

Biooxidation–thiosulfate leaching of refractory gold concentrate

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Abstract: A process of biooxidation followed by thiosulfate leaching of gold from refractory gold concentrate was investigated. Mineralogical studies on the concentrate showed that very fine gold grains (<10 μm) were encapsulated in pyrite and arsenopyrite, while the proportion of monomer gold was only 21%. The gold-bearing sample was identified as a high-sulfur fine-sized wrapped-type refractory gold concentrate. The gold leaching efficiency obtained by direct cyanidation was only 59.86%. After biooxidation pretreatment, the sulfide minerals were almost completely decomposed, 92wt% of the mineral particles of the biooxidation residue were decreased to <38 μm, and the proportion of monomer gold in the biooxidation residue was over 86%. Meanwhile, the gold content in the biooxidation residue was enriched to 55.60 g/t, and the S, Fe, and As contents were reduced to approximately 19.8wt%, 6.97wt%, and 0.13wt%, respectively. Ammoniacal thiosulfate was used for gold extraction from the biooxidation residue of the refractory gold concentrate. The results showed that the optimal reagent conditions were 0.18 M thiosulfate, 0.02 M copper(II), 1.0 M ammonia, and 0.24 M sulfite. Under these conditions, a maximum gold leaching efficiency of 85.05% was obtained.

Keywords: thiosulfate; leaching gold; biooxidation; refractory gold concentrate; process mineralogy

1. Introduction

Over the past 100 years, cyanidation has been the most common method of gold extraction from gold-bearing ore or concentrate due to its chemical stability, high leaching rate, and low cost [1–2]. The risk and toxicity of cyanide, however, have led to growing environmental and public concerns over cyanide usage [3]. Meanwhile, refractory gold ores such as carbon-containing and copper-bearing minerals and encapsulated minerals are difficult to leach using cyanide. On this basis, several non-cyanide leaching reagents such as chloride, thiourea, and thiosulfate have been proposed and widely studied in the past few decades [4]. Thiosulfate in the presence of ammonia and copper(II) has been proven to be a suitable alternative reagent to cyanide for leaching carbon-containing and copper-bearing minerals [5–7]. Thiosulfate is not only cheap and non-toxic but can also form stable gold complexes. It is also suitable for leaching typical oxidized gold ores [8].

With the leaching of certain sulfide minerals with thiosulfate, however, previous studies have shown that while a large amount of thiosulfate is consumed, a poor leaching rate is obtained. Sulfide minerals are believed to have a

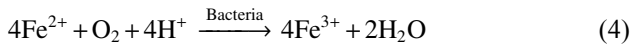
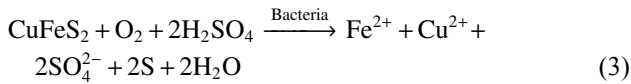
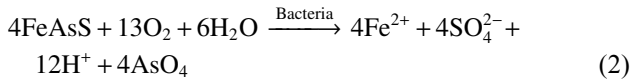
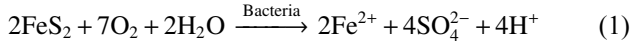
strong affinity for aqueous sulfur species and semiconducting properties, which can accelerate the reaction between thiosulfate and oxygen via the conduction band of the sulfides [9–10]. In particular, the fine gold particles that are encapsulated inside compact sulfide minerals cannot be extracted by cyanide or thiosulfate [11]. The extraction of gold from gold-bearing sulfide mineral samples depends not only on the dissolution behavior of gold but also, critically, on the behavior of the associated minerals [12]. Therefore, to extract gold more easily, the sulfide lattice must be oxidized and decomposed. Pretreatment of refractory gold ores via methods such as roasting, chemical oxidation, pressure oxidation, ultrafine grinding, and biological oxidation can be used to expose the encapsulated gold and cause an increase in the gold recovery rate [11–15].

Extensive laboratory and pilot-scale campaigns have demonstrated the pretreatment ability of biooxidation for refractory encapsulated finely disseminated gold ore. Compared with roasting, chemical oxidation, and pressure oxidation, biooxidation has the advantages of low cost, low equipment requirements, easy control, low energy consumption, and environmental friendliness [12,16–17]. The mechanism of biooxidation is a very complex process; it involves the ox-

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idation of sulfide minerals (such as pyrite (FeS_2), arsenopyrite (FeAsS), and chalcopyrite (CuFeS_2)) to ferric iron, sulfate, or elemental sulfur either directly or indirectly, as expressed in Eqs. (1)–(4) [11]:



Most sulfide minerals and impurities (such as Cu, As, Sb, Zn, and Ni) can be dissolved and removed by biooxidation and multiple washes [18–21].

Although cyanidation and pretreatment with thiosulfate leaching have been studied considering various gold-bearing sulfide ores [11–15,22–24], detailed studies on the process mineralogy of ores/concentrate leaching have barely been reported. Therefore, this paper presents a detailed mineralogical study on the presence and association of gold within concentrate. Furthermore, according to the concentrate characteristics, an environmentally friendly and efficient technology is proposed. Refractory sulfide concentrate was pretreated by biooxidation. Then, thiosulfate was used to re-

place cyanidation for gold extraction. In the thiosulfate leaching tests, the effects of the concentrations of thiosulfate, copper(II), ammonia, and sulfite on the gold extraction were investigated.

2. Experimental

2.1. Materials

The gold-bearing concentrate sample was obtained by the flotation of gold-bearing sulfide ore from the Longnan region, China. The concentrate was milled in a laboratory ball mill. Subsequently, the ball-milled concentrate was dried at 60°C for 24 h. The particle size of the milled sample was analyzed by a Better laser particle size analyzer, which showed that 86wt% of the sample particles were smaller than 38 μm . The gold content was determined by fire assay, and other elements were determined by fire assay or inductively coupled plasma optical emission spectrometry (ICP-OES).

Table 1 shows the elemental composition of the gold-bearing concentrate. The sample was determined to be rich in gold, containing 28.9 g/t Au. The concentrate consisted predominantly of 35.1wt% S, 37.9wt% Fe, and 15.0wt% Si. X-ray diffraction (XRD) analysis (Fig. 1) indicates that pyrite, arsenopyrite, and quartz are the major minerals in the concentrate with traces of chalcopyrite, galena, and sphalerite.

Table 1. Elemental composition of the gold-bearing concentrate

Content / wt%											Content / ($\text{g}\cdot\text{t}^{-1}$)	
Mg	Al	Si	S	Ca	Cr	Fe	Cu	Zn	As	Pb	Au	Ag
2.46	5.68	15.0	35.1	1.68	0.12	37.9	0.07	0.16	1.72	0.11	28.9	20.1

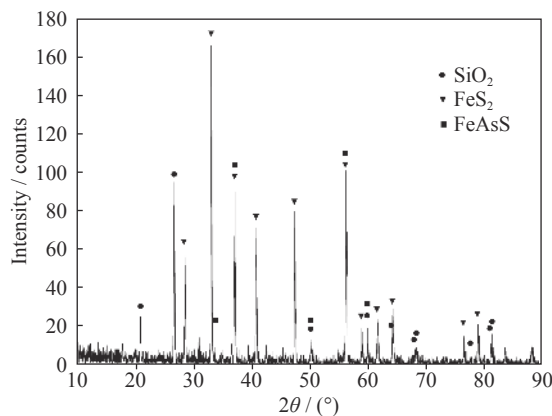


Fig. 1. XRD pattern of the gold-bearing concentrate.

2.2. Mineralogical characterization of the concentrate

The results of the mineragraphic analysis of a polished section of the concentrate samples by scanning electron microscopy–energy-dispersive X-ray spectroscopy (SEM–EDS) are depicted in Fig. 2. The gold minerals were dominantly native gold, whereby particulate and submicroscopic gold were the only two fractions. Pyrite and arsenopyrite were the

main gold-bearing minerals. Micro-grained gold particles (<10 μm) were fully encapsulated in the pyrite, as shown in Figs. 2(a) and 2(b). Part of the monomeric gold was distributed in the form of intergranular gold on the edge of arsenic pyrite (Fig. 2(c)). Meanwhile, only a small amount of gold was present in the concentrate in the form of monomer gold (Fig. 2(d)). According to the statistical analysis, submicroscopic gold with a particle size <1 μm accounts for 39.28%, and 79% of the gold particles were encapsulated in the concentrate. Thus, according to the results of the elemental composition analysis (Table 1), XRD analysis (Fig. 1) and mineragraphic analysis (Fig. 2), the sample was a high-sulfur fine-sized wrapped-type gold concentrate.

Moreover, a large number of minerals were in interlocked and encapsulated forms. Xenomorphic granular pyrite was disseminated in gangue mineral, as shown in Fig. 2. The pyrite particle size in the gangue mineral was uneven, and part of the fine and microfine pyrite was difficult to dissociate into monomer during the grinding process. Numerous bright particles of arsenopyrite were included or intergrown with large granular fragments of pyrite, which further increased the difficulty of leaching the concentrate.

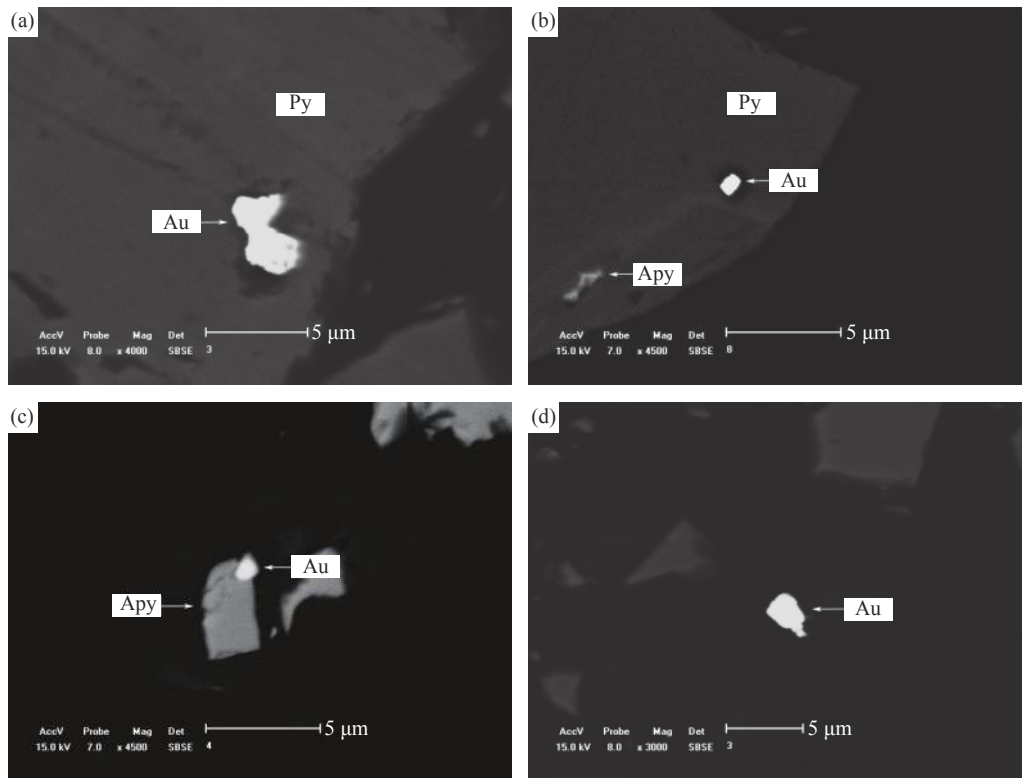


Fig. 2. SEM images of the gold-bearing concentrate (Au—gold; Py—pyrite; Apy—arsenopyrite).

2.3. Leaching tests

A cyanide leaching test was performed to estimate the refractory nature of the concentrate and contrast it with those obtained from other leaching techniques. A 220 g ball-milled concentrate was used for cyanide leaching under the experimental conditions shown in Table 2. Solution samples were taken at regular intervals for measurement of CN^- concentration during 48 h of leaching. The cyanide concentration was maintained at 3.0 g/L. The cyanide concentration was determined by performing a silver nitrate titration, using rhodanine and thymolphthalein as indicators. After 48 h, the cyanide residue was washed and dried for gold analysis.

Bacteria HQ0211, which were cultured and domesticated by Bio-metallurgy Laboratory of Northeastern University, China, were used in the present study. The bacteria were cultured in a 9K medium containing 3.0 g/L $(\text{NH}_4)_2\text{SO}_4$, 0.1 g/L KCl, 0.5 g/L K_2HPO_4 , 0.5 g/L $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 0.01 g/L $\text{Ca}(\text{NO}_3)_2$, and 9 g/L $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ as sources of energy for the bacteria. Biooxidation was carried out in a multi-stage continuous biooxidation reactor for 120 h, with a pulp density of 150 g/L, a temperature of 46°C, an aeration rate of 60 L/min, and a stirring speed of 750 r/min. During the leaching process, the pH value of the pulp was monitored continu-

ously and maintained at 1.5 using H_2SO_4 . After biooxidation, the oxidation residue was filtered through multiple washes and dried at 60°C for 24 h.

The biooxidized concentrate sample was leached with thiosulfate in a 1.0 L three-necked flask as a batch experimental reactor with a working volume of 0.8 L. A moderate amount of sodium thiosulfate pentahydrate was dissolved in distilled water. Then, certain amounts of ammonia solution, ammonium sulfate, and copper sulfate pentahydrate were added to the solution, followed by a weighed amount of sulfite. A digital pH meter (Rex, PHS-3E) was used to measure the pH value. The initial pH value of the leaching solution was adjusted to 10.0–10.5 by sodium hydroxide or sulfuric acid. Then, the mixture was mechanically stirred (IKA, RW20 digital) at 400 r/min.

The reagents used in this study were analytically pure. The gold concentration was determined by atomic absorption spectroscopy (HITACHI, Z-2300, Japan). The thiosulfate concentration was measured using an iodometric method with the addition of EDTA (0.1 M) to eliminate the interference of the cupric tetraamine complex, and formaldehyde (20wt%) to eliminate the interference of the sulfite. The morphologies and qualitative compositions of the concentrate, cyanide residue, and biooxidation residue were determined

Table 2. Experimental conditions for cyanidation of the concentrate

Temperature / °C	Leaching time / h	NaCN concentration / ($\text{g} \cdot \text{L}^{-1}$)	pH	Pulp density / ($\text{g} \cdot \text{L}^{-1}$)	Agitation / ($\text{r} \cdot \text{min}^{-1}$)
20	48	3.0	10.0–10.5	250	950

by X-ray diffraction (Rigaku D/max 2550 VB + 18 kW, Japan) and SEM (SHIMADZU, CamScan SSX-550, Japan).

3. Results and discussion

3.1. Cyanidation test

Cyanidation tests revealed that only 59.86% of gold can be leached from the refractory concentrate, even though 86wt% of the sample particles were smaller than 38 μm . To determine the causes of the low leaching rate, mineragraphic analysis of the cyanide leaching residue was carried out by SEM–EDS. As shown in Fig. 3, a little amount of monomer gold was visible. The remaining gold particles were all trapped in the arsenopyrite and pyrite after 48 h leaching. Meanwhile, dissociating gold from the sulfide minerals was difficult because the size of most of the inclusion gold particles was $<3 \mu\text{m}$. Hence, it was difficult for cyanide to react with the inclusion gold and then dissolve it into the leachate. Finally, the gold grade in the leaching residue was 11.6 g/t.

Based on the above analysis, the sample was a high-

sulfur fine-sized wrapped-type refractory gold concentrate. To recycle the particulate and submicroscopic inclusion gold, the sulfide minerals in the concentrate need to be decomposed.

3.2. Biooxidation pretreatment of the concentrate

A biooxidation process was used to pretreat the concentrate to ensure the gold was amenable to leaching. After biooxidation, 92% of the mineral particles were smaller than 38 μm . The oxidation behavior of the main elements (As, Fe, and S) from the concentrate was evaluated during the biooxidation experiment, as shown in Table 3. Almost all the pyrite and arsenopyrite were oxidized within 120 h. Part of the pyrite was not decomposed because it was trapped in gangue (quartz) and could not contact the solution, as shown in Fig. 4(b). According to Table 3, As is oxidized faster than Fe, because the oxidation potential of arsenopyrite is lower than that of pyrite. After the biooxidation process, the content of S, As, and Fe in the biooxidation residue reached 19.8wt%, 0.13wt%, and 6.97wt%, respectively, and the Au content was 55.60 g/t.

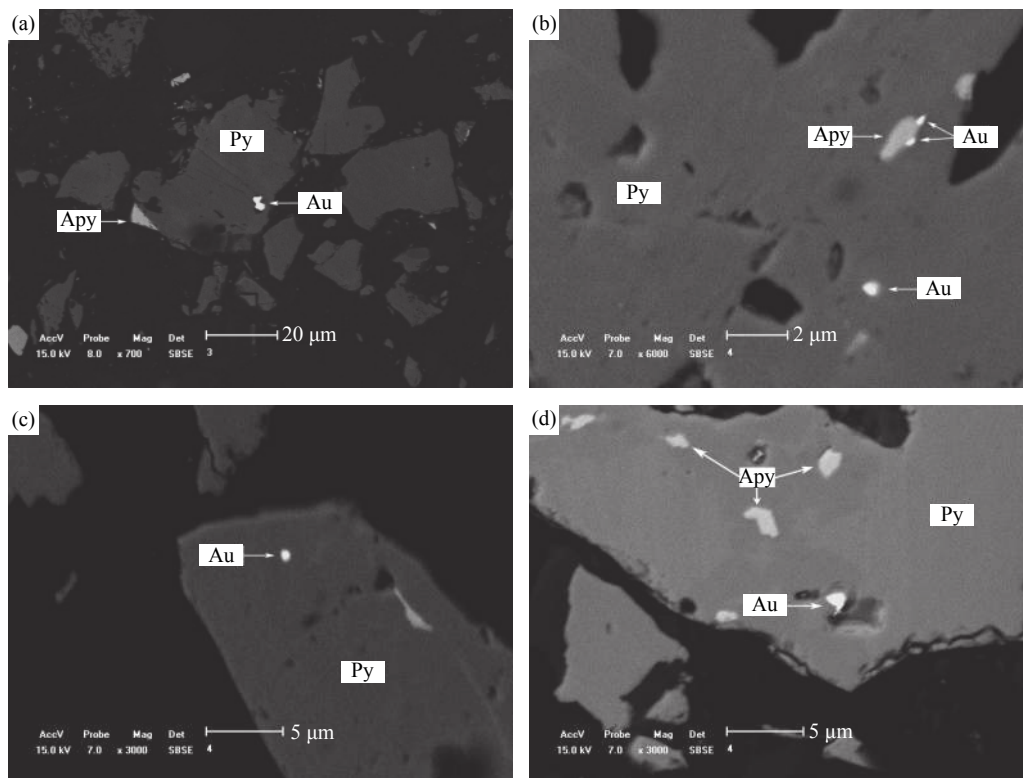


Fig. 3. SEM images of the cyanide leaching residue (Au—gold; Py—pyrite; Apy— arsenopyrite).

Table 3. Biooxidation behavior of main elements (As, Fe, and S) at different stages

Stage	Oxidation time / h	E_h / mV vs. Ag/AgCl	Bacterial concentration / ($10^8 \text{ cell} \cdot \text{mL}^{-1}$)	As oxidation rate / %	S oxidation rate / %	Fe oxidation rate / %
First stage	72	600–627	12.0–13.0	88.56	54.73	68.79
Second stage	24	610–641	13.0–13.8	92.28	61.29	76.87
Third stage	24	652–686	15.3–15.9	96.44	72.80	90.03

Fig. 4 shows the SEM image of a polished section of the biooxidation residue. The particle size of the minerals in the residue was significantly less than that in the original concentrate. The proportion of gangue (quartz) was increased, owing to the oxidation and dissolution of the sulfide minerals.

As shown in Figs. 4(c) and 4(d), pyrite was oxidized into jarosite and almost all the inclusion gold was exposed. The proportion of monomer gold was more than 86% in the biooxidation residue. Reducing the particle size and increasing gold exposure were beneficial to the gold leaching process.

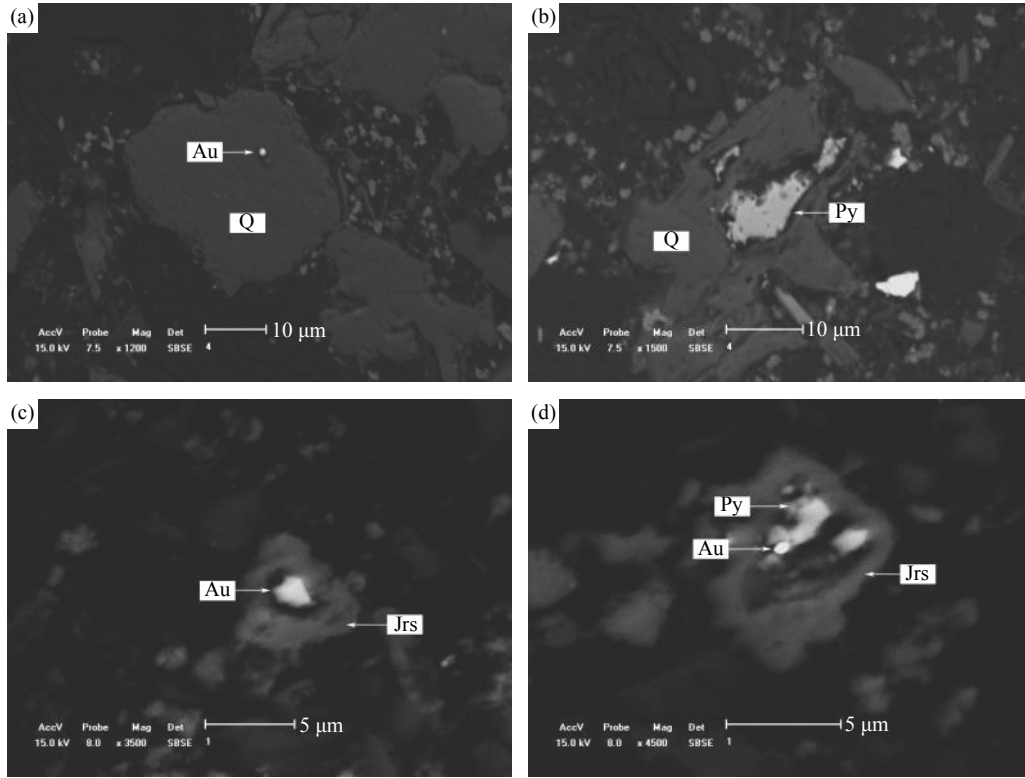
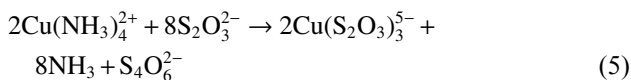


Fig. 4. SEM images of the biooxidation leaching residue (Au—gold; Py—pyrite; Apy—arsenopyrite; Q—quartz; Jrs—jarosite).

3.3. Thiosulfate leaching after treatment

3.3.1. Effect of thiosulfate concentration

The effect of thiosulfate concentration on gold leaching efficiency is illustrated in Fig. 5. The gold leaching efficiency was increased to 80.99% in 0.18 M thiosulfate after 24 h leaching. When the thiosulfate concentration was higher than 0.18 M, the gold leaching efficiency decreased with increasing concentration, while the thiosulfate consumption increased with increasing concentration. As shown in Fig. 5, the thiosulfate consumption was very low when 0.06 M thiosulfate was used. The sulfite amount can be considered excessive compared with the thiosulfate amount, and this greatly inhibits the thiosulfate consumption. When the thiosulfate concentration was 0.18 M, the consumed thiosulfate reached 0.0198 mol (24.55 kg/t). A higher concentration of thiosulfate can react with copper(II) more easily, which increases the consumption of thiosulfate, as shown in the following equation:



Moreover, the decomposition products of thiosulfate, such as polythionate, trithionate, and tetrathionate, can reduce Cu(II) concentration, which in turn leads to a decline in the gold leaching efficiency. On the other hand, the decomposi-

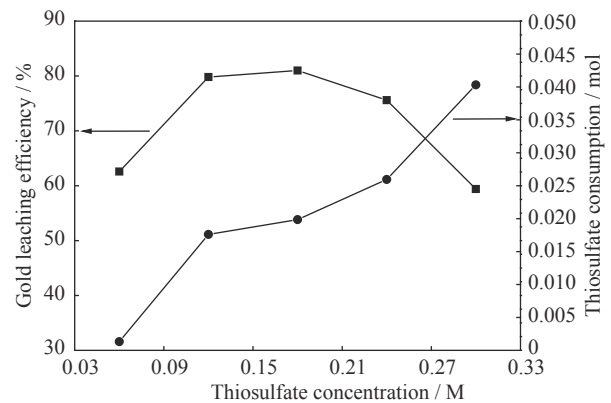


Fig. 5. Effect of thiosulfate concentration on gold leaching efficiency and thiosulfate consumption. Leaching conditions: leaching time, 24 h; pH value, 10.0; pulp density, 250 g/L; ammonium sulfate, 0.50 M; copper(II), 0.020 M; ammonia, 1.0 M; sulfite, 0.18 M.

tion could precipitate Au^+ from the solution and form a precipitated passivation layer on the gold surface to varying degrees [12,25–26]. Consequently, the thiosulfate concentration and decomposition products should be kept low to prevent loss of gold from the solution.

3.3.2. Effect of copper(II) concentration

The effect of copper(II) concentration on gold leaching efficiency was investigated. As can be seen in Fig. 6, when the copper(II) concentration was <0.02 M, the gold leaching efficiency and thiosulfate consumption increased with an increase in copper(II) concentration. This is because the copper(II) is beneficial to the formation of a cupric tetraamine complex, which can increase the oxidation of gold [27–30]. Meanwhile, a higher concentration of copper(II) increases the reaction probabilities with thiosulfate, and thus, more thiosulfate is oxidized and decomposed by copper(II), as shown in Eq. (5). Gold dissolution occurs via the following equation:

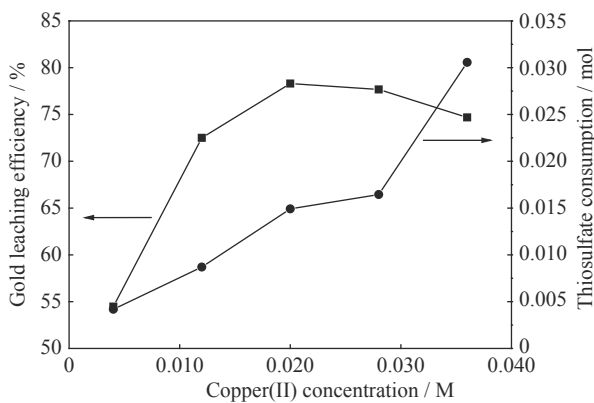
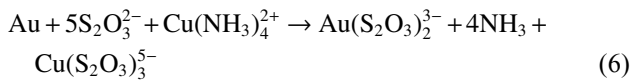


Fig. 6. Effect of copper(II) concentration on gold leaching efficiency and thiosulfate consumption. Leaching conditions: leaching time, 24 h; pH value, 10.0; pulp density, 250 g/L; ammonium sulfate, 0.50 M; thiosulfate, 0.18 M; ammonia, 1.0 M; sulfite, 0.18 M.

When the initial copper(II) concentration further increased from 0.020 to 0.036 M, the gold concentration subsequently decreased and the thiosulfate consumption continuously increased. Particularly, the thiosulfate consumption increased rapidly when the copper(II) concentration increased from 0.28 to 0.36 M. It can be considered that part of the leached gold is precipitated. Copper(II) not only oxidizes thiosulfate to tetrathionate but also further oxidizes tetrathionate to trithionate and sulfide [31–32]. Thus, a higher copper concentration eventually leads to the formation of insoluble precipitates and passivation of the gold surface with products such as Au_2S , CuO , Cu_2O , Cu_2S , tetrathionate, and trithionate [14,26,30]. The experiment indicated that the gold

leaching efficiency reached its maximum when 0.020 M copper(II) was used in the leaching process, and the consumed thiosulfate was only 0.0149 mol (18.48 kg/t).

3.3.3. Effect of ammonia concentration

The effect of ammonia concentration on gold leaching efficiency is illustrated in Fig. 7. The gold leaching efficiency increased significantly with an increase in ammonia concentration up to 1.0 M. Ammonia plays an important role in copper(II) stabilization by forming copper(II)–ammine complexes under alkaline conditions. When the ammonia concentration is low, excess copper(II) is precipitated or reacts with thiosulfate to form insoluble precipitates, resulting in the passivation of the gold surface [33–35]. However, when the ammonia concentration is over 1.0 M, the gold leaching rate declines. This is because excess ammonia decreases the reduction potential of the copper(II)/copper(I) couple, thereby decreasing the oxidizing ability of copper(II) [36]. Meanwhile, a high concentration of ammonia leads to higher pH values, which reduces the thermodynamic stability of $\text{Cu}(\text{NH}_3)_4^{2+}$ and $\text{Cu}(\text{S}_2\text{O}_3)_3^{5-}$, and it widens the thermodynamic stability regions of CuO and Cu_2O [27]. The precipitation of CuO and Cu_2O hinders thiosulfate attack by covering the gold surface. Moreover, excess ammonia can push the equilibrium of Eq. (7) to the left, reducing the stability of gold in aqueous solution [33,36–39]:

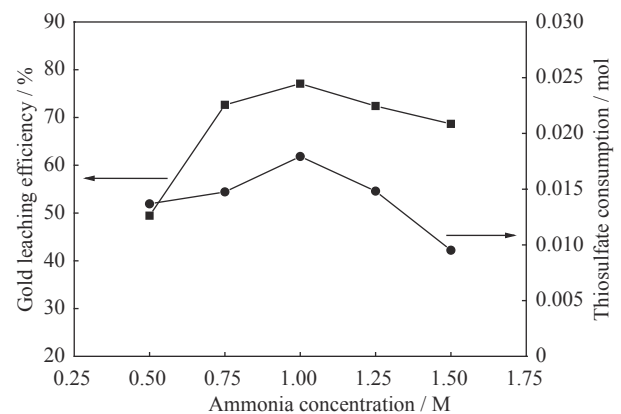
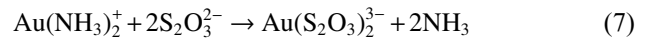


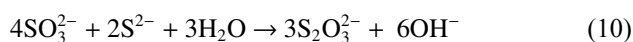
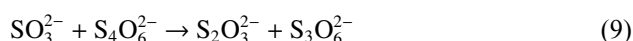
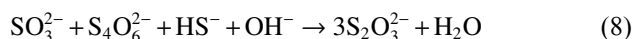
Fig. 7. Effect of ammonia concentration on gold leaching efficiency and thiosulfate consumption. Leaching conditions: leaching time, 24 h; pulp density, 250 g/L; ammonium sulfate, 0.50 M; thiosulfate, 0.18 M; copper(II), 0.020 M; sulfite, 0.18 M.

As shown in Fig. 7, ammonia has little effect on the thiosulfate consumption. Both lower and higher ammonia concentrations are not conducive to the stability of copper(II) in the solution, which ultimately leads to a reduction in the thiosulfate consumption. Copper(II) could form stable copper(II)–ammine complexes under 1.0 M ammonia. Adequate copper ammonia complexes not only increase the gold leaching efficiency but also increase the thiosulfate consumption

to 0.0179 mol (22.20 kg/t). Therefore, 1.0 M ammonia was considered the optimum ammonia concentration and was used for subsequent experiments.

3.3.4. Effect of sulfite concentration

The results of varying sulfite concentrations are depicted in Fig. 8. Gold leaching efficiency increased with an increase in sulfite concentration up to 0.24 M. This was due to the presence of sufficient sulfite to eliminate tetrathionate, thus preventing gold precipitation (Eqs. (8) and (9)). Meanwhile, sodium sulfite can react with S^{2-} , HS^- , and S^0 and then prevent the formation of sulfur and sulfides deposits on the gold surface (Eqs. (10)–(12)) [5–6,40–41].



With an increase in sulfite concentration from 0.24 to 0.30 M, the gold leaching efficiency decreased slightly. This is because sulfite in high concentration decreases redox potential, reducing copper(II) and precipitating gold from the solution [40,42]. According to Fig. 8, the thiosulfate consumption decreased with an increase in the sulfite concentration. Sulfite can maintain the stability of thiosulfate in the leaching system by reaction (Eqs. (8)–(12)). The regulating effect of sulfite is bidirectional; determining whether to use it as well as determining the optimum concentration required based on the mineral species [41]. Thus, the optimum sulfite concentration in this case was considered to be 0.24 M, at which the final gold leaching efficiency and consumed thiosulfate reached 85.05% and 0.0186 mol (23.06 kg/t), respectively.

The optimum conditions of leaching gold from the biooxidized concentrate with thiosulfate are $Na_2S_2O_3$ 0.18 M, $(NH_4)_2SO_4$ 0.1 M, $CuSO_4$ 0.02 M, ammonia 1.0 M, Na_2SO_3

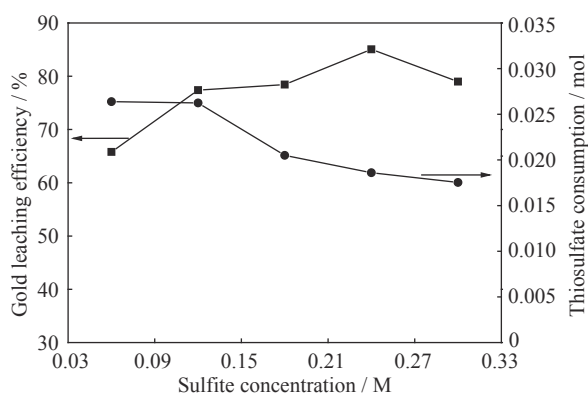


Fig. 8. Effect of sulfite concentration on gold leaching efficiency and thiosulfate consumption. Leaching conditions: leaching time, 24 h; pH, 10.0; pulp density, 250 g/L; ammonium sulfate, 0.50 M; thiosulfate, 0.18 M; copper(II), 0.020 M; ammonia, 1.00 M.

0.24 M, pH 10.0, pulp density 250 g/L, and leaching time 24 h. However, only 28.6% final leaching efficiency was obtained when the optimum condition was used to leach the high-sulfur gold concentrate. The gold leaching efficiency of the biooxidation–thiosulfate process increased by 25.2% and 56.5% compared with those of direct cyanidation and direct thiosulfate leaching of concentrate, respectively.

4. Conclusions

(1) Process mineralogical analysis of the concentrate showed that all the gold particles were $<10 \mu m$ in size and approximately 79% of the gold was encapsulated in pyrite and arsenopyrite.

(2) Biooxidation leaching pretreatment can significantly convert the mineral properties of the concentrate. Almost all the pyrite and arsenopyrite were oxidized. The proportion of monomer gold was more than 86% in the biooxidation residue.

(3) Ammoniacal thiosulfate leaching experiments of bacteria-oxidized gold concentrate were carried out. The concentrations of thiosulfate, copper(II), ammonia, and sulfite in the leaching process were the most important influencing factors affecting the gold leaching efficiency. The highest gold leaching efficiency of 85.05% was obtained after 24 h using thiosulfate, copper(II), ammonia, and sulfite concentrations of 0.18 M, 0.02 M, 1.0 M, and 0.24 M, respectively. The gold leaching efficiency of this biooxidation–thiosulfate process increased by 25.2% and 56.5% compared with those of direct cyanidation and direct thiosulfate leaching of the concentrate.

Biooxidation pretreatment followed by ammoniacal thiosulfate leaching technology is highly suitable for gold extraction from high-sulfur fine-sized wrapped-type refractory gold concentrate.

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