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Gold Extraction from Oxide Ore using Copper Ethylenediamine Thiosulfate

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Abstract—Thiosulfate leaching is considered a green gold extraction approach and a gold mine in Laos was subjected to such treatment in this study. A single factor test was undertaken to determine the optimal leaching conditions, leading to the following parameters: thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; pH value of 10; leaching time of 4 h; stirring speed of 200 rpm. After leaching, the remaining ion concentrations were relatively low, positively affecting gold recovery. To further enhance the leaching rate, the impact of cetyltriethylammonium bromide (CTAB) on the leaching rate of gold was investigated, revealing that the addition of CTAB can increase gold's leaching rate while reducing thiosulfate consumption. In conclusion, a comparison was made between thiosulfate and cyanide leaching, where copper-ethylenediamine-thiosulfate leaching exhibited a shorter duration compared to cyanidation, establishing its potential as a more environmentally sustainable approach.

Keywords: thiosulfate leaching, optimal leaching conditions, CTAB

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INTRODUCTION

Currently, cyanidation serves as the primary process in gold hydrometallurgy. However, due to the highly toxic nature of cyanide and it's detrimental effects on the environment and human health, there is a pressing need to explore alternative, environmentally friendly gold leaching agents [1, 2]. Researchers have investigated various green gold extraction methods utilizing compounds such as thiourea and thiosulfate. Among these alternatives, thiosulfate stands out as a promising non-cyanide leaching agent, forming a stable complex with gold. This method offers advantages such as rapid leaching and shorter processing times, making it the most viable environmentally friendly option for replacing cyanide [3]. While the copper-ammonia-thiosulfate system has been extensively studied for gold extraction [4]. it poses challenges related to the use of volatile ammonia and the consumption of thiosulfate. These issues result in high reagent costs and numerous environmental concerns. To mitigate this, the application of ethylenediamine in place of ammonia has been explored, forming a copper-ethylenediamine-thiosulfate gold leaching system. Ethylenediamine is capable of forming a more stable coordination compound with copper ions, thereby reducing the oxidation of copper ions and theoretically reducing the consumption of sodium thiosulfate [5, 6]. The main reaction is represented by Eq. (1), with $Cu(en)_2^{2+}$ serving as a catalyst in the process. By adopting this approach, the copper-ethylenediamine-thiosulfate system offers a solution with improved efficiency, reducing reagent costs and mitigating environmental impact.

$$\begin{aligned} &Au + 5S_2O_3^{2-} + Cu(en)_2^{2+} \\ &= Au(S_2O_3)_2^{3-} + Cu(S_2O_3)_3^{5-} + 2en. \end{aligned} \tag{1}$$

In this study, a gold ore sourced from Laos was subjected to leaching using a copper-ethylenediamine-thiosulfate system specifically designed for gold leaching. The electrochemical impact of varying concentrations of thiosulfate, copper ion, and ethylenediamine on the leaching process was thoroughly investigated. The obtained research findings hold significant importance in establishing a solid foundation for the theoretical framework and practical implementation of thiosulfate-based gold leaching systems.

THERMODYNAMIC CALCULATIONS

Cu(II) exists as $Cu(en)_2^{2+}$, while Cu(I) mainly exists as $Cu(S_2O_3)_3^{5-}$ in the copper-ethylenediamine-thiosulfate system. The concentration of fixed copper

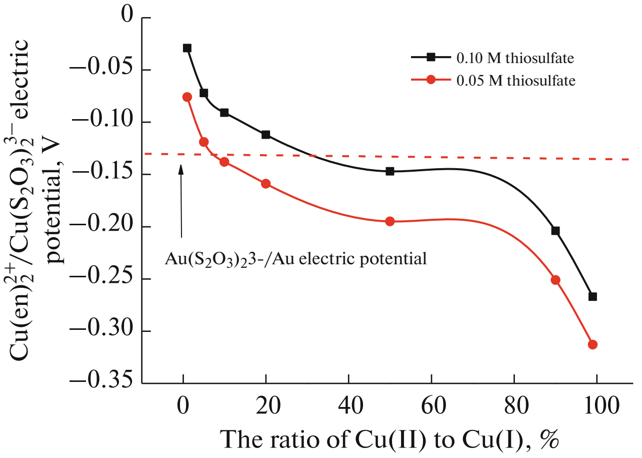

Fig. 1. Potential of the $Cu(en)_2^{2+}/Cu(S_2O_3)_3^{5-}$ electrode pair at different Cu(II)/Cu(I) ratios.

ion is 0.005 M, and the concentration of ethylenediamine is 0.01 M. The Nernst equation (2) is used to calculate the potential, and the results are displayed in Fig. 1.

$$\varphi_{Cu(en)_2^{2+}/Cu(S_2O_3)_3^{5-}} = \varphi^{\Theta}_{Cu(en)_2^{2+}/Cu(S_2O_3)_3^{5-}} + 0.0592\log\frac{[Cu(S_2O_3)_3^{5-}][en]^2}{[Cu(en)_2^{2+}][S_2O_3^{2-}]^3}. \tag{2}$$

As shown in Fig. 1, copper-ethylenediamine-thiosulfat system is able to meet the requirements for dissolving gold, the potential of $Cu(en)_2^{2+}/Cu(S_2O_3)_3^{5-}$ is lower than that of the $S_4O_6^{2-}/S_2O_3^{2-}$ electrode potential. This suggests that the copper-ethylenediamine-thiosulfate system not only meets the requirements for gold dissolution, but also does not oxidize the thiosulfate reagent. This is beneficial as it helps to reduce the consumption of reagents.

Table 1 presents thermodynamic calculations for typical chemical equations [7]. The results demonstrate that the association between divalent copper ions and ethylenediamine forms a complex that exhibits the ability to spontaneously dissolve gold, thus establishing its thermodynamic feasibility. Moreover, through thermodynamic analysis, it has been observed that the copper-ethylenediamine-thiosulfate system exhibits a greater propensity for dissolving gold compared to the copper-ammonia-thiosulfate system. This finding suggests the potential utility of ethylenediamine in enhancing gold leaching processes. Despite the inability of $[Cu(en)_2]^{2+}$ to oxidize thiosulfate to $Cu(S_2O_3)_2^{3-}$, all other reactions exhibit $\Delta_rG_m^{\Theta}$ values below 0, indicating that $S_2O_{33}^{2-}$ can be effectively oxidized by $[Cu(en)_2]^{2+}$. Additionally, both $[Cu(S_2O_3)_2]^{3-}$ and $[Cu(S_2O_3)_3]^{5-}$ are oxidized to $[Cu(en)_2]^{2+}$, indicating the interchangeability of Cu(I) and Cu(II) complexes within this system.

EXPERIMENTAL

Reagents and materials. All reagents used in the study were of analytical grade, while deionized water served as the test water. The ore sample was sourced from a gold mine situated in Laos, specifically selected for its representative characteristics. The gold within the ore is classified as an independent occurrence of natural gold. Based on the X-ray diffraction analysis results (Table 2), it is evident that the ore composition predominantly consists of elevated levels of quartz and muscovite. In addition, a minor proportion of goethite is present within the ore. Furthermore, the ore sample displays a gold and silver content constituting approximately 5% of the total composition.

The results of the multielement analysis for the ore chemistry are detailed in Table 3. The ore exhibits an Au grade of 12.2 g t^{-1}. Considering the relatively higher market price per gram of gold compared to silver, despite its lower abundance, the presence of gold in the ore holds considerable value.

In order to enhance the characterization of gold content within the gold mine, we conducted a detailed

Table 1. Chemical equation thermodynamic calculations

Reactions	$\Delta_rG_m^{\Theta}$, KJ mol^{-1}
$Au + 4S_2O_3^{2-} + Cu(en)_2^{2+} = Au(S_2O_3)_2^{3-} + Cu(S_2O_3)_2^{3-} + 2en$	−59.96
$Au + 5S_2O_3^{2-} + Cu(en)_2^{2+} = Au(S_2O_3)_2^{3-} + Cu(S_2O_3)_3^{5-} + 2en$	−68.41
$4[Cu(en)_2]^{2+} + 12S_2O_3^{2-} = 4Cu(S_2O_3)_2^{3-} + 4en + 2S_4O_6^{2-}$	+148.04
$4[Cu(en)_2]^{2+} + 16S_2O_3^{2-} = 4Cu(S_2O_3)_3^{5-} + 8en + 2S_4O_6^{2-}$	−223.24
$4Cu(S_2O_3)_2^{3-} + 8en + O_2 + 2H_2O = 4[Cu(en)_2]^{2+} + 8S_2O_3^{2-} + 4OH^-$	−215.61
$4Cu(S_2O_3)_3^{5-} + 8en + O_2 + 2H_2O = 4[Cu(en)_2]^{2+} + 12S_2O_3^{2-} + 4OH^-$	−182.40

Table 2. X-ray diffraction data

Mineral name	Formula	Content, %
Quartz	SiO_2	50.45
Muscovite	$(K,Na)(Al,Mg,Fe)_2(Si_{3.1}Al_{0.9})O_{10}(OH)_2$	42.67
Goethite	$FeO(OH)$	1.88
Kustelite	Au/Ag	5.00

Table 3. Ore chemical multi-element analysis

Element	Au	Ag	Fe	S	As	SiO_2	MgO	Al_2O_3	CaO
Content, %	12.20 g t^{-1}	93.51 g t^{-1}	6.40	0.060	0.27	62.27	2.72	16.72	5.03

ore fire assay analysis using the cupellation method. The findings from the fire assay analysis aligned seamlessly with the results obtained from the chemical multi-element analysis, validating an established Au grade of 12.2 g t^{-1} for the ore under examination.

Laboratory apparatus. An AA320 atomic absorption spectrophotometer, a constant-temperature electric stirrer 107 (Guohua JJ-1), and a pH meter (PHS-3C, Shanghai Scientific Instruments Co., Ltd., China) were used for the experiments.

Synthesis. A specified quantity of mineral sample with a grinding fineness of 74 μm (95% passing) was introduced into distinct leaching solutions with varying concentrations of copper sulfate, ethylenediamine, sodium thiosulfate, and deionized water. The pH of each solution was adjusted using either sodium hydroxide or hydrochloric acid to enable leaching under defined liquid-to-solid ratio and leaching duration. After leaching, the filter residue was filtered, and the gold content in the filter residue was determined by AA320 atomic absorption spectrophotometer to evaluate the leaching rate of gold.

RESULTS AND DISCUSSION

Univariate Analysis

The impact of agitation speed on the gold leaching rate was examined within the specified experimental parameters (Fig. 2): thiosulfate concentration of 0.2 mol L^{-1}, copper ion concentration of 0.005 mol L^{-1}, ethylenediamine concentration of 0.01 mol L^{-1}, liquid-to-solid ratio of 3 : 1, pH value of 10, and leaching time of 4 h. The gold leaching rate demonstrates a gradual increase with rising stirring speed. Among the given leaching conditions, the gold leaching rate reaches its peak at 200 rpm. However, when the stirring speed exceeds 200 rpm, the gold leaching rate experiences a gradual decline. Consequently, 200 rpm was chosen as the optimal stirring speed for the experimental investigation.

The impact of pH on the gold leaching rate was examined within the specified conditions (Fig. 3): thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid/solid ratio of 3 : 1; leaching time of 4 h; and stirring speed of 200 rpm. The gold leaching rate remains stable within the pH range of 8 to 11. Based on Eqs. (3)–(6), excessively high pH levels can result in the precipitation of copper ions or the disproportionation of sodium thiosulfate. Consequently, maintaining a pH range between 8 and 11 is considered optimal for the process of gold leaching in the copper-ethylenediamine-thiosulfate system. This system exhibits a broader pH range compared to the copper-ammonia-thiosulfate system, providing better control over leaching pH. These findings are in accordance with literature reports, with a gold leaching rate of 83.14% achieved within this pH range [5]. To investigate the influence of other factors on gold dissolution, a pH of 10 was selected for subsequent experiments.

$$Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2, \tag{3}$$

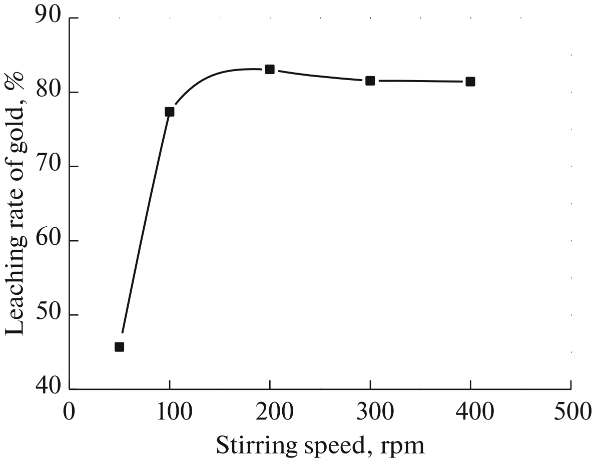

Fig. 2. Effect of stirring speed on the gold leaching rate.

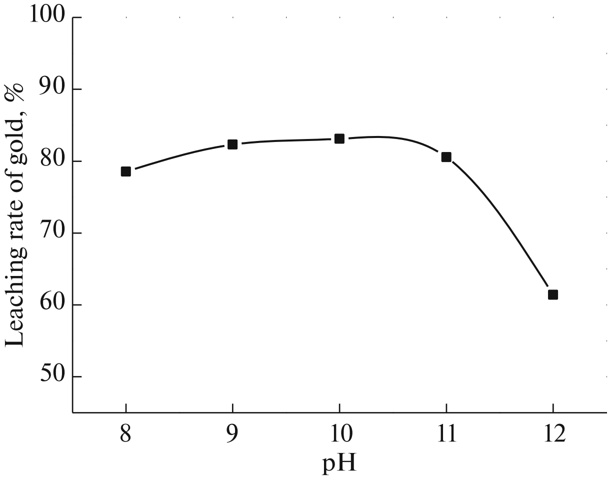

Fig. 3. Effect of pH on the gold leaching rate.

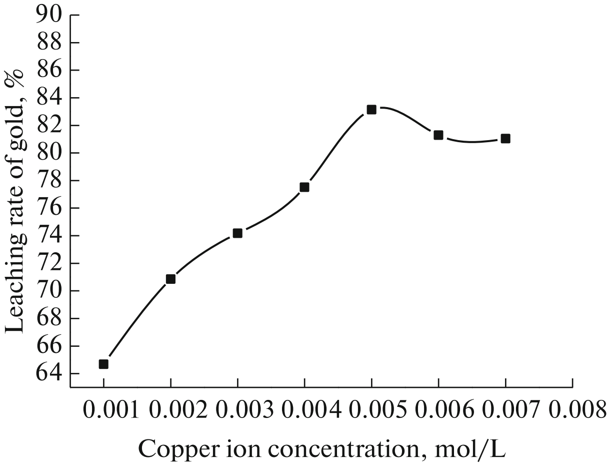

Fig. 4. Effect of copper ion concentration on the gold leaching rate.

$$Cu^{2+} + 4OH^{-} \rightarrow Cu(OH)_4^{2-}, \quad (4)$$

$$Cu(en)_2^{2+} + OH^{-} \rightarrow Cu(OH)_2 + 2en, \quad (5)$$

$$3S_2O_3^{2-} + 6OH^{-} = 4SO_3^{2-} + 2S^{2-} + 3H_2O. \quad (6)$$

Copper Ion Concentration

The influence of copper ion concentration on the gold leaching rate was investigated under the specified conditions (Fig. 4): thiosulfate concentration of 0.2 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid/solid ratio of 3 : 1; pH value of 10; leaching time of 4 h; and stirring speed of 200 rpm. With an increase in copper ion concentration from 0.001 to 0.005 mol L^{-1}, the gold leaching rate exhibited a corresponding rise from 64.67 to 82.33%. A peak in the gold leaching rate was observed within the range of 0.005–0.007 mol L^{-1} copper ion concentration, making approximately 0.005 mol L^{-1} the most suitable concentration for optimal results. During the actual leaching process, copper ions and ethylenediamine form stable coordination complexes, as demonstrated in Eq. (7). This coordination complexes plays a crucial role in facilitating the dissolution of gold. Specifically, when the molar ratio of ethylenediamine to copper ions is maintained at 2, the leaching process exhibits enhanced gold dissolution. Increasing the concentration of copper ions contributes to improved mixing potential within the copper-ethylenediamine-thiosulfate system. It is important to note, however, that excessive copper ions have a pronounced impact on thiosulfate decomposition, which is the primary factor contributing to the higher consumption of thiosulfate in the gold leaching process [8].

When the concentration of copper ions is 0.001 mol L^{-1}, the leaching rate of gold is only 36.3% in the copper-ammonia-thiosulfate system. A lower copper ion concentration is required in the copper-ethylenediamine-thiosulfate system compared to the copper-ammonia-thiosulfate system [9].

$$Cu^{2+} + 2en \rightarrow Cu(en)_2^{2+}. \quad (7)$$

Ethylenediamine Concentration

The impact of ethylenediamine concentration on the leaching rate of gold was also examined in this study, as illustrated in Fig. 5. The experimental conditions involved a thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; liquid/solid ratio of 3 : 1; pH value of 10; leaching time of 4 h; and stirring speed of 200 rpm. Notably, the gold leaching rate exhibited a gradual enhancement corresponding to the incremental ethylenediamine concentrations, peaking when the ethylenediamine concentration reached 0.01 mol L^{-1}. This observation underscores the significance of ethylenediamine in influencing the efficiency of gold leaching in the system under investigation. Given that the primary role of ethylenediamine is to stabilize copper ions within the copper-ethylenediamine-thiosulfate system, it becomes imperative to meticulously control the concentration of ethylenediamine. In instances where the ethylenediamine concentration is inadequate for the formation of stable complexes with copper ions, some of the copper ions may precipitate as copper hydroxide, consequently diminishing the concentration of copper ions in the solution. This decrement in copper ions ultimately hampers the mixing potential of the leaching solution, thereby leading to a discernible reduction in the dissolution rate of gold.

Thiosulfate Concentration

In order to investigate the influence of sodium thiosulfate solution concentration on the rate of gold leaching, the experimental parameters were set as follows (Fig. 6): copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid/solid ratio of 3 : 1; pH value of 10; leaching time of 4 h and stirring speed of 200 rpm. As the concentration of sodium thiosulfate was raised from 0.05 to 0.4 mol L^{-1}, there was a corresponding increase in the leaching rate from 53.54 to 82.05%.

However, further increasing the thiosulfate concentration to 0.5 mol L^{-1} resulted in a decrease in the gold leaching rate to 81.27%. This indicates that a moderate increase in thiosulfate concentration can enhance gold leaching efficiency [10]. Nonetheless, excessive thiosulfate not only leads to thiosulfate consumption but also significantly reduces the concentration of $Cu(en)_2^{2+}$, causing a drop in system potential. Additionally, the decomposition products of thiosulfate play a crucial role in the passivation of the gold surface. Therefore, excessive thiosulfate may lead to gold passivation in solutions containing copper, ethylenediamine, and thiosulfate. Moreover, excessive thiosulfate easily decomposes, generating sulfate, sulfide, and polythionate. This not only increases thiosulfate consumption but also affects subsequent gold recovery due to the formation of polysulfate and sulfide compounds [10, 11].

Based on these findings, we determined that the highest gold leaching rate was achieved with a thiosulfate concentration of 0.2 mol L^{-1}, which was consequently selected as the appropriate concentration of sodium thiosulfate (Fig. 5).

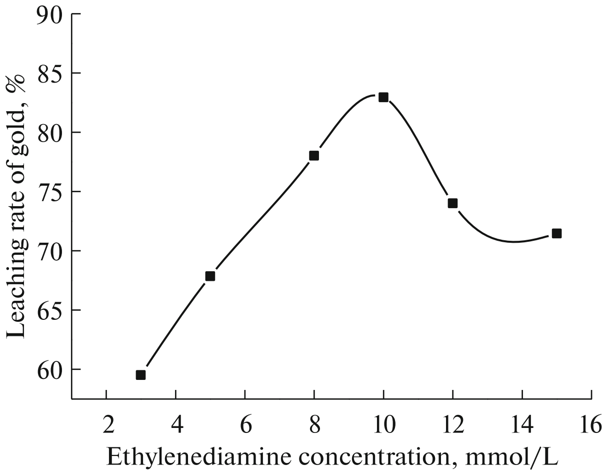

Fig. 5. Effect of ethylenediamine concentration on the gold leaching rate.

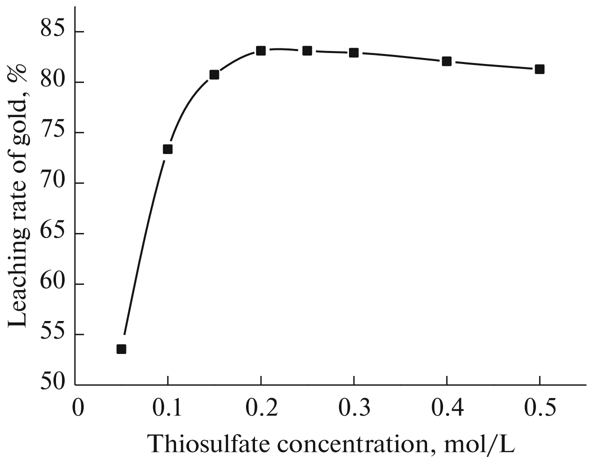

Fig. 6. Effect of thiosulfate concentration on the gold leaching rate.

Leaching Time

We systematically investigated the impact of leaching time on the rate of gold leaching under specific leaching conditions (Fig. 7). These conditions included thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid-to-solid ratio of 3 : 1; pH value of 10; and stirring speed of 200 rpm. We observed that the gold leaching rate remained stable and exhibited no significant changes after 4 h. Consequently, we selected 4 h as the optimal leaching time. By conducting the aforementioned single-factor analysis, we determined the optimal leaching conditions: sodium thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid-to-solid ratio of 3 : 1; stirring speed of 200 rpm; pH value of 10 and leaching time of 4 h. Under these conditions, the gold leaching rate of the ore samples reached 83.19%.

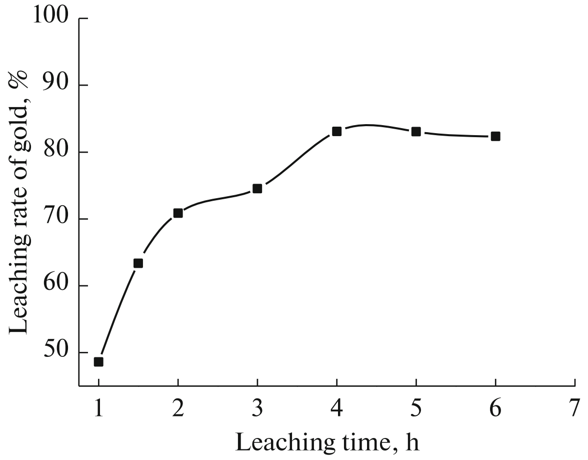

Fig. 7. Effect of leaching time on the gold leaching rate.

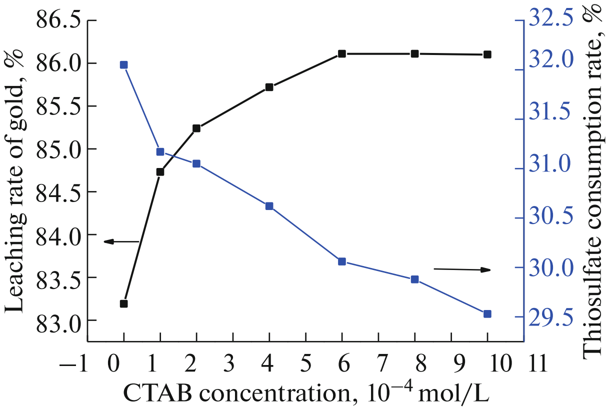

Fig. 8. Effect of CTAB concentration on gold leaching rate and thiosulfate consumption rate.

Cetyltriethylammonium Bromide (CTAB)

CTAB is a cationic surfactant widely used in hydrometallurgy that can improve the surface activity of mineral particles and the adsorption of ore particles to leaching agents. At the same time, the addition of CTAB can significantly reduce the surface tension of the leachate and improve the wettability of the surface of the ore particles.

The effect of CTAB addition on gold leaching rate was investigated under the following leaching conditions and was observed to significantly improve the gold leaching rate (Fig. 8): thiosulfate concentration of 0.2 mol L^{-1}; copper ion concentration of 0.005 mol L^{-1}; ethylenediamine concentration of 0.01 mol L^{-1}; liquid/solid ratio of 3 : 1; pH value of 10 and leaching time of 4 h. When the CTAB concentration was 6×10^{-4} mol L^{-1}, the leaching rate of gold reached a maximum value and then remained basically unchanged. One possible reason for this improvement could be the hydrolysis of CTAB to [CTA^+] and the subsequent attraction of negatively charged mucilage, resulting in changes in the slurry rheology. CTAB causes the gold surface to be exposed, increasing its accessibility to the leaching agent and promoting gold leaching. In addition, the consumption rate of thiosulfate decreased significantly (Fig. 8). CTAB is beneficial to the diffusion of the leaching solution on the gold ore surface, increasing the flow rate of the leaching solution on this surface and accelerating the mass transfer process. The flow rate of the solution will reduce the thickness of the liquid film on the surface, causing the convective diffusion to increase. The above reasons are conducive to the full reaction of various substances in the leaching solution with gold, reducing their interaction and thereby reducing the consumption of thiosulfate. The literature shows that the system has less leaching of other components. Other ions have less influence on the leaching process [13, 14].

Morphology Analysis before and after Leaching

The characterization and comparison of the ore surface before and after leaching was subsequently performed (Fig. 9). After leaching, corrosion points appeared on the surface of the ore sample, the surface of the ore became uneven, and the edges and corners of the flaky ore sample became smoother. This indicated that the ore sample had been severely corroded and that the mineral sample had been leached well in this system.

Cyanidation

Flotation and gravity separation exploratory tests were carried out on the ore samples but could not obtain an ideal beneficiation effect. Therefore, it is not necessary to set up such operations before cyanida-

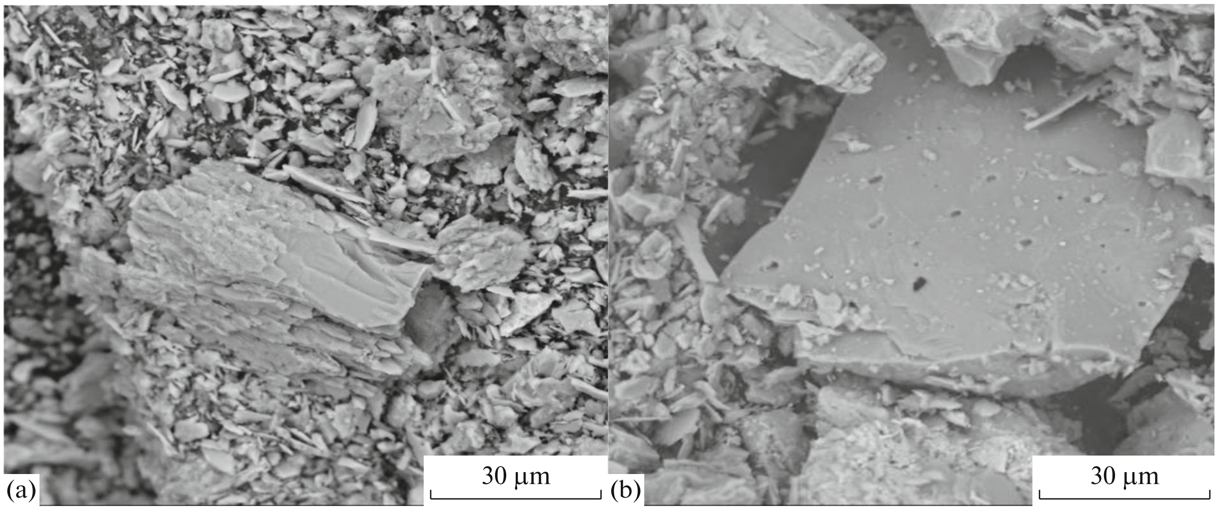

Fig. 9. Scanning electron microscope images of the copper-ethylenediamine-thiosulfate system (a) before and (b) after leaching.

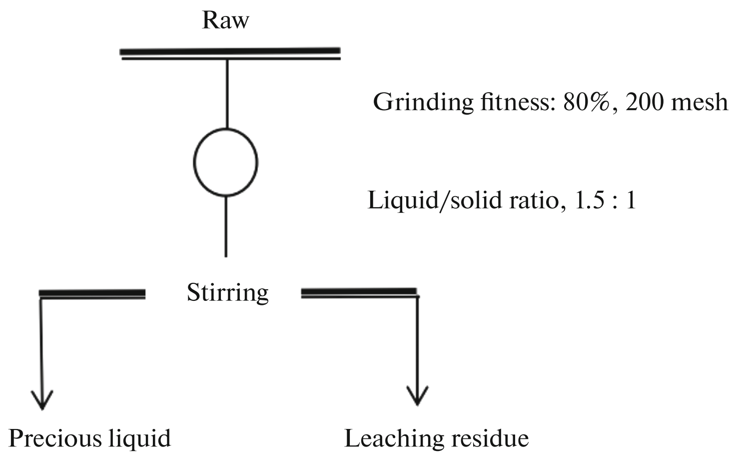

Fig. 10. Cyanidation process and leaching conditions.

tion. In view of the mineralogy characteristics of the raw ore process, and based on the abovementioned flotation and gravity separation exploratory test results, a single gold ore was used to extract gold by cyanidation with full mud. A full-slime cyanidation leaching test was carried out on the ore sample, and the optimal conditions were the following: grinding to 80%, 200 mesh; sodium cyanide dosage, 1 kg t^{-1}; and amount of lime, 8 kg t^{-1}. Under these conditions, the leaching rate of gold in 24 h via stirring cyanidation leaching reached 91.61%. The cyanidation process and conditions are shown in Fig. 10.

Compared with the cyanidation method, The leaching rate using thiosulfate is not as high as that of the cyanidation method, but the leaching time is much shorter and the approach is more environmentally friendly [14–16]. In all, many factors affect the leaching rate in copper-ethylenediamine-thiosulfate solutions, and it is thus necessary to strictly control the leaching conditions [17–19].

CONCLUSIONS

(1) A gold mine sample from Laos underwent leaching, and the optimal leaching conditions were determined via single-factor experimentation: thiosulfate concentration of 0.2 mol L^{-1}, copper ion concentration of 0.005 mol L^{-1}, ethylenediamine concentration of 0.01 mol L^{-1}, pH value of 10, leaching time of 4 h, and stirring speed of 200 rpm.

(2) Furthermore, the ore sample has a good leaching effect in the system, which is conducive to the subsequent recovery of gold from the leaching solution.

(3) The addition of CTAB can increase the leaching rate of gold and reduce the consumption rate of thiosulfate.

(4) Finally, the leaching time using the copper-ethylenediamine-thiosulfate system is shorter than that of the cyanidation method. The leaching rate of thiosulfate is not as high as that of cyanidation, but its use is more environmentally friendly.

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CONFLICT OF INTEREST

The author of this work declare that they have no conflicts of interest.

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