A new extraction process of carbonaceous refractory gold concentrate[①]

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Abstract: A new hydrometallurgical process for a carbonaceous refractory gold concentrate at ambient temperature and pressure was presented, including grinding-leaching, intensified alkaline leaching(IAL), thiosulfate leaching and cementation by zinc powder. The experimental results show that the grinding-leaching and intensified alkaline leaching process result in the selective oxidation of arsenopyrite and pyrite. The oxidation ratio of As is 96.6%, and 46.7% for S. The total consumption of NaOH in alkaline leaching is only 28% of that theoretically calculated under the conditions of full oxidization for the same amount of arsenopyrite and pyrite transforming into arsenates and sulfates, and 83.6% of gold is synchro-dissolved by thiosulfate self-generated during pretreatment. Since the carbonaceous matter in concentrate possesses a strong capability of preg robbing, the cyanidation process is not suitable for the extraction of gold after pretreatment. However, the gold leaching rate by thiosulfate leaching for 24 h is increased to 91.7% from 0 - 3.2% by ultra-fine grinding without the pretreatment. The recovery of gold by zinc cementation gets to 99.6%. Due to the thiosulfate self-generated during alkaline leaching, the reagent addition in thiosulfate leaching afterwards is lower than the normal one.

Key words: carbonaceous refractory gold concentrate; alkaline leaching; self-leaching of gold; preg robbing; thiosulfate leaching

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1 INTRODUCTION

The treatment of refractory gold ores(or concentrates)[1, 2] attracts more concerns of metallurgists. If there is not only arsenopyrite (FeAsS) bearing gold but also carbonaceous matter contained in the refractory ores, it could be more difficult for the extraction of gold[3~6]. Under many conditions, the carbonaceous matter in refractory ores could possess a strong capability of preg robbing[7~9] so that the cyanide leaching is not suitable for the extraction of gold after pretreatment. Moreover, the cyanide is very toxic. Therefore it is important to develop the non-cyanide method for gold extraction. Thiosulfate is an alternate to cyanide[10~12]. However, due to the high reagent cost as well as requiring heating the pulp[12, 13] in conventional thiosulfate leaching, its application has been defined to some extent.

In this paper, a hydrometallurgical method is described for the processing of carbonaceous refractory gold-bearing arsenopyrite concentrate under ambient temperature and pressure, including alkaline leaching pretreatment[14~16] and thiosulfate leaching. During alkaline leaching, the oxidation of arsenopyrite and pyrite(FeS₂), which always took place under high temperature and pressure,

was induced to occur under ambient temperature and pressure by ways of the mechanical activation of an ultra-fine pulverizing-leaching tower mill and the intensification of enhanced agitation tanks. The arsenopyrite was oxidized faster than the pyrite, owing to the lower oxidation potential of FeAsS. Due to the constituent change, lattice distortion and dislocation, and other mineralogical reactions caused by the gold born in minerals, the difference of oxidation distribution happened on the pyrite surface and the pyrite was selectively oxidized as well. Under the condition of low consumption of NaOH, a high oxidation of As (FeAsS was almost completely oxidized), a low oxidation of S(FeS₂ was partially oxidized) and a high exposition or liberation of gold born in arsenopyrite and pyrite were got. In the meantime, a portion of gold was synchro-dissolved by thiosulfate which self-generated during alkaline leaching[14~16].

2 EXPERIMENTAL

2.1 Experimental flowsheet and apparatus

The experimental flowsheet is shown in Fig.1, in which the main apparatus used includes feeder, stirring-type pulverizing-leaching tower

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mill of d 159 mm × 840 mm, enhanced agitation tank of d 190 mm × 285 mm, laser particle size analyzer (Mastersizer Ver. 2. 15), temperature gauge, pH meter, voltameter, air compressor, flow meter, leaching tank, filter, baking oven and XRD.
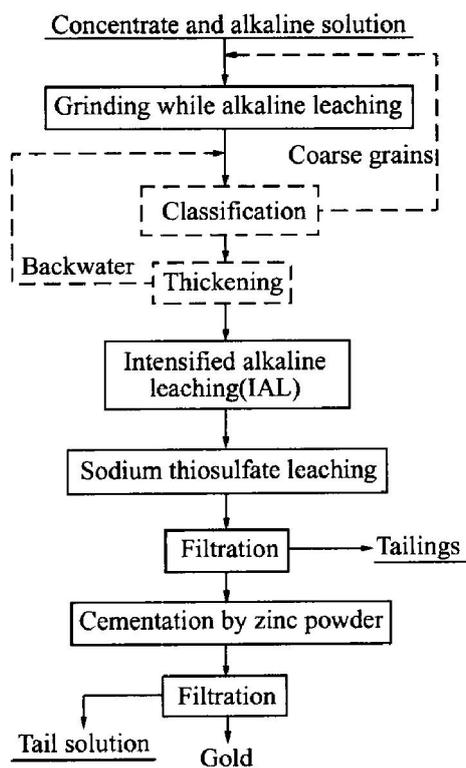

Fig. 1 Flowsheet of gold extraction

2. 2 Experimental materials

The raw material used in this work was a refractory gold concentrate, produced from Guangxi Province of China, whose chemical composition is shown in Table 1. The carbonaceous matter in the concentrate included the graphite and some organic compounds. The specific mass was 3. 17. The gold was not visible, mainly born in FeAsS(78. 3%), FeS$_2$(14. 2%) and gangue. Under the condition of ultra-fine grinding size (99. 9% < 30. 5 μm), the dissolution of gold by cyanide was about 0, or only 3. 2% by sodium thiosulfate. This is one of the typical refractory gold concentrates.

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

Au	Ag	As	S	Fe
1. 123 × 10^{-2}	2. 71 × 10^{-3}	10. 3	27	25. 7

Cu	Pb	Zn	C(total)	C(organic)
0. 14	0. 9	0. 8	3. 3	1. 0

The reagents used were NaOH (IR), NaCN (AR), CuSO$_4$ • 5H$_2$O(AR), NH$_3$ • H$_2$O(AR), Na$_2$SO$_3$(AR), HCl(AR), Na$_2$S$_2$O$_3$ • 5H$_2$O(AR),

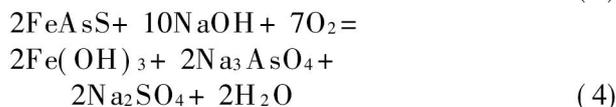CaO(IR), Zn(AR), Fe$_2$(SO$_4$)$_3$(AR) and activated carbon(AR). The water came from the city water pipe.
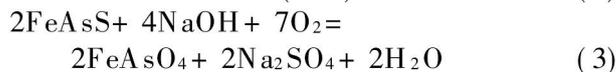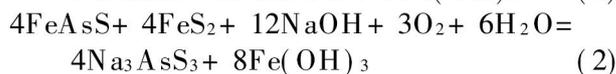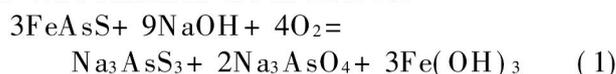
3 RESULTS AND DISCUSSION

3. 1 Grinding and alkaline leaching

The pulverizing-leaching tower mill mainly utilizes the abrasive action to carry out grinding, which possesses not only a strong capability of fine grinding with high efficiency but also a function of mechanical activation. It enables some chemical reactions to be intensified and sped up[14-16] with reduction of the size, and it can be used as a pre-reactor during pulverizing in the fields of activation, cyanidation, alkaline leaching pretreatment, and floatation[14-17].

During alkaline leaching, the active and intensive functions are as follows[14-16]: 1) The coarse arsenopyrite particles are well ground to increase their surface area as well as the active points of reaction; 2) The freshly liberated arsenopyrite particles are subjected to reaction with O$_2$ and NaOH; 3) The enhanced mass transfer conditions increase the contact probabilities among arsenopyrite particles, NaOH and O$_2$; 4) The surface coating and tarnishing are removed from the arsenopyrite particles under the high shear conditions so that the passivation is impossible; 5) The diffusion film is smashed or thinned to reinforce the diffusion; 6) The heat generated from grinding and oxidation increases the oxidation rate.

Thus, the combination of grinding-leaching in the tower mill with intensified alkaline leaching in the enhanced agitation tanks can substantially increase the reaction kinetics of alkaline leaching so that the oxidation probabilities of FeAsS, which always takes place under high temperature and pressure, can be induced to occur under ambient ones. The reactions are as follows:

$$3FeAsS + 9NaOH + 4O_2 =$$
$$Na_3AsS_3 + 2Na_3AsO_4 + 3Fe(OH)_3 \qquad (1)$$
$$4FeAsS + 4FeS_2 + 12NaOH + 3O_2 + 6H_2O =$$
$$4Na_3AsS_3 + 8Fe(OH)_3 \qquad (2)$$
$$2FeAsS + 4NaOH + 7O_2 =$$
$$2FeAsO_4 + 2Na_2SO_4 + 2H_2O \qquad (3)$$
$$2FeAsS + 10NaOH + 7O_2 =$$
$$2Fe(OH)_3 + 2Na_3AsO_4 +$$
$$2Na_2SO_4 + 2H_2O \qquad (4)$$
$$2FeS_2 + 4NaOH + 3O_2 =$$
$$2Na_2S_2O_3 + 2Fe(OH)_2 \qquad (5)$$
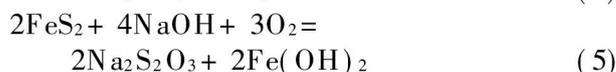
Under certain conditions, the anions $S_2O_3^{2-}$ and AsS_3^{3-} can be further oxidized to SO_4^{2-} and AsO_4^{3-}, Fe(OH)$_2$ is oxidized to Fe(OH)$_3$, and Fe(OH)$_3$ is decomposed to Fe$_2$O$_3$. The occurring extent of reaction(5) will depend on the reactivity of FeS$_2$.

Under certain conditions, the activated degree of a material corresponds to the grinding size. In this work the grinding size (listed in Table 2) is suitable for the intensified alkaline leaching(IAL) afterwards, which can be economically obtained by the pulverizing-leaching tower mill[16]. When the grinding-leaching is completed, i. e. at the beginning of IAL, the oxidation of As and S are 13. 3% and 4. 8% in mass fraction, respectively. The contribution to arsenopyrite oxidation by grinding-leaching in the tower mill is large.

3. 2 Intensified alkaline leaching(IAL)
3. 2. 1 Selective oxidation of arsenopyrite and pyrite

After grinding-leaching and with IAL(listed in Table 3) going on in the enhanced agitation tanks, the arsenopyrite and pyrite are selectively oxidized, and the arsenopyrite is oxidized faster than the pyrite. As shown in Fig. 2, after IAL for 48 h, the oxidation ratios of As and S are 96. 6% and 46. 7% in mass fraction, respectively.

During IAL, the color of the solid phase changes fast. After IAL for 3 h, the solid appears yellow obviously. In the end of IAL, the solid becomes brownish yellow contrasted to the blackish gray of raw material. And the specific mass decreases to 2. 61 from 3. 17 before pretreatment. Due to the precipitation of Fe^{3+} and Ca^{2+} with AsO_4^{3-} in the solution, most of the oxidized As is turned into the solid phase again. In the end of IAL, the concentration of As in solution is 3. 8 g/L.

Due to the selective oxidation of arsenopyrite and pyrite in IAL, the arsenopyrite is almost completely oxidized, but the pyrite is partially oxidized and the non-oxidized pyrite still keeps its primitive phase. This is further verified by the analysis result of XRD, as shown in Fig. 3. After IAL, there is no FeAsS but FeS_2 in the solid residue. Thus, most skeletal structures of particles could be
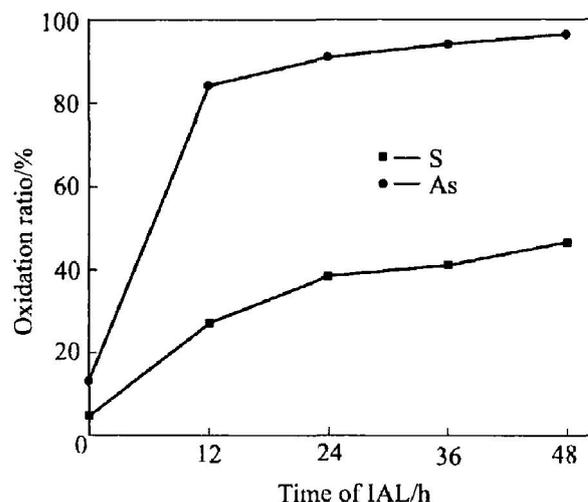

Fig. 2　Selective oxidation kinetics of arsenopyrite and pyrite

reserved[14-16]. As shown in Fig. 4, the particle distributions between 10 μm and 30 μm before and after IAL are well matched. This is profitable for the subsequent extraction of gold.

3. 2. 2 φ_h change of pulp

Fig. 5 shows the φ_h change of the pulp. After IAL for 12 h, φ_h rises quickly from initial − 378 mV to − 163 mV. Then it increases gradually. After IAL for 48 h, it is − 97 mV. In fact, the changing tendency of φ_h of the pulp is the objective reflection of the oxidation progress of arsenopyrite and pyrite. The more the arsenopyrite and pyrite are oxidized, the weaker the capacity for the pulp to be oxidized, and the higher the φ_h of the pulp. At the end of IAL, the refractory concentrate becomes easily extracted for the gold.

3. 2. 3 Consumption kinetics of NaOH and variation of pulp temperature

As much heat comes out from the oxidation of arsenopyrite and pyrite in IAL, the temperature of pulp rises quickly, as shown in Fig. 6. After IAL for 1. 25 h, the temperature is increased from

Table 2　Conditions for grinding-leaching

| Concentrate/ kg	Water/ L	Particle size of Product/ μm	Aerating air/ ($m^3 \cdot h^{-1}$)	NaOH/ ($kg \cdot t^{-1}$)	Temperature/ ℃	Pressure/ Pa	Concentration/ %
2	2	< 30. 5(99. 9%)	0. 1	5	21 − 24	1×10^5	50(solids)

Table 3　Conditions for intensified alkaline leaching(IAL)

Water of regulation/ L	Concentration/ %	Temperature/ ℃	Pressure/ Pa	Aerating air/ ($m^3 \cdot h^{-1}$)	Speed of agitation/ ($m \cdot s^{-1}$)	NaOH/ ($kg \cdot t^{-1}$)	pH	Time of leachig/ h	Temperature of pulp* / ℃
1	40(solids)	21 − 24	1×10^5	0. 2 − 0. 3	10 − 13	130	11	48	33(beginning), 58(peak). 42(end)

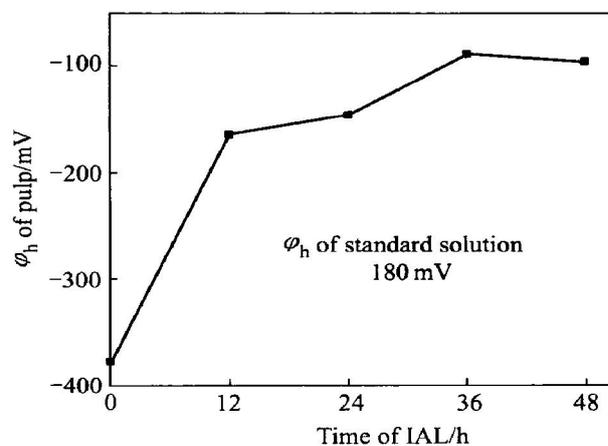① * —Without preheating and incubation; ② 50 kg/t of CaO is added when IAL for 47 h
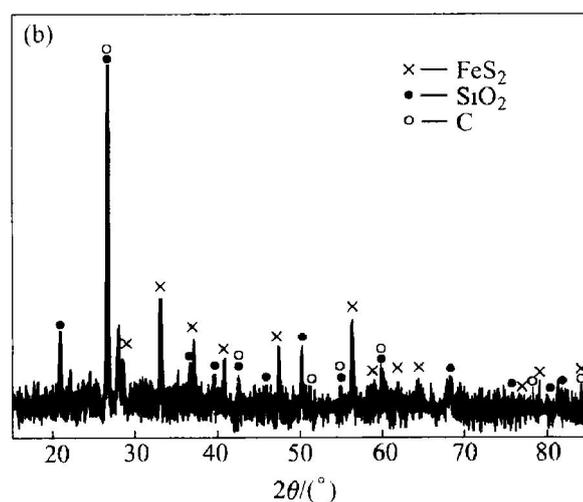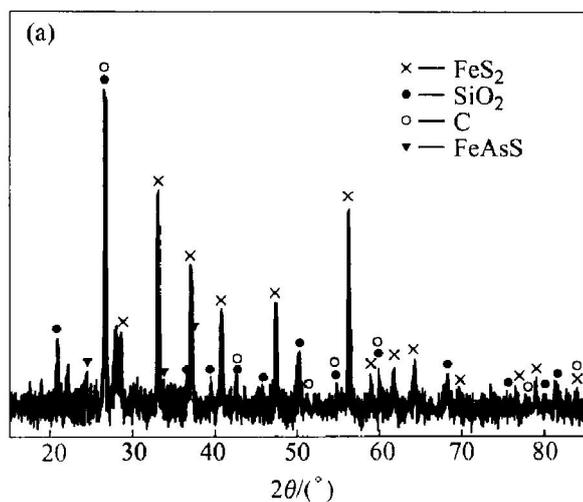
Fig. 3 XRD patterns of solids
(a) —Before IAL; (b) —After IAL
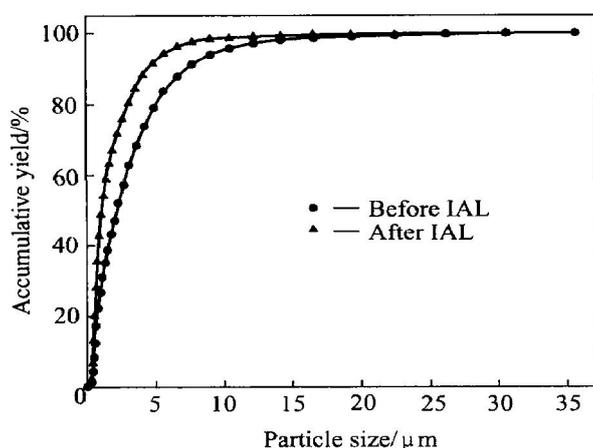
Fig. 4 Change of particle distribution after IAL

Fig. 5 φ_h change of pulp under
pH= 11 at 21 ℃

Fig. 6 Consumption kinetics of NaOH and
variation of pulp temperature

initial 33 ℃ to the peak value of 58 ℃. The rising speed of pulp temperature gets to 20 ℃/ h, and the consumption rate of NaOH for 1 t ore reaches the maximum value of 32 kg/ h. During 1. 25 ~ 2. 5 h of IAL, the temperature drops to 45 ℃ quickly, and the consumption rate of NaOH falls to 8 kg/ h. After IAL for 3 h, both the pulp temperature and the consumption rate of NaOH (about 1. 8 kg/ h) vary
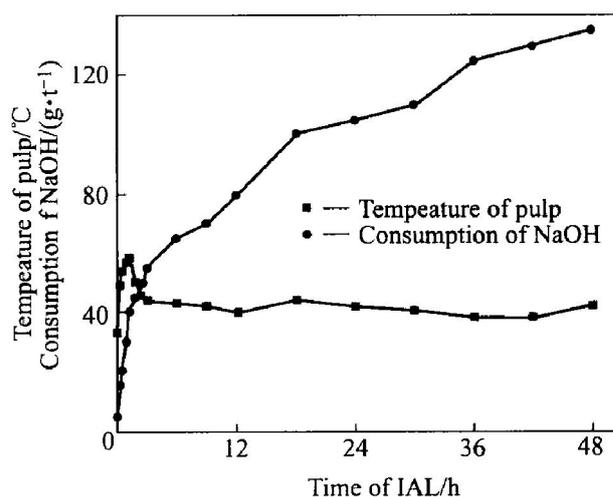
slowly. At the end of IAL, the pulp temperature is 42 ℃.

Due to the selective oxidation of arsenopyrite and pyrite as well as the generation of thiosalts shown in reactions(1), (2) and(5) during alkaline leaching, the amount of sulfur which needs to be fully oxidized is decreased and a smaller amount of NaOH is required. After IAL for 48 h, the total consumption of NaOH for 1 t ore is 135 kg that includes 5 kg NaOH consumed during the grinding-leaching process. This is only 28% of those theoretically calculated under the conditions of full oxidization for the same amount of arsenopyrite and pyrite transforming into arsenates and sulfates.

3. 2. 4 Concentration change of $S_2O_3^{2-}$ in solution
  and self-leaching of gold

The thiosulfate is a good and non-toxic reagent for the extraction of gold[10-13]. During alkaline leaching, some thiosalts, including thiosulfate, can be generated in the oxidation of arsenopyrite and pyrite(shown in reactions (1), (2) and (5)). With the alkaline leaching going on, the

gold born in arsenopyrite and pyrite is progressively exposed or liberated, and simultaneously some of them can be synchro-dissolved by the self-generated thiosulfate. This phenomenon is called synchro-self-leaching of gold during alkaline leaching pretreatment[14–16].

Fig. 7 gives the concentration change of $S_2O_3^{2-}$ in solution and the result of synchro-self-leaching of gold during alkaline leaching. When the grinding-leaching is completed, i. e. at the beginning of IAL, the concentration of $S_2O_3^{2-}$ in solution is 0.98 g/L, and the self-leaching rate of gold is 10.6%. After IAL for 12 h, the concentration of $S_2O_3^{2-}$ in solution rises to 36.2 g/L quickly, and the self-leaching rate of gold is 65.7%. Then, the self-leaching rate of gold slowly increases to 83.6% in the end. However, for the contentration of $S_2O_3^{2-}$ in solution, after it reaches the maximum value of 57.9 g/L for 36 h IAL, it drops a little to 50.4 g/L in the end. This is because that the amount of sulfur oxidized during 36 – 48 h IAL was much less (only 5.6%) and the oxidation speed of $S_2O_3^{2-}$ to SO_4^{2-} is higher than its generation rate. Anyhow, this little decrease for the concentration of $S_2O_3^{2-}$ in solution does not affect the self-leaching of gold under the conditions of IAL.
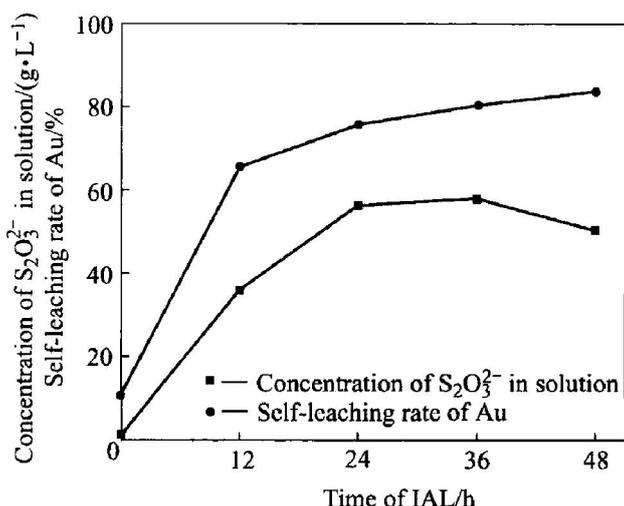

Fig. 7 Concentration change of $S_2O_3^{2-}$ in solution and corresponding self-leaching rate of gold

3.3 Gold extraction

3.3.1 Preg robbing of carbonaceous matter in cyanidation

Some activated carbon ingredients in the carbonaceous matter can adsorb the gold from the cyanidation solution, resulting in a low rate of recovery of leaching. This behavior is called preg robbing[7]. After pretreatment, the action of preg robbing is tested in cyanidation, as shown in Fig. 8. For 3 h cyanide leaching, the leaching recovery of gold gets to the maximum value of 89.0%. Afterwards, it drops gradually. For 24 h cya-

nide leaching, it further drops to 79.6%, which is lower than the self-leaching rate of 83.6% during alkaline leaching pretreatment. These results show that the carbonaceous matter in the concentrate possesses a strong capability of preg robbing.
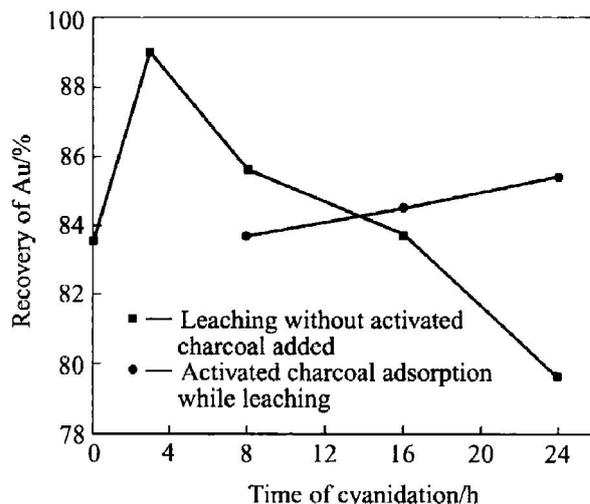

Fig. 8 Preg robbing of carbonaceous matter in cyanidation

If 30 g/L activated carbon is added in the pulp at the beginning of cyanidation in order to generate a competitive adsorption together with the carbonaceous matter, the effect of preg robbing could be cut down to some extent. However, the recovery of gold by activated carbon for 24 h absorption while cyanide leaching is 84.7%, only 1.1% higher than the self-leaching rate during pretreatment and yet is lower than the maximum cyanide leaching rate of 89.0% after pretreatment. It is shown that the competitive adsorption of activated carbon could not completely eliminate the preg robbing effect of carbonaceous matter. Moreover, the consumption of NaCN is very high, about 18 kg/t.

Based on the results above, it is shown that the cyanidation process is not suitable for the extraction of gold from the oxidized pulp.

3.3.2 Sodium thiosulfate leaching

After pretreatment, the recovery of gold by cyanide and activated carbon is low because of the preg robbing effect of carbonaceous matter in the gold-bearing materials. Besides, the cyanide is very toxic, which can cause serious poison problem to the environment. Therefore it is important to develop the non-cyanide method for gold extraction. The thiosulfate is a non-toxic reagent that possesses a strong capability of gold extracting from ores[10–13]. Different from the anions $Au(CN)_2^-$, the anions $Au(S_2O_3)_2^{3-}$ cannot be adsorbed by carbonaceous matter[12]. Since there is an amount of self-generated thiosulfate and a portion of synchro-dissoluted gold during alkaline leaching, it might be more reasonable for

thiosulfate leaching process just following the pre-treatment. On one hand this can eliminate the preg robbing effect of carbonaceous matter in the gold-bearing materials; on the other hand it can result in not only a non-cyanide extraction of gold but also a less reagent consumption. In fact, due to the main defect of high reagent cost in conventional thiosulfate leaching, its application has been defined to some extent[12, 13].

Under the conditions listed in Table 4, Fig. 9 shows the dissolution results by an additional $Na_2S_2O_3$ in the oxidized pulp for 24 h leaching at the ambient temperature of 21 ~ 24 ℃. When the addition of $Na_2S_2O_3$ in solution increases to 0. 11 mol/ L, the dissolution of gold is the highest, reaching 91. 7%. When it further increases to 0. 22 mol/ L, the dissolution of gold drops a little to 88. 6%. More addition of $Na_2S_2O_3$ is disadvantageous to the gold extraction. This is because that it can cause the stabilizing equilibrium of reaction (6) generated by the additional Na_2SO_3 to move towards the left:

$$6H^+ + 4SO_3^{2-} + 2S_2^- = 3S_2O_3^{2-} + 3H_2O \qquad (6)$$
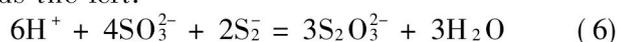
However, the anions S_2^- could give rise to the sulfide film precipitated on the surface of gold, impeding the extraction process. Moreover, it is noticeable that the dissolution of gold under the condition of no $Na_2S_2O_3$ addition in the pulp is only 86. 6%. These results show that a high dissolution of gold can be obtained at ambient temperature without heating the pulp in contrast with leaching at 40 ~ 60 ℃ for some other conventional thiosulfate methods[10-13], and the consumptions of $Na_2S_2O_3$, $CuSO4$, $NH_3 \cdot H_2O$ and Na_2SO_3 listed in Table 4 are several times lower than those in other thiosulfate leaching processes[10-13].

Table 4   Conditions for conventional mixing of sodium thiosulfate leaching

Concentration of pulp/ %	Temperature/ ℃	Speed of mixing/ ($m \cdot s^{-1}$)
38 ~ 40(solids)	21 ~ 24	1. 7 ~ 2. 1
$c(CuSO_4)/$ ($mol \cdot L^{-1}$)	$c(NH_3 \cdot H_2O)/$ ($mol \cdot L^{-1}$)	$c(Na_2SO_3)/$ ($mol \cdot L^{-1}$)
0. 02	0. 25	0. 05
$c(HCl^*)/$ ($mol \cdot L^{-1}$)	pH	$c(Na_2S_2O_3)/$ ($mol \cdot L^{-1}$) · Time/ h
0. 15	~ 9	0. 11 · 24

* —Addition in regulation of pH from 11 to 9

3. 3. 3   Cementation by zinc powder

After thiosulfate leaching, if 30 g/ L activated carbon is added in the filtrate, the adsorption of gold for 24 h is zero. This further confirms the point of view that the anions $Au(S_2O_3)_2^{3-}$ can not
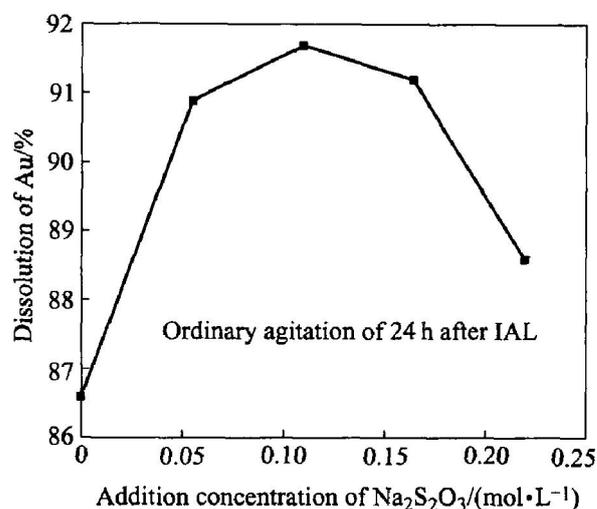
Fig. 9   Sodium thiosulfate leaching of gold

be adsorbed by carbonaceous matter[14]. However, when 0. 18 kg/ m^3 zinc powder is added in the filtrate, the recovery of gold by zinc cementation for 0. 5 h gets to 99. 6%.

3. 4   Environmental control and techno-economic assessment

After zinc cementation and filtration, 6. 5 kg/ m^3 $Fe_2(SO_4)_3$ is added step-by-step to the solution in a conventional agitation tank. The arsenide in the solution is precipitated to form a stable phase of ferric arsenate. The concentration of As in the solution is decreased below 0. 5 mg/ L, which meets the requirement of the environmental pollution control system.

A preliminary techno-economic assessment shows that processing of the objective concentrate is about RMB 430 yuan per ton.

4   CONCLUSIONS

1) After grinding-leaching in a pulverizing-leaching tower mill and intensified alkaline leaching for 48 h in the enhanced agitation tanks at ambient temperature(21 ~ 24 ℃) and pressure(10^5 Pa), the dissolution of gold by thiosulfate of the objective carbonaceous refractory concentrate is enhanced to 91. 7% from 0 ~ 3. 2% by ultra-fine grinding without the pretreatment, and the recovery of gold by zinc powder cementation in the solution gets to 99. 6%.

2) Due to the selective oxidation of arsenopyrite and pyrite as well as the generation of thiosalts during alkaline leaching, the total consumption of NaOH in the present processing is only 28% of those theoretically calculated under the conditions of full oxidization for the same amount of arsenopyrite and pyrite transforming into arsenates and sulfates. And the thiosulfate self-generated during

alkaline leaching results in 83. 6% of gold synchro-dissoluted during the pretreatment.

3) The carbonaceous matter in the gold-bearing materials possesses a strong capability of preg robbing. The cyanidation process is not suitable for the extraction of gold after the pretreatment. However, small addition of thiosulfate afterwards can make a high recovery of gold at ambient temperature without pulp heating.

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