



Selection of reductants for extracting selenium and tellurium from degoldized solution of copper anode slimes



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Abstract: This paper focused on investigating high-efficient reductants of recovering selenium and tellurium from degoldized solution of copper anode slimes. Firstly, the effect of various reductants on recovery rates of Se and Te was investigated based on thermodynamic analysis of various metallic ions in degoldized solution. Secondly, the single factor experiments were made to investigate the effect of the process parameters on recovering Se and Te with hydrazine hydrate. Finally, the hydroxylamine hydrochloride was added to intensify the extraction efficiencies of Se and Te. The results indicated that hydrazine hydrate was the most suitable reductant, and the recovery rates of Se and Te are 71.23% and 76.50%, respectively; the recovery rates of Se and Te were 92.07% and 97.81%, respectively, under the optimal process conditions of hydrazine hydrate dosage of 0.2133 mol/L, H⁺ concentration of 4.305 mol/L, reaction temperature of 85 °C and reaction time of 5 h; the recovery rate of Se was 97.59%, and that Te reached up to 100% when hydroxylamine hydrochloride dosage was 1.5116 mol/L.

Key words: degoldized solution; hydrazine hydrate; hydroxylamine hydrochloride; selenium; tellurium; recovery rate

1 Introduction

Copper anode slimes are valuable by-products generated in copper electrolytic refining process, which account for 0.2%–1.0% (mass fraction) of anode copper [1–5]. Approximately 90% of selenium (Se) and tellurium (Te) are obtained from copper anode slimes, which are also one of the most important materials for extracting gold and silver [6–11]. Generally, the conventional process of treatment of copper anode slime includes the following steps [12–15]. Copper anode slimes are firstly pretreated with different methods to remove copper which is harmful for the subsequent procedure. Then, the decopperization residue is used to recover gold and silver by either hydrometallurgy or pyrometallurgy process. During the process, gold is sometimes separated from the leached residue by chlorination followed by precipitating gold. The obtained products are gold dust with high purity and degoldized solution, which still contains large amount of selenium, tellurium and certain amount of gold and platinum group

metals.

So far, several techniques have been developed for recovering valuable metals from the degoldized solution, and the most popular ones are zinc dust replacement process [16,17] and sulfur dioxide reduction process [18,19]. The zinc dust replacement process is usually designed with the purpose to recover precious metals, especially gold and platinum group metals with low recovery rates on selenium and tellurium. Meanwhile, zinc dust tends to react with H⁺ in degoldized solution to generate H₂, which is the potential security danger for production. The sulfur dioxide reduction process can not only efficiently capture gold and platinum group metals, but also recover Se and Te in colloidal state. However, sulfur dioxide tends to escape from strong acidic solution, which pollutes the environment.

With the increasing demand of valuable metals and more strict requirements of environmental protection, it is necessary to explore suitable reductant with high extraction efficiencies for Se and Te from degoldized solution. The aim of this work is to investigate the novel

reduction system for efficiently extracting both Se and Te from degoldized solution. Firstly, the effect of various reductants on recovery rates of Se and Te was investigated to determine suitable reductant for extracting Se and Te. Then, the process parameters in hydrazine hydrate reduction system were studied in a systematic manner. Finally, the combined system of hydrazine hydrate and hydroxylamine hydrochloride was used to intensify the extraction efficiencies of Se and Te.

2 Experimental

2.1 Materials

A sample of copper anode slimes was provided by one of the copper smelters in China. Copper anode slimes were firstly decopperized by high pressure leaching process, and then followed by chlorination process to separate Au and Ag. In subsequent steps, Au was precipitated from the gold-bearing solution with sodium sulfite and the obtained degoldized solution was used as the raw materials in all experiments, wherein $[H^+]=3.755$ mol/L. Chemical analysis of the solutions was carried out by inductively coupled plasma-atomic emission spectroscopy (Intrepid II XSP, Thermo Electron, USA), and the results are given in Table 1.

Table 1 Main chemical composition of degoldized solution (mmol/L)

Se	Te	Au	Pt	Pd	Cu	Fe	Cl
3.92	23.60	0.18	0.0095	0.11	8.95	2.34	6536

It can be seen from Table 1 that the degoldized solution was rich in Se (3.92 mmol/L) and Te (23.60 mmol/L), while a negligible amount of precious metals (0.18 mmol/L Au, 0.0095 mmol/L Pt and 0.11 mmol/L Pd) was dissolved in degoldized solution.

2.2 Methods

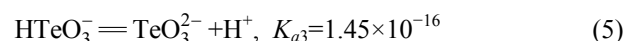
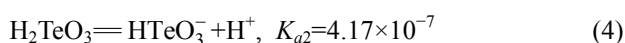
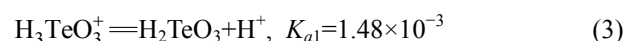
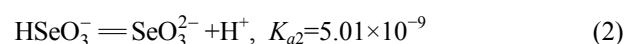
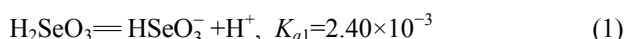
Accurate amount of degoldized solution was added to the flask, a measured amount of reductant was charged to the flask and heated to a specific temperature under magnetically stirring and the reaction initiated. Two reflux condensers were installed at the top of the flask to minimize the loss of solution due to evaporation. After the required time, the slurries were separated by vacuum filtration immediately. The residues were washed with distilled water and then dried in air oven at 105 °C for weighing. The recovery rates of valuable metals were estimated on the basis of materials balance. AR-grade chemical reagents were used in all experiments.

Chemical compositions of the reduction products were analyzed by X-ray fluorescence analysis (ARL Perform'X, Thermo Electron, USA). The crystalline

lattice and the micro morphology of the reduction products were analyzed by X-ray diffraction (D/max-TTR III, Rigaku, Japan) and scanning electron microscope (JSM-6300, JEOL, Japan).

3 Thermodynamic analysis

In copper anode slimes, Se and Te occur as silver and copper selenides and tellurides, while in degoldized solution Se mainly exists as SeO_3^{2-} , $HSeO_3^-$, H_2SeO_3 , and Te mainly exists as TeO_3^{2-} , $HTeO_3^-$, H_2TeO_3 , $H_3TeO_3^+$ [18–21]. The dissociation equations of selenides and tellurides are presented as follows:



The total concentration of $[M(IV)]_T$ in degoldized solution is

$$[M(IV)]_T = [MO_3^{2-}] + [HMO_3^-] + [H_2MO_3] + [H_3MO_3^+] \quad (6)$$

where $[M(IV)]_T$ is the total concentration of $[Se(IV)]_T$ or $[Te(IV)]_T$. The effects of pH value on the distribution of Se(IV) and Te(IV) species are depicted respectively in Figs. 1 and 2 according to Eqs. (1)–(6).

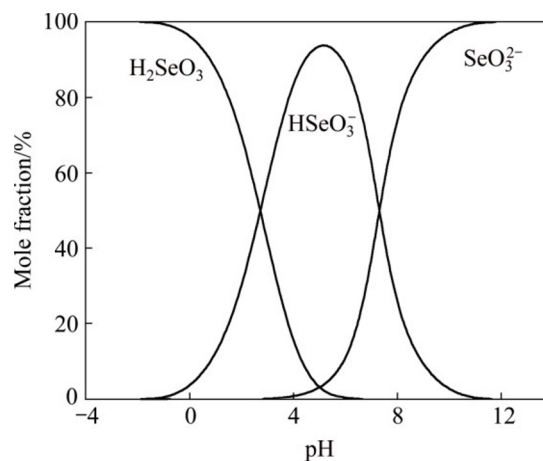


Fig. 1 Relationship between mole fraction of Se(IV) species and pH

Apparently, in strongly acidic solution, the main existing form of Se(IV) is H_2SeO_3 , and that of Te(IV) is $H_3TeO_3^+$ when $pH \leq 0$. In weakly acidic and alkaline solutions, the main existing forms of Se(IV) are $HSeO_3^-$ and SeO_3^{2-} , and those of Te(IV) are H_2TeO_3 , $HTeO_3^-$ and TeO_3^{2-} .

The results of Table 1 indicate that degoldized solution contains large amounts of Se and Te, and small

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