



Original Research Article

A simple method for the recovery of selenium from copper anode slime sample using alkaline roasting process

Hoda Pasdar*, Bahare Hedayati Saghavaz, Masoumeh Masoumi

Department of Chemistry, North Tehran Branch, Islamic Azad University, Tehran, Iran.

*Corresponding author Tel.: +98 (912) 1754903

*E-mail: h_pasdardar@iau-tnb.ac.ir

ABSTRACT

The recovery of selenium from Iranian sar-cheshmeh copper anode slime has been investigated. Copper anode slimes are containing varying precious metals, such as: gold, silver, selenium and tellurium. They are being extracted as a by-product in the production process. Arsenic and antimony that present in anode slimes dissolved in 0.4 M KOH. Then, the alkaline roasting of anode slimes in the presence of Na₂CO₃ solubilized selenium. Various parameters such as time and temperature of alkaline roasting were studied. The results indicated that the optimum condition for the temperature and time of roasting found to be T=600°C and time= 8 h respectively, and that optimum recovery of selenium was 98%.

Keywords: Selenium, Recovery, Sar-cheshmeh, Copper anode slimes, Alkaline roasting.

Introduction

Electrolytic refinery slimes contain those constituents that remain insoluble during the refining process and ultimately sink to the bottom of the electro refining tank [1]. Copper anode slimes from sar-cheshmeh (a mine in the southeast of Iran) are black in color and are extremely fine-grained. The copper anode slimes from sar-cheshmeh are composed mainly of 9.41% selenium, 12.86% copper, 3.35% silver, 0.01% gold and about 45% barite, plus lead, tellurium, antimony, sulphur, silica with small amounts of nickel, iron, zinc, bismuth and trace of arsenic, cadmium, cobalt and manganese [2].

Since selenium is positioned between sulphur and tellurium in the periodic table, they have similar chemical properties [3]. Selenium's proximity to tellurium can result in separation problems when these two elements co-exist in solution. Both are readily precipitated from solution with sulphur dioxide. However, the reduction of tellurium from solution with sulphur dioxide is inhibited in strongly acidic chloride solution. Dilute sulphuric and hydrochloric acid do not attack selenium [4].

Although selenium minerals are scarce, there are several different techniques for recovering selenium from them [5-7]. In the beginning of electrolytic copper refining there was no interest in the recovery of selenium from anode slimes in the copper refining process. Most of the refineries used to gather the precious metals in to one big bullion and then separate them. All unwanted elements were then just slugged off [8]. Today most refineries have one or more roasting or hydrometallurgical step for recovery of the selenium [9]. Hydrometallurgical processes are designed to recover precious metals, such as gold, silver, selenium and tellurium etc, from the anode slimes with small amount of impurities. These processes are economic; energy saving, as well as pollution free [10]. They comprise three stages: 1) leaching, where the valuable elements are dissolved in the aqueous solution, 2) solvent extraction where metals are recovered and purified, 3) reduction where metallic ions are precipitate from the solution [11].

In this study, in order to recover the selenium from anode slime, the optimum conditions for time and temperature were investigated.

Experimental

Material and procedure

A single batch of copper anode slime used in all the experiments which was collected from sarcheshmeh, Iran. Table 1 shows the chemical assays of the sample. The sample ground to-100 mesh after drying at 100 °C for 4 h. Quantitative analyses were carried out using Inductively Coupled Plasma mass spectrometry (ICP-MS) method.

Table 1. Assay of copper anode slime.

Element	Amounts (%)
Cu	1.882
Se	6.377
Te	0.566
Sr	1.042
Ag	6.012
Ba	51.95
Sb	3.219
Au	0.193

Selenium recovery

To remove arsenic and antimony from the sample, 100 ml of 0.4 M solution of KOH was added to 20g of copper anode slime. Then the reaction mixture was refluxed for 5 h. After the reaction mixture reached at room temperature, the precipitate was isolated by filtration and dried in an oven at 100 °C.

After leaching in KOH, the residue obtained was subjected to a wide range of roasting conditions, by varying temperature, time and sodium carbonate/ anode slime ratios, in order to study the amount of selenium. The amount of selenium in the leaching process was determined by ICP-MS.

Experiments were carried out by weighing 5 g of anode slime and mixing the sample with a suitable amount of sodium carbonate. All the experiments were performed by covering the Na₂CO₃/slimes mixture with a 3 mm thick carbonate coat to prevent volatilization. As can be

seen in Table 2, experiments were carried out at various temperatures (400, 600 and 800°C). The mixture was leached with distilled water at room temperature for 4h. In the next step, the participate leached with 0.5 M H₂SO₄ to remove copper from anode slime. The precipitate and solution were then separated with filtration.

Table 2. The effect of various parameters on selenium recovery.

Experiment no.	Temperature	Time	Na ₂ CO ₃ /anode slime	Se recovery (%)
1	400	8	0.6	90
2	600	8	0.6	98
3	800	8	0.6	80
4	600	4	0.6	85
5	600	6	0.6	93
6	600	8	0.6	98

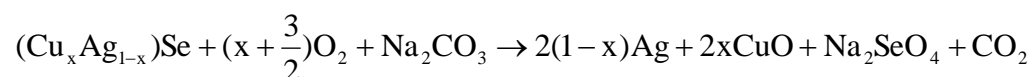
Results and discussion

Leaching in KOH

If anode slimes have not been previously leached in KOH, Arsenic and antimony are also solubilized with selenium after roasting.

Alkaline roasting after KOH leached

The study of 0.6 Na₂CO₃/slime weight ratios was performed in different time and temperature conditions. After roasting, tellurium remained in the solid. The solutions obtained after leaching the roasted residues in water contain selenium as sodium selenate. The optimum conditions for alkaline roasting for temperature and time are 600°C and 8 h, respectively. The alkaline roasting reaction of selenides can be written as:



Effect of temperature

The effect of temperature was studied over the range 400-800 °C. The maximum recovery of selenium was achieved at 600 °C. Effect of the temperature on selenium recovery is shown in Figure 1. It should be mentioned that the time of roasting was 8 h and the Na_2CO_3 /anode slime ratio was 0.6.

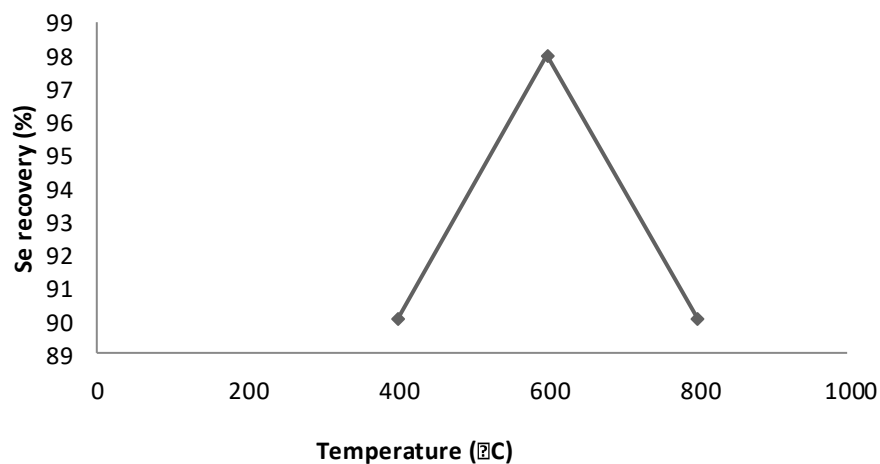


Figure 1. Effect of temperature on selenium recovery

Effect of time

Figure 2 shows the time needed to complete the roasting process. According to the experiments maximum selenium recovery was carried out for 8 h of roasting.

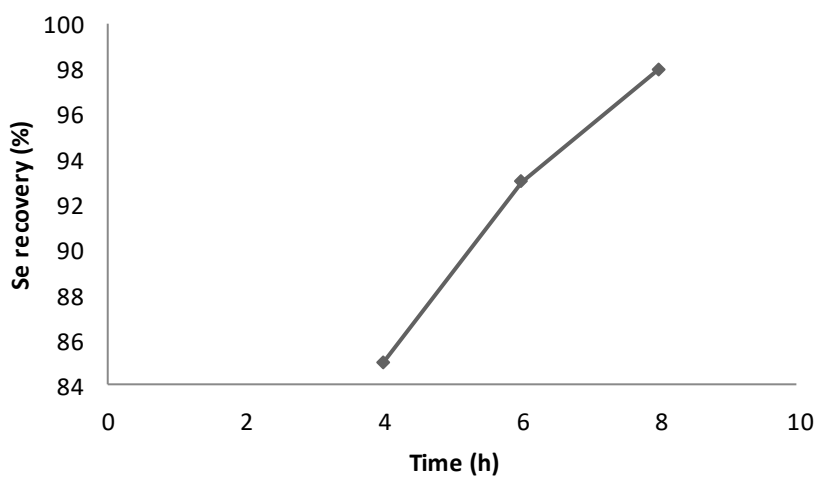


Figure 2. Effect of time on selenium recovery

Proposed flowsheet

Figure 3 shows the proposed flowsheet for slime treatment. By leaching in 0.4 M KOH at 100 °C, arsenic and antimony can be dissolved. The solid resulting from this step is mixed with sodium carbonate (Na_2CO_3 /slime 0.6) and roasted at 600 °C for 8 h. Then the solid is leached with water at room temperature. The leaching solution contains selenium in the form of selenate. To remove copper and tellurium, the solution was leached with 0.5 M H_2SO_4 .

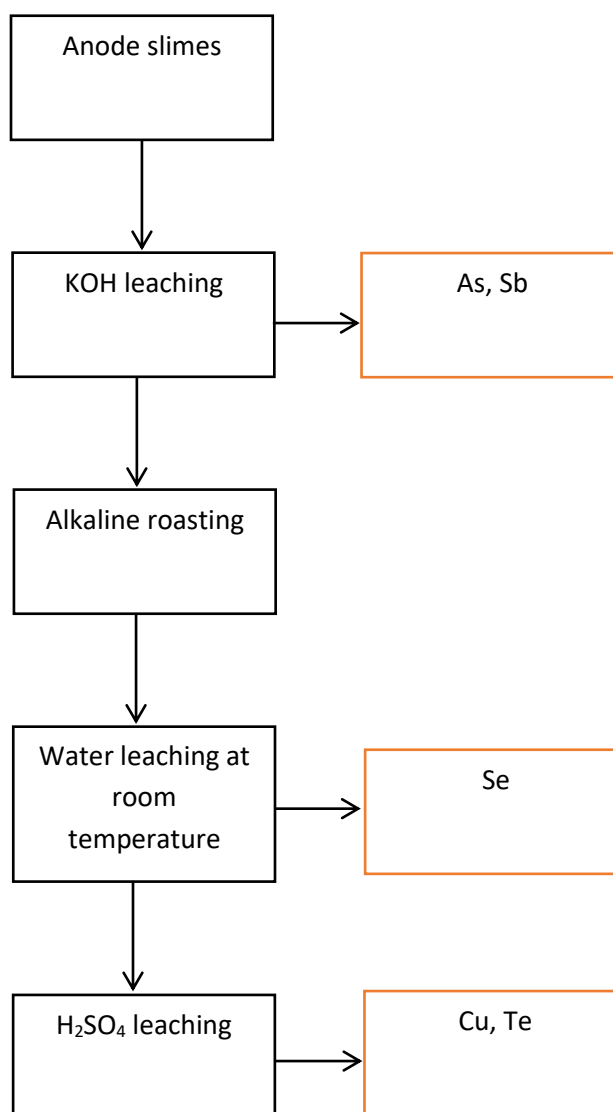


Figure 3. Proposed flowsheet for the treatment of the anode slime.

Conclusion

In this study, the recovery of selenium from copper anode slimes was investigated in a laboratory scale and the results can be summarized as the following:

The alkaline roasting of copper anode slime is an efficient method to recover precious metals such as selenium, tellurium, silver and gold. In this work we focus on recover selenium. In alkaline roasting processes different parameters such as Na_2CO_3 /anode slime ratio, temperature and time can influence the process. The optimum conditions for alkaline roasting found to be Na_2CO_3 /anode slime= 0.6, temperature =600 °C, time= 8h. Under these conditions 98% of the selenium was recovered.

Tellurium can be removed from the solution by H_2SO_4 leaching, in order to increase the yield of selenium recovery.

Acknowledgments

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