

Study of silver leaching with the thiosulfate–nitrite–copper alternative system: Effect of thiosulfate concentration and leaching temperature



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ABSTRACT

Thiosulfate system is considered an interesting alternative leaching process for precious metals. Nevertheless, most of the literature published on these conventional thiosulfate leaching solutions has been focused on the use of ammonia and copper to generate the cupric tetraamine complex, which acts as a catalytic oxidant for silver. However, ammonia toxicity is also a detrimental issue in terms of the process sustainability. For that reason, thiosulfate–nitrite–copper solutions were studied as an alternative less toxic system for silver leaching.

In this work, the effect of the thiosulfate concentration (0.07 M, 0.1 M and 0.15 M) and temperature (room temperature, 30, 35, 40 and 45 °C) on the metallic silver leaching kinetics is presented for the S_2O_3 – NO_2 –Cu system. The results show that the thiosulfate concentration plays an important role in the S_2O_3 – NO_2 –Cu–Ag system since it controls the silver leaching kinetics. On the other hand, an increase in temperature favors the silver recovery.

Finally, the SEM–EDS analysis, the X-ray mapping and the X-ray diffractograms show that the solid silver particles are coated by a Cu, S and O layer for the 0.07 M and 0.1 M thiosulfate experiments, which is consistent with the formation of antlerite ($Cu_3(SO_4)(OH)_4$); while the 0.15 M thiosulfate scenario produced a layer composed only of Cu and S, revealing the formation of stromeyerite (CuAgS). The UV–Visible technique confirmed the in-situ generation of copper–ammonia complexes for the 0.07 M leaching condition; however, these complexes are not formed at the 0.15 M condition.

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1. Introduction

At present time, the mining industry has experienced new technological (e.g. leaching of carbonaceous refractory ores) and environmental (e.g. cyanide toxicity) challenges for the extraction of silver and gold (Aylmore and Muir, 2001; Ahern et al., 2006). In this sense, the thiosulfate has become an excellent alternative to face these challenges.

The thiosulfate system has been studied extensively in the last years as an efficient and economic system for gold and silver leaching due to its ability to produce strong complexes with these metals and to leach them easily from their ores when the system is catalyzed with cupric ions (Aylmore and Muir, 2001).

The catalytic effect of the cupric ions on the leaching solution of the thiosulfate–copper–ammonia system increases the silver and

gold leaching kinetics up to 20 times; this increase is mainly attributed to the formation of a copper–ammonia complex, the so-called cupric tetraamine $Cu(NH_3)_4^{2+}$ (Aylmore and Muir, 2001). Nevertheless, an excessive increase of the cupric ions concentration in the solution has the disadvantage of promoting the thiosulfate degradation into polythionates (Aylmore and Muir, 2001; Zhang and Dreisinger, 2002; Ahern et al., 2006), compromising the precious metals recovery and the process profitability (Ji et al., 2003).

In addition to the formation of the cupric tetraamine, ammonia has another important effect on the leaching kinetics: the ammonia prevents the gold passivation due to its preferential adsorption on the gold surface generating a gold–ammonia complex (Aylmore and Muir, 2001); its absence causes a decrease in the leaching kinetics due to the accumulation of sulfur layers on the precious metal particles as a result of the thiosulfate degradation.

The temperature effect on the silver and gold leaching kinetics has been reported extensively in the literature. Abbruzzese et al. (1995) found that an increase in temperature adversely affect the gold recovery due to the passivation of the gold particle by copper sulfide. This passivation is presumably caused by the excessive loss

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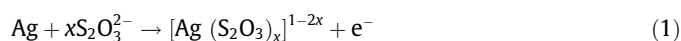
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of ammonia at high temperatures (Aylmore and Muir, 2001; Abbruzzese et al., 1995).

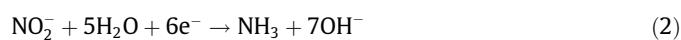
In contrast, Zipperian et al. found that the initial rate of silver and gold extraction is enhanced by an increase in the leach temperature; but the rate falls dramatically after a processing time of one hour (Zipperian et al., 1988).

In the last decades, the nitrogen species catalyzed (NSC) pressure leaching system has been proposed for the recovery of silver, copper, zinc and iron from refractory sulfide ores, as an alternative method to the cyanidation process (Anderson, 1995, 2003; Anderson et al., 1996a,b; Anderson and Nordwick, 1996). This process uses a leaching aqueous solution that contains sulfuric acid and sodium nitrite under oxygen pressure.

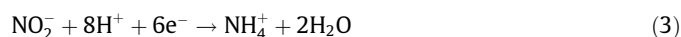
The combination of sodium thiosulfate and sodium nitrite has been used in the United States since the 1930's as the primary antidote for cyanide intoxication (Baskin et al., 1992); however, the combination of these reactants has not been used for silver leaching. In 2015, Alvarado-Macías et al. reported the use of these two reactants for silver dissolution. The reported results revealed that the thiosulfate ions act as the complexing agent for the silver ions:



where x corresponds to 1, 2 or 3 thiosulfate molecules, while the nitrite ions act as the oxidizing agent:



As a result of the nitrite reduction, ammonia will be predominantly formed at pH values above 9, while ammonium will be predominantly formed at pH values below 9.



The addition of copper ions in the leaching system is important in order to form cupric ammonia complexes (Eq. (4)) which catalyze the silver dissolution; their formation in situ was possible due to the presence of the ammonia produced by the system (Eq. (2)).



where x corresponds to 1, 2, 3 or 4 ammonia molecules.

According to the results reported by Alvarado-Macías et al. (2015), the best silver leaching system was a 0.1 M thiosulfate, 1.5 M nitrite and 0.05 M copper solution with a natural pH of 7.2 and a natural redox potential of 0.369 V vs SHE. This leaching solution dissolves 96% of silver after 70 min. It was also found in that research that a molar ratio of 30:1 nitrite:copper is necessary to maintain the best conditions for the in-situ generation of cupric ammonia complexes.

The main objective of this research was to evaluate the effect of the thiosulfate concentration and the temperature on the silver leaching kinetics employing the thiosulfate–nitrite–copper alternative leaching system in order to find the best conditions to accelerate the silver leaching.

2. Materials and methods

This section describes the materials, equipment, reagents, leaching solution composition and the experimental methodologies used in this research work.

Leaching solutions were prepared with analytical grade reagents and deionized water. The reagents were: metallic silver (99.999%, 1–3 μm , spherical, Alfa Aesar), sodium nitrite (97.2%, Analytika), cupric sulfate pentahydrate (99%, Merck) and sodium thiosulfate pentahydrate, crystals (100%, J.T. Baker).

In order to elucidate the predominant species in the thiosulfate–nitrite–copper–silver system, species distribution diagrams were

constructed at room temperature using the Medusa Software and considering the equilibrium data contained in the Hydra database of the same software (Puigdomenech, 2004). These diagrams were used to determine all the aqueous and solid species that can be formed in the leaching solutions at different pH and potential conditions.

All silver leaching experiments were performed in an agitated batch reactor (250 mL glass beaker); the reactor was provided with a pH meter (pH 538 WTW), a pH electrode (SENSOREX), a redox potential electrode with Ag/AgCl reference (Cole-Parmer) and a magnetic stirring plate. The general procedure for the leaching experiments was as follows: 200 mL of the leaching solution with sodium thiosulfate, sodium nitrite, cupric sulfate and 0.1266 g of metallic silver were placed in the reactor at room temperature. The effect of thiosulfate concentration (0.07 M, 0.1 M and 0.15 M) and temperature (room temperature, 30, 35, 40 and 45 °C) on the silver leaching was tested during 6 h. The pH and redox potential were measured before the silver addition and during the leaching experiments (every 30 min). Samples of the leaching solution were withdrawn each 5 min or 30 min, during 6 h for the silver analysis by atomic absorption spectrophotometry (Varian SpectrAA 220FS). All samples were kept in dark vessels to prevent the silver precipitation caused by light. Once the experiments were concluded, the residues were filtered, rinsed with deionized water and left to air dry.

The solid residues i.e., unreacted silver and solid products formed at different times (15 min, 30 min and 6 h) were microstructurally characterized (superficially and in cross section) in order to elucidate their morphology and local chemical composition by scanning electron microscopy (SEM) (Phillips, XL30E-SEM), X-ray mapping and Energy-dispersive X-ray spectroscopy (EDS) (EDAX, Genesis). Some of the solid residues were also analyzed by X-ray diffraction (XRD) (Phillips, X'PERT PW3040) in order to identify the predominating species.

The formation of cupric–ammonia complexes in the leaching solutions was studied by UV/Vis spectroscopy. A 3 mL quartz cuvette (10 mm path length) was used to characterize the solutions in the wavelength range from 500 to 950 nm (UV-1201S, Shimadzu).

3. Results and discussion

In this section are presented the results obtained for the silver leaching tested at room temperature and different temperatures. These results are discussed taking into consideration the thermodynamic diagrams, the solid residue characterization and the liquid UV/Vis spectroscopy analysis of the solutions.

3.1. Effect of thiosulfate concentration on the silver leaching kinetics

3.1.1. Leaching test at room temperature

In order to understand the thiosulfate concentration effect on the silver leaching kinetics for the $\text{S}_2\text{O}_3\text{--NO}_2\text{--Cu--Ag}$ alternative system, three different concentrations were tested (0.07 M at natural pH of 6.6; 0.1 M at natural pH of 7.27, and 0.15 M at natural pH of 8.6). Fig. 1 presents the progress of the silver recovery for the three thiosulfate concentrations at room temperature with 1.5 M NO_2^- , 0.05 M Cu^{2+} and 0.1266 g Ag.

In this figure the result of the optimal condition for the $\text{S}_2\text{O}_3\text{--NO}_2\text{--Cu--Ag}$ solution reported by Alvarado-Macías et al. (2015) is shown to make a comparison with the other two thiosulfate concentrations. In the case of 0.1 M concentration reported by Alvarado-Macías et al. (2015), the silver recovery reaches a 96% at 6 h; this result shows an efficient and promising process to be employed as an alternative silver leaching system. For the test with 0.07 M thiosulfate, the silver recovery reached 89% at 6 h; this

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