using a multi-stage mixer–settler extractor was studied. Systematic solvent extraction tests were performed using TBP as an extractant diluted in kerosene. The optimum extraction conditions were as follows: pH = 0, temperature 25 °C, 500 rpm agitation rate, 6 min contact time and 40% v/v TBP. The results showed that the extraction reaction is exothermic. A flow sheet was developed for the process. Three different extraction ways were studied in a laboratory size mixer–settler: Namely, counter-current, cross-current and co-current. The results showed that counter-current was the best. In addition, it was found that 2 h of continuous operation was necessary to reach a steady state condition for the process. Extraction as high as 95.43% was obtained.

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1. Introduction

Rhenium is important industrially as high-temperature super alloys and as catalysts, as Pt-Re, for example, for the production of highoctane, lead free gasoline (Nebeker and Brent Hiskey, 2012; USGS, 2012; Abisheva et al., 2011). Re world resources reach 6 million kg and only 49,000 kg was produced in 2011, mainly in Chile, in which more than 1.3 million kg Re reserves are reported. World consumption, according to Roskill Information Services Ltd (2010), was mostly for super alloy making (78%). 14% for catalyst production and 8% for other applications. Re is recovered from molybdenite concentrates through roasting or direct reduction of concentrates (Sutulov, 1970) and separated by different methods such as precipitation, chlorination (Snell et al., 1973), adsorption on activated carbon (Seo et al., 2012), solvent extraction (Leszczyńska-Sejda et al., 2009) and ion exchange (Nebeker and Brent Hiskey, 2012). Several purification methods are used to produce Re and Mo. Among them, liquid–liquid extraction provides an effective and simple separation method (Jordanov et al., 1968; Karagiozov and Vasilev, 1979; Almela et al., 1998). Also, it is used especially for separation of rhenium from other components, such as arsenic, tungsten and molybdenum (Sawant et al., 1997). Tri-n-octylamine, bis-isododecylamine, pyridine, Aliquat 336, tributyl phosphate, trioctyl

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phosphine oxide, cyklohexanon, ethyl xanthate and mesityl oxide are frequently employed as extractants for Re extraction (Kertes and Beck, 1961; Kinjakov and Korszunov, 1976; Gerhardt et al., 2001).

Tributyl phosphate (TBP), a widely applied extractant in hydrometallurgical processes, is a major extractant and modifier for Re separation (Leszczyńska-Sejda et al., 2009; Zhan-fang et al., 2009).

Over the past 20 years, mixer–settlers have been intensively used in chemical, pharmaceutical and hydrometallurgical industries. The main advantages of this equipment are: strong operational loads, easy operation and maintenance and simple start-up (Hadjiev and Paulo, 2005).

This study investigates solvent extraction of Re from roasting dust leaching solution of molybdenite using a multi-stage mixer–settler. Systematic studies were performed using TBP as an extractant diluted in kerosene. For an efficient separation of Re, parameters such as pH, extractant concentration, phase ratio, contact time, stirring speed and temperature were studied. Three different ways of extraction (countercurrent, cross-current and co-current) (Fig. 1) (Rydberg, 2004) and the number of extraction stages were studied using a constant phase ratio of V_a/V_o = 1.

2. Extraction procedure

2.1. Reagents

Dust required from the molybdenite roasting furnace of the Karmania Co. (Kerman, Iran) was obtained. Tributyl phosphate (Fluka,

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Fig. 1. Three different ways of connecting in liquid-liquid extraction: (a) counter-current; (b) cross-current and (c) co-current.

Switzerland) was an extractant, Kerosene (Tehran Refinery, Tehran) was diluents and sulfuric acid and ammonium hydroxide (Merck, Germany) were pH modifier.

2.2. Feed solution preparation

The aqueous solutions were prepared by leaching the roasting dust. The samples were analyzed using X-ray diffraction (XRD). The compositions of dust sample are given in Table 1.

The leaching experiments were performed under the conditions that the liquid-to-solid ratio, stirring speed, temperature and leaching time were 3.5, 400 rpm, 85 °C and 90 min, respectively. The bath temperature was digitally controlled within \pm 0.5 °C. The obtained pulp was filtered and analyzed for Re, Mo and the other compositions. The amount of Re in the leaching solution was obtained at 600–700 mg/L. Then, the leach liquor was prepared to solvent extraction experiments.

2.3. Batch solvent extraction studies

Extraction equilibrium experiments were carried out by agitating equal volumes (50 mL) of feed solution and organic phase for 60 min to approach the equilibrium at room temperature (25 ± 1 °C). All the experiments were conducted at room temperature. Other than temperature effect, where the temperature was investigated in the range of 25 to 60 °C. After phase separation, the pH and ICP concentrations of Re

 Table 1

 Elemental compositions of the roasting dust used in

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this study.		

Element	wt.%
Re	0.61
Мо	61.8
Cu	0.45
Fe	0.62
W	0.56
Rh	0.032
Nb	0.016
S	23
Mn	0.1
Te	0.028
Ti	0.23
Hf	0.012
Bi	0.077

and Mo were measured in the aqueous phase. The distribution ratio, D, was calculated as the concentration ratio of metal in the organic phase to that in the aqueous phase at equilibrium ($D = [Re]_{org}/[Re]_{aq}$). From the D values, the extraction percentage was calculated:

$$\%E = D \times 100 / \left[D + \left(V_{aq}/V_{org}\right)\right]$$

where V_{aq} and V_{org} are the volumes of aqueous and organic phases, respectively.

2.4. Multi-stage procedure

2.4.1. Experimental apparatus

Fig. 2 shows one unit of a horizontal type mixer–settler extractor, with 25.0 cm \times 5.0 cm \times 8.2 cm dimensions. The aqueous and organic phases flowed into the bottom of the mixer and were discharged from different points of the settler bottom.

2.4.2. Continuous multi-stage separation process

For the multi-stage experiments, the mixer–settler units were assembled for counter-current, cross-current or co-current experiments. The aqueous and organic phases were transported to the system inlets by separate peristaltic pumps. All the experiments were done at room temperature. The liquid temperature, T, was measured by an alcohol thermometer immersed in the settler. The aqueous phase in each system outlet was sampled at specific time intervals. The ratio of the flow rates, Q_a/Q_o , was 1:1 at 4 stages.

3. Results and discussion

3.1. Extraction equilibrium results

3.1.1. Extraction isotherm

Fig. 3 shows the extraction curves of Re and Mo using 40% (v/v) TBP as an extractant against pH. It was found that with increasing H^+ in the leaching solution, extraction percentage of Re increased. On the other hand, the lowest amount of Mo was extracted at pH close to zero.

The difference in extraction behavior can be attributed to the extraction reactions of Re and Mo (Lee et al., 2008):

$$\operatorname{ReO}_{4}^{-} + \operatorname{H}^{+} + \operatorname{TBP} = \operatorname{ReO}_{4}^{-} \cdot \operatorname{H}^{+} \cdot \operatorname{TBP}$$

$$\tag{1}$$

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