

Electrochemical Pretreatment for Improved Leaching Rate of Refractory gold Ore with Large Cyanide Consumption

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Abstract

Original Research Article

In this paper, the combined treatment of sodium peroxide addition and electro-oxidation for the pretreatment of refractory gold concentrate improved gold extraction. The conventional gold extraction rate was 17% with 40 kg/t of sodium cyanide without pretreatment, and the refractory gold concentrate with little cyanide concentration at 2h cyanide leaching time. Pretreatment and cyanide leaching were carried out at normal temperature and atmospheric pressure in alkaline media, in a single leaching bath, and a simple method without conventional separation operation. After pretreatment, the gold extraction increased from 17% to 88%.

Keywords: refractory gold concentrates, pretreatment, electro-oxidation, peroxide.

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Introduction

As the depletion of the deposit from which gold can be easily leached, recovery of gold from refractory ores has been widely investigated. The most common pretreatment techniques of refractory gold ores include roasting, high pressure oxidation, biological oxidation and chemical pretreatment. [1, 3] The use of roasting, high pressure oxidation and biological oxidation has been an effective method of pretreatment, but the high investment cost and the strict process control have led to the development of alkaline pretreatment. [2, 5] Sandra L. Mesa Espitia et al. [2] used hydroxyl ion for alkaline pretreatment of arsenopyrite. The gold ore used in the experiments include mainly arsenopyrite, and in addition pyrite, sphalerite, silver-bearing tetrahedrite, boulangerite, galenite, a small quantity of chalcopyrite, heteromorphite, and pyrrhotite. The gold leaching rate of the conventional cyanide leaching method without pretreatment was 23%, and the gold leaching rate after alkaline pretreatment was 81%. Richmond K. Asamoah et al. [1] investigated the alkaline cyanide leaching of refractory gold concentrates and

bio-oxidised products and H. Kasaini et al. [4] investigated the effect of alkaline pretreatment of jarosite minerals. The pretreatment was treated with $\text{Ca}(\text{OH})_2$ and heated at 90°C for 2 h to dissolve silver-bearing minerals (Ag , $\text{PbFe}_3(\text{SO}_4)_2(\text{OH})_6$). The leaching rate of gold and silver without alkaline pretreatment was 41% Au and 25% Ag, and the leaching rate of gold and silver was improved to 69% and 90% after pretreatment. Nan, Junfang et al. [5] investigated the pretreatment of refractory gold ore using Na_2S - NaOH . Pretreatment at 85.66 g/L Na_2S , 45.5 g/L NaOH and 90°C temperature increased gold extraction. In another study, Na_2S - NaOH was used to increase the pretreatment gold leaching rate from 49% to 78% [6]. Oktay Celep et al. [7] investigated alkaline pretreatment using sodium hydroxide to improve the gold and silver recovery of refractory gold silver ores containing antimony minerals such as antimonite, andorite, and zinkenite. The removal of antimony by alkaline pretreatment was more than 77.5%, while the silver leaching rate from cyanide leaching was increased from 18.7% to 90% and the gold leaching rate from 49.3% to 85.4%.



MENG Yu-qun et al. [8] studied the alkaline pretreatment-cyanidation leaching process for refractory gold concentrates under normal and normal pressure conditions. This method can provide high gold leaching rates by oxidation of arsenic and sulfur during alkaline pretreatment in a grinding-leaching column mill, and the cyanide consumption is reduced due to the new self-leaching of gold. The pretreatment was carried out in a column mill with 12 kg/t NaOH in a continuous flow with a particle size of -35 mm 95.5%. Then, the alkaline leaching was carried out in a stirred tank at 40% pulp concentration, 9.5-13.5 °C, and 105 Pa pressure for 48 h at 108 kg/t NaOH. The oxidation rate of As in pretreatment was 94.9% and 47.6% S, and the gold leaching rate was improved from 9.2% before pretreatment to 94.2%. Dowd, Peter A et al. [9] proposed a model for pyrite oxidation in the direct cyanidation of refractory gold ores, and Sun, Chunbao et al. [10] reported an improvement of gold recovery from 34.8% to 57.08% using 15 kg/t of disyanamide as gold leaching agent. Ehsan Bidari et al. [11] studied the alkaline pre-oxidation of pyrite in the Karin-type gold deposit of Zarshuran, Iran.

Another research is being carried out to improve gold extraction by adding hydrogen peroxide and heavy metal ions to cyanide leaching process. Heavy metal ions such as Pb^{2+} , Bi^{3+} , Tl^+ , Hg^{2+} and Ag^+ accelerate the anodic process of gold dissolution, and peroxides such as hydrogen peroxide promote the cathod dissolution process, thus improving gold leaching. [12] Terzi et al. [13] reported the use of hydrogen peroxide in thiourea leaching and the use of hydrogen peroxide in cyanide leaching to improve the leaching efficiency. E.Elorza-Rodri'guez et al. [14] studied the pretreatment using oxygen/ozone before cyanidation, and Qingcui Li et al. [22] reported the pretreatment of refractory gold ore with high sulfur and arsenic contents using ozone and ferrous sulfate. LI Qian et al. [15] investigated the effect of mercury ions on cyanide leaching of gold. In the absence of mercury ions and hydrogen peroxide, the gold leaching rate was 45.68% at 24 h of leaching. After 4 h of leaching with mercury ions under co-enhanced conditions, the leaching efficiency increased to 72.33%.

Many researchers have investigated the use of ammonia for the treatment of copper-bearing gold ores to reduce cyanide consumption and improve gold extraction. In cyanidation, copper minerals are reported to reduce gold leaching while 1% copper consumes about 30 kg/t of sodium cyanide [17]. A. D. BAS et al. [16] investigated direct cyanidation, ammonia pretreatment, and ammonia cyanidation of copper-bearing gold ores. Xie, Boyi et al. [18] investigated the use of ammonium bifluoride and sulfuric acid solutions for the pretreatment of refractory gold concentrate, and Esmail Mohammadi et al. [24] investigated the effect of ammonia on thiosulfate leaching.

In addition, the improvement of gold extraction by electro-oxidation of cyanide leaching is also being investigated. Evelien Martens et al. [19] studied the electrokinetics of gold leaching and Lundström, Mari et al. [20] improved gold leaching by using electrodeposition material redox method for the treatment of refractory tellurium gold ore. L.M. Abrantes et al. [21] used the electro-oxidation for the pretreatment of gold ore.

Thus, the alkaline pretreatment method has high NaOH consumption, high process temperature, and solid-liquid separation is necessary. In addition, the oxidizing agents containing hydrogen peroxide were used as co-additives in cyanide leaching process, and the electrooxidation process was partially investigated.

In this study, the combination of alkaline pretreatment and electrolytic oxidation treatment under normal temperature and pressure conditions was investigated. After stirring with sodium peroxide and ammonium salt in the pretreatment, the electro-oxidation pretreatment was carried out sequentially to investigate the method to electrodeposit impurities at the cathode and improve gold leaching rate.

Materials and methods

Materials

The gold ore sample was obtained from the West Coast Mine of the Democratic People's Republic of Korea with high sulfur and lead content

as a flotation concentrate. The gold concentrate sample purchased has a particle size of 50µm 80%. Chemical composition analysis of the ore was performed using an atomic absorption spectrometer (Z-5000 Hitach, Japan) and an inductively coupled plasma atomic emission spectrometer (iCAP 7000 ThermoFisher Scientific, USA). The mineralogical characterization was carried out with X-ray fluorescence (ZSX PrimsIII + Rigaku, Japan) and X-ray diffraction (D8 ADVANCE Bruker, Germany).

The electrode material used in the experiments was stainless steel.

The experiment used industrial water as in the workshop conditions. The experimental values are the average of three replicates.

The main chemical composition of the gold concentrate used in the experiments are given in Table 1.

Table 1 The main chemical composition of the gold concentrate, %

element	Au, g/t	Ag, g/t	Pb	S	Fe	Zn	Cu	As	SiO ₂	Al ₂ O ₃
content, %	35	263	7.38	16.3	20.85	4.47	1.68	0.67	35.4	7.16

The gold grade and S, Pb content reached 35g/t, 16.3%, 7.38%, respectively, indicating high content of valuable metals.

The mineral composition revealed by X-ray diffraction (XRD) (figure 1).

The main sulfides of gold concentrates are pyrite, galena, sphalerite, arsenopyrite, chalcocopyrite and a small quantity of pyrrhotite, and gangue contains quartz, muscovite and microcline.

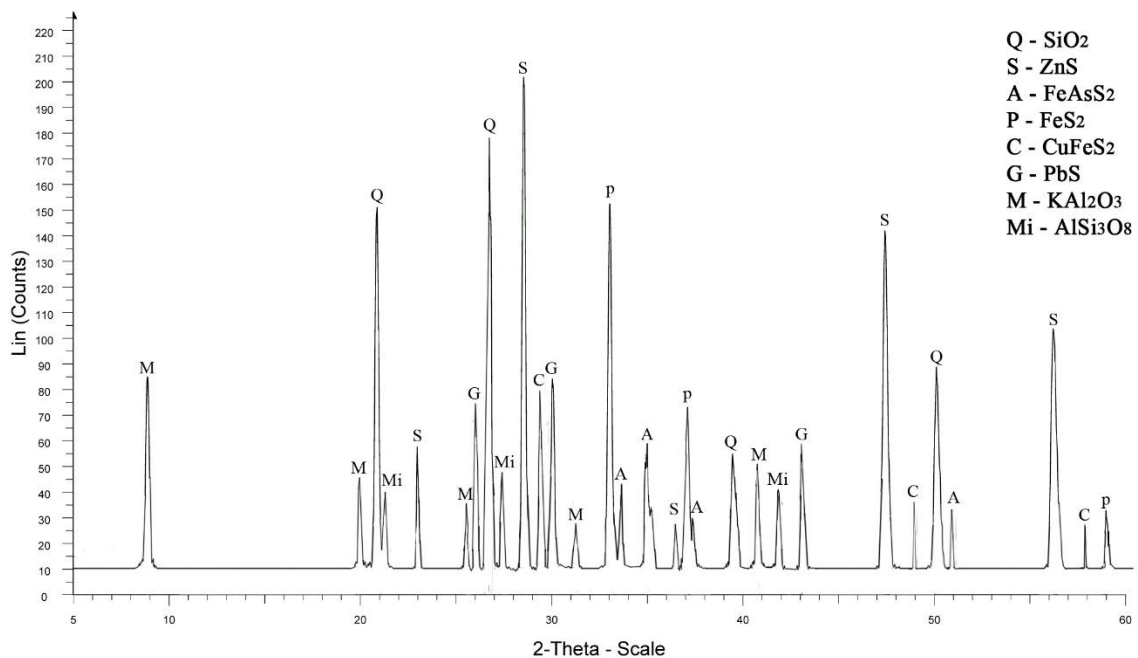


Figure 1 XRD pattern of gold concentrate

Methods

In the experiments, the effects of adding sodium peroxide, ammonium bifluoride and sodium

carbonate in the pretreatment and the effect of the electro-oxidation pretreatment were investigated. The experimental process diagram is shown in Figure 2.

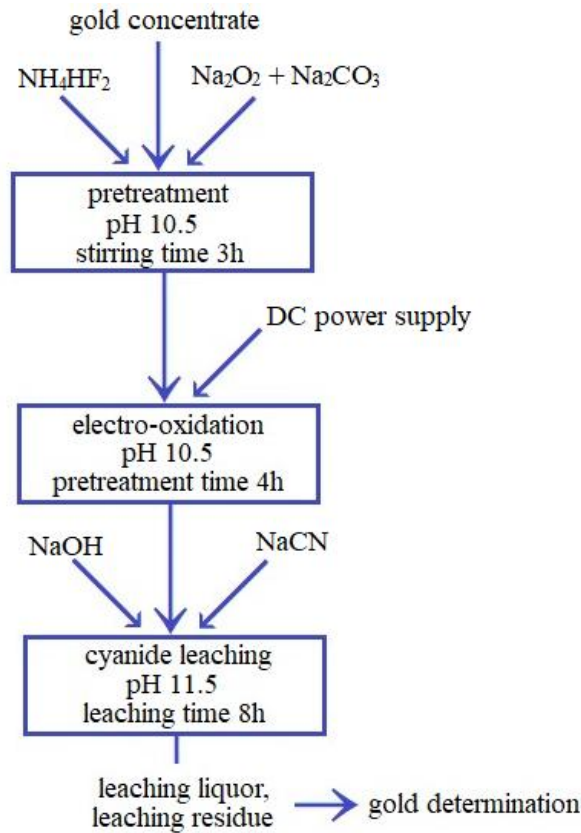


Figure 2 Schematic of gold leaching process with pretreatment.

The leaching experiments were carried out in a 1 L plastic stirrer using a mechanical stirrer (JJ-1A, China). The plastic stirrer was equipped with two stainless steel cathodes and four anodes. The dc

power supply was used as a rectifier transformer (WYJ 40A 15V DC REGULATED POWER SUPPLY, China).

The experimental stirrer is shown in Figure 3.

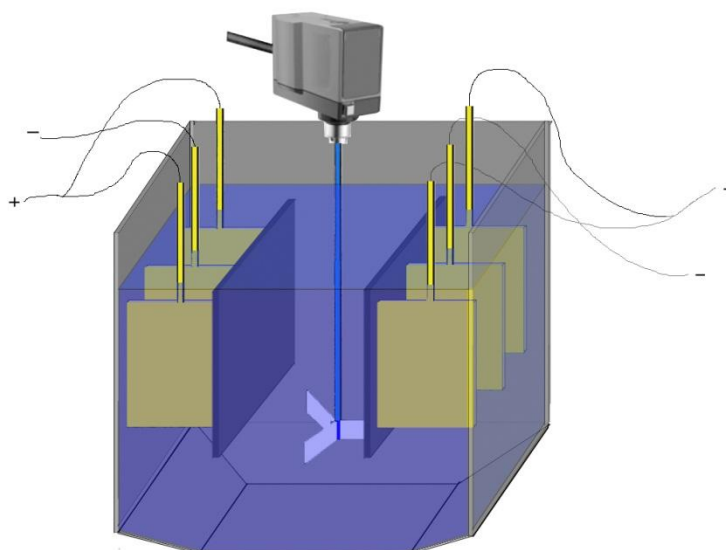


Figure 3 Experimental stirrer

The leaching experiment stirrer was equipped with 300 g concentrate sample and 900 mL of industrial water to give a liquid-solid ratio of 3:1. Add a certain quantity of sodium peroxide, ammonium bifluoride (NH_4HF_2), sodium carbonate, stir for 3~5 h and then apply a DC power supply to the plate without solid-liquid separation. The condition is 10 V of voltage and 100 A/m^2 of cathodic current density. Electro-oxidation treatment shall be done for 3~5 hours before power-up is stopped and stirring continues. The pH of the pulp is adjusted to 10.5, 40 kg/t of sodium cyanide is added and cyanide leaching is carried out for 2~24 h.

Chemical composition analysis of the pretreated liquor, cyanide leachate and leach residue was

carried out using Atomic Absorption Spectrometry and Inductively Coupled Plasma Atomic Emission Spectrometry, and cyanide concentration analysis of the leachate was performed using silver nitrate titration.

Result and discussion

General cyanide leaching without pretreatment

The results of the gold concentrate samples examined by the conventional cyanidation are given in Table 2. The experiments were carried out by varying the NaCN dosage from 5 kg/t to 40 kg/t at a particle size of 50 mm 80%, liquid-solid ratio of 3:1, initial pH of the pulp = 10 and leaching time of 30 h.

Table 2 Results of cyanide leaching without pretreatment

quantity of added cyanide, kg/t	gold content in cyanide residue, g/t	gold leaching rate, %
5	33.8	3.4
10	33.8	3.4
15	29.3	16.3
20	29.2	16.6

25	29.2	16.6
30	28.9	17.4
35	28.9	17.4
40	28.9	17.4

As shown in Table 2, the conventional method, even with high cyanide addition, has a gold extraction rate of less than 20%. Furthermore, the cyanide concentration is zero from the leaching time of 2h and the cyanide concentration is zero from the

leaching time of 2 h or more at the higher sodium cyanide dosage of 40 kg/t. The results of several components analysis of the leachate are given in Table 3.

Table 3 Variation of the concentration of the components with leaching time

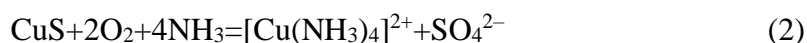
time, h	1	2	3	5
Cyanide content, %	0.05	0.0	0.0	0.0
Fe, mg/L	574.5	722.9	825.3	1056.6
Cu, mg/L	132.6	157.5	186.4	203.5
Pb, mg/L	160.9	372.5	475.0	676.4

As shown in Table 3, the cyanide concentration reaches zero above the leaching time of 2 h, and the Fe, Cu and Pb contents increase with time. This could be attributed to the high S, Fe, Pb, Cu, etc. contents of the concentrate samples, which increased oxygen and cyanide consumption and decreased gold extraction. The cathodic electrodepositon material of the pulp electrolysis process after cyanide leaching has 49.5% Fe, 38.9% Pb, 7.2% Zn, 2.4% Cu, and the quantity of electrodepositon material is 1.7g. Since the quantity of cathodic electrolytic slime treated with 300g concentrate is 1.7g, the total electrolytic slime content is very high, corresponding to 5.7kg for 1t concentrate. This can be attributed to the high dissolution of Fe and other impurities in the leachate, which increases the cyanide consumption

and the quantity of electrodepositon material.

Pretreatment with Peroxide

In this experiment, we investigated the improvement of gold extraction by using alkaline pretreatment with peroxide, ammonium salt and sodium carbonate. In the experiments, ammonium salt was added to improve the negative effect of copper during cyanide leaching. During cyanide of gold, leaching of copper minerals not only consumes a lot of cyanide and consumed oxygen, but also reduces the gold leaching rate, severely affecting gold leaching. Ammonia in cyanide of copper-bearing gold ores can suppress the negative effect of copper on cyanide [17, 18, 25].



In the leaching solution, NH_3 and Cu^{2+} form the copper ammonia complex ion $\text{Cu}(\text{NH}_3)_4^{2+}$, which lowers the ability of Cu^{2+} to bind cyanide and to some extent prevents the consumption of cyanide due to Cu^{2+} . In the leachate, the copper cyanide ions gradually displace to the cuprammonium complex ion form. The addition of NH_3 improves gold extraction by increasing free cyanide in solution by substituting the cyanide in the metal complex entirely or partially. In our experiments, 5 kg/t ammonium bifluoride was added based on the research and empirical data [17]. In general, peroxide is commonly used as a co-leaching agent in cyanide leaching processes [12, 13, 14]. In the experiments, the use of peroxide as a pre-treatment process was investigated to improve the pretreatment efficiency and improve the cyanide leaching process. The

results of the experiments with hydrogen peroxide, calcium peroxide and sodium peroxide are shown in Figure 4.

The experimental conditions were particle size $>50\mu\text{m}$ 80%, liquid solid ratio of 3:1 and ammonium bifluoride of 5 kg/t, and the addition quantity of sodium peroxide was 3 kg/t and the pretreatment time was 3 h. After pretreatment, the concentration was leached for 12 h by the addition of sodium cyanide (40 kg/t) without solid solution separation.

As shown in Figure 4, the use of peroxide in the pretreatment improved the gold leaching rate. Hydrogen peroxide did not show significant effect due to its high decomposition rate but the effect of sodium peroxide was higher.

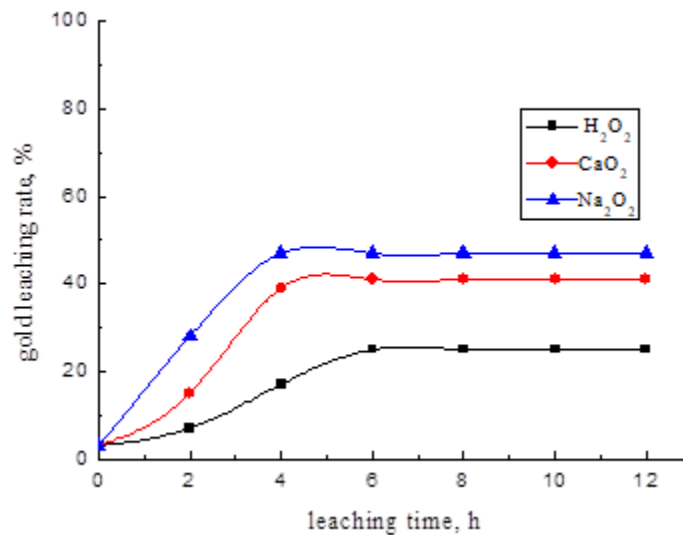


Figure 4 Effect of oxidants on gold leaching rate

The free energy profiles of the reactions of sodium peroxide and sulfides are shown in Figure 5. It can be seen from Figure 5 that the decomposition reaction of sulfide is possible. As can be seen from equation (4) to equation (6), it is believed that sodium

hydroxide is produced in the reaction products, which can have an effect on the alkaline pretreatment. It can also be seen that sodium peroxide improved gold leaching by partially exposing gold surrounded by sulfides during the pretreatment.



In the experiment, a comparison experiment was carried out by mixing various additives with sodium peroxide during the pretreatment. The effect of mixing was investigated by adding 5 kg/t ammonium

bifluoride in the pretreatment and adding additives in the same ratio as in Table 4. The leaching conditions after pretreatment were the same as above.

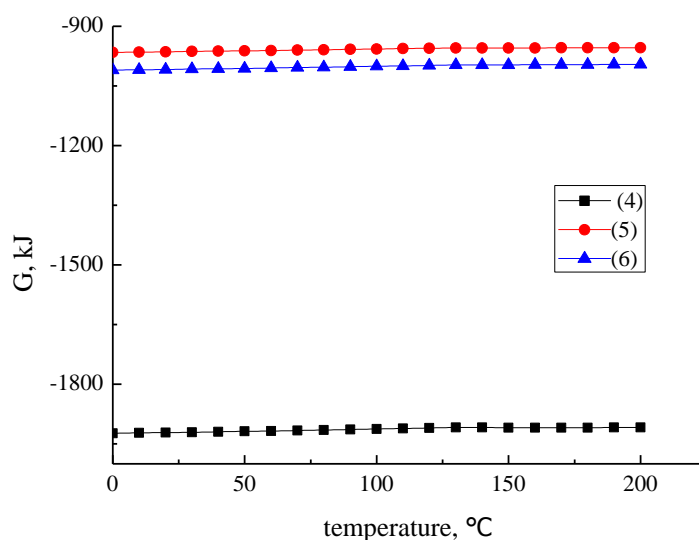


Figure 5 Free energy change curve of reaction.

Table 4 Effect of various additives

Additives	Quantity of additives, kg/t	Gold leaching rate, %
NaOH	5	24
Na ₂ CO ₃	5	23.1
Na ₂ O ₂ - NaOH	3:5	47
Na ₂ O ₂ - Ca(OH) ₂	3:5	46
Na ₂ O ₂ - Na ₂ CO ₃	3:5	76
NaOH - Na ₂ CO ₃	3:5	52
Ca(OH) ₂ - Na ₂ CO ₃	3:5	39

As shown in Table 4, the pretreatment was the most effective in the combination of ammonium bifluoride, sodium peroxide and sodium carbonate. The changes in the concentrations of NaCN, Fe, Cu and Pb with leaching time

after pretreatment with the mixture of ammonium bifluoride, sodium peroxide and sodium carbonate are given in Table 5.

Table 5 Variation of concentration of the components with pretreatment time

time, h	1	2	3	4	5
NaCN, %	0.09	0.08	0.07	0.05	0.02
Fe, mg/L	174.5	322.9	425.3	556.6	667.3
Cu, mg/L	132.6	197.5	216.4	253.6	314.2
Pb, mg/L	60.9	172.5	175.0	176.4	176.8

As shown in Table 5, after pretreatment with three mixed additives, the cyanide concentration remained low after 3 h of leaching. Comparing Table 5 and Table 3, it can be seen that the concentration of Fe, Cu and Pb in the solution is lower. The free energy profiles of several reactions with the addition of

sodium peroxide and sodium carbonate are shown in Figure 6. Figure 6 shows that the reaction is possible. This can be explained by the formation of precipitates of Fe, Pb, etc. by the action of sodium peroxide and sodium carbonate.

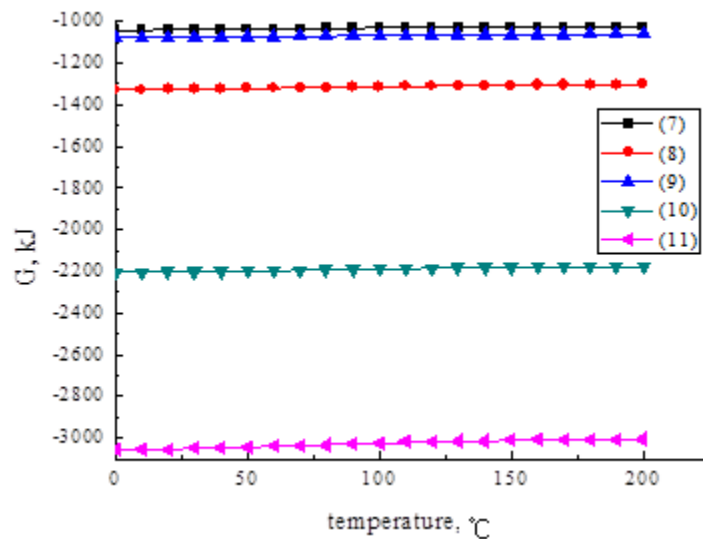
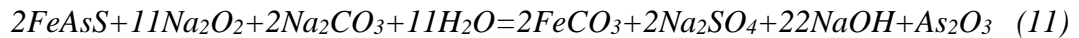
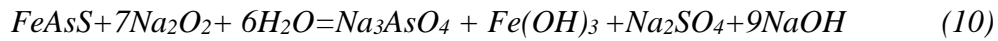
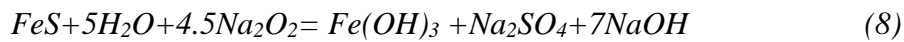


Figure 6 Free energy evolution curve of the reaction.

The effect of sodium carbonate addition and pretreatment time on gold extraction rate with 5 kg/t ammonium bifluoride and 3 kg/t sodium peroxide is shown in figure 7 and figure 8.

As shown in Figure 7, there is no significant change from sodium carbonate dosage up to 6 kg/t. As shown in Figure 8, there is no significant change over the 3 h pretreatment time. Therefore, the quantity of sodium carbonate was set at 6 kg/t and the pretreatment time was 3h. On the other hand, after

pretreatment with this method, the pulp color changed from black sulfide to light brown, indicating that the pretreatment process oxidized the iron of sulfide. The pulp color and the results of the X-ray diffraction analysis of residue before and after pretreatment are shown in Figure 9 and Figure 10. As shown in Figure 10, after pretreatment, the contents of FeS₂ and FeAsS decreased slightly. This suggests that the peroxide pretreatment partially decomposes the sulfide.

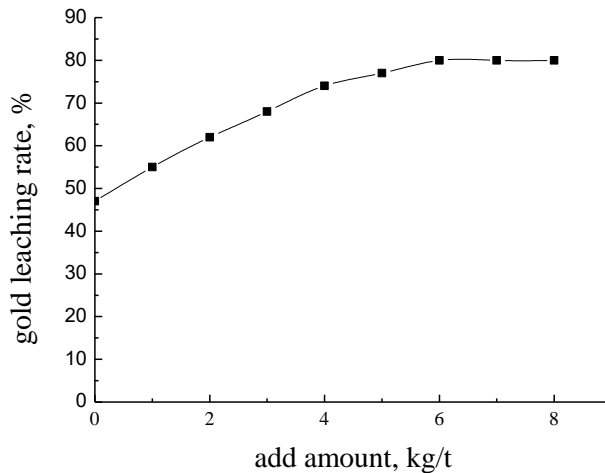


Figure 7 gold leaching rate with sodium carbonate addition

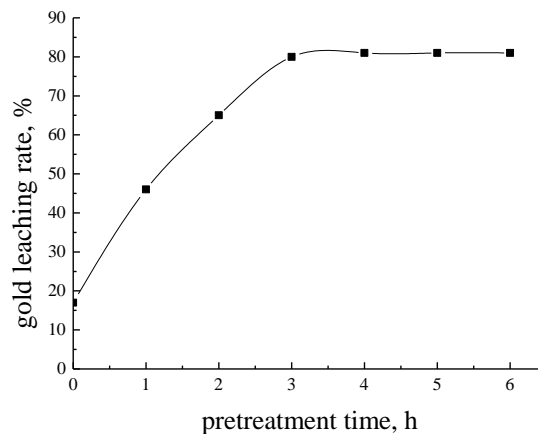


Figure 8 gold leaching rate with pretreatment time

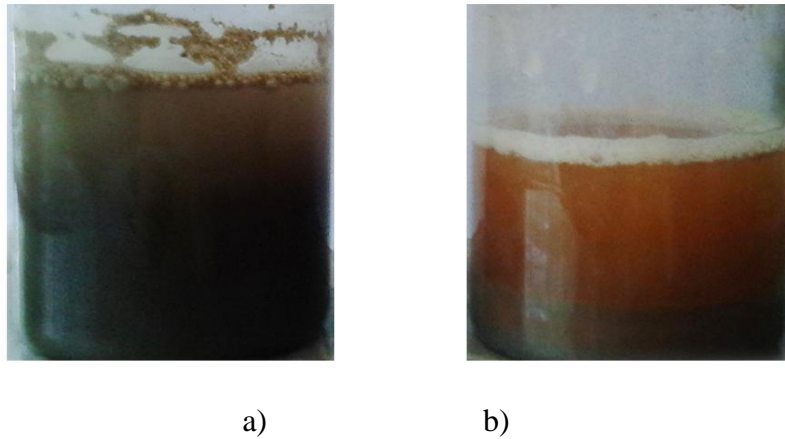


Figure 9 pulp colour before and after peroxide pretreatment

(a: pulp colour before peroxide pretreatment b: pulp colour after peroxide pretreatment)

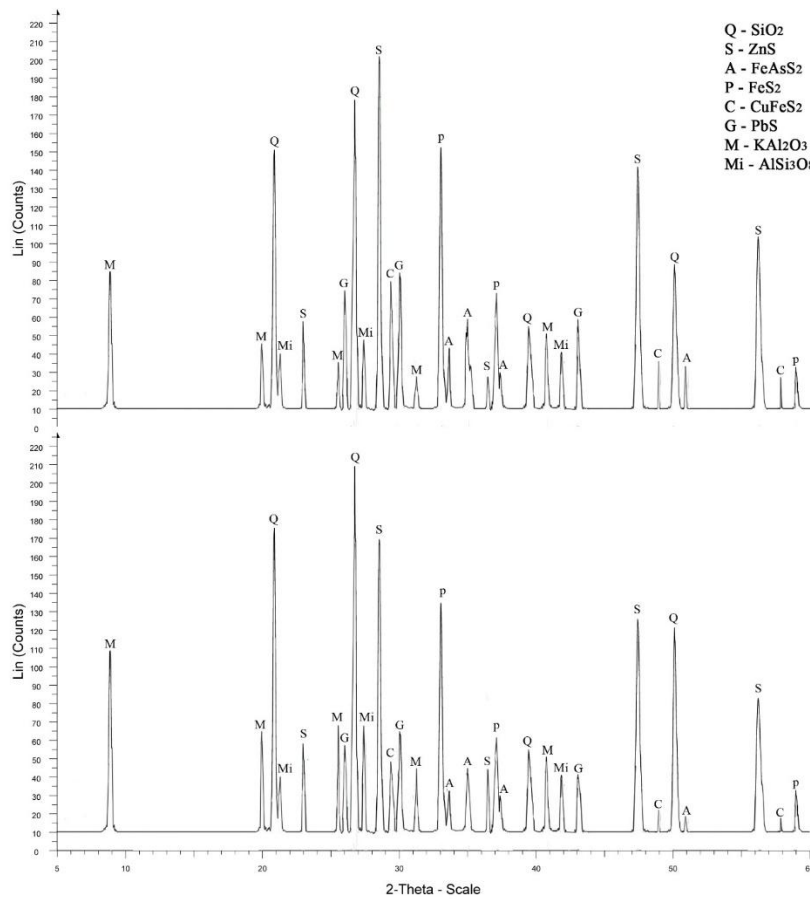


Figure 10 XRD pattern of gold concentrate and residue after pretreatment with Peroxide (Top-gold concentrate, Down-residue after pretreatment)

Electro-oxidation pretreatment

In the experiments, pre-oxidation of the pulp with alkaline pretreatment by electro-oxidation pretreatment was carried out to further improve the gold extraction. When the process of gold concentrate is subjected to an external electric field, the gold mineral surface is polarized and the excited electrons are transferred by an electric field. That is,

the cation is transferred in the opposite direction, and to inhibit this movement, the accumulated cation (e.g., H^+) on the cathode is deposited by hydrogen gas, and the anion (e.g., OH^-) accumulated on the anode is deposited by oxygen gas. The deposited oxygen gas can oxidize gold minerals and provide oxygen for cyanidation of gold. This physical and chemical action accelerates the dissolution rate of gold.

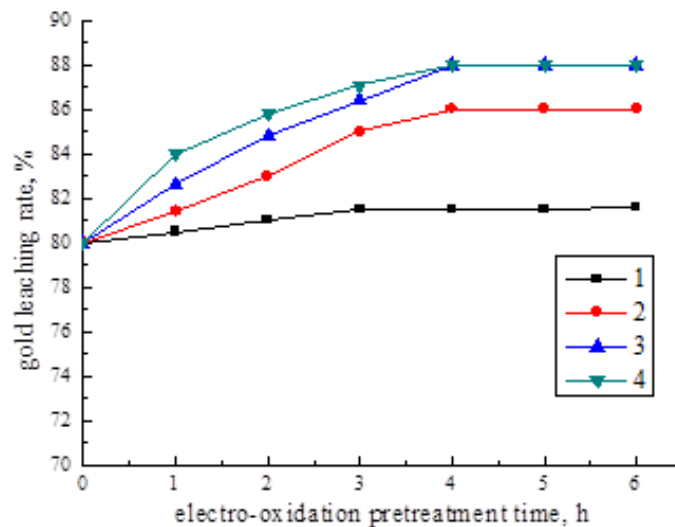


Figure 11 gold leaching rate with electro-oxidation pretreatment time
(1: 30A/m², 2: 50A/m², 3: 80A/m², 4: 100A/m²)

Figure 11 shows the effect of gold leaching rate on the current density. After peroxide treatment, electrooxidation pretreatment was performed and the pH of the pulp was adjusted to 9.5-10.5 with NaOH solution. Gold leaching rate was investigated by varying the cathodic current density at a distance of 1.5cm and a cell voltage of 8V at 30A/m², 50A/m², 80A/m² and 10 A/m². As shown in Figure 11, the gold leaching rate does not increase significantly from the current density above 80A/m². Also, the leaching rate was high from 4 h before pretreatment

and no change was observed above.

Therefore, the current density was set to 80A/m² and the electro-oxidation pretreatment time was set to 4 h.

The results of some elemental analyses of the leach residue before and after pretreatment are shown in Table 6, and the XRD patterns are shown in Figure 12. This indicates that the dissolution reaction of sulfide during pretreatment is favourable for cyanide leaching.

Table 6 Component analysis results of gold concentrate and pretreatment residue, %

elements	Pb	S	Fe	Zn	Cu	As	SiO ₂	Al ₂ O ₃
concentrate	7.38	17.41	22.67	4.47	1.68	0.67	35.4	7.16
residue after pretreatment	5.94	12.5	16.11	3.97	0.53	0.24	46.2	8.68
residue after electro-oxidation	5.12	10.33	12.86	3.86	0.42	0.09	55.74	9.25

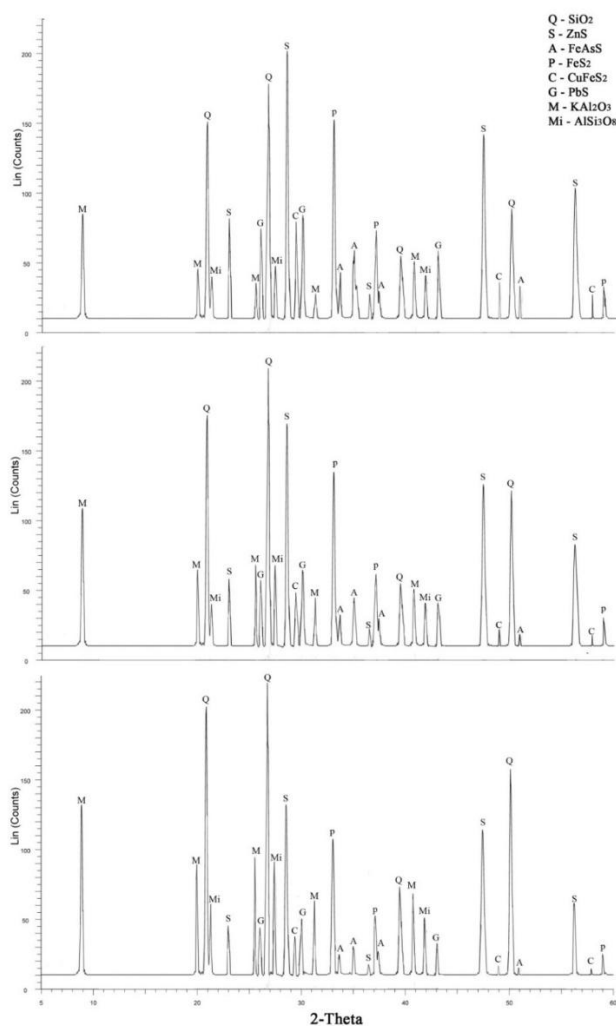
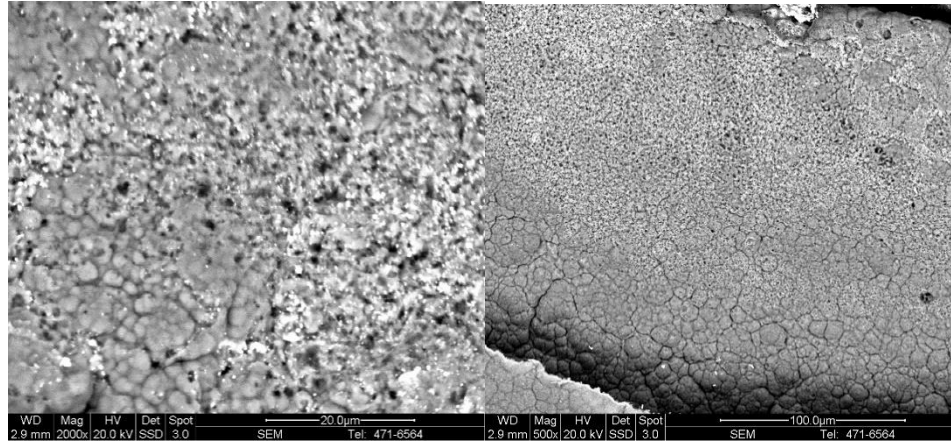


Figure 12 XRD pattern of gold concentrate and residue after pretreatment. (Top-gold concentrate, middle- Cyanide leaching residue after peroxide pretreatment, Down- Cyanide leaching residue after electro-oxidation pretreatment)



After pretreatment, the cathodic electrodeposition was reduced from 1.7g to 0.05g before pretreatment. That is, 0.18kg for 1t concentrate. It is shown that the effect of impurities causing cyanide consumption was minimized when the cathodic electrodeposition

was reduced. SEM images of the cathode electrodeposits before and after pretreatment are shown in the figure13 and figure 14. This indicates that the quality of the electrodeposits was improved.

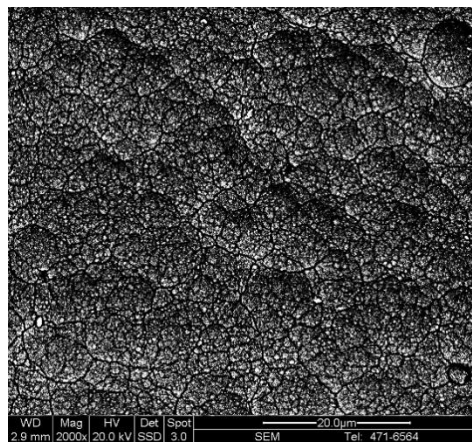


Figure 13 SEM images of the electrodeposit before pretreatment

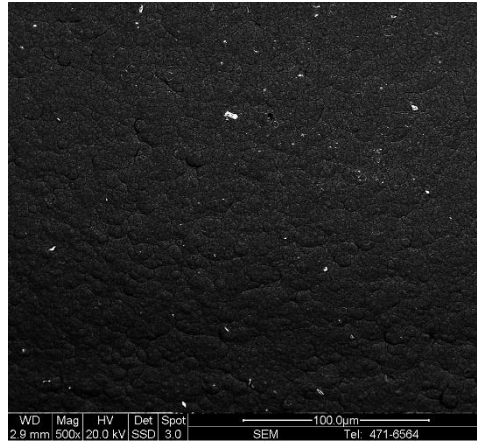


Figure 14 SEM images of the electrodeposit after pretreatment

Cyanide leaching after pretreatment

After pretreatment, the pH of the pulp was 9.5-10.5.

Then, the negative plates were removed and cyanide

leaching was carried out continuously. The effect of the quantity of sodium cyanide and the leaching time on the cyanide leaching process was investigated (Figure 15).

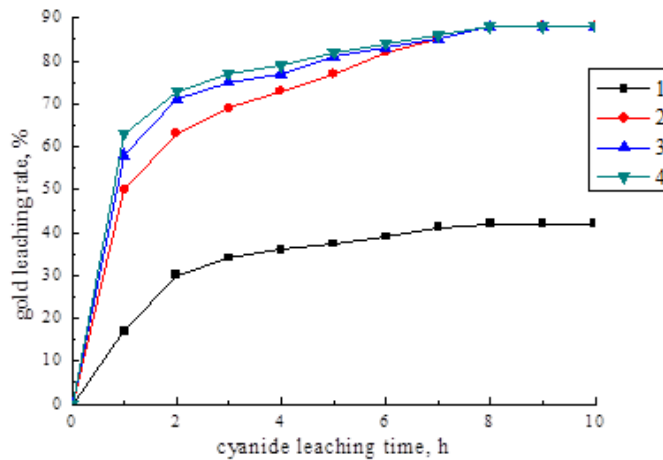


Figure 15 gold leaching rate with leaching time

(1: NaCN 5kg/t, 2: NaCN 10kg/t, 3: NaCN 15kg/t, 4: NaCN 20kg/t)

As shown in Figure 15, the gold leaching rate was 88% and higher at 8h of leaching from 10kg/t of sodium cyanide.

Therefore, cyanide leaching conditions were set at 10

kg/t of sodium cyanide and 8h of leaching time.

The concentration of sodium cyanide and the concentration of several components are given in Table 7. As shown in Table 7, after the peroxide

pretreatment and the electro-oxidation pretreatment, the cyanide concentration in the leachate was 0.11%, and the impurity content decreased.

This indicates that the proposed pretreatment method significantly reduces cyanide consumption and improves gold leaching.

Table 7 Concentrations of some components in cyanide leachate after pretreatment

NaCN, %	Fe, mg/L	Cu, mg/L	Pb, mg/L	Gold leaching rate,%
0.11	44.68	327.47	9.2	88

Comparison of the efficiency before and after pretreatment

The comparison of the efficiency before and after pretreatment is given in Table 8.

Table 8 Comparison of the efficiency before and after pretreatment

component	pretreatment time, h	quantity of added cyanide, kg/t	cyanide leaching time, h	NaCN, %	quantity of the electrodeposit, kg/t	gold leaching rate, %
before pretreatment	-	40	8	0	5.7	17.4
after pretreatment	7	10	8	0.11	0.18	88

Conclusions

The experimental results show that the combination of pretreatment with sodium peroxide, sodium carbonate and ammonium bifluoride and electrical pretreatment can reduce cyanide consumption and improve gold extraction. After 5kg/t ammonium bifluoride, 3kg/t sodium peroxide, 6kg/t sodium carbonate, stirred for 3h, and then electro-oxidation for 4 h at a current density of 80A/m² and a cell voltage of 10V, the cyanide leaching was directly carried out to increase the gold extraction from 17.4% to 88%.

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Conflicts of interest

The authors declare that there exists no competing financial interest or personal relationships that could have appeared to influence the work reported in this paper.

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