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## Effect of operational parameters and internal recycle on rhenium solvent extraction from leach liquors using a mixer-settler

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## ABSTRACT

The extraction of rhenium from molybdenite roasting dust leach solution was performed using a mixer-settler extractor by tributyl phosphate (TBP) diluted in kerosene as the extractant. In the single-stage extraction experiments, effect of the aqueous to organic phase ratios,  $Q_a/Q_o$ , and the number of extraction stages,  $N$ , on the rhenium extraction was studied. It was found that using the phase ratio of 1:1 in a two-stage extraction, 87.5% depletion of rhenium was obtained. The comparison of experimental results with the continuous co-current extraction showed a good agreement. The effect of internal recycle of organic phase was investigated in the phase ratio of 1:1 by changing the flow rate ratio of recycle-to-fresh organic phase,  $Q_{ro}/Q_{fo}$ . The optimum performance was achieved in the phase ratio,  $Q_{ro}/Q_{fo}$ , equal to 3:7. It was found that improvement in the performance of the mixer-settler for the rhenium-TBP system can be obtained in the phase ratio of 1:1 when  $Q_{ro}/Q_{fo} = 3:7$ .

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### 1. Introduction

Rhenium (Re) is one of the most interesting transition elements due to its properties that have been useful mainly in the petroleum-reforming industry and in the production of special metal alloys (e.g. nickel-based super alloys, refractory alloys, high performance alloys for aerospace application, among others) [1]. This element and its compounds (ammonium perrhenate- $\text{NH}_4\text{ReO}_4$  and perrhenic acid- $\text{HReO}_4$ ) are produced in Chile (53% of the world production in 2010) as a byproduct from porphyry copper-molybdenum ores [2]. Nevertheless, rhenium is present in nature in very low concentration (0.4 mg/t) which makes it a very valuable metal [3]. Hydrometallurgical techniques such as solvent extraction [4], adsorption [5], ion exchange [6], supported liquid membrane [7], precipitation [8], etc., have been used extensively for the recovery of rhenium from various solutions. Amongst these, solvent extraction method is one of the most effective methods and especially used for separation of rhenium from other

components, such as arsenic, tungsten or molybdenum [9]. Solvent extraction has been used in the minerals processing industry since the 1950s. It involves contacting two immiscible phases, usually an organic phase as an extractant and an aqueous phase containing the metal values. With energy input into the solvent extraction system, the two immiscible phases form an emulsion to provide an interfacial area for chemical reaction or mass transfer. As a result, the metal ions are extracted from the aqueous phase into the organic phase to achieve separation, purification, and concentration [10].

Over the past 20 years mixer-settlers have been intensively used in chemical, pharmaceutical, and hydrometallurgical industries [11]. Mixer-settler extractor has been widely used in the rare earth element separation industry [12]. The main advantages of this equipment are: strong operational loads, easy operation and maintenance, and simple start-up [11]. In hydrometallurgical solvent extraction systems, in addition to purification, a metal value concentration effect between the feed and strip aqueous streams are usually required. This means that the feed-to-strip flow rate ratio ( $A/A'$ ) is usually large. A large  $A/A'$  value would result in either a small  $O/A$  or a large  $O/A'$  value or both ( $O$  is the organic phase flow rate). In such cases the resulting mixture in a mixer-settler will tend to emulsify and be difficult to separate. One way to overcome this problem is to recycle the minor phase from the settler back to the mixer. Recycle is also often practiced

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to reduce the entrainment of the organic phase in the aqueous stream or of the aqueous phase in the organic stream, or both [13]. In the present work, the separation of rhenium from molybdenite roasting dust leach solution using solvent extraction technique by tributyl phosphate (TBP) as an extractant diluted in kerosene was studied. The investigation was carried out in a laboratory-scale mixer-settler. The objectives of the present work are: (i) optimization of operation parameters such as flow rate ratio of the two phases,  $Q_a/Q_o$ , and the number of extraction stages,  $N$ , in a single-stage mixer-settler; (ii) to study the effect of internal recycle on rhenium extraction; and (iii) eventually to investigate the agreement between single-stage experimental results with continuous co-current extraction experiments in a multi-stage mixer-settler.

## 2. Materials and methods

### 2.1. Sample and reagents

The outgoing dust of the roasting furnace was obtained from the Karmania Co. (Kerman, Iran). Chemical analysis of the dust used in this research is listed in Table 1. The organic phase was composed of Tributyl phosphate (TBP) and kerosene. TBP is produced by the Fluka Factory in Switzerland and the kerosene was the product of the Tehran Refinery in Tehran. For pH adjusting, sulfuric acid and ammonium hydroxide (Merck, Germany) were used in this study.

### 2.2. Leaching

Operational conditions used for the leaching of dust in this research were as following: distilled water as leachant at the solid to liquid ratio of 1:3.5 at 85 °C for 90 min with the agitation rate of 400 rpm. The bath temperature was digitally controlled within  $\pm 0.5$  °C. Under these conditions, the value of rhenium in the aqueous solution 600–700 mg/L was obtained. Then, the leach liquor was prepared for the solvent extraction experiments of rhenium in the mixer-settler.

### 2.3. Solvent extraction experiments

For selective extraction of rhenium from the leach liquor, the solvent extraction experiments were performed by 40 vol.% TBP diluted in kerosene as organic solvent and leach liquor at pH = 0 and ambient temperature for 60 min to approach the equilibrium. The solvent extraction data were selected based on previously published results [14–17]. According to previous study [14,15]; small amounts of Mo were extracted in these conditions which can

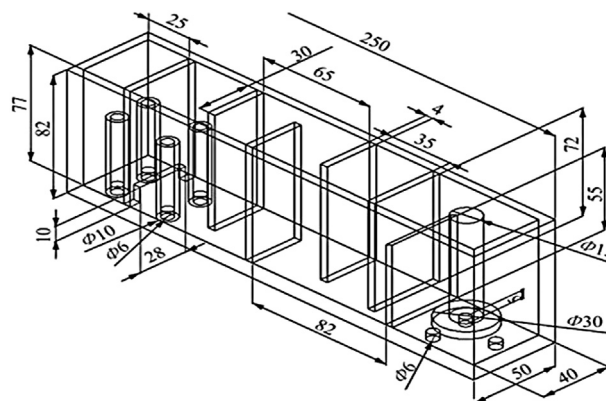


Fig. 1. Schematic diagram of the mixer-settler used in the experiments (unit of length: mm).

be removed later by selective stripping. Also, it was found that Cu and Fe extraction is impossible by TBP in this media. In the case of Mn, Mg, Pb, As, and Se presence, TBP is useless. After the two phases were separated, the aqueous phase was analyzed for determination of rhenium. The concentration of rhenium in the aqueous phase was determined by an Inductively-Coupled Plasma (ICP) instrument.

### 2.4. Experimental apparatus

A box-type mixer-settler made of plexiglas was used as the extraction equipment. The dimension of the mixer-settler is 25 × 5 × 8.2 cm (Fig. 1). The aqueous and organic phases flowed into the bottom of the mixer and were discharged from different points of the settler bottom.

### 2.5. Operating conditions

All the solvent extraction experiments were performed through a single-stage mixer-settler other than continuous co-current extraction experiments, which was carried out through a four-stage mixer-settler. The experiments were conducted at the flow rate ratios,  $Q_a/Q_o$ , 1:1, 3:2, 2:1, and 5:2. Based on our previous study [data not published], the extraction time in the mixer-settler was selected 6 min. In these experiments, in each phase ratio the values of volumetric flow rates of aqueous and organic phases were set at one hour. Therefore, with the completion of the feed of phases in each vessel, the raffinate and pregnant organic phase was returned

Table 1  
Elemental compositions of roasting dust used in this research.

Element	wt.%
Re	0.61
Mo	61.80
Cu	0.45
Fe	0.62
W	0.56
Rh	0.03
Nb	0.02
S	23.00
Mn	0.10
Te	0.03
Ti	0.23
Hf	0.01
Bi	0.08

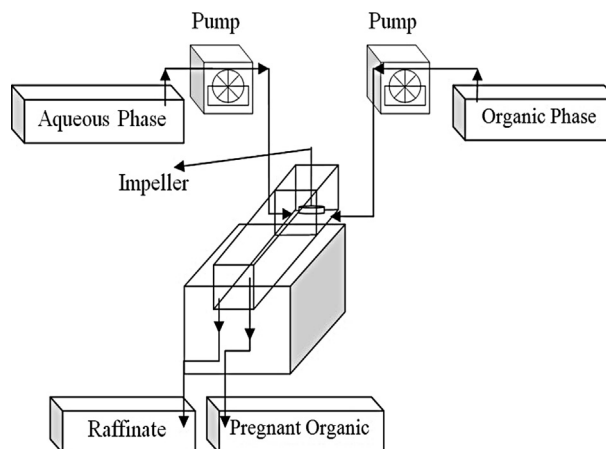


Fig. 2. Process scheme for operational parameters optimization on Re extraction.

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