



Effect of arsenopyrite on thiosulfate leaching of gold

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Abstract: Arsenopyrite was artificially added into the thiosulfate leaching solution to clarify the role of arsenopyrite on the thiosulfate leaching of gold. The effect of arsenopyrite on the thiosulfate leaching of gold was studied by the thermodynamic calculation, mineral dissolution test, leaching test and XPS analysis. The results show that the thiosulfate consumption slightly increases with increasing the concentration of arsenopyrite, but the gold dissolution is obviously hindered. This may mainly attribute to the catalytic effect of arsenopyrite on the thiosulfate decomposition and the formation of passivation layer on the gold foil surface. The passivation layer likely consists of Cu_2S or $\text{Cu}(\text{S}_2\text{O}_3)_3^{5-}$, element S, FeOOH and iron arsenate, which is deduced from the XPS analysis. However, the negative effect of arsenopyrite can be eliminated by adding additives. It is found that both additives of sodium carboxymethyl (CMC) and sodium phosphate (SHPP) can not only decrease the thiosulfate consumption but also improve the gold dissolution.

Key words: gold leaching; thiosulfate; arsenopyrite; passivation

1 Introduction

Thiosulfate has shown considerable promise as an alternative non-cyanide lixiviant due to the increasing awareness of environment [1]. However, the industrial application of thiosulfate method is rare at present. The high consumption of thiosulfate and the passivation of gold are two limitations [2–4].

Since gold always associates with various minerals, the dissolution of minerals impacts the composition of the thiosulfate leaching solution and then impacts the thiosulfate leaching of gold. So, the effects of associated minerals on thiosulfate leaching of gold have great research value.

The dissolution of associated minerals influenced the thiosulfate leaching of gold [5]. Pyrite catalyzed the thiosulfate decomposition [6] and passivated the gold dissolution. It was the formation of coated layer which includes cuprous sulfide, sulfur, iron hydroxide, etc., on gold surface that hindered the gold dissolution [7]. Moreover, the heavy metal ions in thiosulfate solution reacted with thiosulfate by competing with gold, and thus, hindering gold leaching [8,9]. It was reported that arsenopyrite was also detrimental to thiosulfate leaching

of gold. It inhibited gold dissolution, but had unobvious effect on the thiosulfate consumption [10]. However, the results are controversial and the reason for the inhibited effect of arsenopyrite on the gold dissolution is not clear.

Based on the above, the effect of arsenopyrite on the thiosulfate leaching of gold was unclear, especially on the chemical components of the coated layer on the gold surface. This study focused on the identification of species on the leached gold foil surface by X-ray photon spectroscopy (XPS) analysis, and further investigated the role of arsenopyrite on the thiosulfate leaching of gold. In addition, the improving measures to relieve the deteriorate impact of arsenopyrite on the thiosulfate leaching of gold were also studied.

2 Experimental

2.1 Minerals and reagents

Arsenopyrite and quartz samples were obtained from Mineral Specimen Supplies, Wuhan, China. The chemical compositions of them are listed in Tables 1 and 2, respectively. Quantitative X-ray diffraction (XRD) was also used to determine the mineralogy of the samples. The results indicate that arsenopyrite and quartz samples are with purity of 97.8% and 99%, respectively.

Table 1 Chemical composition of arsenopyrite (mass fraction, %)

O	F	Al	Si	S	Cl	K	Ca	Cr
11.039	0.576	4.284	1.259	13.673	0.021	0.047	2.216	0.008
Mn	Fe	Cu	Zn	As	Se	Sb	Pb	Bi
0.208	28.772	0.035	0.194	37.338	0.015	0.017	0.189	0.109

Table 2 Chemical composition of quartz (mass fraction, %)

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	P ₂ O ₅	CaO	MgO	MnO ₂
0.962	98.447	0.027	6×10^{-8}	0.025	1.11×10^{-5}	2.2×10^{-3}
V	S	Sr	Zn	Cu	Pb	K ₂ O
4×10^{-4}	0.127	9×10^{-4}	1.8×10^{-3}	1.6×10^{-3}	1.2×10^{-7}	1×10^{-7}

The samples were dry-milled in a planetary ball mill to the sizes of 90% less than 44 μm , and stored in air-tight plastic bags to avoid further oxidation. Gold foil (Au 99.99%) was used in the experiments with a surface area of 0.38 cm^2 . Sodium thiosulfate pentahydrate, ammonia water (25%, mass fraction), cupric sulfate, hydrogen peroxide (30%, mass fraction) and nitric acid (50%, volume fraction) were all in analytically pure grade. De-ionized water was used throughout all experiments.

2.2 Leaching experiment

The gold foils were polished by a 4000 grid sand paper and washed with acetone twice and then rinsed with de-ionized water before each test. After that, 200 mL de-ionized water containing desired quantities of reagents was added into a 250 mL beaker with a mechanical agitator (IKA EURO-STPCUS25) at a rotating speed of 200 r/min. A gold foil was then suspended in the middle of the solution. All experiments were performed at $(25 \pm 0.5)^\circ\text{C}$ with the solution pH value maintained in the range of 9.5–10.5 during 24 h leaching. The samples were taken continuously at certain intervals from the solution during the 24 h leaching, and the obtained samples were centrifuged and filtered for the subsequent analysis. All experiments were conducted with the reagent concentrations of 0.1 mol/L $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, 0.012 mol/L $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 0.5 mol/L $\text{NH}_3 \cdot \text{H}_2\text{O}$.

2.3 Detection methods

The concentrations of gold, iron and arsenic in solutions were analyzed by atomic absorption spectrum (AA-6800, SHIMADZU). The thiosulfate concentration was determined by iodometric method. In order to eliminate the effects of cupric-ammonia complex and sulfite on the iodine titration, a certain amount of EDTA-2Na and formaldehyde water were added prior to the titration with the indicator Vitex. A platinum electrode was used to measure the redox potential with a double junction reference electrodes (Ag/AgCl, saturated

KCl). All potentials were given with respect to standard hydrogen electrode (SHE).

X-ray photon spectroscopy (XPS) (ESCALAB 250Xi, Thermo Fisher) was used to identify the surface species on the leached gold foils and minerals. It was conducted with a monochromatic aluminum excitation (1486.6 eV) operating at 200 W. Cu 2p, Fe 2p, S 2p and As 3d spectra were collected using a pass energy of 20 eV and an energy step of 0.1 eV. The binding energy calibration was based on C1s at 284.6 eV. Advantage5.52 software was used to fit the XPS peaks. A summed Gaussian–Lorentzian (SGL) function was used to describe the convoluted Gaussian–Lorentzian line shapes of the peaks. The weighting used was 70% Gaussian and 30% Lorentzian for S 2p. The background of the spectrum was obtained using the Shirley method. The gold foils and arsenopyrite samples were rinsed with distilled water and dried under vacuum overnight for XPS testing.

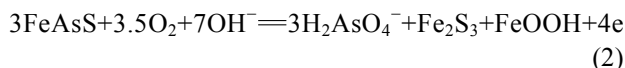
3 Results and discussion

3.1 Thermodynamic calculation

A thermodynamic program HSC chemistry was used to plot the ϕ –pH diagram of Fe–As–S–H₂O system to visualize the chemical reactions in terms of potential and pH value for the thiosulfate leaching of gold in the presence of arsenopyrite. Under normal temperature and pressure, the most relevant ϕ –pH area of thiosulfate leaching is in the potential range of -0.5 to $+0.5$ V and in the pH value range of 6 to 12. The sulfur, iron and arsenic concentrations used in plotting the diagrams were determined, which are 0.2 mol/L, 0.0002 mol/L and 0.002 mol/L, respectively.

Figure 1 shows that the oxidation potential of arsenopyrite is negative, so it is unstable in the most relevant pH value and potential ranges of thiosulfate leaching under normal temperature and pressure. The oxidation of arsenopyrite firstly generates pyrrhotite and pyrite, which can oxidize to iron hydroxide with the

increase of redox potential, as shown in Fig. 1. The possible reactions of arsenopyrite in thiosulfate leachate are shown in Eqs. (1) and (2).



However, other than iron hydroxide, the precipitate of iron arsenate is supposed to coat on the leached gold surface in the presence of arsenopyrite [8,11]. If iron hydroxide is out of calculation in plotting the ϕ -pH diagram, a stable region of iron arsenate exists. As shown in Fig. 2, the species of FeAsO_4 or $\text{Fe}_3(\text{AsO}_4)_2$ are the ultimate oxidation products of arsenopyrite when iron hydroxide is out of calculation in plotting the diagram. This indicates that iron hydroxide is more easily to form than iron arsenate, thermodynamically. It may be the dynamic factors that are responsible for the formation of iron arsenate.

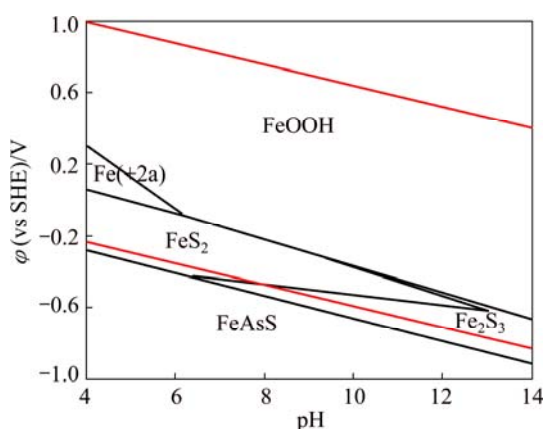


Fig. 1 ϕ -pH diagram of Fe-As-S-H₂O system

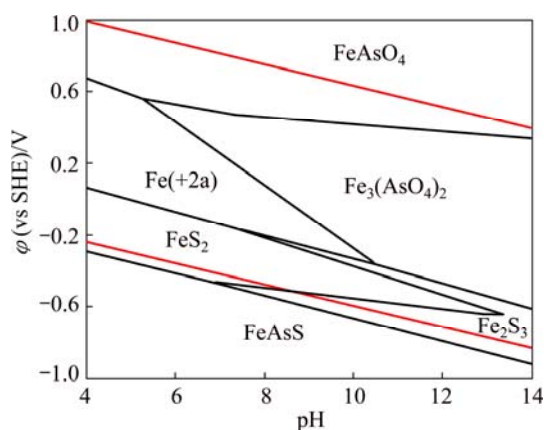
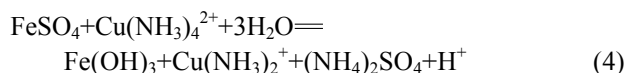
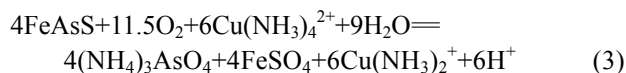


Fig. 2 ϕ -pH diagram of Fe-As-S-H₂O system without consideration of FeOOH

3.2 Dissolution behavior of arsenopyrite

The dissolution characteristics of arsenopyrite can be represented by the iron and arsenic contents in thiosulfate solution. As shown in Fig. 3, both the concentrations of iron and arsenic are low during the

entire leaching period. The arsenic content first increases to the highest point of 275.6×10^{-6} and then goes down with time, while the iron content is consistently lower than 30×10^{-6} during the entire leaching period. The possible reactions of arsenopyrite during thiosulfate leaching of gold are shown in Eqs. (3)–(5) [5]:



The above reactions mainly focus on the oxidation of arsenopyrite where the sulfur compounds are all simplified to sulfate. As shown in Eqs. (3) and (5), arsenopyrite first reacts with copper ammonia to generate ammonium arsenate which then reacts with iron hydroxide to form iron arsenate. This is consistent with the changing trend of arsenic content which first rises and then drops, as shown in Fig. 3. The iron precipitates form along with the dissolution of arsenopyrite, which may follow the order of $\text{Fe}_2\text{S}_3 \rightarrow \text{Fe}_2\text{S} \rightarrow \text{FeOOH}$, as shown in Fig. 1.

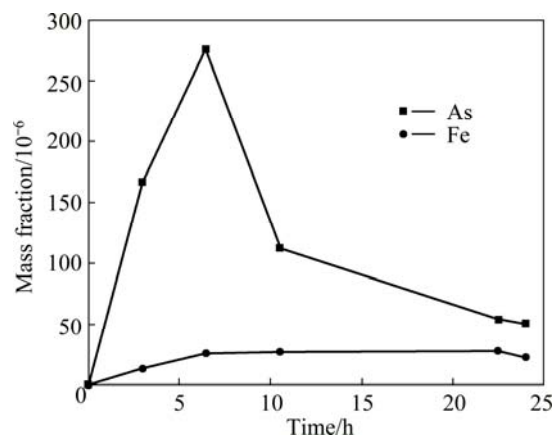


Fig. 3 Variation of iron and arsenic contents with time in presence of 10 g/L arsenopyrite

In summary, only if iron hydroxide is out of thermodynamic calculation, the precipitate of iron arsenate forms as a result of arsenopyrite oxidation in thiosulfate leaching solution, as shown in Figs. 1 and 2. But, the dissolution behavior and the possible reactions of arsenopyrite indicate the likely presence of iron arsenate during the thiosulfate leaching of gold. So, the role of arsenopyrite on the thiosulfate leaching of gold is further studied according to the leaching test and the chemical component of the leached gold foil surface.

3.3 Effect of arsenopyrite on thiosulfate leaching

Based on the inert properties of quartz, it can be used as standard to compare with arsenopyrite [2]. As

shown in Fig. 4(a), 35% of thiosulfate is consumed after 24 h leaching in the absence of arsenopyrite. However, the consumptions of thiosulfate are 42.5%, 46% and 48% when the concentrations of arsenopyrite are 5, 10 and 20 g/L, respectively. Hence, arsenopyrite accelerates the decomposition of thiosulfate, and the negative effect slightly increases with increasing the concentration of arsenopyrite. This is different from the research carried out by CHEN [10] where arsenopyrite had almost no impact on the stability of thiosulfate.

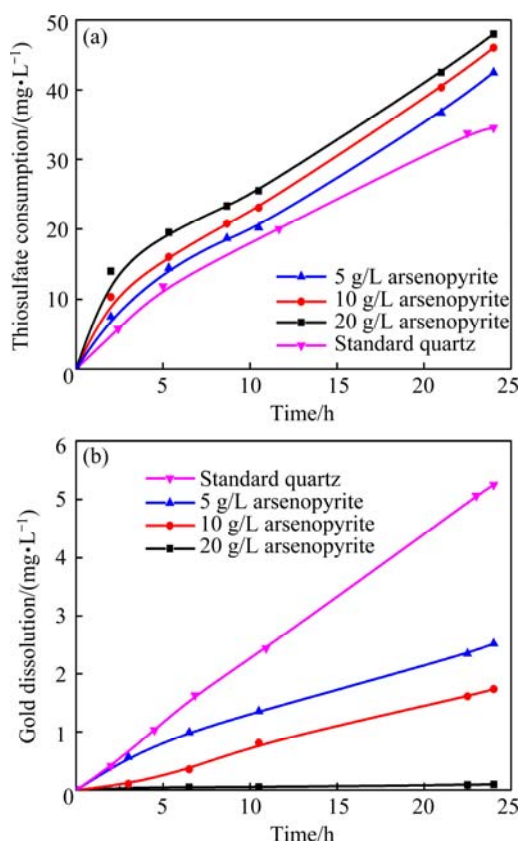


Fig. 4 Variation of thiosulfate consumption (a) and gold dissolution (b) with time in thiosulfate leaching of gold in presence of 10 g/L quartz

The redox potential of the solution was also measured by a platinum electrode with a Ag/AgCl reference electrode. In the initial 5 h leaching, the redox potential of the solution is in the range of 270–229 mV in the presence of 10 g/L quartz, but decreases to the range of 238–213 mV in the presence of 10 g/L arsenopyrite.

Even though the initial range of the redox potential decreases in the presence of arsenopyrite, it accelerates the decomposition of thiosulfate (which is an oxidation reaction) yet. This may be caused by the semi-conductive properties of arsenopyrite, which accelerates the transfer of electrons from thiosulfate to oxygen. So, it is the catalytic effect of arsenopyrite that increases the thiosulfate decomposition.

As shown in Fig. 4(b), the gold dissolutions are 5.25, 2.52, 1.74 and 0.1 mg/L when the concentrations of arsenopyrite are 0, 5, 10 and 20 g/L, respectively. It is worth noting that gold leaching is almost completely inhibited when the addition of arsenopyrite increases to 20 g/L. This reveals that arsenopyrite hinders the gold dissolution in the thiosulfate leaching of gold, and the gold dissolution is significantly hindered at high concentrations of arsenopyrite. It may be the coated layer on the gold surface that passivates gold leaching.

In summary, arsenopyrite influences the thiosulfate leaching of gold. It catalyzes the thiosulfate consumption and hinders the gold dissolution, simultaneously. The catalytic effect of arsenopyrite on the thiosulfate consumption slightly increases when the concentration of arsenopyrite increases, but the gold dissolution significantly reduces.

3.4 XPS analysis

In order to further investigate the component of the passivation layer, the arsenopyrite and gold foil were analyzed using XPS. The spectra of Cu 2p, As 3d, S 2p and Fe 2p were recorded for these samples.

Figure 5(a) shows Cu 2p spectra for arsenopyrite before and after the thiosulfate leaching of gold. As

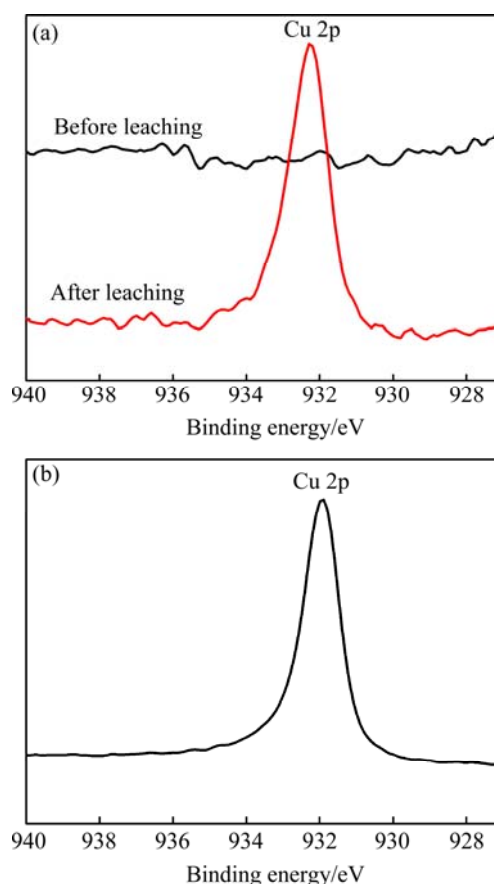


Fig. 5 Cu 2p spectra for arsenopyrite before and after 24 h leaching in thiosulfate solution (a) and Cu 2p spectrum for leached gold after 24 h leaching (b)

shown in Fig. 5(a), there is no peak of Cu $2p_{3/2}$ before leaching. But, an obvious peak of Cu $2p_{3/2}$ centers at 932.35 eV (FWHM=1.27 eV) after leaching. It is a typical character of Cu(I), which can be originated from Cu_2S or $Cu(S_2O_3)_3^{5-}$ [12]. So, the copper precipitate on the surface of arsenopyrite is mainly in the form of Cu(I). The copper species on the surface of leached gold are also detected, as shown in Fig. 5(b). The Cu $2p_{3/2}$ centered at 932 eV (FWHM=1.17 eV) on the leached gold surface is consistent with that on the arsenopyrite surface.

Figure 6(a) shows the S 2p peaks of gold foil. The S 2p peaks occur as doublets as a result of spin-orbit splitting. After the XPS-peak-differentiation-imitating analysis, the doublets peak in Fig. 6(a) can be converted into three doublets, as shown in Fig. 6(b). The three doublets of S $2p_{3/2}$ peaks center at 161.5 (FWHM=0.78 eV), 162.3 (FWHM=0.82 eV) and 163.9 eV (FWHM=0.8 eV), which are assigned to monosulfide [13,14], disulfide or thiosulfate [15,16] and element sulfur [17], respectively. The dominant sulfur specie is disulfide or thiosulfate according to the peak area.

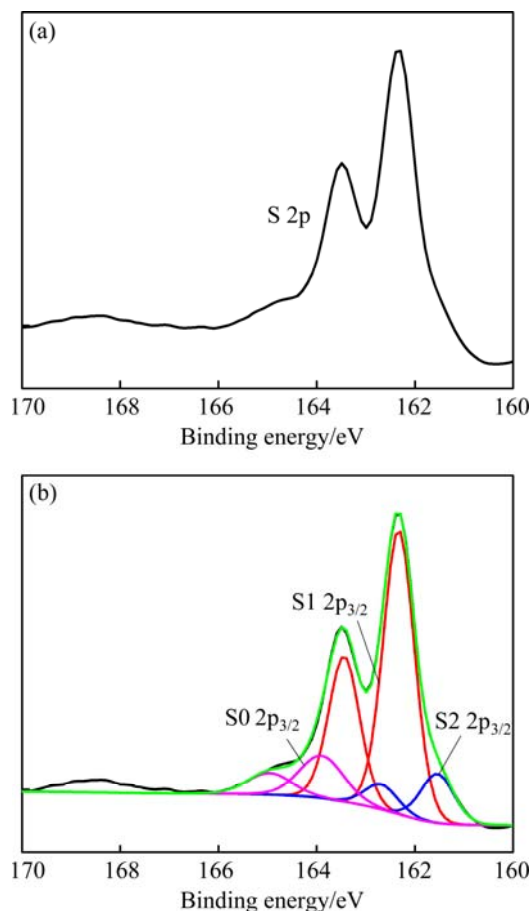


Fig. 6 S 2p spectrum for leached gold in presence of 10 g/L arsenopyrite (a) and S 2p spectra after peak-differentiation-imitating analysis (b)

Figure 7 shows the As 3d spectrum for the leached gold foil in the presence of arsenopyrite, which reflects a typical peak of arsenate at 44.3 eV [18]. As discussed in Section 3.1, the precipitate of iron hydroxide is more easily to form than that of iron arsenate from thermodynamic calculation. But, the detection of arsenate species on the leached gold surface supports the possible oxidation reactions, as shown in Eqs. (3) and (5). So, arsenate species on the leached gold surface may be in the form of ion arsenate. This may be ascribed to the low activation energy barrier, which is a dynamic reason for the formation of iron arsenate.

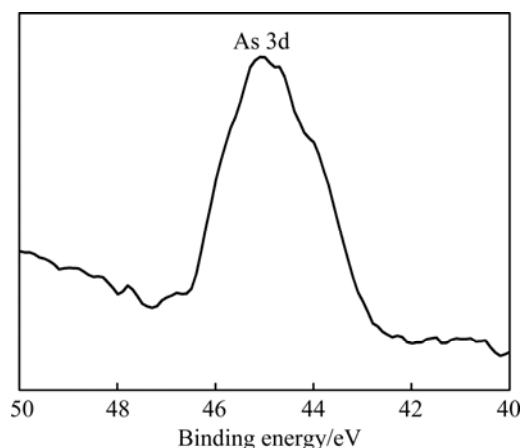


Fig. 7 As 3d spectrum for leached gold after 24 h leaching in thiosulfate solution

As shown in Fig. 8, the obvious peak near 711.8 eV in Fe 2p electron spectrum is assigned to FeOOH [19,20]. This is consistent with the φ -pH diagrams of Fe-As-S- H_2O system (Section 3.1), which indicates easy formation of FeOOH during the thiosulfate leaching of gold.

The mole fractions of sulfur, copper, iron and arsenic on the leached gold surface are 35.68%, 37.31%, 19.06% and 7.95%, respectively. This indicates that the coating layers on the leached gold surface are mainly composed of copper, sulfur and iron species, and significant amount of arsenate species.

In summary, according to the XPS analysis, the arsenate species form on the surface of gold in the form of iron arsenate. The composition of the passivation layer is likely Cu_2S or $Cu(S_2O_3)_3^{5-}$, element S, FeOOH and iron arsenate. It is the passivation layer on the leached gold foil surface that largely inhibits gold leaching.

3.5 Thiosulfate leaching with additives

Among the additives that have been studied, sodium carboxymethyl (CMC) and sodium phosphate (SHPP) are universal and well-adapted to minerals. So, additives of CMC and SHPP were used to relieve the detrimental impact of arsenopyrite on the thiosulfate leaching of gold.

Figure 9 reveals the impacts of additives on the thiosulfate leaching of gold.

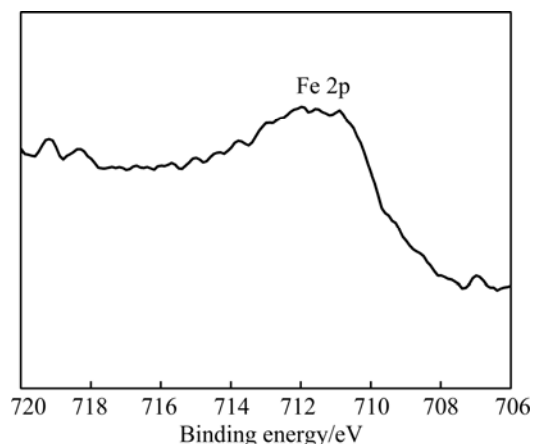


Fig. 8 Fe 2p spectrum for leached gold after 24 h leaching in presence of 10 g/L arsenopyrite

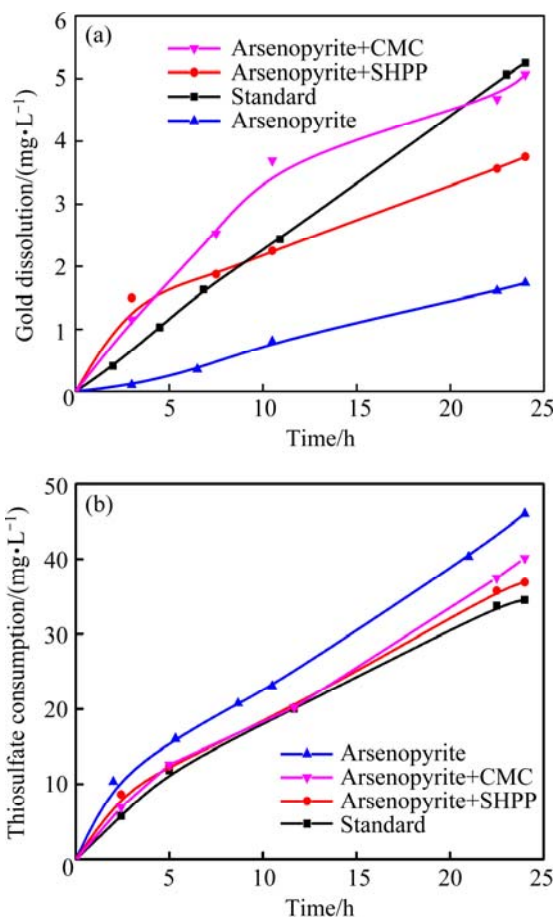


Fig. 9 Effect of CMC and SHPP on gold dissolution (a) and thiosulfate consumption (b) in presence of 10 g/L arsenopyrite with 5 g/L SHPP and 120 mg/L CMC

As shown in Figs. 9(a) and (b), proper concentrations of CMC and SHPP improve the gold dissolution from 1.74 to 5.06 and 3.75 mg/L, and reduce the thiosulfate consumption from 46% to 40% and 37%, respectively.

The roles of CMC and SHPP on reducing the detrimental effect of arsenopyrite are proposed as follows: the negatively charged functional groups of CMC and SHPP compete with thiosulfate to complex cupric ions [9] and thus stabilize thiosulfate; the non-selective adsorption of additives prevents the precipitates from coating on the gold surfaces by electrostatic repulsion and hence improves gold leaching [21,22].

4 Conclusions

1) Arsenopyrite increases the thiosulfate decomposition, and inhibits the gold dissolution due to the passivation layer formed on the gold surface during the thiosulfate leaching. The thiosulfate consumption slightly increases when the concentration of arsenopyrite increases, but the gold dissolution is significantly hindered.

2) According to the thermodynamic calculation, mineral dissolution test and XPS analysis, it is Cu_2S or $\text{Cu}(\text{S}_2\text{O}_3)_3^{5-}$, element S, FeOOH and iron arsenate species on the leached gold foil surface that passivate the gold leaching.

3) The negative effect of arsenopyrite is relieved by adding additives. The gold dissolution increases from 1.74 to 5.06 and 3.75 mg/L, and the thiosulfate consumption decreases from 46% to 40% and 37% by adding 120 mg/L CMC and 5 g/L SHPP, respectively.

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砷黄铁矿对硫代硫酸盐浸金的影响

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摘要: 在硫代硫酸盐浸出液中人工加入砷黄铁矿以了解砷黄铁矿在硫代硫酸盐浸金过程中发挥的作用。通过热力学计算、矿物溶解行为实验、浸出实验和 XPS 分析, 研究砷黄铁矿对硫代硫酸盐浸金过程的影响。结果表明: 砷黄铁矿对硫代硫酸盐分解过程有催化作用, 随着砷黄铁矿用量的增加, 硫代硫酸盐的消耗量增加, 而在金表面生成的钝化膜则会显著降低金的溶解量。XPS 分析结果表明: 金表面的钝化膜由 Cu_2S 或者 $\text{Cu}(\text{S}_2\text{O}_3)_3^{5-}$ 、元素 S、 FeOOH 和砷酸铁构成。向浸出液中加入添加剂可以降低砷黄铁矿对硫代硫酸盐浸金过程的不利影响。研究结果表明: 在含有砷黄铁矿的硫代硫酸盐浸金过程中, 添加剂羧甲基钠(CMC)和磷酸钠(SHPP)都可以在降低硫代硫酸盐的消耗的同时增强金的溶解。

关键词: 浸金; 硫代硫酸盐; 砷黄铁矿; 钝化

(Edited by Mu-lan QIN)