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## Basic theory and optimization of gold containing antimony concentrate leaching by alkaline sulfide

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**Abstract:** As sodium sulfide is easily oxidized to polysulfide and thiosulfate which have a gold leaching effect, gold would dissolve in leaching solution when extracting stibium from gold containing antimony concentrate by alkaline sulfide. Through leaching test and kinetics analysis, the decomposition regularity of sodium sulfide and leaching rate were studied under different leaching conditions. The results indicated that the gold content in antimony concentrate was 28.41g/Mg, and the content of antimony and sulfur was 36.01% and 14.04%, respectively. The main metallic minerals were native gold, arsenopyrite, stibnite, and the gangue minerals were mainly quartz. Anodic polarization curve shows reduced iron powder can increase the peak potential of the oxidation of the leaching solution and it can effectively prevent the decomposition of sodium sulfide and the dissolution of gold. Optimized stibium-extraction efficiency was achieved under the following conditions: a concentration of sodium sulfide and sodium hydroxide at 110g/dm<sup>3</sup> and 20g/dm<sup>3</sup>, respectively; a ratio of iron powder to concentrate of 1:30; a ratio of liquid to solid of 5:1; agitation speed of 600rad/min; reaction temperature of 353.15K; and a reaction time of 3 h. Under the optimized conditions, high antimony recovery (97.35%) and low gold dissolution (1.32%) were achieved.

**Keywords:** antimony concentrate, leaching process, alkaline leaching, kinetics

### 1. Introduction

Gold containing antimony ore is a type of refractory gold in which gold is intimately associated with stibnite (Guo et al., 2017). When the traditional cyanidation method is used for gold extraction, thioantimonate by stibnite decomposition reacts with oxygen to form antimony acid salt, or antimony reacts with sodium cyanide to form a stable compound. Due to a large amount of cyanide and oxygen consumption, the effect of cyanide leaching on gold is adversely affected (Raschman et al., 2012; Multani and Smincakova, 2016;).

In order to avoid the adverse effects of stibnite on cyanide leaching, the pre-treatment process of hydrometallurgy to recover antimony before gold extraction can be adopted (Mahlangu et al., 2006). Because leaching has the advantages of strong adaptability and low environmental pollution, there are two well-reputed methods of leaching antimony using acid or alkali. Owing to acid mist volatilization, equipment corrosion, and low leaching rate, the process of acid leaching has not been applied in industry so far (Guo et al., 2016). The most effective and frequently-used method is the alkaline leaching by sodium sulfide. Its essential procedure includes antimony dissolved into leach solution, and the antimony metal of 96-99% purity is produced by electrowinning from sulfantimonate solution. The tailing of pretreatment process enters the cyanide process as raw material for gold extraction (Yang et al., 2017).

However, when gold containing antimony ore is pre-leached by sodium sulfide, sodium sulfide will oxidize and hydrolyze easily to form polysulfide and thiosulfate which have the ability to leach gold

(Anderson, 2012). These substances will react with gold, so that a lot of dissolved gold is contained in the antimony solution. The separation and recovery of antimony and gold is therefore be impacted (Yang et al., 2014; Yu et al., 2016; Huang et al., 2003). At the same time, with the recycling of the leaching solution, the accumulation of sodium polysulfide, sodium sulphate, sodium thiosulfate and sodium sulfite in the leaching process is serious, creating difficulties to the electrowinning of antimony (CELEP et al., 2011), and also making the treatment of waste liquid complicated.

In order to prevent the oxidation of sodium sulfide, the influence of leaching parameters on the sodium sulfide oxidation has been explored by electrochemical measurement. Change regulations of the leaching rate of antimony and gold were investigated under different leaching conditions (Janusz and Skwarek, 2018). Based on these findings, the decomposition of sodium sulfide and the dissolution of gold in the leaching process of antimony were avoided by adding an auxiliary agent, and the comprehensive recovery of valuable metal was realized.

## 2. Materials and methods

### 2.1. Materials and characterization

Gold containing antimony concentrate in Shandong, China was selected as the research object in this study. The main chemical composition of the ore sample is shown in Table 1. The leaching rate of gold will be weak due to the influence of antimony if direct gold leaching is carried out by cyanide. As is well known, As-bearing gold ore is leached ineffectively. Thus, the ore was refractory gold ore because the content of As was 2.73%.

Table 1. Multi-element analysis results of antimony ore

Element	O	Sb	S	Si	Fe	As
Content/ %	36.47	36.01	14.04	4.00	3.09	2.73
Element	Ca	Al	Pb	Zn	K	Au*
Content/ %	0.99	0.90	0.63	0.47	0.31	28.41

\* Unit g/Mg.

The main phases of the material were analyzed by XRD (Fig. 1). According to Table 1 and Fig. 1, the metallic minerals are mainly composed of native gold (Au), arsenopyrite (FeAsS), stibnite (Sb<sub>2</sub>S<sub>3</sub>), and the gangue minerals were mainly quartz (SiO<sub>2</sub>).

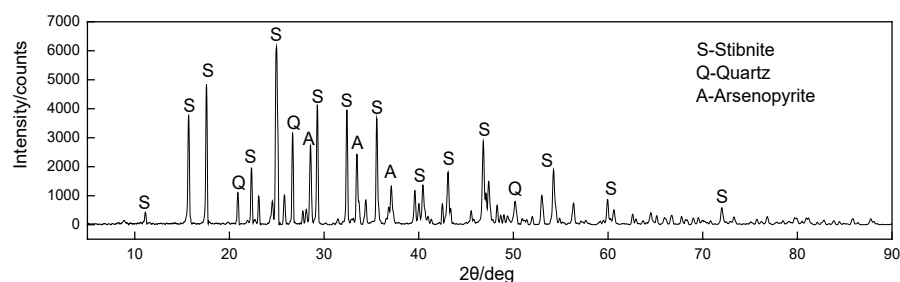


Fig. 1. XRD curve of the test sample

### 2.2. Leaching test

The samples were leached in a 250 cm<sup>3</sup> conical flask, which was magnetically stirred at 600 rpm and placed in a water bath to control the leaching temperature on the HJ-6A magnetic heating agitator. A 150 cm<sup>3</sup> leach solution (Na<sub>2</sub>S+NaOH) was added to the conical flask, followed by a 30 g ore sample. Through single-factor experiments, the effects of sodium sulfide concentrations (90-130 g/dm<sup>3</sup> Na<sub>2</sub>S), sodium hydroxide concentrations (10-40 g/dm<sup>3</sup> NaOH), temperature (333.15-363.15 K) and auxiliary agent on gold and antimony leaching were investigated under the liquid-solid ratio of 5:1. After a leaching period of 180 min, solid and liquid phases were separated by filtration and the leaching residue

was analyzed for antimony and gold. Finally, the leaching rates of gold and antimony were calculated (Ubaladini et al., 2000).

### 2.3. Dynamic test

The objective of the kinetics study was to reveal the intrinsic relationship between the decomposition of sodium sulfide and the technological parameters of antimony leaching (Sayiner, 2015). And, through researching the effects of sodium sulfide, sodium hydroxide, temperature and auxiliary agent on polarization curve, the polarization properties and electrode process of sodium sulfide decomposition in the process of alkaline leaching of antimony were studied. The three-electrode system and CHI660D electrochemistry tester were used in the experiment, CHI104 glassy carbon electrodes were used as the auxiliary electrode and working electrode, and a saturated calomel electrode was used as the reference electrode. The surfaces of the electrodes were cleaned before each measurement. During the experiment, the reaction medium was heated to a specified temperature and stirred, and the selected agents were added according to the test conditions. In order to measure the oxidation behavior of sodium sulfide during alkaline leaching, steady-state polarization curves in reaction medium were measured. The scanning speed of the steady-state polarization curve was determined to be  $1 \text{ mV s}^{-1}$  of the experiment. The experimental device is illustrated in Fig. 2.

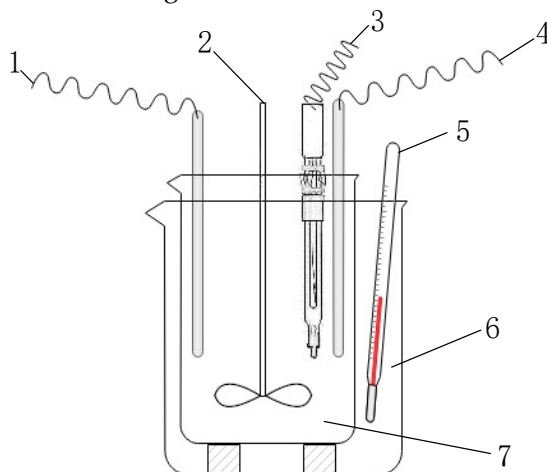
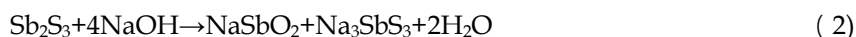


Fig. 2. Diagram of experimental device: 1. Counter elect rode, 2. Blender, 3. Working elect rode, 4. Reference electrode, 5. Thermometer, 6. Water-bath, 7. Slurry

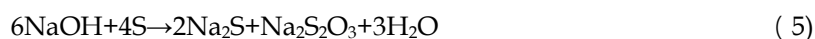
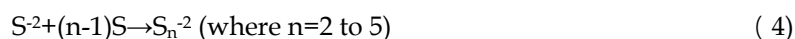
## 3. Results and discussion

### 3.1. Anodic polarization curve of sodium sulfide solution

The alkaline pre-leaching of antimony sulfide proceeds in accordance with the following reaction (Eq. (1), Eq. (2)):



According to the analysis of the reaction, the complex decomposition reaction mainly occurs in the solution. There is no redox reaction in the process of leaching antimony. Due to the oxidation and hydrolysis of sodium sulfide, the following reactions are inevitable in the antimony leaching solution (Anderson et al., 2016).



The formation of polysulfide and thiosulfate will dissolve gold as the following reaction. As a result, the separation of gold and antimony is not thorough, which increases the difficulty of gold recovery and leads to the loss of gold.

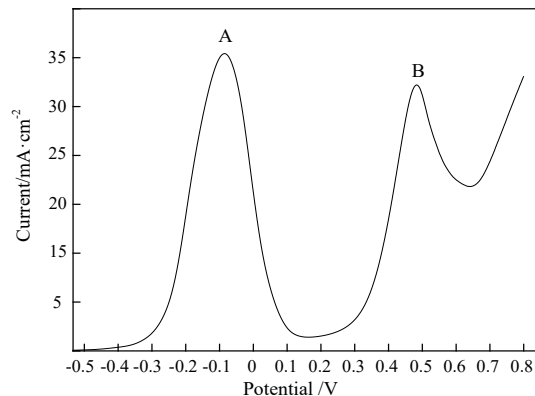
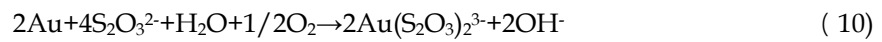
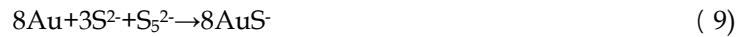


Fig. 3. Anodic polarization plot in solutions of  $\text{Na}_2\text{S}$ . Scanning rate 1.0 mV/s, concentration of  $\text{Na}_2\text{S}$  100 g/dm<sup>3</sup>, concentration of  $\text{NaOH}$  10 g/dm<sup>3</sup>, and temperature 353.15 K

In order to study the rule of oxidation decomposition of sodium sulfide during the antimony leaching process, the anode polarization plot of sodium sulfide in the solution was measured. In the potential range from -0.5 to 0.8 V, it is clear that there were two main oxidation reactions carried out in the solution as evidenced by the two current peaks in the anode curve. It is well known that peak current represents the maximum rate of oxidation reaction, and peak potential represents the anode potential at the maximum oxidation rate. It can be noted from Fig. 3 that the first peak current at -80 mV is slightly higher than the second peak current at 480 mV. This shows that the rate of the first reaction was slightly greater than that of the second.

About the reaction process of the two peaks, it is generally believed that the first peak A means sodium sulfide is oxidized to elemental sulfur and polysulfide (Qin et al., 2000; Zhang et al., 2012; Yi, 2002; Zhang et al., 2017), then the surface of the electrode is passivated. The reaction of the first peak A corresponded to Eq. (3) and Eq. (4), and the analysis of the anode electrode products confirmed this conclusion. The second peak B implied that the passivation layer of elemental sulfur was dissolved by the high concentration of sulfur ion  $\text{S}^{2-}$ . The increase of the electron transfer amount caused the increase of the current value. The reaction of the second peak B corresponded to Eq. (5), and sodium thiosulfate was produced.

In the alkali leaching process of antimony, the dissolution of antimony sulfide is not a redox reaction. It is mainly the oxidation decomposition of sodium sulfide corresponding with the reaction of the anodic curve. In order to observe and contrast the polarization curve succinctly, the rule of the current and potential of the first peak must be measured to preliminarily examine the oxidation process of sodium sulfide.

### 3.2. Effect of sodium sulfide concentration

The solubility of stibnite in sodium sulfide solution increases with the increase of concentration of sodium sulfide. But, excessive sodium sulfide will be oxidized into sodium thiosulfate and sodium polysulfide, so that gold in stibnite will be lost (Wen et al., 2017). The effect of sodium sulfide

concentration on the leaching rate of antimony and gold was studied under the condition of sodium hydroxide concentration at 20 g/dm<sup>3</sup>, stirring speed at 600 rad/min, temperature at 353.15 K, leaching time of 3 h and a liquid-solid ratio of 5:1. At the same time, the oxidation decomposition law of sodium sulfide in the leaching process was obtained by combining the kinetic polarization curve. The leaching results are shown in Fig. 4, and the anodic polarization plots are shown in Fig. 5.

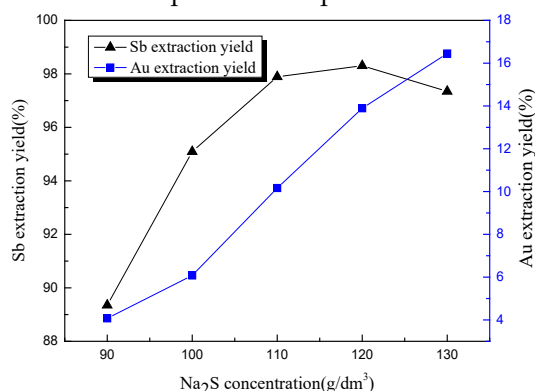


Fig. 4. The effect of the Na<sub>2</sub>S concentration on extraction

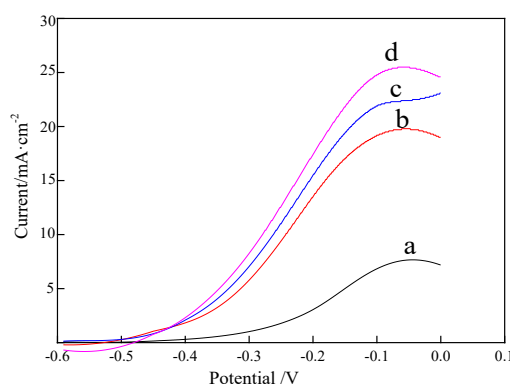


Fig. 5. Anodic polarization plots in solutions of various Na<sub>2</sub>S concentration. (a) - Na<sub>2</sub>S=90 g/dm<sup>3</sup>, (b) - Na<sub>2</sub>S=100 g/dm<sup>3</sup>, (c) - Na<sub>2</sub>S=110 g/dm<sup>3</sup>, (d) - Na<sub>2</sub>S=120 g/dm<sup>3</sup>. Scanning rate 1.0 mV/s, concentration of NaOH 20 g/dm<sup>3</sup>, and temperature 353.15 K

It can be noted from Fig. 4 that the antimony leaching increased firstly and then decreased with increasing sodium sulfide concentration, from 90 g/dm<sup>3</sup> to 130 g/dm<sup>3</sup>. The antimony leaching rate had a turning point when sodium sulfide concentration was 120 g/dm<sup>3</sup>. But the gold leaching rate increased from 4.07% to 16.45% with an increase in sodium sulfide concentration. When the concentration of sodium sulfide increased from 110 g/dm<sup>3</sup> to 120 g/dm<sup>3</sup>, the leaching rate of antimony increased only 0.41%, while the loss rate of gold increased 3.73%. As a result, the optimum dosage of sodium sulfide was determined to be 110 g/dm<sup>3</sup>.

It is shown from the curve in Fig. 5 that the current and the current peak value increased at the same potential with the increase of sodium sulfide. It is indicated that the process of anodic oxidation was controlled by the electron transfer of sulfur ions on the anode surface, and by the diffusion of sulfur ions in the electrolyte to the surface of the electrode. Remarkably, the change in sodium sulfide concentration affected the peak of the polarization curve. With the increase of sodium sulfide concentration, the peak current of the polarization curve increased and the peak potential reduced slightly. The current density was positively correlated with the velocity of electron migration when the redox reaction occurs in the solution. Therefore, the current density can be used to indicate the rate of oxidation reaction. The phenomenon showed that increasing sodium sulfide concentration was beneficial to the formation of polysulfide ions in the solution, and also it stimulated the oxidation rate of sodium sulfide.

### 3.3. Effect of sodium hydroxide concentration

When using sodium sulfide to leach stibnite, sodium hydroxide was added to inhibit the hydrolysis and decomposition of sodium sulfide. Moreover, stibnite can also be dissolved by sodium hydroxide as the concentration of sodium sulfide was low. But excessive sodium hydroxide would affect stibnite dissolution and mass transfer to reduce the leaching rate of stibnite (Li et al., 2010). The effect of sodium hydroxide concentration on the leaching rate of antimony and gold was studied under the condition of sodium sulfide concentration of 110 g/dm<sup>3</sup>, stirring speed at 600 rad/min, temperature at 353.15 K, leaching time of 3 h and a liquid-solid ratio of 5:1. Similarly, the oxidation decomposition law of sodium sulfide under different sodium hydroxide concentrations was obtained by combining the kinetic polarization curve. The leaching results are shown in Fig. 6, and the anodic polarization plots are shown in Fig. 7.

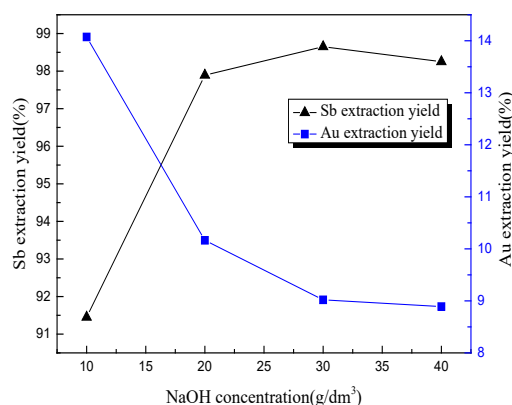


Fig. 6. The effect of the NaOH concentration on extraction

The following phenomena can be observed in Fig. 6: As the concentration of sodium hydroxide increased from 10 g/dm<sup>3</sup> to 40 g/dm<sup>3</sup>, the leaching rate of antimony increased and the leaching rate of gold reduced initially, then both leaching rates remained relatively stable with further increasing. Whereas the change of the leaching rate of antimony and gold had only negligible change when the concentration of sodium hydroxide increased from 20 g/dm<sup>3</sup> to 30 g/dm<sup>3</sup>, the concentration of sodium hydroxide was determined to be 20 g/dm<sup>3</sup>.

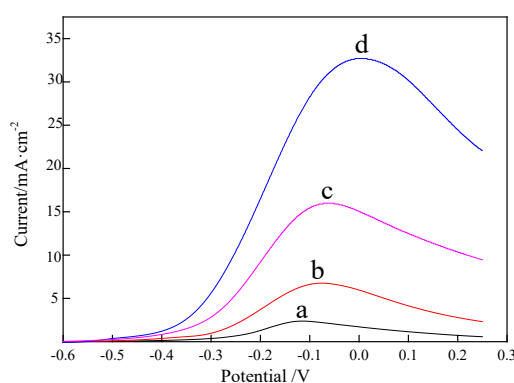


Fig. 7. Anodic polarization plots in solutions of various NaOH concentration. (a) - NaOH =40 g/dm<sup>3</sup>, (b) - NaOH =30 g/dm<sup>3</sup>, (c) - NaOH =20 g/dm<sup>3</sup>, (d) - NaOH=10 g/dm<sup>3</sup>. Scanning rate 1.0 mV/s, concentration of Na<sub>2</sub>S 110 g/dm<sup>3</sup>, and temperature 353.15 K

It is shown that the peak potential of oxidation increased with an increase in the concentration of sodium hydroxide. This indicated that an increasing amount of alkali could increase the electrode potential of the oxidation reaction and avoid the oxidation decomposition of sodium sulfide within certain limits. But, an excessive amount of sodium hydroxide would increase the peak current, and then lead to an increase in the oxidation rate of sodium sulfide. That is because the excessive OH-

concentration would promote the Eq. (5), thus the oxidation rate of sodium sulfide into elemental sulfur was increased (Eq. (3)).

### 3.4. Effect of temperature

Temperature has a positive influence on leaching velocity and sodium sulfide oxygenolysis. (Kowalczuk et al., 2018). The effect of temperature on the leaching rate of antimony and gold was obtained under the condition of sodium sulfide concentration of 110 g/dm<sup>3</sup>, sodium hydroxide concentration of 20 g/dm<sup>3</sup>, stirring speed at 600 rad/min, leaching time of 3 h and a liquid-solid ratio of 5:1. Also, the effect of temperature on extraction was researched (Fig. 8), and anodic polarization plots in solutions of various temperature were researched and shown in Fig. 9.

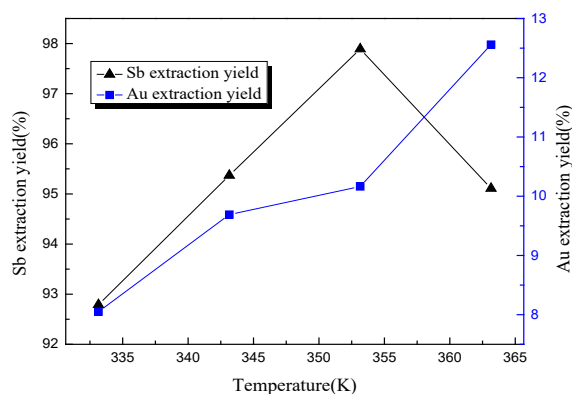


Fig. 8. The effect of temperature on extraction

From the data in Fig. 8, it can be seen that the extraction rate of antimony increased firstly from 92.79% to 97.89%, and then decreased to 95.11% with the increase of leaching temperature from 333.15 K to 363.15 K. The trend of the extraction rate of antimony reversed at 353.15 K. Exorbitant temperature was beneficial to the oxygenolysis of sodium sulfide to reduce the antimony extraction rate. The dissolution rate of gold increased from 8.05% to 12.56% with the increase of temperature. Due to the increase of the concentration of polysulfide and thiosulfate with increase of temperature in the solution, the rate of gold dissolution was accelerated. Considering antimony extraction and gold dissolution, 353.15 K was determined to be the optimum leaching temperature.

It can be noted from Fig. 9 that the anodic current peak and polarization current density for sodium sulfide oxidation significantly increased with increasing temperature from 333.15 K to 363.15 K. But the peak potential had a non-obvious change. It is indicated that the temperature could promote the oxygenolysis rate of sodium sulfide, but impacted only slightly on the electrode potential of the oxidation reaction.

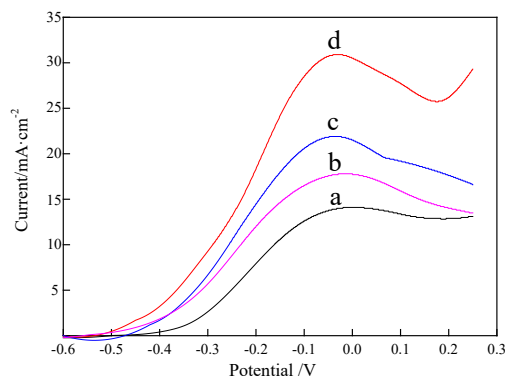


Fig. 9. Anodic polarization plots in solutions of various temperature. (a) - 333.15 K, (b) - 343.15 K, (c) - 353.15 K, (d) - 363.15 K. Scanning rate 1.0 mV/s, concentration of Na<sub>2</sub>S 110 g/dm<sup>3</sup>, and concentration of NaOH 20 g/dm<sup>3</sup>

### 3.5. Effect of reduced iron powder

In view of the oxidation of sodium sulfide in the antimony leaching process, reduced iron powder was added into the antimony leaching solution. The influence of reduced iron powder on the leaching rate of antimony and gold was analyzed, and the oxygenolysis law of sodium sulfide under different iron powder dosages was obtained. The results are presented in Fig. 10 and Fig. 11. The experimental conditions were sodium sulfide addition of 110 g/dm<sup>3</sup>, sodium hydroxide addition of 20 g/dm<sup>3</sup>, leaching temperature of 353.15 K, stirring speed of 600 rad/min, leaching time of 3 h, and a liquid-to-solid ratio of 5:1.

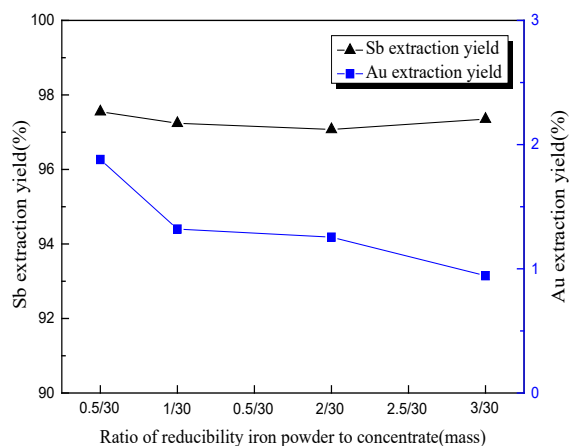


Fig.10. The effect of the reduced iron powder on extraction

According to the data in Fig. 10, gold dissolution can be relieved effectively by adding reduced iron powder into the alkali leaching. The rate of gold dissolution can be efficaciously reduced from 10.16% to 1.88% with only 0.5 g of iron powder added to the leach solution. But, the extraction rate of antimony did not vary obviously with the addition of the iron powder. The leaching efficiencies of gold obviously decreased with the increase of iron powder and reached 1.32% with 1 g of iron powder. With further increase in iron powder, the dissolution efficiency remained relatively steady. Based on the above results, the appropriate addition of iron powder was chosen to be 1/30 of the leaching ore amount. At this time, the antimony leaching rate was 97.35%, and the gold leaching rate was 1.32%.

It is indicated from Fig. 11 that the anodic current peak and polarization current density of sodium sulfide oxidation obviously decreased with increasing iron powder addition from 0.5 g to 3 g. As iron powder addition increased, the peak potential increased. The addition of iron ions increased the potential of sodium sulfide oxidation, thus avoiding the formation of sulfur and polysulfide.

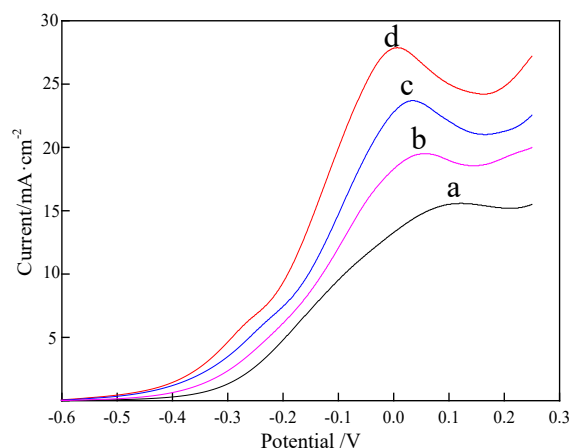


Fig. 11. Anodic polarization plots in solutions of reduced iron powder. (a) - 3 g, (b) - 2 g, (c) - 1 g, (d) - 0.5 g. Scanning rate 1.0 mV/s, concentration of Na<sub>2</sub>S 110 g/dm<sup>3</sup>, concentration of NaOH 20 g/dm<sup>3</sup>, and temperature 353.15 K



The standard electrode potential is an important criterion for determining the direction of redox reaction and judging the strength of redox agent (Kiviluoma et al., 2016). The standard electrode potential of the related reaction in the alkaline leaching is given in Table 2 (Speight, 2005), for the sake of discussing the effect of the reduced iron powder in the leaching solution in terms of electrochemistry.

Table 2. Standard electrode potential of related reactions

Electrode	Electrode reaction	Standard electrode potential $E^\circ/V$
$O_2/O^{2-}$	$O_2 + e^- = O^{2-}$ (11)	0.401
$S/S^{2-}$	$S + 2e^- = S^{2-}$ (12)	-0.476
$Fe^{2+}/Fe$	$Fe(OH)_2 + 2e^- = Fe + 2OH^-$ (13)	-0.877
$Fe^{3+}/Fe^{2+}$	$Fe(OH)_3 + e^- = Fe(OH)_2 + OH^-$ (14)	-0.56

Remarkably, the standard cell voltages of sodium sulfide oxidation are calculated as follows.

$$E_1^\circ = E^\circ(11) - E^\circ(12) = 0.877 \text{ V.} \quad (11)$$

When adding the reduced iron powder into the leaching solution, the iron powder will react with the oxygen in the solution into ferrous iron. The reacting standard cell voltage is  $E_2^\circ$ .

$$E_2^\circ = E^\circ(11) - E^\circ(13) = 1.278 \text{ V.} \quad (12)$$

Ferrous iron will continue to react with oxygen into ferric iron due to instability, the reacting standard cell voltage is  $E_3^\circ$ .

$$E_3^\circ = E^\circ(11) - E^\circ(14) = 0.961 \text{ V.} \quad (13)$$

It is deduced that  $E_2^\circ > E_3^\circ > E_1^\circ$  by comparing the cell voltages of the three reactions. Owing to adding the reduced iron powder to the leaching solution, the order of reaction in the solution is as follows: The oxidation of the reduced iron; the oxidation of  $Fe^{2+}$  into  $Fe^{3+}$ ; the oxidation of sodium sulfide. The oxidizing agent in the solution is consumed by adding the reduced iron powder. Thus, the oxidation decomposition of sodium sulfide is avoided effectively, and the stability of sodium sulfide is enhanced so as to prevent gold dissolution during the antimony leaching process.

#### 4. Conclusions

1) Mineralogical analysis of the ore revealed that the concentrates were gold contained stibnite concentrates which contain 28.41 g/Mg Au and 36.01% Sb. There mainly existed stibnite ( $Sb_2S_3$ ), arsenopyrite ( $FeAsS$ ), and quartz ( $SiO_2$ ) concentrates.

2) It can be summarized from anode polarization plots that the increasing of sodium sulfide concentration was beneficial to the oxidation rate of sodium sulfide. The increasing amount of alkali could avoid the oxidation decomposition of sodium sulfide within certain limits. But, an excessive amount of sodium hydroxide would lead to an increase in the oxidation rate of sodium sulfide. Temperature could promote the oxygenolysis rate of sodium sulfide. And, reduced iron powder could effectively prevent the decomposition of sodium sulfide and the dissolution of gold.

3) Based on the results of factorial experiments and anodic polarization plots, the optimum conditions for the extraction of antimony from gold containing stibnite concentrates were determined: the concentration of sodium sulfide and sodium hydroxide at 110 g/dm<sup>3</sup> and 20 g/dm<sup>3</sup>, ratio of iron powder to concentrate of 1:30, ratio of liquid to solid of 5:1, Agitation speed at 600 rad/min, reaction temperature of 353.15 K, reaction time of 3 h. Under these optimized conditions, high antimony recovery (97.35%) and low gold dissolution (1.32%) were achieved. The addition of iron ions enhanced the stability of sodium sulfide so as to prevent gold dissolution during the antimony leaching process.

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