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 the pyrite and arsenopyrite minerals while the amount of monomer gold was only 21%. It was identified as a high-sulfur fine-sized wrapped type refractory gold concentrate. The direct cyanide leaching efficiency of gold was only 59.86%. After biooxidation pretreatment, the sulfide minerals were almost completely decomposed, 92% of the mineral particles of the biooxidation residue were decreased to < 38 μm, and the amount 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 contents of S, Fe, and As were reduced to approximately 19.8, 6.97, and 0.13 wt%, 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.

Key words: thiosulfate; leaching gold; biooxidation; refractory gold concentrate; process mineralogy;

1. Introduction

Over the past one hundred years, cyanidation has been the most common method for 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 its use [3]. Meanwhile, refractory gold ores such as carbon-containing and copper-bearing minerals and encapsulated minerals are difficult

to leach by 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]. It was proved that thiosulfate in the presence of ammonia and copper(II) is 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 also can 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, only a poor leaching rate is obtained. It is believed that sulfide minerals have a strong affinity for aqueous sulfur species and semi-conducting 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, such as roasting, chemical oxidation, pressure oxidation, ultrafine grinding, or biological oxidation can be used to expose the encapsulated gold and enable 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 to roasting, chemical oxidation, or 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 in which sulfide minerals (such as pyrite (FeS₂), arsenopyrite (FeAsS) and chalcopyrite (CuFeS₂)) can be oxidized to ferric iron, sulfate or elemental sulfur either directly or indirectly, as expressed in Eqs. (1–4) [11].

$$2\text{FeS}_2 + 7\text{O}_2 + 2\text{H}_2\text{O} \xrightarrow{\text{bacteria}} 2\text{Fe}^{2+} + 4\text{SO}_4^{2-} + 4\text{H}^+ \quad (1)$$

$$4FeAsS+13O_2+6H_2O \xrightarrow{bacteria} 4Fe^{2+} + 4SO_4^{2-} + 12H^+ + 4AsO_4 \quad (2)$$

$$CuFeS_2+O_2+2H_2SO_4 \xrightarrow{bacteria} Fe^{2+}+Cu^{2+}+2SO_4^{2-}+2S+2H_2O$$
 (3)

$$4Fe^{2+} + O_2 + 4H^+ \xrightarrow{bacteria} 4Fe^{3+} + 2H_2O \quad (4)$$

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].

Despite cyanide and pretreatment with thiosulfate leaching having been studied on various gold-bearing sulfide ores [11-15, 22-24], rarely has detailed research on the process mineralogy of ores/concentrates leaching been reported. Therefore, the presence and association of gold within concentrate were investigated by a detailed mineralogical study. Furthermore, according to concentrate characteristics, an environmentally friendly and efficient technology is proposed. Refractory sulfide concentrate was pretreated by biooxidation. Then thiosulfate was used to replace cyanidation for gold extraction. In the thiosulfate leaching tests, the effects of concentrations of thiosulfate, copper ion, ammonia, and sulfite on the extraction of gold were investigated.

2. Experimental

2.1 Materials

The gold-bearing concentrate sample was obtained by flotation of gold-bearing sulfide ore originally 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. Particle size analysis of the milled sample by a Better laser particle size analyzer showed that 86 wt% of the sample particles were smaller than 38 µm. Gold content was determined by fire-assay, and other elements were determined by fire-assay or inductively couple plasma-optical emission spectrometry (ICP-OES).

Table 1 shows the elemental composition analysis 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 35.1% S, 37.9% Fe, and 15% Si. X-ray diffraction 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 analysis of the concentrate

Element Mg Al Si S Ca Cr	Element Mg Al	S	Ca Cr	
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Sample (%)	2.46	5.68	15	35.1	1.68	0.12
Fe	Cu	Zn	As	Pb	Au (g/t)	Ag (g/t)
37.9	0.07	0.16	1.72	0.11	28.9	20.1

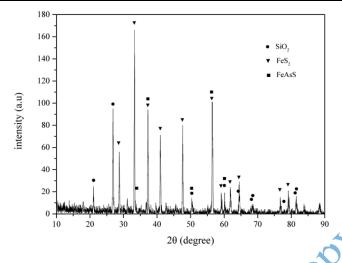


Fig. 1 XRD pattern of the concentrate

2.2 Mineralogical characterization of the concentrate

The results of a mineragraphic analysis of a polished section of concentrate samples by SEM-EDS are depicted in Fig. 2. Gold minerals are dominantly native gold, which particulate and submicroscopic gold were the only two fractions. Pyrite and arsenopyrite are the main gold-bearing mineral. Micro-grained gold particles (< 10 µm) were fully encapsulated in the pyrite, as shown in Fig. 2(a and b). Part of the monomeric gold was distributed in the form of intergranular gold on the edge of arsenic pyrite (Fig. 2c). Meanwhile, only a small amount of gold was present in the concentrate in the form of monomer gold (Fig. 2d). 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 concentrate. Thus, according to the results of the elemental composition analysis (Table 1), X-ray diffraction analysis (Fig. 1) and mineragraphic analysis (Fig. 2), it can be seen that the sample was a high-sulfur fine-sized wrapped type gold concentrate.

Moreover, there were a large number of minerals in interlocked and encapsulated forms. Xenomorphic granular pyrite was disseminated in gangue mineral, as shown in Fig. 2 and Fig. 3. The pyrite particle size in 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 concentrate.

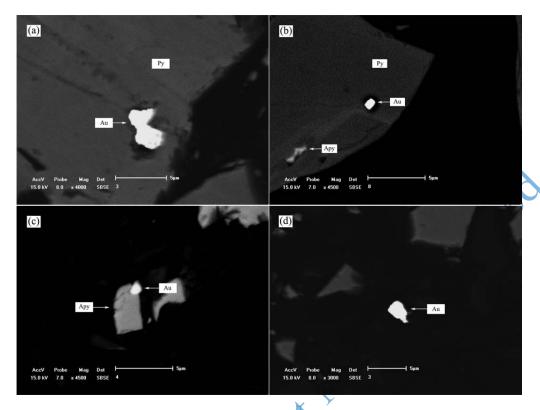


Fig. 2 Scanning electron microscopy (SEM) images of the concentrate (Au: Gold; Py: Pyrite; Apy: arsenopyrite;)

2.3 Leaching tests

A cyanide leaching test was performed to estimate the refractory nature of the concentrate and contrast it with 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. Cyanide concentration was determined by performing a silver nitrate titration, using rhodanine and thymolphthalein as an indicator. After 48 h, the cyanide residue was washed and dried for gold analysis.

Bacteria HQ0211 were used in the present study, which were cultured and domesticated by Northeast University Bio-metallurgy Laboratory. The bacteria were cultured in a 9K medium containing 3.0g/L (NH₄)₂SO₄, 0.1 g/L KCl, 0.5 g/L K₂HPO₄, 0.5 g/L MgSO₄·7H₂O, 0.01 g/L Ca(NO₃)₂, and 9 g/L FeSO₄·7H₂O as sources of energy for the bacteria. Biooxidation was carried out in a multi-stage continuous biooxidation reactor for 120 h with a density of 15% (w/v), 46 °C, aeration of 60 L·min⁻¹ and a stirring speed of 750 rpm. During the leaching

process, the pH of the pulp was monitored continuously and maintained at 1.5 using H₂SO₄. 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. The initial pH of the leaching solution was adjusted to 10-10.5 by sodium hydroxide or sulfuric acid. Then the mixture was mechanically stirred (IKA, RW20 digital) at 400 rpm.

The reagents used in this study were all analytically pure. The gold concentration was determined by atomic absorption spectroscopy (AAS) (HITACHI, Z-2300). The thiosulfate concentration was measured by an iodometric method with the addition of EDTA (0.1 M) to eliminate the interference of the cupric tetra-amine complex and formaldehyde (20%) to eliminate the interference of the sulfite. The morphologies and qualitative compositions of concentrate, cyanide residue, and biooxidation residue were determined by X-ray diffraction (RigakuD/max 2550VB+18kW) and scanning electron microscopy (SHIMADZU, CamScan SSX-550).

Table 2 Experimental conditions for cyanidation of the concentrate

Parameter	Cyanidation	
Temperature, °C	20	
Leach time, h	48	
NaCN concentration, g/l	3.0	
pH	10-10.5	
Pulp density, (w/v), %	25	
Agitation, rpm	950	

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 86 wt% of the sample particles were smaller than 38 µm. To find out the causes of the low leaching rate, mineragraphic analysis of the cyanide leaching residue was carried out by SEM-DES. As shown in Fig. 3, there

was little monomer gold visible. The remaining gold particles were all trapped in the arsenopyrite and pyrite after 48 h leaching. Meanwhile, it was difficult to dissociate gold from the sulfide minerals, because the size of most of the inclusion particles of gold was \leq 3 μ m. Hence, it was thus difficult to react cyanide with inclusion gold and then dissolve it into the leachate. Finally, the gold grade in the leaching residue was still 11.6 g/t.

Based on the analysis above, 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.

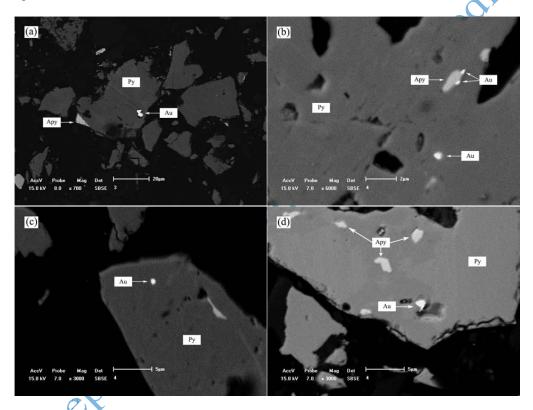


Fig. 3 Scanning electron microscopy (SEM) images of the cyanide leaching residue (Au: Gold; Py: Pyrite; Apy: arsenopyrite;)

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 μ m. The oxidation behavior of the main elements (As, Fe, S) from the concentrate was evaluated during the biooxidation experiment, as shown in Table 3. Almost all of the pyrite and arsenopyrite were oxidized within 120 h. Part of the pyrite was not decomposed because it was

trapped into 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 19.8, 0.13, and 6.97 wt%, respectively and the Au content was 55.60 g/t.

Fig. 4 shows an SEM image of a polished section of 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 oxidation and dissolution of the sulfide minerals. As shown in Fig. 4(c) and Fig. 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.

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

	First stage	Second stage	Third stage
Oxidation time/h	72	24	24
Eh (Ag/AgCl)/mV	600-627	610-641	652-686
Bacterial concentration/(×10 ⁸ cell/mL)	12-13	13-13.8	15.3-15.9
As oxidation rate/%	88.56	92.28	96.44
S oxidation rate/%	54.73	61.29	72.8
Fe oxidation rate/%	68.79	76.87	90.03

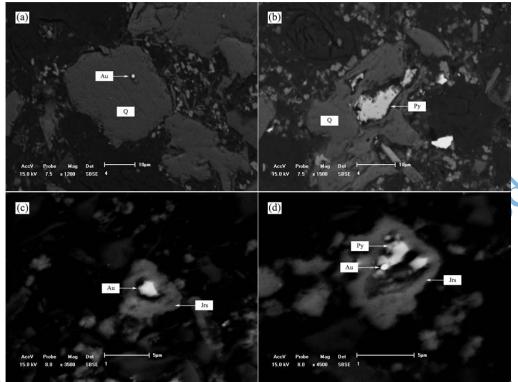


Fig. 4 Scanning electron microscopy (SEM) images of the biooxidation leaching residue (Au: Gold; Py: Pyrite;

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 extraction is shown 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 extraction efficiency decreased with increasing concentration. Meanwhile, 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. It can be consider that the amount of sulfite is excessive compared to that of thiosulfate, which greatly inhibits the consumption of thiosulfate. When the thiosulfate concentration was 0.18 M, consumption of thiosulfate reached to 0.0198 mol. A higher concentration of thiosulfate can react with copper(II) more easily which increases the consumption of thiosulfate, as shown by Eq. (5). Moreover, the decomposition products of thiosulfate, such as polythionate, trithionate and tetrathionate, can reduce Cu(II) concentration, which in its turn leads to a decline in gold leaching efficiency. On the other hand, the decomposition could product

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

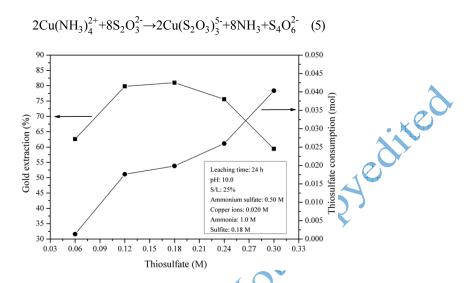


Fig. 5 Effect of thiosulfate concentration on gold extraction and thiosulfate consumption

3.3.2 Effect of copper ion concentration

The effect of copper ion concentration on gold extraction was investigated. As can be seen in Fig. 6, when the copper ion concentration is < 0.02 M, gold leaching efficiency and thiosulfate consumption increases with concentration. This is because copper ion is beneficial to the formation of 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 by Eq. (5). Gold dissolution occurs via the following equation:

$$Au+5S_2O_3^{2-}+Cu(NH_3)_4^{2+} \longrightarrow Au(S_2O_3)_2^{3-}+4NH_3+Cu(S_2O_3)_3^{5-} \quad (6)$$

When the initial copper ion concentration further increased from 0.020 to 0.036 M, the gold concentration subsequently decreased and the thiosulfate consumption continuously increased. Particularly, the consumption of thiosulfate increased rapidly when the concentration of copper(II) increased from 0.28 M 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, higher copper concentration eventually

leads to the formation of insoluble precipitates and passivation of the gold surface with products such as Au₂S, CuO, Cu₂O, Cu₂S, tetrathionate, and trithionate [14, 26, 30]. The experiment indicated that the gold leaching efficiency reached the maximum when 0.020 M copper ion was used in the leaching process, and the consumption of thiosulfate was only 0.0149 mol.

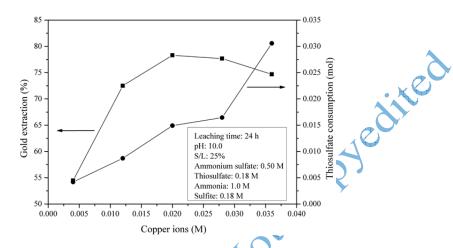


Fig. 6 Effect of copper ion concentration on gold extraction and thiosulfate consumption

3.3.3 Effect of ammonia concentration

The effect of ammonia concentration on gold extraction is shown in Fig. 7. It is evident that gold extraction increases 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 concentration of ammonia is low, excess copper(II) is precipitated or reacts with thiosulfate to form insoluble precipitates and 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 Cu(NH₃)₄²⁺ and Cu(S₂O₃)₃⁵⁻, and it widens the thermodynamic stability regions of CuO and Cu₂O [37]. The precipitation of CuO and Cu₂O hinders thiosulfate attack by covering the gold surface. Also, excess ammonia can push the equilibrium of Eq. 7 to the left, reducing the stability of gold in aqueous solution [33, 36, 38-40]. As shown in Fig. 7, ammonia has little effect on the consumption of thiosulfate. Both lower and higher ammonia concentrations are not conducive to 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 consumption of thiosulfate to 0.0179 mol. Therefore, 1.0 M ammonia was considered the optimum ammonia concentration and was used for subsequent experiments.

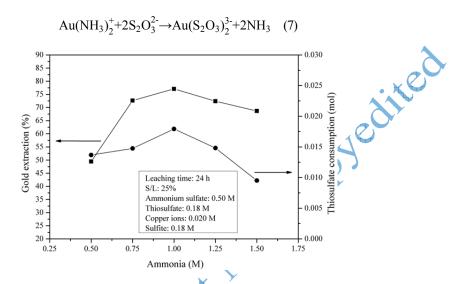


Fig. 7 Effect of ammonia concentration on gold extraction and thiosulfate consumption

3.3.4 Effect of sulfite concentration

The results of varying sulfite concentration are depicted in Fig. 8. Gold extraction 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. 11, 12). Meanwhile, sodium sulfite can react with S⁰, HS⁻ and S²⁻, and then prevent the formation of deposition of sulfur and sulfides on the surface of the gold (Eqs. 8–10) [5, 6, 41, 42].

$$4SO_{3}^{2-}+2S^{2-}+3H_{2}O \rightarrow 3S_{2}O_{3}^{2-}+6OH^{-} (8)$$

$$4SO_{3}^{2-}+2HS^{-}+H_{2}O \rightarrow 3S_{2}O_{3}^{2-}+4OH^{-} (9)$$

$$SO_{3}^{2-}+2S \rightarrow S_{2}O_{3}^{2-} (10)$$

$$SO_{3}^{2-}+S_{4}O_{6}^{2-}+HS^{-}+OH^{-}\rightarrow 3S_{2}O_{3}^{2-}+H_{2}O (11)$$

$$SO_{3}^{2-}+S_{4}O_{6}^{2-}\rightarrow S_{2}O_{3}^{2-}+S_{3}O_{6}^{2-} (12)$$

With an increase in sulfite concentration from 0.24 to 0.30 M, gold leaching efficiency decreased slightly. This is because sulfite in high concentration decreases redox potential, reducing copper(II) and precipitating gold from

solution [41, 43]. According to Fig. 8, the consumption of thiosulfate 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, a decision on whether to use it and determination of the optimum concentration need to be based on the mineral species [42]. Thus, the optimum sulfite concentration in this case was considered to be 0.24 M, at which the final gold leaching efficiency and consumption of thiosulfate reached to 85.05% and 0.0186 mol respectively.

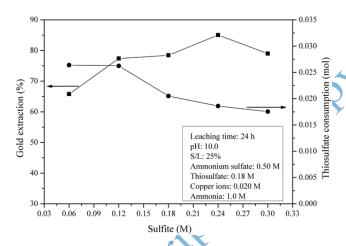


Fig. 8 Effect of sulfite concentration on gold extraction and thiosulfate consumption

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 1.0M, pH 10.0, S/L 25% 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 29% and 56.5% compared with direct cyanidation and direct thiosulfate leaching of concentrate, respectively.

4. Conclusions

According to the results of the present study, the following conclusions were obtained:

(1) Process mineralogical analysis of the concentrate showed that all the gold particles were < 10 μm 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 of 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 ion, ammonia, and sulfite in the leaching process were the most important influencing factors for the extraction of gold. 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 29% and 56.5% compared with direct cyanidation and direct thiosulfate leaching of 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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