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Research Article

Thermodynamic Behavior of Pyrite and Arsenopyrite in Preoxidation for Chlorination Leaching of Refractory Gold Concentrate

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Recently, preoxidation is an effective pretreatment method of refractory gold ore, which has been widely used due to the high desulfurization, arsenic removal, low environmental pollution, and rapid reaction rate. In this paper, we describe the thermodynamic considerations of preoxidation of pyrite and arsenopyrite, the main minerals of the refractory gold ore, and the effect of the pressure oxidation, one of the preoxidation processes on the chlorination leaching. The thermodynamic results indicated that arsenopyrite under acidic conditions is easier to oxidize compared to pyrite and the oxidation decomposition of pyrite and arsenopyrite-type gold ore can be considered mainly for pyrite. The experiment has shown that the arsenic removal rate was higher than the desulfurization rate; it is confirmed that the thermodynamic conclusion of the oxidation of pyrite and arsenopyrite was correct. Comparing the XRD, SEM, and EDX analyses of gold concentrate and pressurized oxidation residue, it can be seen that the surface of pressurized oxidation residue is a fine porous structure and the dense and durable structure of sulfide ore is mainly destroyed.

1. Introduction

In recent years, the amount of gold production all over the world increases continuously, the manageable gold ore resources which adapt to cyanidation have been depleted, and the refractory ore of low grade and impregnation deposit, containing harmful impurities such as sulphur, arsenic, and copper, become the main resources for the gold production. Generally, in the sulphur and arsenic-rich refractory gold ore, the gold leaching rate is very low because the ultrafine gold particle is wrapped with the pyrite and arsenopyrite; though the ore is finely grinded, gold particles are not exposed [1–3].

Also, during the leaching process, because arsenide reacts with cyanide and consumes the leaching agent and oxygen, it prevents gold leaching. In order to extract the gold from the refractory gold ore effectively, the oxidation pretreatment must be conducted prior to leaching to oxidize the sulfide-bearing gold particles and convert their physical and chemical properties [4, 5]. On the other hand, cyanide leaching, one of the main methods for gold production, has been used widely due to the high extraction rate and the low production cost, but nowadays, the requirements of environment protection and ecological balance become exacting day by day, and the cyanidation of gold extraction has been further limited [6–8].

Therefore, the pretreatment of refractory gold ore and noncyanidation technology without poison and pollution will be the focus of future research. The pretreatment of refractory gold ore includes roasting oxidation, bio-oxidation, chemical oxidation, and others [9–11]. The roasting oxidation is the traditional pretreatment method of the refractory gold ore and is the accomplished process, and the operation is simple, but the gold leaching rate is not high and the cost of waste gas treatment is expensive [12, 13]. Recently, the wet preoxidation such as the chemical oxidation and bio-oxidation is widely used because the leaching is effective, the productivity is high, the environmental pollution is low, and so on [14–17]. The preoxidation of pyrite and arsenopyrite is important for the pretreatment of the refractory gold ore, especially to illustrate the oxidation mechanism and the oxidation-reduction potential of sulphur and arsenic, and the effect of desulfitation and arsenic removal on the gold leaching ratio is significant for the treatment of the refractory gold ore containing pyrite and arsenopyrite mainly.

At the present, only chlorination leaching is available in practice for refractory gold concentrates [18–20]. The chlorination leaching method is mainly used in the processing of gold mud and gold refining and is also partially used in the leaching of the gold ore [21–23]. The advantage of chlorination leaching is that the reaction is fast, the cost is low, the process is simple, the pollution is little, and the treatment of waste water is easy.

Preoxidation of refractory gold concentrate studied so far is mostly for cyanide leaching, and there are few studies on preoxidation for chloride leaching, one of the kinds of noncyanidation leaching.

In this paper, we describe the thermodynamic considerations of preoxidation of pyrite and arsenopyrite, the main minerals of the refractory gold ore, and the effect of the pressure oxidation, one of the preoxidation on chlorination leaching.

2. Thermodynamics of Oxidation of Pyrite and Arsenopyrite

The potential-pH diagram is an important instrument of thermodynamic study in hydrometallurgy, and it represents the possibility and the stability of reaction that can occur in the given aqueous solution system as the function of potential, pH, and ion activity. Gold ores containing pyrite and arsenopyrite are commonly recognized as refractory ores, and especially, the gold leaching rate is very low, when these minerals exist together, because ultrafine gold particles are locked up in sulphur and arsenic mineral matrix [24, 25]. Although the thermodynamic study on the oxidation of pyrite and arsenopyrite was conducted, there are not abundant data representing the difference of oxidation-reduction potential and the effect of desulfurization and arsenic removal on the gold leaching in the pyrite-arsenopyrite-water system.

Therefore, in this paper, when the pyrite and arsenopyrite exist together, thermodynamic properties of the oxidation are observed by studying on the E-pH diagram of FeS_2-H_2O, FeAsS-H_2O, and FeS_2-FeAsS-H_2O systems.

The $\Delta_f G^0$ of FeS_2-H_2O and FeAsS-H_2O systems are listed in Table 1.

2.1. E-pH Diagram of the FeS_2-H_2O System.

Based on the calculation of oxidation-reduction potential E and pH, we can calculate $\triangle_r G^0$ of each reaction possible in the FeS_2-H_2O system at 298.15K and then obtain the E-pH diagram under the standard condition. Herein, the ionic concentration of the reaction system is set to 1.0 mol/L, and the activity coefficient is assumed to be 1; the potential-pH diagram of the 298.15 K FeS_2-H_2O system is shown in Figure 1.

It shows that elemental sulphur is stable at high acidity and the stable region is relatively narrow during the oxidation of pyrite. Once elemental sulphur is produced, the coating is formed on the surface of gold and has a negative effect on gold leaching so that the generation of elemental sulphur if possible during the pretreatment should be avoided. Also, although pyrite is a refractory mineral in the stable region of water, it can be oxidized by the oxidant such as O_2 at high oxidation potential.

When the potential or pH increases, sulphur in pyrite can be oxidized to stable HSO^{4-} and $SO_4{}^{2-}$ type. Meanwhile, iron can be oxidized to Fe^{3+} and Fe^{2+} in the acidity condition, and when pH increases, it is precipitated as $Fe(OH)_3$. $Fe(OH)_3$ is an insoluble matter, but gold can be exposed in the oxidation decomposition of pyrite, and so, it does not affect the gold leaching process. The result of Figure 1 shows that thermodynamically acid oxidation is favorable compared to alkaline conditions.

2.2. E-pH Diagram of the FeAsS-H_2O System.

As mentioned above, when it is assumed that the ion concentration is 1.0 mol/L and ion activity coefficient is 1, the E-pH diagram of FeAsS-H_2O system at 298.15K is shown in Figure 2.

The stable oxidation number of arsenic is +5, +3, 0, and −3, but it can exist only as As^{5+} and As^{3+} in the aqueous solution. Figure 2 shows that arsenic exists as arsenious acid and arsenite in the low oxidation-reduction potential, and also, arsenic exists as arsenic acid and arsenate in the high potential. The element arsenic has some stable region but their oxidation-reduction potential is very low, so it becomes unstable under the general process condition, and it is continuously oxidized.

H_3AsO_3 has a large stable region. Thus, it is possible to extract As_2O_3 from the solution, and it is necessary to maintain the high acidity and potential to avoid the negative effect of its hydrate on gold leaching.

Also, Figure 2 shows that FeAsS is oxidized by the oxidant such as O_2 when oxidation potential is increased in the stable region of water. The oxidation potential of FeAsS is not higher than that of the pyrite. The stable region of the element sulphur in the FeAsS-H_2O system is larger than that in the FeS_2-H_2O system. Due to the kinetic factor, the formed sulphur is difficult to be oxidized continuously, so it negatively affects the gold leaching process. Thus, in the low

Table 1: Values of the $\Delta_f G^0$ FeS_2-H_2O system and FeAsS-H_2O system at 298.15K.

Component	$\Delta_f G^0$ (kJ/mol)	Component	$\Delta_f G^0$ (kJ/mol)	Component	$\Delta_f G^0$ (kJ/mol)
H_2	0	Fe	0	H_3AsO_3	−639.8
H^-	0	Fe^{2+}	−78.9	$H_2AsO_3^-$	−587.1
O_2	0	Fe^{3+}	−4.7	$HAsO_3^{2-}$	−517.2
H_2O	−237.1	$Fe(OH)_2$	−479.3	AsH_3	168.9
S	0	$Fe(OH)_3$	−696.5	FeS_2	−166.9
HSO_4^-	−755.9	As	0	FeS	−100.4
SO_4^{2-}	−744.5	H_3AsO_4	−766.0	FeAsS	−109.6
H_2S	−26.9	$H_2AsO_4^-$	−753.2	$FeAsO_4$	−768.6
HS^-	12.1	$HAsO_4^{2-}$	−714.7		
S^{2-}	85.8	AsO_4^{3-}	−648.4		

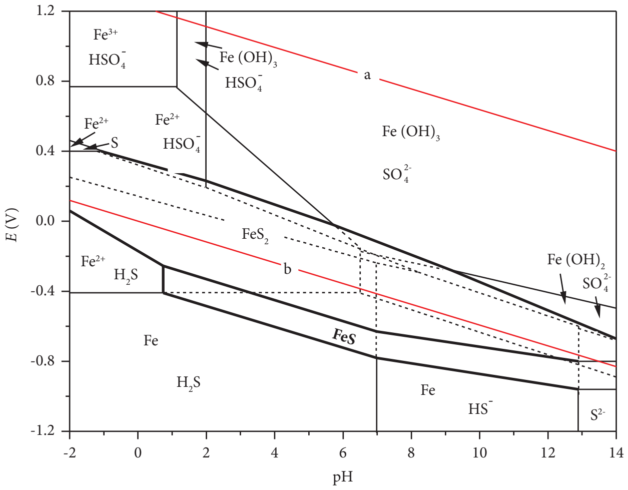

Figure 1: E-pH diagram of the FeS_2-H_2O system at 298.15K (p = 1.0).

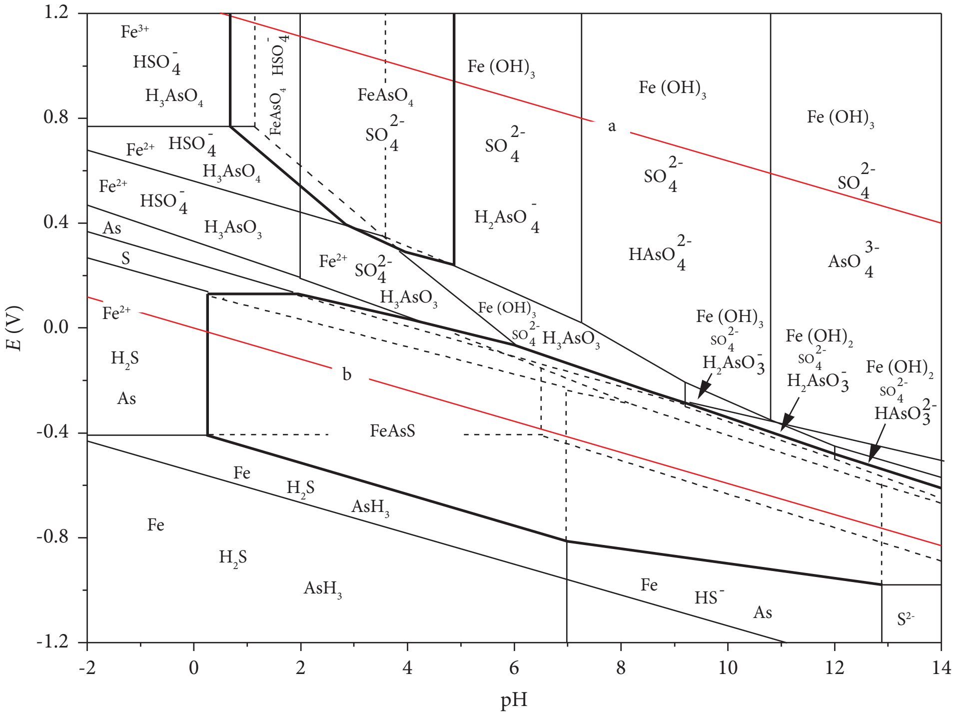

Figure 2: E-pH diagram of the FeAsS-H_2O system at 298.15K (p = 1.0).

acidity and high potential as possible, the sulphur should be converted to SO_4^{2-} and HSO_4^-.

In the stable region of water, the stable one of $FeAsO_4$ is relatively large at $pH > 0.12$, and if potential gets high, its precipitation acidity is increased. Thus, the oxidation pretreatment should provide suitable acidity to prevent the discharge of arsenic-containing waste water. At $pH > 5.3$, $FeAsO_4$ is converted to $Fe(OH)_3$ and arsenic is eluted at the same time.

2.3. *E-pH Diagram of the FeS_2-FeAsS-H_2O System.*

The *E*-pH diagram of the FeS_2-FeAsS-H_2O system can be described from combination of the diagrams of FeS_2-H_2O system and FeAsS-H_2O system, and also, some expanded diagram is shown in Figure 3 in the acidic region.

Figure 3 shows that the stable region of FeS_2 is larger than FeAsS and the stable region of FeS_2 includes the stable region of FeAsS. It means that arsenopyrite is easily oxidized than pyrite. Thus, the oxidation pretreatment of the gold ore including pyrite and arsenopyrite can be mainly considered as the pyrite. Also, as Figure 3 shows, $FeAsO_4$ is stable in the region consisting of As^{5+} and Fe^{3+}. In other words, the formed As^{5+} and Fe^{3+} in the oxidation process react with each other and are precipitated as $FeAsO_4$. Due to fact that the solubility product of $FeAsO_4$ at 298.15K is 5.7×10^{-21}, so the elimination of arsenic from the solution containing As is possible. Therefore, the reasonable control of the potential and acidity during pretreatment to produce $FeAsO_4$ can prevent the effluence of solution containing arsenic.

Based on the *E*-pH diagram, the possibility of the oxidation pretreatment of the refractory gold ore containing high sulphur and arsenic and the process condition are established thermodynamically, so the pretreatment for gold leaching and the environmental protection are guaranteed theoretically.

3. The Effect of Preoxidation on Gold Chlorination Leaching

Based on thermodynamic studies of the oxidation process of pyrite and arsenopyrite, the effect of the actual oxidation degree of pyrite and arsenopyrite on gold chlorination leaching was examined experimentally. The wet preoxidation of pyrite and arsenopyrite-type gold ore includes pressure oxidation (acidic and alkaline pressure oxidation) and normal pressure oxidation (ARSENO, NITROX, REDOX, COAL, and mechanical activation leaching).

Pressure oxidation is one of the promising pretreatment technologies that have developed rapidly in the treatment of refractory gold ores. The advantages of pressure oxidation are that the reaction rate is fast, environmental pollution is low, decomposition of sulfide minerals is perfect, mass reduction of raw materials during oxidation is large, and the gold leaching rate is high. Acid pressure oxidation is applied to the acidic minerals such as pyrite and arsenopyrite. Based on the thermodynamic study of the oxidation of pyrite and arsenopyrite, the reactions that can occur during wet oxidation of pyrite and arsenopyrite under acidic conditions are as follows:

$$\begin{aligned}
2FeS_2 + 7O_2 + 2H_2O &= 2FeSO_4 + 2H_2SO_4 \\
FeS_2 + 2O_2 &= FeSO_4 + S \\
2FeAsS + \frac{13}{2O_2} + 3H_2O &= 2FeSO_4 + 2H_3AsO_4 \\
2FeSO_{4+} + \frac{1}{2O_2} + H_2SO_4 &= Fe_2(SO_4)_3 + H_2O \\
FeS_2 + 7\,Fe_2(SO_4)_3 + 8H_2O &= 15\,FeSO_4 + 8H_2SO_4 \\
2FeAsS + 13Fe_2(SO_4)_3 + 16H_2O &= 28FeSO_4 + 2H_3AsO_4 + 13H_2SO_4 \\
2H_3AsO_4 + Fe_2(SO_4)_3 &= 2FeAsO_4\downarrow + 3H_2SO_4
\end{aligned} \qquad (1)$$

Thus, pyrite and arsenopyrite are oxidized, leached into sulfate or precipitated as ferric arsenate, which is favorable for gold leaching by the liberation or exposure of the gold that was wrapped. The pressure oxidation process is a complex multiphase reaction involving gaseous O_2, and desulfurization and the arsenic removal rate play important roles in gold leaching.

The experiments of pressure oxidation and chlorination leaching with pyrite and arsenopyrite-type gold concentrate were performed to examine the correctness of

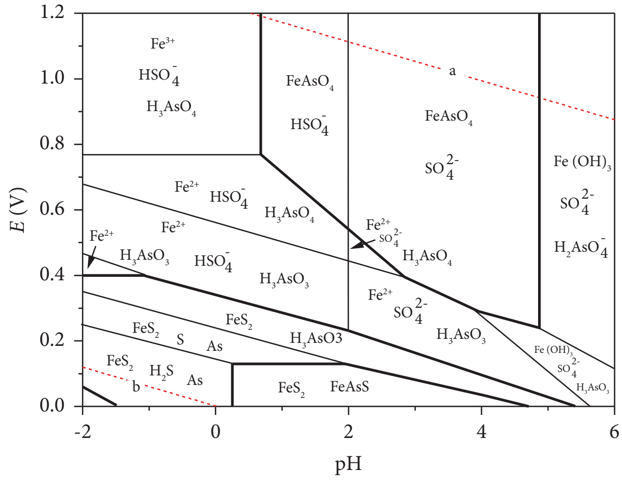

Figure 3: E-pH diagram of the FeS_2-FeAsS-H_2O system at 298.15K (p = 1.0).

thermodynamic results of oxidation of pyrite and arsenopyrite. The effect of the desulfurization and derate on gold leaching was investigated by oxidizing pyrite and arsenopyrite gold ore under acidic pressure, determining the desulfurization rate and derate, and again after chlorination leaching.

3.1. Experimental Materials and Methods. The main chemical and mineral compositions of pyrite and arsenopyrite-type gold concentrate used in the experiments are listed in Tables 2 and 3, and XRD patterns, SEM, and EDX images are shown in Figures 4 and 5, respectively.

As shown in Tables 2 and 3, the main metal minerals in the concentrate are pyrite and arsenopyrite, and gold in the concentrate is mainly dispersed in these minerals as the form of microscopic or submicroscopic particle.

This gold concentrate is a typical refractory gold concentrate with the gold leaching rate of 34.63% and 42.65% in direct cyanidation and chlorination, respectively.

The effect of the desulfurization and arsenic removal in pressure oxidation pretreatment on gold leaching in chlorination is investigated experimentally. The experiments were carried out by measuring the desulfurization rate and the arsenic removal rate according to time during pressure oxidation of the gold concentrate and followed by measuring the gold leaching rate according to the desulfurization rate and the arsenic removal rate during chlorination of the residue.

First, gold concentrate was oxidized under the suitable conditions. The pressure oxidation pretreatment was performed at a temperature of 180°C, oxygen partial pressure of 0.8 MPa, concentrate size of 0.061 mm~0.075 mm (240 mesh-200 mesh), initial sulfuric acid concentration of 60 g/L, liquid/solid ratio of 4 : 1, and stirring speed of 600 rpm to measure desulfurization and arsenic removal rates.

Then, the pressure oxidized gold concentrate was leached in the chloride solution. The chlorination leaching was carried out at pH 4, sodium hypochlorite concentration, 75 g/L NaCl concentration, 30°C leaching temperature, 3 : 1 liquid–solid ratio, and 120 min reaction time to measure gold leaching.

3.2. Effect of Desulfurization and Arsenic Removal Rate on Gold Leaching. The desulfurization rate, arsenic removal rate, and gold leaching rate according to pressure oxidation time are shown in Figure 6.

As shown in Figure 6, the desulfurization and arsenic removal rate were 93.8% and 95.9%, respectively, and after pretreatment, the gold leaching rate was 96.5%. After the pressure oxidation pretreatment of gold concentrate, pyrite and arsenopyrite decomposed almost entirely, and the arsenic removal rate was higher than the desulfurization rate, and this shows that the experimental result is consistent with the thermodynamic result.

The XRD and SEM analysis results of the pressure oxidation residue of gold concentrate under given conditions are shown in Figures 7 and 8, respectively.

XRD analysis shows that the pretreated gold concentrate contains only a small amount of FeS_2 but does not contain other sulfide minerals such as FeAsS. That is, this confirms the thermodynamic result that the oxidation of FeAsS proceeds better than FeS_2. This suggests that the effectiveness of pressure oxidation pretreatment of pyrite and arsenopyrite-type gold concentrates can only be evaluated by desulfurization, and the effect on gold leaching after pressure oxidation can only be considered by desulfurization.

Comparing the SEM diagrams of gold concentrate before and after pretreatment, the surface of after-pretreatment concentrate has fine porous structure, and the dense and stable structure of sulfide mineral is mainly destroyed so that gold particles are exposed and leaching reactivity is

TABLE 2: The main chemical composition of gold concentrate.

Chemical composition	Au	Ag	S	Fe	As	Zn	Cu	Pb	SiO_2	Al_2O_3
w (%)	17.9 g/t	25.0 g/t	28.07	27.23	3.2	0.77	0.04	0.14	26.33	4.52

TABLE 3: The major mineral compositions of gold concentrate.

Mineral compositions	Pyrite	Arsenopyrite	Blende	Galenite	Quartz
w (%)	49.17	7.06	1.15	0.16	26.33

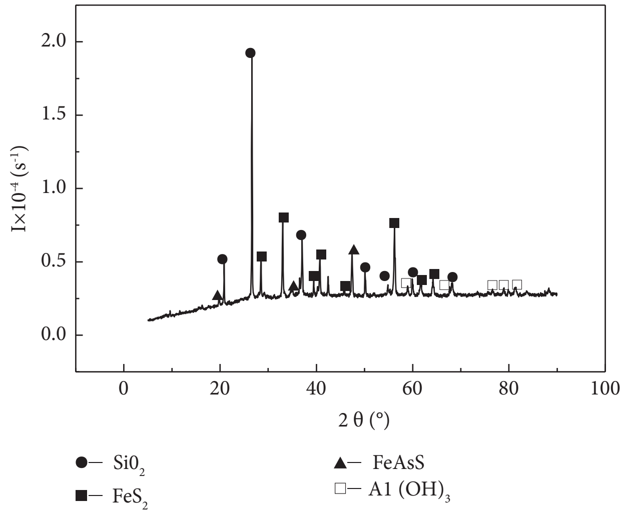

FIGURE 4: XRD patterns of the gold concentrate.

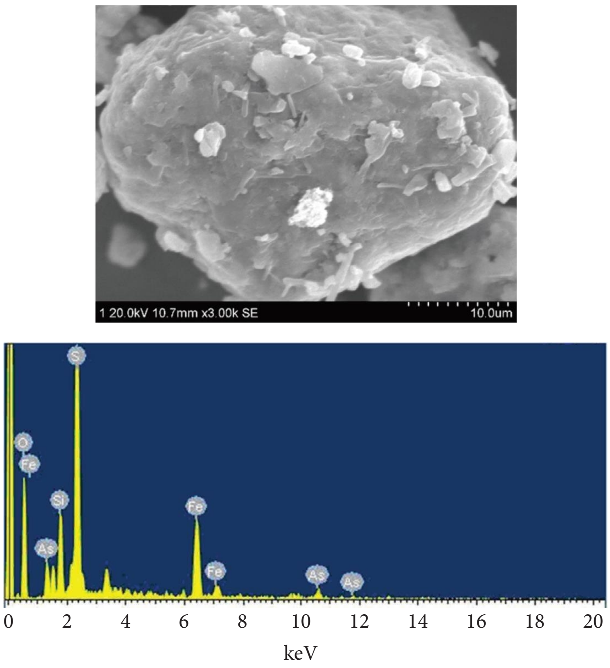

FIGURE 5: SEM and EDX images of the gold concentrate.

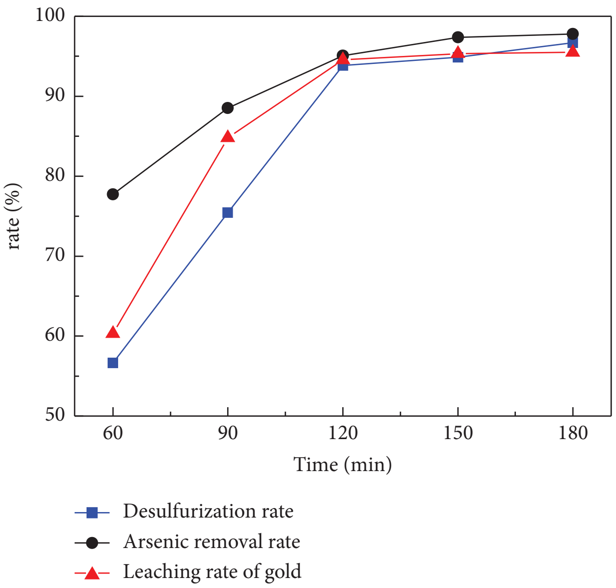

Figure 6: The desulfurization rate, arsenic removal rate, and gold leaching rate according to pressure oxidation time.

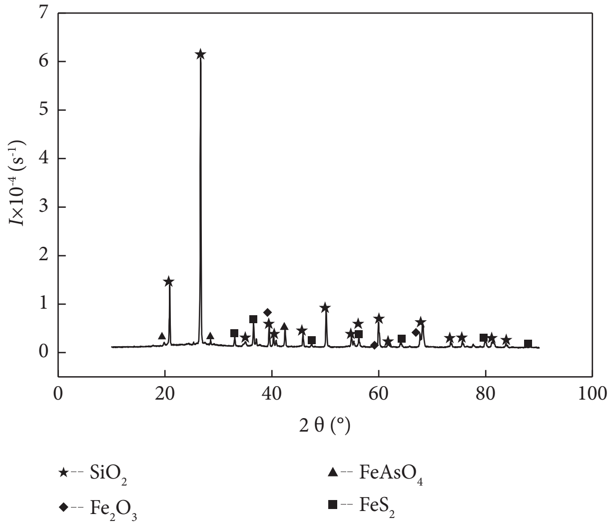

Figure 7: XRD pattern of pressure oxidized gold concentrate.

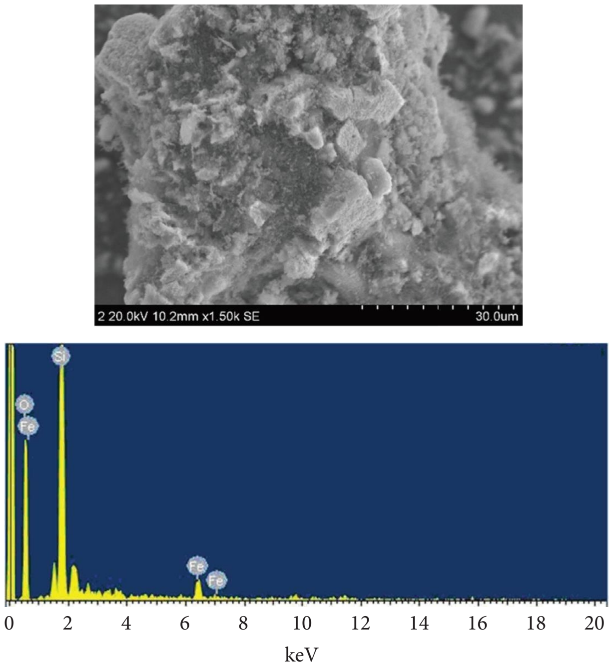

Figure 8: SEM and EDX images of pressure oxidized gold concentrate.

improved. That is, sulfide minerals, including FeS_2 and FeAsS, undergo different degrees of physical and chemical changes during the pressure oxidation process to create favorable conditions for gold leaching.

4. Conclusions

In the present study, the thermodynamic behavior of pyrite and arsenopyrite in preoxidation for chlorination leaching of refractory gold concentrate was carried out.

First, by studying the thermodynamic behavior of their wet oxidation process with the E-pH diagram of the pyritic-arsenopyrite-H_2O system, arsenopyrite under acidic conditions are easy for wet oxidation compared to pyrite, and the preoxidation process of pyrite and arsenopyrite-type gold ore can be considered as the basis of pyrite.

Second, based on the thermodynamic study of the wet oxidation process of pyrite and arsenopyrite, the actually pyrite and arsenopyrite oxidation degree was experimentally observed to influence the gold leaching rate of gold concentrate, which confirmed that the arsenic removal rate is higher than the desulfurization rate, confirming that the thermodynamic result of the wet oxidation process of pyrite and arsenopyrite was correct.

Third, in the preoxidation of pyrite and arsenopyrite-type gold concentrate, the main factor is the desulfurization rate and a certain degree of desulfurization can improve the gold leaching efficiency sufficiently.

Data Availability

The data used to support the findings of the study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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