

Article

# Leaching Behavior of Gold and Silver from Concentrated Sulfide Ore Using Ammonium Thiosulfate

Mooki Bae <sup>1,2</sup>, Sookyung Kim <sup>1,2,\*</sup>, Jeongsoo Sohn <sup>2</sup>, Donghyo Yang <sup>2</sup> and Hyunju Lee <sup>2,\*</sup>

<sup>1</sup> Resources Recycling, University of Science and Technology (UST), 217, Gajeong-ro, Yuseong-gu, Daejeon 34113, Korea; muki.bae@kigam.re.kr

<sup>2</sup> Mineral Resources Research Division, Korea Institute of Geoscience and Mineral Resources (KIGAM), 124 Gwahak-ro, Yuseong-gu, Daejeon 34132, Korea; jss@kigam.re.kr (J.S.); ydh@kigam.re.kr (D.Y.)

\* Correspondence: skkim@kigam.re.kr (S.K.); hlee@kigam.re.kr (H.L.);  
Tel.: +82-42-868-3020 (S.K.); +82-42-868-3151 (H.L.)

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**Abstract:** Ammonium thiosulfate is an alternative lixiviant for the hydrometallurgical treatment of sulfide gold ores. The present study is primarily focused on ammonium thiosulfate leaching of gold (Au) and silver (Ag) from the sulfide ore (Sunshin mine in Korea). The main chemical composition of the concentrate was Au (84 ppm), Ag (852 ppm), Fe (18.9%), Si (23.2%), and S (21.1%). The effects of various parameters on the process, such as leaching time (1–4 h), ammonium thiosulfate concentration (0.05–0.5 M), copper sulfate (CuSO<sub>4</sub>) concentration (0.05–0.25 M), solid to liquid ratio (0.2–0.5), and reaction temperature (40–60 °C) were systematically examined. Optimum Au leaching efficiency (>99%) was obtained under the following leaching conditions: 0.5 M ammonium thiosulfate with 0.05 M CuSO<sub>4</sub> concentration, 0.2 S/L ratio at 60 °C for 2 h. The results indicate that the behavior of Ag was similar to that of Au. Almost complete dissolution of Ag occurred under following leaching conditions: 0.5 M ammonium thiosulfate with 0.05 M CuSO<sub>4</sub> concentration at 60 °C for 4 h. This study would be useful in understanding the eco-friendly leaching systems of Au and Ag during the hydrometallurgical process of sulfide gold ore.

**Keywords:** concentrated sulfide ore; ammonium thiosulfate; copper sulfate; leaching; gold; silver

## 1. Introduction

For over 100 years, cyanide has been the most common leaching reagent for gold (Au) recovery from ores and concentrates. Although cyanide has advantages including high Au recoveries, robustness, and relatively low costs, it is highly toxic and thus harmful to human health and the environment [1,2]. Various alternative lixivants, such as halide, thiourea, and ammonium thiosulfate, have been extensively researched in the last few years [1]. Among them, ammonium thiosulfate, as an alternative to cyanide, has received much attention recently for a relatively cheap and nontoxic reagent solution [3].

In the 1990s, few researchers conducted Au leaching studies using ammonium thiosulfate with cupric ions acting as an oxidant [4–7]. Several studies to recover Au using ammoniacal thiosulfate have been reported in the past [8–12]. Senanayake explained the leaching behavior of Au by Cu (II) in ammoniacal thiosulfate solutions in the presence of additives [8,9]. The chemistry of the ammonia-thiosulfate-copper system is complex due to the simultaneous presence of complexing ligands such as ammonia and thiosulfate, Cu(II)-Cu(I) redox couples in a solution. Hence, the practical application also requires consideration of the oxidation and stability of various sulfur species [13]. Aylmore and Muir conducted a thermodynamic analysis to provide a range of pH and Eh conditions for reagent concentrations [13]. In addition, the thiosulfate leaching process for Au extraction has

been investigated in terms of leaching mechanism and kinetics [14–17]. Lampinen et al. investigated the ammoniacal thiosulfate leaching of pressure oxidized sulfide gold concentrate [18]. As a result, the highest Au extraction (89%) was achieved in 6 h with the following leaching solutions: 0.2 M thiosulfate concentration, 0.2 M ammonia ( $\text{NH}_3$ ), 0.1 g/L Cu [18].

In the case of silver (Ag), several studies of leaching behavior have also been carried out by applying thiosulfate [19–21]. Ficeriova et al. investigated the mechanical activation and mechanochemical pretreatment step for thiosulfate leaching of Ag, Au, and bismuth (Bi) from sulfide concentrate [19]. It was possible to achieve more than 90% recovery of the examined metals. Alonso-Gómez and Lapidus carried out thermodynamic analyses for the different compositions of the leaching solution to determine their ability to extract Ag, lead (Pb), and Au [20]. Ayata and Yildiran suggested optimized conditions for the extraction of selective Ag from Ag sulfide concentrates by thiosulfate leaching [21].

The application of thiosulfate leaching behavior for Au recovery depends on the associated minerals. Pyrite, chalcopyrite, chalcocite, bornite, pyrrhotite, and arsenopyrite are the main sulfide minerals responsible for locking Au in refractory sulfide gold ores [22]. The leaching behavior of these minerals could play an important role in the thiosulfate leaching of Au sulfide ores [22]. Few researchers have studied the effect of associated minerals on Au leaching [10,23]. However, the composition of precious metals (including Au and Ag) and base metals is highly dependent on the sulfide ores, and hence their properties are also different. Thus, it becomes necessary to study the leaching behavior of such sulfide ores.

The aim of the present study is to develop ammonium thiosulfate leaching of Au and Ag from the sulfide ore (Sunshin mine in Korea). This study is mainly focused on the leaching behavior of Au and Ag with respect to changes in ammonium thiosulfate and copper sulfate ( $\text{CuSO}_4$ ) concentrations. The parameters studied for the optimization of the process conditions were ammonium thiosulfate (0.05–0.5 M), reaction time (1–4 h), reaction temperature (40–60 °C), solid to liquid ratio (0.2–0.5), and  $\text{CuSO}_4$  concentration (0.05–0.25 M).

## 2. Materials and Methods

### 2.1. Materials

The gold ore sample was supplied from Sunshin Mine (or Moisan Mine), which in Haenam epithermal gold mineralized area, Korea. Those ore was concentrated by crushing, ball mill, hydro-cyclone, and flotation. Samples were then collected for chemical analysis and size distribution characterization. Quantitative analysis by XRD was carried out by estimation of XRD with SRM (Standard Reference Material) using PDXL (Ver. 2.8.4.0., Rigaku Ltd., Akishima, Tokyo, Japan) to determine the mineralogy of the concentrated sulfide ore, as shown in Figure 1. It indicates that the ore contains the quartz: 40.5%, pyrite: 40.1%, and muscovite: 19.3%.

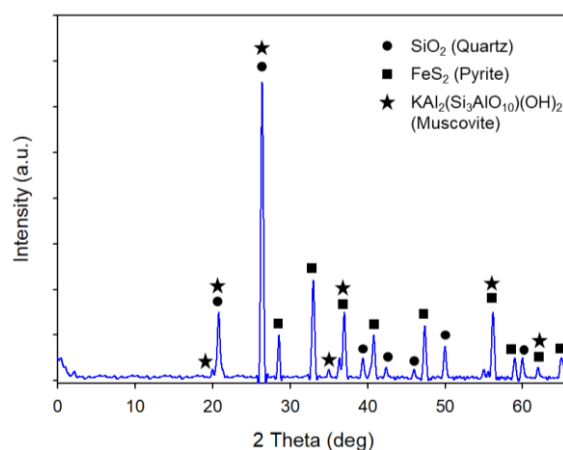
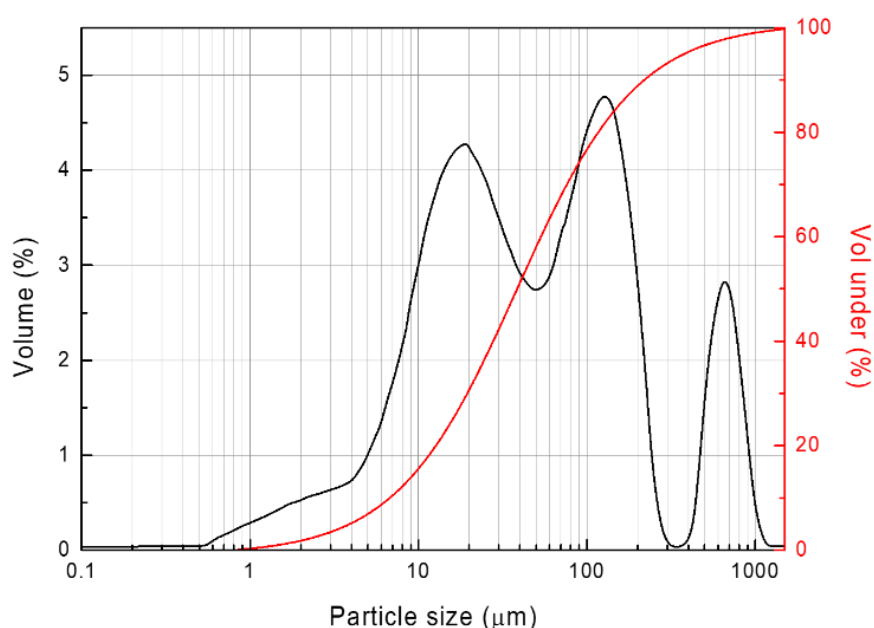


Figure 1. XRD pattern of concentrated sulfide gold ore.

The particle size distribution of the gold concentrates by a particle size analyzer (Mastersizer 2000, Malvern Panalytical Ltd., Malvern, UK) is seen in Figure 2. The particle size analysis showed that the D50 value of the concentrated gold ore after flotation was 45.3  $\mu\text{m}$ . Table 1 shows the results of chemical analysis of the ore. Au and Ag content was determined as 84 ppm and 852 ppm, respectively, by fire-assay, and other elements were determined by acid digestion followed by atomic absorption spectroscopy (A400 model, Perkin Elmer Ltd., Waltham, MA, USA). Sulfur content determined by a Carbon/Sulfur Determinator (CS230 Series, LECO Ltd., St. Joseph, MI, USA) was 21.1 wt.%.



**Figure 2.** Particle size distribution of concentrated sulfide gold ore.

**Table 1.** The chemical analysis of concentrated sulfide ore (Au\*, Ag\*: ppm).

Element	Au*	Ag*	Al	Fe	Mg	Na	Pb	Cu	Zn	Ti	Si	S
Contents (wt.%)	84.0	852	2.42	18.9	0.0820	0.160	0.270	0.380	0.440	0.170	23.2	21.1

For the leaching experiment, ammonium thiosulfate ( $(\text{NH}_4)_2\text{S}_2\text{O}_3$ ), Copper (II) sulfate ( $\text{CuSO}_4$ ), and sodium hydroxide ( $\text{NaOH}$ ) were used as the reagents. A pH meter (Orion 3-Star Benchtop, Thermo Scientific Ltd.) was used for the pH measurements of the leaching solutions.

## 2.2. Methods

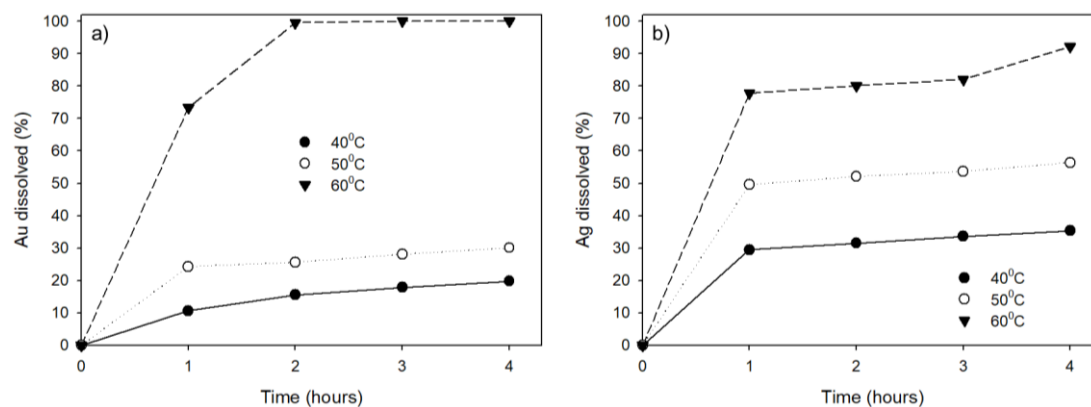
The as-received sulfide ore was dried in an oven at 80  $^\circ\text{C}$ . For the thiosulfate leaching tests, the ammonium thiosulfate (0.05–0.5 M) with  $\text{CuSO}_4$  (0.05–0.25 M) was dissolved in distilled water. Experiments were carried out in a 150 mL glass reactor located in a heating mantle equipped with a pH meter and mechanical stirrer. As soon as the temperature of the flask reached the set temperature, 30–75 g of the given ore (S/L ratios of 0.2, 0.33, and 0.5) were added to it. A stirrer with the rotational speed of 300 rpm was used to mix the solution. Samples of the solution were withdrawn at different time intervals (1–4 h). After suitable dilution, Au and Ag contents were determined by AAS in combination with the fire assay method for analysis of residues in order to estimate the leaching efficiency. The leaching experiments were performed twice to minimize the sampling error.

### 3. Results and Discussion

#### 3.1. Effect of Temperature on Au and Ag Leaching Efficiency

For investigating the leaching behavior, the temperature variation was carried out from 40 to 60 °C for 1 h, 0.2 S/L ratio, pH 9.5 using 0.5 M ammonium thiosulfate, and 0.05 M CuSO<sub>4</sub>.

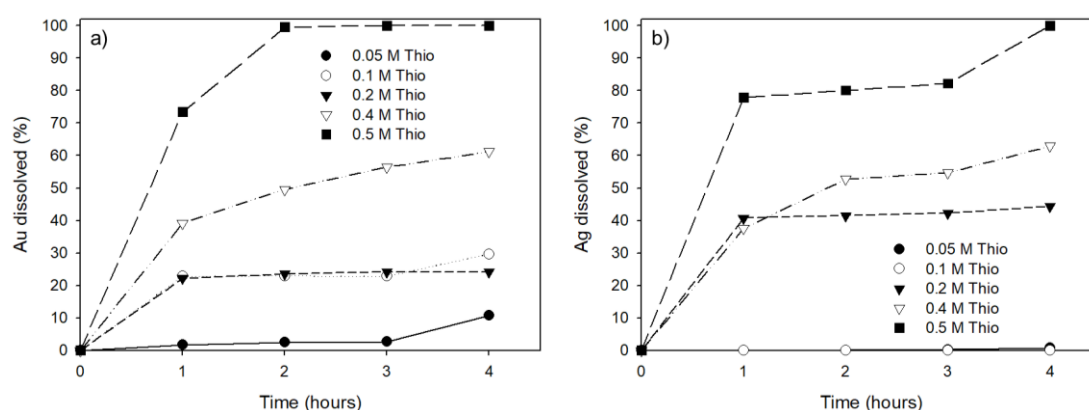
The effect of temperature and time on Au and Ag leaching by ammonium thiosulfate solutions is shown in Figure 3. As shown in Figure 3a, the Au leaching efficiency increases with the increase in temperature in the range of 40 °C to 60 °C, and >99% leaching efficiency is reached at 60 °C after 2 h. However, in case of Ag (Figure 3b), the leaching efficiency rapidly increased up to 1 h of reaction at all temperatures, and then gradually increased over next time intervals. The results of the present study indicate that, with leaching process conditions at 60 °C for 4 h, the Au and Ag were found to be almost dissolved. Contrary to our results, Abbruzzese et al. found that Au recovery decreases with increasing temperature from 25 to 60 °C [6]. They further suggested that the kinetics of cupric sulfide film formation is very high at 60 °C, which hinders Au dissolution [6]. However, in this study, no decrease in Au recovery was observed throughout the temperature range (40 to 60 °C). Based on the results obtained, the temperature of the leaching experiment was selected as 60 °C.



**Figure 3.** Effect temperature and time on metals leaching efficiency: (a) Au, (b) Ag. (Experimental conditions: 0.5 M ammonium thiosulfate, 0.05 M CuSO<sub>4</sub>, 0.2 S/L ratio, pH 9.5).

#### 3.2. Effect of Ammonium Thiosulfate Concentration on Au and Ag Leaching Efficiency

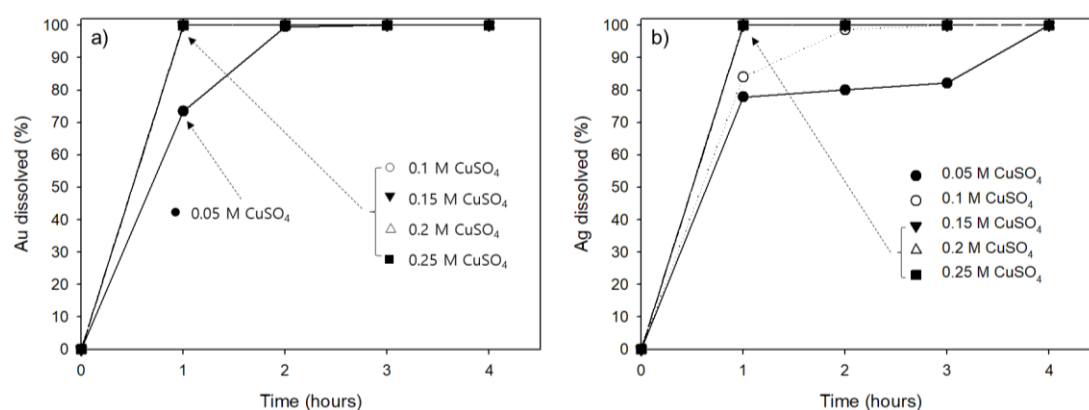
Figure 4 shows the effect of ammonium thiosulfate concentration (in the range of 0.05–0.5 M) on the leaching efficiency of Au and Ag, as a function of time (from 1 to 4 h) with 60 °C temperature, 0.2 S/L ratio, and 0.05 M CuSO<sub>4</sub> concentration. It is observed that the leaching efficiency of Au and Ag increases with increasing concentration of ammonium thiosulfate and the chemical reaction time. As shown in Figure 4a, in the case of Au, there was no leaching of Au for 0.05 M concentration of ammonium thiosulfate until 3 h. In contrast, as the ammonium thiosulfate concentration was increased from 0.4 M to 0.5 M, Au dissolved gradually with increasing order, and, in particular, a leaching efficiency of >99% was observed for Au at 0.5 M ammonium thiosulfate for 2 h. The leaching behavior of Ag was found to be similar to that of Au. Ag was not leached for 0.05 to 0.1 M concentrations of ammonium thiosulfate (Figure 4b); however, Ag was almost completely dissolved with 0.5 M concentration of ammonium thiosulfate for 4 h. This clearly indicates that the leaching efficiency of both Au and Ag increased with the increasing concentration of ammonium thiosulfate, and at least 0.4 M or more of ammonium thiosulfate is required to leach Au and Ag in this system. In this study, Au and Ag were almost completely dissolved by ammonium thiosulfate. It is probably due to not trapping Au and Ag into the pyrite or quartz.



**Figure 4.** Effect of ammonium thiosulfate concentration on metals leaching efficiency: (a) Au, (b) Ag. (Experimental conditions: 0.05 M  $\text{CuSO}_4$ , 0.2 S/L ratio, pH 9.5, 60 °C).

### 3.3. Effect of Copper Sulfate Concentration on Au and Ag Leaching Efficiency

Figure 5 shows the leaching efficiency of Au and Ag with the variation in  $\text{CuSO}_4$  concentration. These studies were carried out at 60 °C temperature and 0.2 S/L ratio using the 0.5 M ammonium thiosulfate solution. As shown in Figure 5a, the leaching efficiency of Au during the first hour increased rapidly. At higher Cu ion concentrations (from 0.25 M to 0.1 M), the leaching efficiency of Au reached up to >99% in 1 h. With 0.05 M  $\text{CuSO}_4$  concentration, the leaching efficiency of Au was increased from 73.4% to >99% during the 1–2 h interval. In the case of Ag (Figure 5b), an increase in the leaching efficiency of Ag was observed, as the concentration of  $\text{CuSO}_4$  was increased from 0.05 M to 0.25 M. From 0.15 M to 0.25 M of Cu ion concentrations, the leaching efficiency of Ag reached up to >99% in the 1 h. Similar to Au, even for a small  $\text{CuSO}_4$  concentration (0.05 M), almost 77.8% Ag was leached in the 1 h and >99% Ag dissolution was found in 4 h.



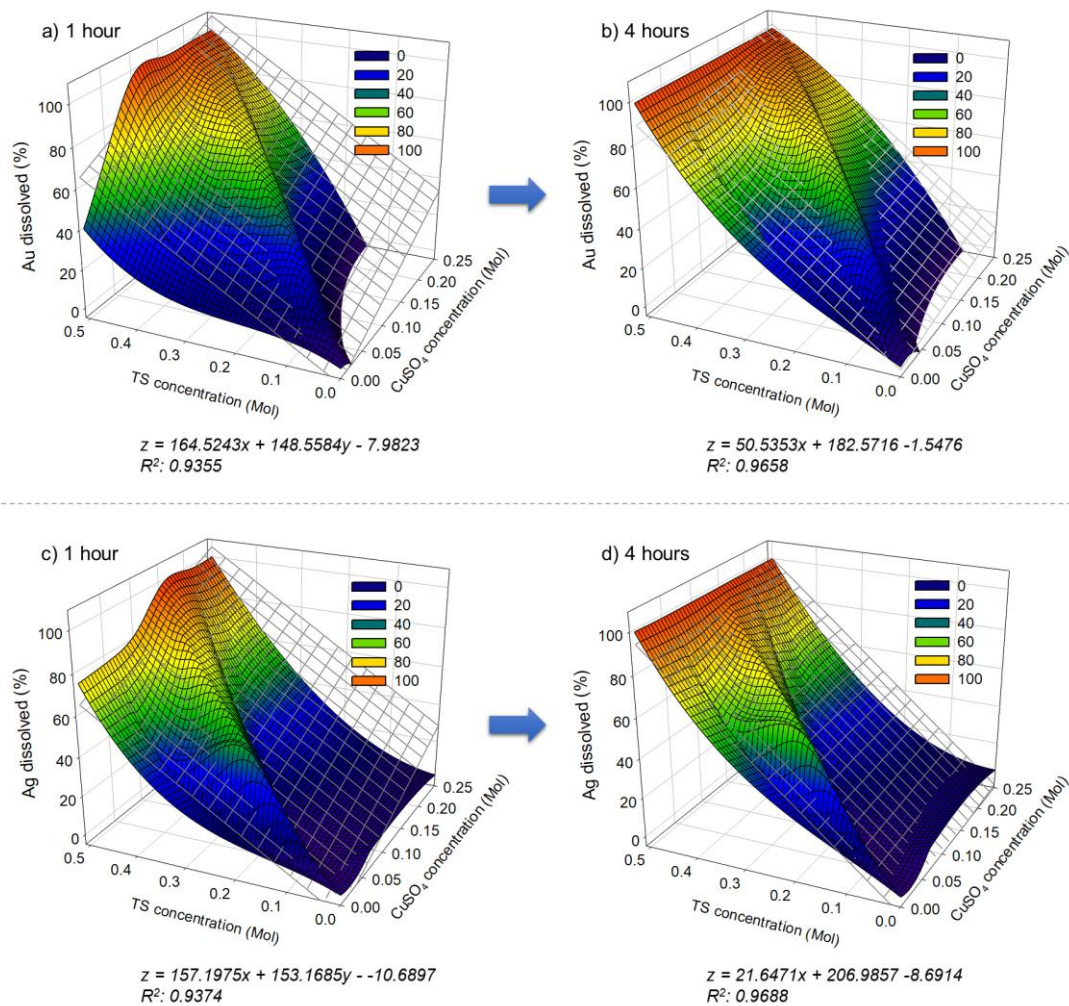
**Figure 5.** Effect of  $\text{CuSO}_4$  concentration on metals leaching efficiency: (a) Au, (b) Ag. (Experimental conditions: 0.5 M ammonium thiosulfate, 0.2 S/L ratio, pH 9.5, 60 °C).

### 3.4. Correlation between the Concentration of Ammonium Thiosulfate and Copper Sulfate for Au and Ag Leaching

The three-dimensional plot has been analyzed to investigate the effect of concentrations of ammonium thiosulfate and  $\text{CuSO}_4$  on the leaching efficiency of Au and Ag. Figure 6 shows a 3D plot of the ammonium thiosulfate concentration,  $\text{CuSO}_4$  concentration, and leaching efficiency of Au and Ag. The z-value represents the leaching efficiency for 1 and 4 h, respectively, x is the  $\text{CuSO}_4$  concentration, and y is the ammonium thiosulfate concentration. As visible in Figure 6, the leaching efficiency of Au and Ag increases with increase in the concentrations of ammonium thiosulfate and  $\text{CuSO}_4$ . For the initial 1 h leaching behavior of Au (Figure 6a), the leaching efficiency of the Au was more affected



by the change in  $\text{CuSO}_4$  concentration than that of ammonium thiosulfate. However, the effect of ammonium thiosulfate concentration increased as the leaching time increased (Figure 6b). In the case of Ag (Figure 6c,d), it was observed that the leaching efficiency of the Ag was more affected by the change in ammonium thiosulfate concentration than that of  $\text{CuSO}_4$ . Interestingly, the trend of Au and Ag leaching efficiency with  $\text{CuSO}_4$  and ammonium thiosulfate followed the plane graph with increasing leaching time (Figure 6b,d). The ideal plane graph was calculated using the given data, and the values are shown in Figure 6. The  $R^2$  (coefficient of determination) values of the two graphs (Figure 6b,d) were found to be 0.9658 and 0.9688, respectively, which were relatively consistent with the plane graphs.

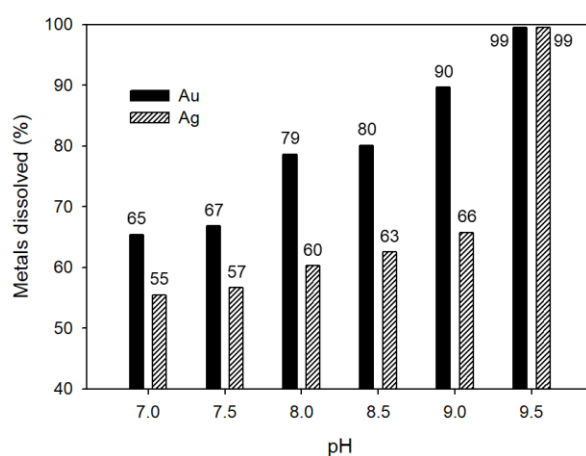


**Figure 6.** Correlation between the concentration of ammonium thiosulfate and  $\text{CuSO}_4$  on metals leaching efficiency: (a,b) Au and (c,d) Ag. (Experimental conditions: 0.2 S/L ratio, pH 9.5, 60 °C).

### 3.5. Effect of pH on Au and Ag Leaching Efficiency

For the effective dissolution of Au and Ag, leaching experiments were conducted under the pH variation (from 7.0 to 9.5), with experimental conditions of 0.2 S/L ratio, 0.5 M ammonium thiosulfate concentration, 0.05 M  $\text{CuSO}_4$  concentration, at 60 °C temperature for 4 h. As shown in Figure 7, the leaching efficiency of both Au and Ag increased with increasing pH value. The leaching efficiency of Au increased from approximately 65% to 99%, whereas leaching efficiency of Ag increased from approximately 55% to 99%, for the corresponding pH range of 7 to 9.5. The pH of the ammonium thiosulfate leaching process is generally maintained between 9 and 10 such that the presence of ammonia is ensured in order to dissolve Cu as a Cu(II)-ammonia complex. Molleman and Dreisinger

reported that ammonium thiosulfate leaching for Au recovery should preferably be performed at a pH of 9–10 [24]. As a result, the pH of 9.5 was found suitable for Au and Ag leaching in the present study.

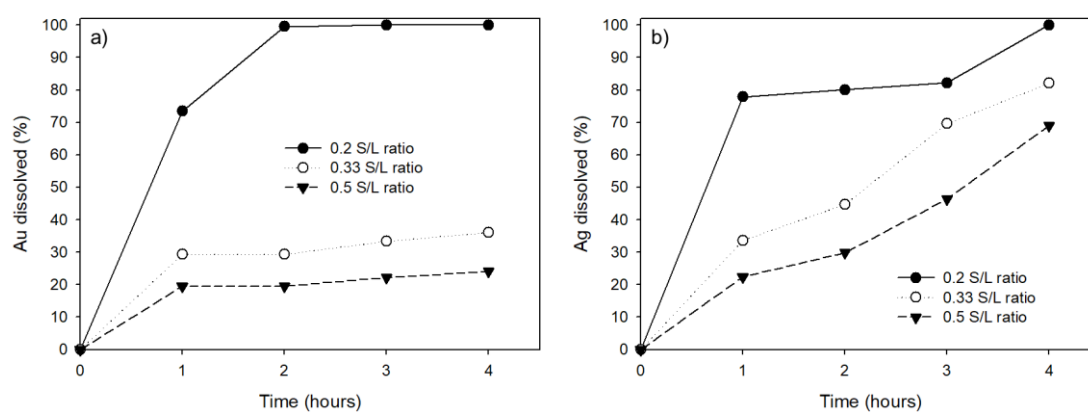


**Figure 7.** Effect of pH on Au and Ag leaching efficiency. (Experimental conditions: 0.5 M ammonium thiosulfate, 0.05 M  $\text{CuSO}_4$ , 0.2 S/L ratio, 60 °C).

### 3.6. Effect of Solid/Liquid ratio on Au and Ag Leaching Efficiency

Leaching is a solid–liquid reaction, wherein the two phases are in intimate contact; the solute can diffuse from the solid to the liquid phase, which causes a separation of the components originally present in the solid. In addition, pulp density is a key factor influencing mass transfer kinetics in Au metallurgical operation [25].

Experiments were carried out at various S/L ratios (0.2, 0.33, and 0.5) using 0.5 M ammonium thiosulfate concentration with 0.05 M  $\text{CuSO}_4$  concentration. These experiments carried out at 60 °C temperature and leaching periods ranging from 1 to 4 h are presented in Figure 8. The leaching efficiency of Au (Figure 8a) has been significantly decreased from >99% to 24% when the S/L ratio was increased from 0.2 to 0.5 for 4 h. It can be observed that the lower S/L ratio enhances the leaching efficiency of Au, while, at higher S/L ratios, it decreased, which can be attributed to the need for greater thiosulfate concentrations as the pulp density increases [26,27]. Navarro et al. reported that the maximum leaching efficiency of Au can be achieved by increasing the thiosulfate concentration when the pulp density is increased [26]. In case of Au, the results suggest that a high leaching rate of Au at 60 °C and 0.2 S/L ratio with preferred concentrations of ammonium thiosulfate (0.5 M) and  $\text{CuSO}_4$  (0.05 M) leads to high leaching efficiency. Unlike Au, the leaching efficiency of Ag (Figure 8b) has been decreased from >99% to 69% with an increase in the S/L ratio from 0.2 to 0.5 for 4 h. It was confirmed that Au is more sensitive to pulp density than Ag in terms of leaching efficiency.



**Figure 8.** Effect of solid/liquid ratio for metals leaching efficiency: (a) Au, (b) Ag. (Experimental conditions: 0.5 M ammonium thiosulfate, 0.05 M  $\text{CuSO}_4$ , pH 9.5, 60 °C).

#### 4. Conclusions

In this study, the ammonium thiosulfate leaching process has been investigated for the recovery of Au and Ag from concentrated sulfide ore. In particular, the present study was mainly focused on the leaching behavior of Au and Ag with the ammonium thiosulfate and  $\text{CuSO}_4$  concentration changes. Improvements in the leaching efficiency of Au and Ag were found with the increase in concentrations of ammonium thiosulfate and  $\text{CuSO}_4$ . Interestingly, the trend of leaching efficiency with  $\text{CuSO}_4$  and ammonium thiosulfate followed the plane graph. Furthermore, the temperature and pH of the leaching experiment were important factors in determining the leaching efficiency. Optimal leaching efficiency was obtained at 60 °C and pH 9.5. On the other hand, the leaching efficiency decreased to 24% for Au and 69% for Ag from 99%, when the S/L ratio was increased from 0.2 to 0.5 for 4 h. Based on obtained results, it was found that the leaching process for the recovery of Au and Ag can achieve >99% efficiency with the following optimum leaching conditions: 0.5 M ammonium thiosulfate, 0.05 M  $\text{CuSO}_4$  concentration, S/L ratio of 0.2, temperature at 60 °C, and pH of 9.5.

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**Conflicts of Interest:** The authors declare no conflicts of interest.

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