Hydrometallurgy 146 (2014) 138–141

Contents lists available at ScienceDirect

Hydrometallurgy

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

Technical note

Purification and stripping of tantalum from organic phase and elimination of emulsification by ultrasound

Xiuli Yang [a,b,c,*], Junwei Zhang [a], Xihui Fang [a], Xiaohui Wang [c]

[a] Faculty of Resource and Environmental Engineering, Jiangxi University of Science and Technology, Jiangxi, 341000, China
[b] Jiangxi Key Laboratory of Mining Engineering, Jiangxi University of Science and Technology, Jiangxi 341000, China
[c] Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100190, China

ARTICLE INFO

Article history:
Received 12 September 2013
Received in revised form 2 April 2014
Accepted 6 April 2014
Available online 18 April 2014

Keywords:
Tantalum
Stripping
Emulsification
Ultrasonic

ABSTRACT

The scrubbing of impurities from tantalum-loaded MIBK was investigated using dilute sulfuric acid as scrubbing agent in a cross-current system. The effects of various scrubbing parameters such as sulfuric acid concentration and scrubbing time have been investigated. Stripping studies of tantalum from the scrubbed tantalum-loaded MIBK were carried out using distilled water as stripping agent in a cross-current system. The elimination of emulsification was investigated using ultrasound in the scrubbing and stripping processes. The effect of ultrasonic power on demulsification was studied. After a six-stage cross-current scrubbing with 5 mol/L dilute sulfuric acid and single-stage cross-current stripping, pure tantalum-loaded MIBK can be obtained that yields 99.6% tantalum oxide after precipitation, filtration and roasting. The overall recovery of tantalum was 96.21%. Ultrasound technology can be used for demulsification with the optimal output power range being from 0.44 to 0.56 W/mL.

1. Introduction

World tantalum resources are scarce and of low-grade and tantalum is therefore classed as a rare metal (Zhang, 2008). Tantalum has good corrosion resistance, a high melting point (3269 K), good thermal conductivity (0.57 J/cm/K at room temperature), and is compatible with the human body and is therefore used widely in metallurgy, nuclear, aerospace, electronics, and medical equipment industries (Guo and Wang, 2009; Zhang, 2008).

During the metallurgical processing of tantalumniobium ores, raw materials are roasted under atmospheric conditions to transform tantalum and niobium into high valence (+5) compounds. After leaching, the tantalum and niobium compounds are separated by solvent extraction. Several solvents, including tributyl phosphate, acetamide, methyl isobutyl ketone (MIBK), and octanols, have been evaluated for the extraction of tantalum and niobium (Han and Zhou, 2004; Kabangu and Crouse, 2012). MIBK has been suggested to be the most effective solvent because of its high selectivity for tantalum and niobium (Sanda and Taiwo, 2012; Yang et al., 2013a, 2013b; Zhu and Cheng, 2011).

Recently, a new method for the separation of tantalum and niobium has been proposed where tantalum and niobium are separated from the pulp using MIBK with reduced fluorine pollution (Yang et al., 2013a, 2013b). In this new process, tantalum and niobium are extracted sequentially, and when compared with the traditional process, the hydrofluoric acid concentration used in the new process decreases from 6 mol/L to 1.6 mol/L. However, because of the lower hydrofluoric acid concentration, emulsification occurs in the scrubbing and stripping processes. Although many researchers have studied tantalum extraction using a variety of extractants and extraction methods, information regarding emulsification in the scrubbing and stripping processes has rarely been reported.

In this work, the scrubbing of impurities from tantalum-loaded MIBK and the stripping of tantalum from scrubbed tantalum-loaded MIBK were investigated in a cross-current system. The effects of different parameters on tantalum and impurity concentrations in the aqueous phase were studied in the scrubbing and stripping process. The elimination of emulsification was investigated using ultrasound technology in the scrubbing and stripping processes. The effect of ultrasonic power on demulsification was studied.

2. Experimental techniques

2.1. Reagents

A stock organic phase (composition in Table 1) containing tantalum in the form of TaF_7^{2-} was obtained from a low acid concentration extraction process (Agulyansky, 2004; Yang et al., 2013b). Concentrated sulfuric acid (98%, Beijing Beihua Fine Chemical Co. Ltd, China) was diluted as required for the scrubbing tests and distilled water was used in the stripping tests.

* Corresponding author at: Faculty of Resource and Environmental Engineering, Jiangxi University of Science and Technology, Jiangxi 341000, China.
E-mail address: yangxiuli21@126.com (X. Yang).

http://dx.doi.org/10.1016/j.hydromet.2014.04.005
0304-386X/

Table 1
Composition of organic phase.

Composition	Ta	Nb	Sn	Si	Na
Concentration, g/L	8.79	0.049	0.174	0.081	0.035

2.2. Analytical

The tantalum content in the aqueous phase after contact was analyzed by inductively coupled plasma spectroscopy. The niobium and content of other impurities was analyzed by mass spectrometry. Micrographs were obtained by cold field emission scanning electron microscopy (SEM). X-ray diffraction (XRD) spectra were obtained using a multi-function X-ray diffractometer. During the experiments, the pH was measured using a Mettler Toledo 320 pH meter. The concentration of tantalum in the organic phase was calculated by mass balance.

2.3. Experimental apparatus

A plastic (PTFE) extraction funnel (Beijing Beihua Fine Chemical Co. Ltd) and high frequency ultrasonic cleaner with digital control (Kunshan Hechuang Ultrasonic Instrument Co., Ltd) were used.

3. Results and discussion

To remove impurities such as silicon, tin, sodium and niobium from the tantalum-loaded MIBK, multistage cross-current scrubbing was carried out using dilute sulfuric acid at room temperature. The stripping of tantalum from the scrubbed tantalum-loaded MIBK was carried out using distilled water as stripping agent at room temperature.

3.1. Effect of sulfuric acid concentration in scrubbing process

To reduce tantalum losses in the tantalum-loaded MIBK by scrubbing, the tantalum content in the aqueous phase was measured at different sulfuric acid concentrations, with results summarized in Fig. 1. The tantalum content in the aqueous phase decreased with increasing sulfuric acid concentration. This is believed to occur because hydrolysis of the tantalum salt ($H_2TaF_7 \cdot 2MIBK$) is inhibited with an increase in sulfuric acid concentration. Therefore, a soluble fluorine–tantalum complex cannot be generated from hydrolysis of the tantalum salt ($H_2TaF_7 \cdot 2MIBK$). Thus, a sulfuric acid concentration of 5 mol/L was used to ensure that the lowest tantalum concentration possible was obtained in the aqueous phase.

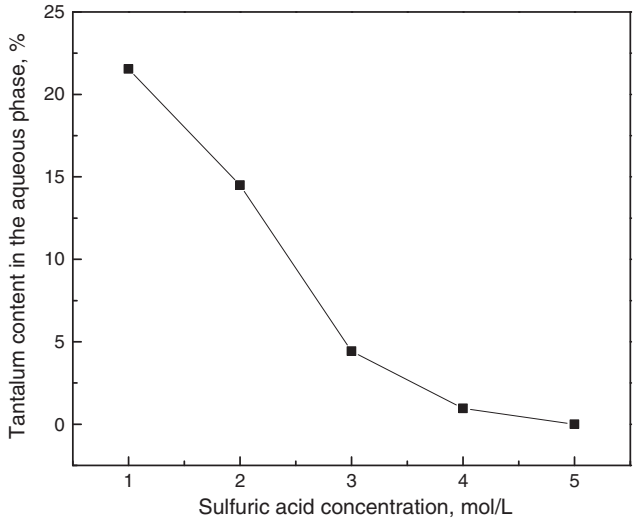

Fig. 1. Effect of sulfuric acid concentration on tantalum content in aqueous phase after scrubbing. A/O (aqueous phase/organic phase, volume) ratio = 1, contact time = 8 min, speed = 80 times/min, room temperature.

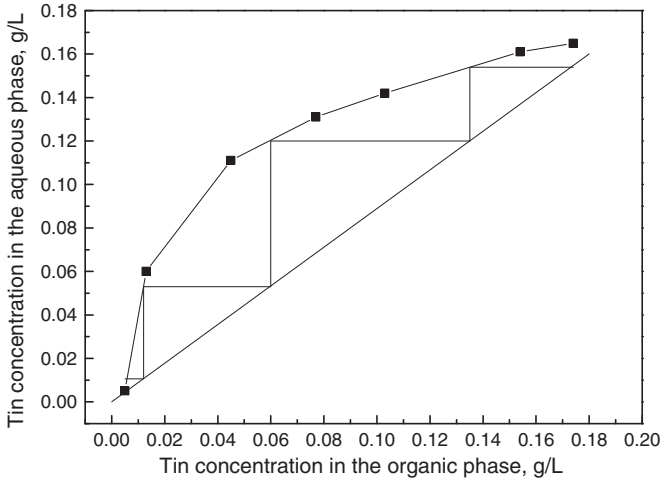

Fig. 2. McCabe–Thiele diagram for tin scrubbing. A/O ratio = 1, contact time = 8 min, speed = 80 times/min, room temperature.

The tin concentration was highest among the impurities. A McCabe–Thiele diagram was constructed for the scrubbing of tin using dilute sulfuric acid at room temperature. Fig. 2 shows that four stages are required theoretically for the complete scrubbing of tin.

The recoveries of tantalum at different sulfuric acid concentrations are given in Fig. 3. The maximum recovery of tantalum occurred at approximately 5 mol/L sulfuric acid.

3.2. Effect of scrubbing times in scrubbing process

To remove impurities completely, cross-current scrubbing stages were investigated with results summarized in Fig. 4. The impurity concentrations decreased with an increase in the number of scrubbing stages. Pure tantalum-loaded MIBK was obtained after six-stage cross-current scrubbing.

3.3. Preparation of tantalum oxide

Stripping of tantalum from the scrubbed tantalum-loaded MIBK was carried out using distilled water at room temperature at a constant aqueous/organic phase ratio (A/O) of 1:1. After stripping, tantalum was extracted from the organic solvent into the aqueous solution,

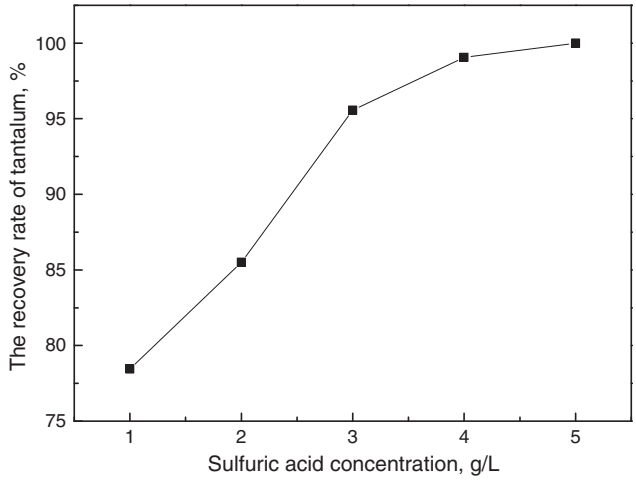

Fig. 3. Effect of sulfuric acid concentration on tantalum recovery rate. A/O ratio = 1, contact time = 8 min, speed = 80 times/min, room temperature.