



## An electrochemical study of silver recovery in thiosulfate solutions. A window towards the development of a simultaneous electroleaching-electrodeposition process



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### ABSTRACT

The simultaneous electroleaching-electrodeposition of silver was studied systematically to develop an alternate process for the recovery of metallic silver. The system was assessed employing electrochemical and microstructural characterization techniques in a thiosulfate-ammonia medium. The electrochemical studies revealed the dependence of silver electroleaching rate in thiosulfate-ammonia solutions on different processes controlled by anodic potential. At the anodic potential below 200 mV<sub>SHE</sub>, the silver is electro-oxidized and complexed by the applied potential and thiosulfate, respectively. Increasing the potential up to 445 mV<sub>SHE</sub>, the thiosulfate is oxidized and the silver leaching current is decreased due to the diffusional control of the leaching. It was also found that the sulfite species formed from the thiosulfate decomposition can leach small amounts of metallic silver. Finally, at potentials from 695 to 795 mV<sub>SHE</sub>, the thiosulfate is further decomposed and a silver sulfide film is generated which inhibits the silver dissolution. The simultaneous electroleaching-electrodeposition of silver was also studied in an electrolytic cell with graphite electrodes at room temperature. The results showed that a 74% of silver can be recovered at a low applied current density of 0.066 mA/cm<sup>2</sup> with a negligible thiosulfate decomposition and a current efficiency of 76.54%. On the other hand, when the current is increased the silver recovery and current efficiency is decreased, due to the oxidative decomposition of thiosulfate which promotes the formation of silver sulfide. These results are corroborated by the X-ray diffraction and SEM-EDS analyses.

### 1. Introduction

Currently cyanidation is the most used process in the hydrometallurgical industry to carry out the leaching of precious metals from different sources (Zhang, 2004; Aylmore and Muir, 2001). However, the main disadvantage of this process is the environmental risk associated with the cyanide toxicity (Eisler and Wiemeyer, 2004; Aylmore, 2016a, 2016b). In view of this, the alternative hydrometallurgical techniques that have of late been investigated involve the use of thiourea and thiosulfate as the lixiviants (Abbruzzese et al., 1995; Xu et al., 2015; Deutsch and Dreisinger, 2013a, 2013b; Grosse et al., 2003; Senanayake, 2004, 2012; Örgül and Atalay, 2000, 2002; Yang et al., 2008; Aylmore et al., 2014; Puente-Siller et al., 2013, 2014, 2017; Alvarado-Macías et al., 2015, 2016; Breuer and Jeffrey, 2000).

As regards the metallic silver, it can be found in different wastes, e.g., the so-called urban ores. From the sustainability point of view, it is

extremely important to recover metallic silver from these urban ores. In this context, it is important to mention the attempts made on the dissolution of metallic silver and silver compounds from the printed circuit boards (PCBs), electronics, spent catalysts, fabrics containing silver nanoparticles, jewelry, and X-ray and photographic films (Ficeriová et al., 2008; Gurung et al., 2013; Jadhav and Hocheng, 2012; Pasricha et al., 2012; Pan-Pan et al., 2014; Ajiwe and Anyadiegwu, 2000; Atia et al., 2014). The dissolution of metallic silver has been reported using the alternative leaching systems in the presence of thiosulfate or ozone (Viñals et al., 2005; Puente-Siller et al., 2013, 2014, 2017; Alvarado-Macías et al., 2015, 2016; Rivera et al., 2015). From a kinetic viewpoint, the thiosulfate systems seem to be the most promising for the extraction of precious metals including the silver (Aylmore and Muir, 2001). Recently, Barrick developed a thiosulfate leaching process on commercial scale at Goldstrike, Nevada, U.S.A. (Aylmore, 2016a; Aylmore, 2016b). However, the main disadvantage of the thiosulfate

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systems relates to the susceptibility of thiosulfate to the oxidative degradation to different polythionates due to the interaction of thiosulfate ions with the oxidizing agents (e.g. cupric tetraamine), commonly found in the thiosulfate systems (Aylmore and Muir, 2001). Actually, Puente-Siller et al. (2013, 2014) and Alvarado-Macías et al. (2015, 2016) have recently shown that Cu–S coatings are formed on the silver particles due to the degradation of thiosulfate. The coating of the Cu–S inhibits the direct contact between the lixiviant and unreacted silver particles.

In order to reduce the thiosulfate degradation, different researchers have proposed the use of complexing agents for cupric ions to lower the redox potential thereby moderating the oxidizing ability of the system (Puente-Siller et al., 2013, 2014, 2017; Feng and Van Deventer, 2010). This suggests that silver may be leached in thiosulfate solutions using a device such as an electrolytic cell which can ensure the control of the oxidizing ability of the system and can minimize the thiosulfate degradation. Several authors have reported the fundamental electrochemistry of gold in thiosulfate media (Pedraza et al., 1988; Zhang and Nicol, 2003; Baron et al., 2011, 2013; Breuer and Jeffery, 2002; Jeffrey et al., 2003, 2008; Zelinsky and Novgorodtseva, 2013; Gudkov et al., 2010a; Zhang and Nicol, 2005; Zelinsky, 2014, 2015; Chu et al., 2003). However, only a few works related to the electrochemistry of silver in thiosulfate solutions are reported in the literature, stressing the electro-dissolution of silver electrodes (Bek and Shevtsova, 2011; Yavors'kyi et al., 1999).

On the other hand, various electrolytic processes have been developed to electro-leach the metals such as copper, nickel, cadmium etc., contained in different materials, e.g., minerals or e-wastes (Fuentes-Aceituno et al., 2008a; Martínez-Gómez et al., 2016; Guimaraes et al., 2014; Hazotte et al., 2016). One of the main advantages of the electrolytic processes is related to the possibility of achieving the selective leaching of a metal of choice (Guimaraes et al., 2014) depending upon the electrode potential (Fuentes-Aceituno et al., 2008a). Furthermore, it has been demonstrated that an electrolytic process can be versatile; i.e., it is possible to carry out the simultaneous electroleaching-electrodeposition of metals such as copper, nickel and cadmium (Guimaraes et al., 2014; Hazotte et al., 2016).

In this research, the electroleaching of silver with thiosulfate solutions has been studied in a single chamber-three electrodes electrochemical cell, using carbon paste electrodes at different applied anodic potentials to ensure the desired oxidizing conditions instead of a chemical oxidant; this is attempted to gain a better understanding of the electroleaching behavior of silver and establish the most suitable conditions to accelerate the leaching kinetics. This novel concept was employed to maximize the silver recovery and minimize the energy consumption and thiosulfate decomposition in the simultaneous electroleaching-electrodeposition (SEE) process at room temperature. This information can be used in future to develop an efficient SEE reactor for the recovery of metallic silver contained in different wastes, using thiosulfate solutions without using any chemical oxidizing agents (e.g., cupric ions).

## 2. Materials and methods

### 2.1. Materials

Carbon paste electrodes (CPE) with and without electro-active species (Ag, Ag<sub>2</sub>S) were prepared using graphite powder (Alfa Aesar, 99.9995%, 2–15 μm), silicone oil (Sigma Aldrich, ρ = 0.96 g/mL) as binder, silver powder (Alfa Aesar, 99.99%, 1.3–3.2 μm), silver sulfide powder (Alfa Aesar, 99%, 5–15 μm) as the electro-active species. These materials in the desired proportions were thoroughly mixed in an agate mortar to prepare the carbon paste while using a polyethylene syringe (0.7 cm length and 0.2 cm inner diameter) as the container for the paste. A platinum wire immersed in the mixture was welded to a copper wire with silver solder to establish the electrical contact with the carbon

paste electrode. A 1200-grit silicon carbide paper was used for re-newing and polishing the carbon paste electrode surface in all cyclic voltammetry tests.

All electrolytic media were prepared in a 50 mL volumetric flask using reagent grade chemicals (sodium thiosulfate, sodium sulfite and ammonium hydroxide). All solutions used in this study employed deionized water. High purity nitrogen was bubbled into the solutions to eliminate the dissolved O<sub>2</sub> and CO<sub>2</sub>.

Voltammetry and chronoamperometry tests were carried out in a conventional single chamber-three electrodes electrochemical cell which consisted of a 100 mL beaker (Pyrex) with a three holes Teflon cap, a calomel reference electrode, SCE (Radiometer Analytical), a graphite counter-electrode (Alfa Aesar, 99.99%), a carbon paste CPE with or without electro-active species (Ag and Ag<sub>2</sub>S), as the working electrode, and a magnetic stirrer. The electrochemical cell was connected to a potentiostat/galvanostat (Versastat 4-400, PAR).

### 2.2. Preparation of the carbon paste electrodes with electro-active species (CPE-Ag or CPE-Ag<sub>2</sub>S)

The procedure for preparing the carbon paste electrode containing the electro-active species is as follows: 0.8 g of graphite powder and 0.2 g of the electro-active species (silver or silver sulfide) were mixed in an agate mortar for 40 min, adding 0.20 mL of silicone oil to obtain a homogeneous paste (Fuentes-Aceituno et al., 2008b). Carbon paste electrodes without the electro-active species were prepared in the same manner. The mixture was then placed in the polyethylene syringe and was compacted using the plunger. The exposed area of the carbon paste electrodes to the electrolytic medium was about 0.0314 cm<sup>2</sup>.

### 2.3. Electrochemical experiments for the dissolution of silver

Since one of the objectives of the present investigation is to study the dissolution of silver as well as the oxidative destruction of thiosulfate at different conditions of anode potential employing carbon paste electrodes with and without electro-active species, several solutions of thiosulfate and sulfite were assessed by cyclic and linear voltammetry or chronoamperometry techniques.

The electrolytic medium consisting of 50 mL of a 0.1, 0.2 and 0.3 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> or 0.2 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> with 0.05 M Na<sub>2</sub>SO<sub>3</sub> solution at pH 10.2, adjusted with ammonium hydroxide, and room temperature was poured in the cell, and a stream of nitrogen was bubbled into the solution for 30 min. Then the CPE (with or without silver) was placed in the cell as the working electrode. The cell was connected to the potentiostat/galvanostat and open circuit potential (OCP) measurements were performed until obtaining stationary potential readings. Then cyclic voltammograms were recorded with a potential sweep velocity of 25 mV/s from the OCP in the positive direction at different vertex (inversion) potentials (E<sub>v</sub>) (800 and 1000 mV<sub>SHE</sub>), finishing the cycle at the OCP. In order to electrochemically characterize the solid products formed on the CPE-Ag surface during the anodic scan, cyclic voltammograms were recorded with the pre-treated CPE-Ag and a fresh CPE-Ag<sub>2</sub>S, sweeping the potential at 25 mV/s from the OCP into the cathodic direction up to –800 mV<sub>SHE</sub>, and then finishing the cycle at the OCP.

Stirred chronoamperometric experiments were carried out using a CPE with 8.1 mg Ag impregnated on the exposed CPE surface. This electrode was prepared as follows: the CPE surface was placed putting pressure on the silver powder (Alfa Aesar, 99.99%, 1.3–3.2 μm) contained in a plate. A solution containing 0.2 M Na<sub>2</sub>SO<sub>3</sub> at pH 10.2 (adjusted with ammonia) was assessed at room temperature. The chronoamperometric curves were recorded applying an anodic potential of 425 mV<sub>SHE</sub> during 133 min. Samples of the leaching solution were withdrawn at the end of the experiment to analyze silver by atomic absorption spectrometry (VARIAN Spectra AA 240).

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