

Review

Pretreatment and Extraction of Gold from Refractory Gold Ore in Acidic Conditions

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Abstract: As high-grade gold deposits are progressively depleted, the proportion of refractory gold ores in total reserves is continuously increasing, making gold recovery from refractory ores an inevitable trend in the future development of the gold industry. This study briefly analyzes the challenges faced during the leaching process of refractory gold ores under ambient conditions, and provides a detailed discussion on two acidic pretreatment technologies—pressure oxidation and bio-oxidation—as well as three acidic gold recovery technologies—thiosulfate leaching process, halogen leaching process, and thio-cyanate leaching process. Additionally, this paper compares and analyzes the advantages and limitations of these acidic pretreatment and hydrometallurgical gold recovery technologies. The goal is to provide a comprehensive review of pretreatment technologies and leaching agents for refractory gold ores under acidic conditions (pH = 1–5), offering a reference for selecting appropriate treatment processes in the future, and to explore the potential development of acidic pretreatment and recovery technologies for refractory gold ores.

Keywords: refractory gold ores; acidic processing; pretreatment and leaching



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1. Introduction

Gold, as a strategic metal, not only symbolize wealth but is an important material for scientific research. With technological advancements and human societal development, the demand for gold is constantly increasing, while high-quality gold reserves are becoming increasingly scarce, making the development and utilization of refractory gold ores a research focus.

Several challenges arise in processing refractory gold ores. The first challenge is the presence of sulfide minerals, such as arsenopyrite and pyrite, which lock gold within their structures [1]. Conventional grinding processes struggle to break down the encapsulation of sulfide minerals, hindering the interaction between lixiviants and gold, and thus complicating the gold extraction process. In addition, sulfide minerals, like pyrite and siderite, consume oxygen, alkali, and cyanide in the leach solution during cyanidation [2], reducing the leaching efficiency; minerals, like chalcopyrite, limonite, and sphalerite, will compete with gold and generate inhibitory substances, such as sulfuric acid [3–5]. Bismuthinite forms galvanic cells with gold, leading to gold passivation. Likewise, gangue minerals, such as clay [6], carbon [7,8] and highly active graphite, are present in the ore, which have a preg-robbing behavior that can adsorb the leached gold, reducing the gold recovery.

Applying appropriate pretreatment techniques and gold leaching processes to different types of refractory gold ores can effectively increase gold recovery efficiency [9].

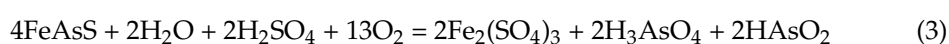
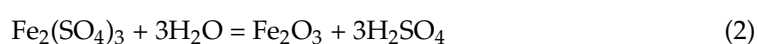
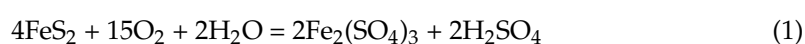
Numerous scholars have been committed to enhancing the efficiency of gold extraction from refractory gold ores. To this end, they have conducted extensive research on the pretreatment technologies and cyanidation processes for refractory gold ores. These studies aim to optimize the gold recovery process, thereby achieving the efficient utilization of resources and sustainable environmental development [10]. In the current field of refractory gold ore processing, the classification of pretreatment technologies and leaching processes can be based on the acid-base environment during the treatment process. Specifically, these technologies can be systematically categorized according to their acidic or alkaline characteristics exhibited during the application. This classification process aids researchers in selecting the appropriate pretreatment and leaching technologies based on specific chemical properties and reaction conditions to achieve optimal processing outcomes. In the gold industry, the pretreatment–cyanidation process is widely adopted [11], with acid-base conversion being a necessary step following acidic pretreatment methods such as bio-oxidation and pressure oxidation. Acid-base conversion neutralizes the acids generated during pretreatment, thereby facilitating subsequent alkaline cyanidation. However, this conversion process also introduces some challenges in the following gold extraction stages. Firstly, excessive neutralization can lead to the formation of unwanted precipitates, such as calcium sulfate or iron hydroxide, which may clog equipment and reduce the efficiency of the extraction process [12]. Secondly, the high consumption of caustic agents during acid-base conversion increases operational costs, making the process less economically viable. Additionally, the pH adjustment may alter the chemical properties of the leach solution, affecting the solubility of gold and the stability of reagents, which can lower extraction efficiency [13,14].

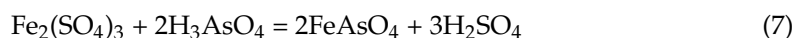
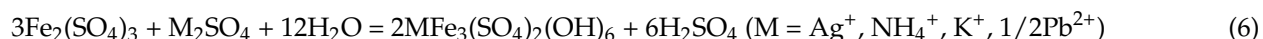
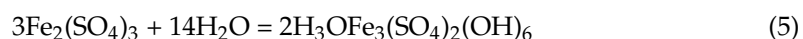
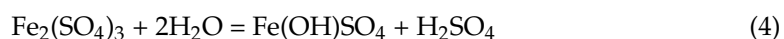
Building on the above discussion, the development of fully acidic pretreatment and gold extraction processes is gaining increasing attention. The purpose of this article is to review existing acidic pretreatment technologies and acidic gold leaching methods, as well as to discuss recent advancements in these processes. Common acidic pretreatment techniques include acidic pressure oxidation (180–225 °C) and acidic bio-oxidation (15–25 °C). Non-cyanide acidic leaching processes, such as the thiosulfate, chloride, and thiocyanate processes, are also widely studied. Additionally, our research group has made significant progress in thiocyanate leaching, particularly through the use of cyclic gold extraction methods.

2. Pretreatment in Acidic Condition

2.1. Pressure Oxidation

The acidic pressure oxidation process is conducted under high temperature, high pressure, and oxygen-rich conditions (the oxygen partial pressure is generally controlled at 350–700 kPa, and oxygen is commonly used as the oxidant). The ore is ground before being sent into a reaction vessel [12,15], where the encapsulation undergoes a series of physical and chemical reactions in an acidic medium, exposing the gold. This process is mainly used to treat acidic or weakly alkaline gold ores, when the encapsulation is a sulfide, such as pyrite, arsenopyrite, or chalcopyrite. When the encapsulation is pyrite or arsenopyrite, the following reactions occur: sulfur, arsenic, and iron mainly enter the solution, while gold is exposed and retained in the residue. The main reactions are as follows:





As a recently developed pretreatment process for refractory gold ores, the pressure oxidation process (POX) offers significant advantages over traditional oxidative roasting techniques by effectively mitigating the environmental issues associated with the release of sulfur dioxide and arsenic oxides [16,17]. Acidic pressure oxidation has garnered considerable research attention and exhibits promising development prospects. This process achieves superior treatment efficiency when the associated minerals are sulfides, such as pyrite and pyrrhotite. In the processing of gold–silver associated ores, the acidic pressure oxidation process is also conducive to the liberation of silver [18]. Furthermore, this technology facilitates the comprehensive recovery of metals, such as copper and zinc, from the ore, thereby achieving efficient resource utilization [19–21].

Gudyanga et al. [22] applied acidic pressure oxidation to refractory gold ores in Zimbabwe. In Zimbabwe, roasting is the main process for treating refractory gold ores before cyanidation, but it only achieves a 75% gold recovery rate. Acidic pressure oxidation in an autoclave offers a higher recovery rate (>90%) and rapid silver recovery (62% in 12 min). These processes depend on factors like temperature, time, pulp density, and oxygen pressure. At Kwekwe's Roasting Plant, pressure leaching is viable, with most gold associated with pyrite, requiring complete oxidation for liberation.

Li et al. [23] studied the gold concentrate before and after acidic pressure oxidation. Under the conditions of 0.5 mol/L thiosulfate ions, 0.03 mol/L copper ions, and 0.50 mmol/L humic acid, leaching experiments were conducted on gold concentrates with and without acidic pressure oxidation. The results indicated that the pre-oxidation process significantly enhanced the leaching efficiency, achieving a gold extraction rate of approximately 86.21%, compared to a direct leaching gold extraction rate of only 21.22%. This outcome suggests that the mineral crystal structure was disrupted, releasing gold that was originally encapsulated within sulfides, thereby enabling the gold to react with the leaching agents and dissolve into the solution, which in turn increased the gold recovery.

Soleymani et al. [24] adjusted the ratio of acidic to alkaline pressure oxidation products in order to achieve the maximum gold recovery rate in the subsequent thiosulfate gold leaching process. Contrary to the conclusions drawn from electrochemical results, the experimental results indicated that the alkaline pressure oxidation method is more conducive to the dissolution of gold, while the acidic pressure oxidation method is more favorable for the subsequent gold leaching.

Zhang et al. [25] proposed a high-pressure oxidation–sodium jarosite decomposition–polythionide leaching method for gold recovery from typical Carlin-type gold concentrate (the gold content is 40.0 g/t). The acidic high-pressure oxidation pre-treatment process can effectively open the host matrix of gold; the exposed gold increased from 4.23% to 80.4%. Under the most suitable conditions, the gold leaching efficiency increased to 90.2%, which is 8.4% higher than that of the direct cyanidation of oxygen pressure leach residue. Figure 1 illustrates the process flow of the leaching system. The flotation concentrate is subjected to pre-acidification, resulting in an oxidized residue after POX which follows the jarosite decomposition treatment. Finally, gold is extracted through PSR leaching, yielding a gold-bearing solution.

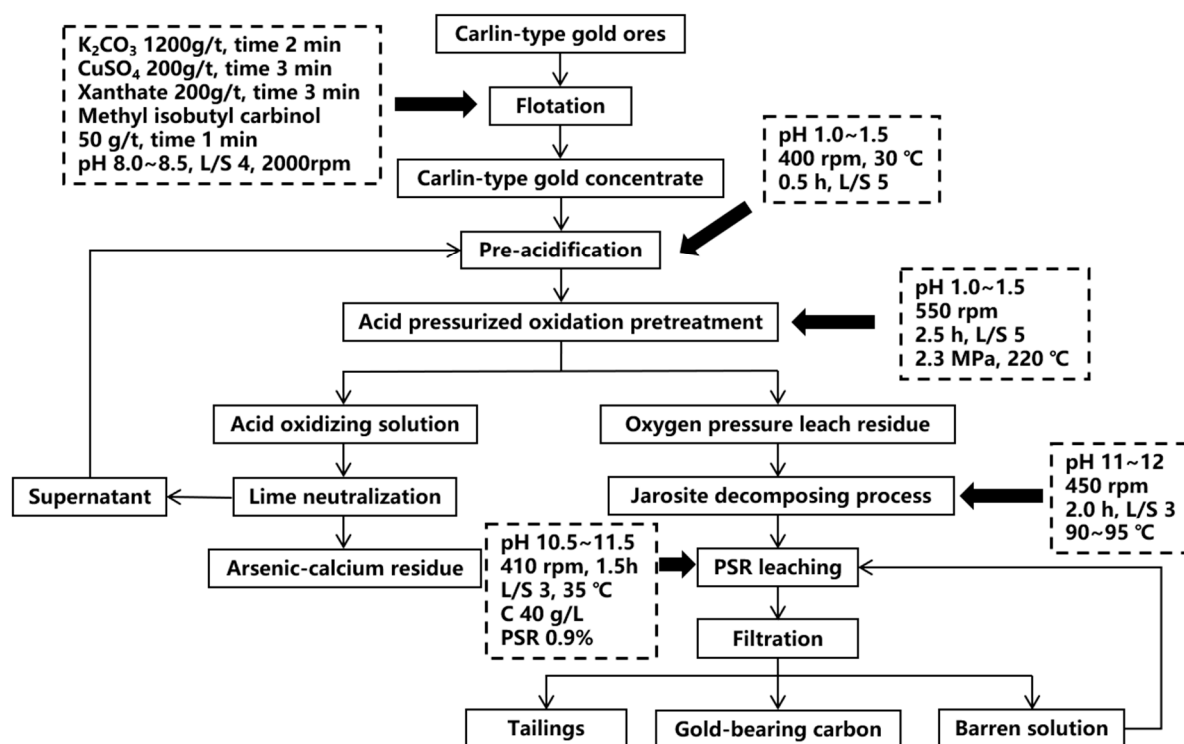


Figure 1. Schematic diagram of pressure oxidation–sodium jarosite decomposing and non-cyanide extraction of gold from polysulfides in typical Carlin-type gold ores [25].

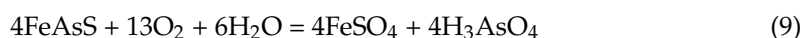
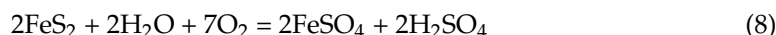
The acidic pressure oxidation process sets high demands for engineering implementation, production operations, and management, as the process requires equipment capable of withstanding extremely high temperatures and pressures, resulting in higher costs in large-scale industrial production. The application range of pressure leaching is relatively broad, with high leaching recovery and minimal environmental pollution. However, it requires equipment with high corrosion resistance, leading to higher maintenance costs, which limits the use of this process. Enhancing equipment corrosion resistance and reducing equipment investment are key to promoting the adoption of the pressure leaching process.

2.2. Bio-Oxidation

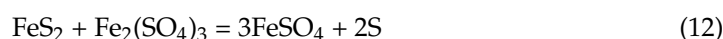
Bio-oxidation is a pretreatment process that involves the oxidation and decomposition of encapsulation under aerobic conditions through the action of microorganisms that destroy the encapsulation and exposes the encapsulated gold [26]. The microbial strains currently used in industrial production include *Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans*, *Acidithiobacillus ferrivorans*, *Leptospirillum ferrooxidans*, *Sulfolobus species*, and *White-rot fungi*, among others [27]. Bio-oxidation is an economical and environmentally friendly pre-treatment process that has been widely used to handle complex refractory gold ores and has become an irreplaceable process for pre-treating such low-grade gold concentrates [28–30]. Compared to the high costs of pressure oxidation, bio-oxidation is more cost-effective, making it economically more suitable for treating high-sulfur, high-arsenic, low-grade refractory gold ores [31–34]. At the same time, this pretreatment can decrease the impact of carbonaceous matter on gold leaching recovery in double refractory gold ores [35–37].

Bio-oxidation mainly has two mechanisms: direct action and indirect action. Bio-oxidation primarily promote the oxidation of the ore through indirect actions, such as providing Fe^{3+} and sulfate ions, thereby enhancing gold recovery [38]. Regarding direct action, it is thought that microorganisms directly oxidize sulfides or metal sulfides in the ore

through the action of enzymes (sulfur oxidase or iron oxidase) on their cell membranes or through the action of extracellular polymeric substances (EPS) [39–41]. The main reactions are as follows:



In the indirect mechanism of the bio-oxidation process, ferrous ions (Fe^{2+}) in the solution are oxidized to ferric ions (Fe^{3+}) by the metabolic activity of the microorganisms. Subsequently, these ferric ions (Fe^{3+}) react with sulfides in the minerals and are reduced back to ferrous ions (Fe^{2+}), during which the sulfides are oxidized, leading to the release of sulfur into the solution. When the encapsulated minerals are primarily composed of FeAsS and FeS_2 , the main chemical reactions occurring during the oxidation process are as follows: sulfur, arsenic, and iron mainly enter the solution, while gold is retained in the residue. The main reactions are as follows:



Fomchenko et al. [42] conducted comparative experiments employing single-stage and two-stage protocols for the bio-oxidation of gold-bearing arsenopyrite and pyrite concentrates, thereby verifying that bio-oxidation primarily functions through indirect actions. In the experiments, the single-stage protocol directly conducted bio-oxidation, whereas the two-stage protocol initially employed ferric (Fe^{3+}) solutions produced by microorganisms for chemical oxidation, followed by bio-oxidation. The results indicated that the two-stage protocol significantly enhanced oxidation efficiency and gold recovery rates.

In the context of bio-oxidation processes, the pH of the solution is typically acidic. This is attributed to the fact that certain microorganisms, such as *Acidithiobacillus ferrooxidans*, exhibit enhanced efficacy in oxidizing Fe^{2+} to Fe^{3+} and oxidizing sulfur to SO_4^{2-} under acidic conditions, with optimal performance occurring at a pH range of 2.0 to 3.0. Furthermore, a lower pH value is conducive to maintaining the solubility and bioavailability of iron ions, thereby augmenting the efficiency of the bio-oxidation process [43,44].

Amankwah et al. [45] conducted a two-stage pretreatment on gold ores with high sulfur and high carbon content. In the first stage, after desulfurization by bacteria such as sulfur-oxidizing bacteria, *Acidithiobacillus ferrooxidans*, and *Leptospirillum ferriphilum*, the gold recovery rate was 81.1%. In the second stage, after decarbonization treatment with *Streptomyces setonii*, the gold recovery rate increased to 94.7%. Fomchenko et al. [46] proposed a two-stage treatment scheme, which involves pre-oxidizing the leachate containing trivalent biogenic iron after treatment with *Acidithiobacillus ferrooxidans*, *Leptospirillum ferriphilum*, and thermophilic sulfur-oxidizing bacteria, followed by treatment of the pre-oxidized minerals with sulfur bacteria. Compared to the one-stage treatment method, the two-stage treatment method has a kinetic that is 1.5 to 2 times faster, and achieves a higher gold extraction rate, increasing from 82.4% in the one-stage method to 94.1%.

Xu et al. [47] investigated the effectiveness of pre-bio-oxidation in decomposing sulfides and deactivating carbonaceous matter in double refractory gold ore (DRGO) using specific bacteria. Direct thiourea leaching of untreated double refractory gold ore resulted in only a 28.7% gold recovery rate, due to sulfide encapsulation and carbon adsorption.

However, after bio-oxidation, thiourea leaching improved the gold extraction rate to over 75%–80%, with bio-oxidation also reducing the carbon's affinity for gold.

Boduen et al. [48] suggested a dual approach of bacterial oxidation and pressure oxidation for high-sulfur refractory gold concentrate treatment. The first group of gold concentrate was subjected to direct cyanidation leaching. The second one was leached after STRB. The third one was leached after POX. The fourth group was leached after STRB followed by POX. Leaching residues and gold-bearing leaching solutions were obtained for each group. Using a pyrite–arsenopyrite concentrate as a model, cyanidation of untreated material achieved a 58% gold recovery rate. Bio-oxidation for 2, 4, and 6 days resulted in 43%, 74%, and 79% sulfide sulfur oxidation, with gold recovery rates of 68%, 82%, and 88%, respectively. Pressure oxidation further increased sulfide sulfur oxidation to 97%–99% and the gold recovery rate to 96%–97%. Two days of bio-oxidation reduced sulfide sulfur and improved the liquid-to-solid ratio for subsequent pressure oxidation. Figures 2 and 3 show the flowchart of the process studied in the present work.

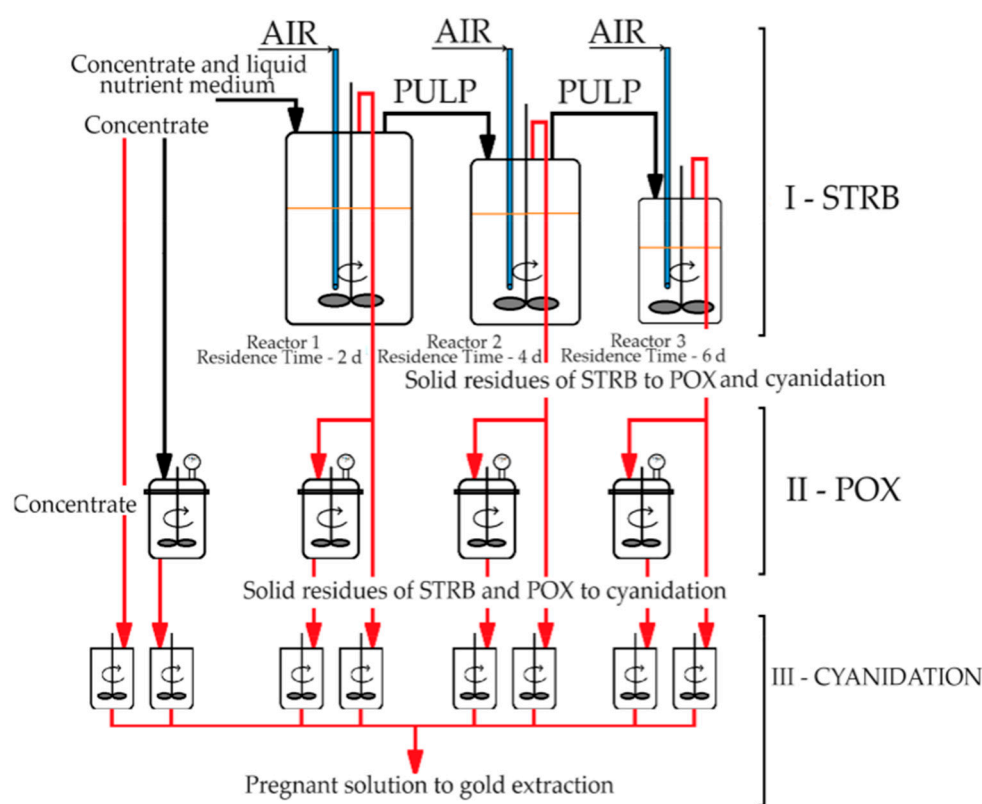


Figure 2. Flowsheet of the process studied in Boduen's present work, the effectiveness of direct cyanidation leaching of concentrate, cyanidation leaching following individual bacterial oxidation, cyanidation leaching following individual pressure oxidation, and cyanidation leaching following combined bacterial and pressure oxidation was compared [48].

Zhang et al. [49] proposed a two-stage oxidation process to treat low-grade refractory gold ore with high arsenic and sulfur, aiming to reuse ferric iron from bio-oxidation waste. The process involves secondary high-temperature oxidation followed by bio-oxidation. Analysis reveals that this approach increases the particle surface area and nitrogen content, which enhances microbial adsorption and growth. As a result, the activity of iron-oxidizing bacteria improves, significantly boosting the efficiency of bio-oxidation. Element extraction and cyanidation yields for gold and silver are substantially higher compared to single-stage bio-oxidation. Figure 4 presents the schematic diagram of the two-stage oxidation process for refractory gold concentrate with high arsenic and sulfur content. Figure 4

presents a schematic diagram of the two-stage oxidation process for treating refractory gold concentrate with high arsenic (45.0%) and sulfur (12.5%) content.

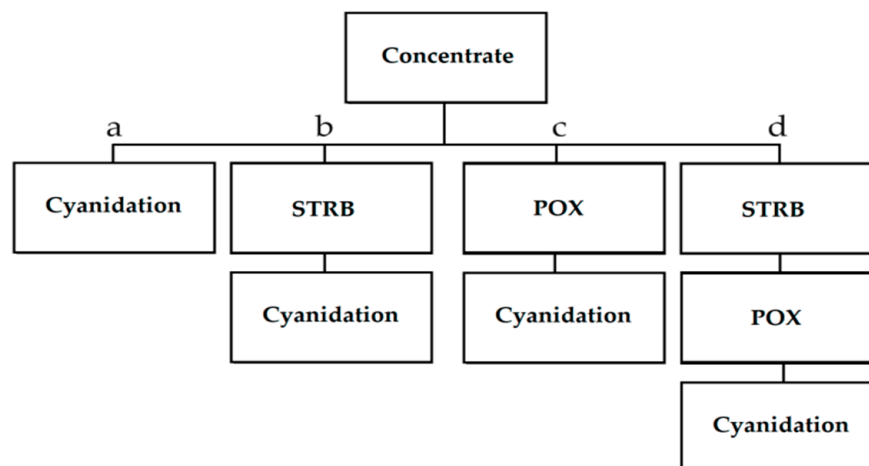


Figure 3. Variants of the experiment: (a) cyanidation of the initial concentrate; (b) STRB of the concentrate and cyanidation; (c) POX of the concentrate and cyanidation; and (d) combined oxidative pretreatment of the concentrate (STRB and POX) and cyanidation [48].

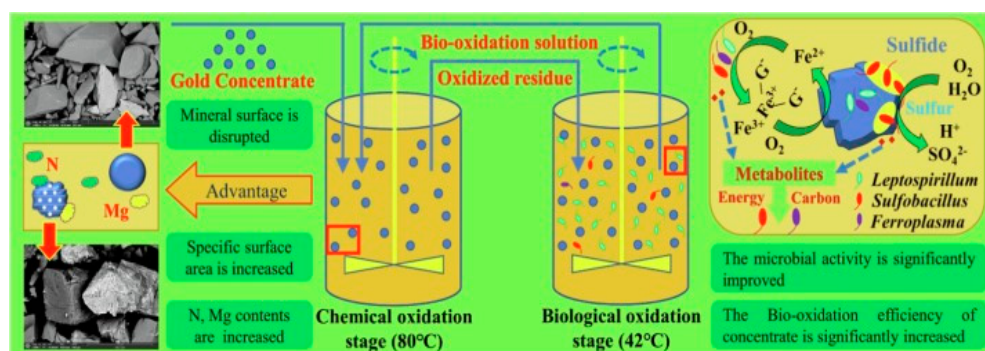


Figure 4. The schematic diagram of two-stage oxidation process for refractory gold concentrate with high arsenic and sulfur [49].

Ahn et al. [32,50] compared the Sand Farming process with conventional tank bio-oxidation, focusing on the differences in gold recovery. The Sand Farming process achieved a gold recovery rate of 75% from high-grade ores with high sulfur content and 68% from low-grade ores with low sulfur content, slightly lower than the 83% and 85% recovery rate achieved by tank bio-oxidation, respectively. The experimental results suggest that, due to its more environmentally friendly nature and better economic benefits, the Sand Farming process is a highly promising bio-oxidation method for treating sulfides in refractory gold ores, offering advantages over pressure oxidation, roasting oxidation, and traditional tank bio-oxidation.

As an emerging pretreatment technology for gold ores, bio-oxidation has the advantages of low cost, low pollution, low energy consumption, high gold extraction, simple processes, and easy operation [29,51]. The disadvantages include a high pulp density that can reduce processing efficiency, the treatment time is significantly affected by the growth of the microorganisms, and selectivity is greatly impacted [52–54]. Overall, the bio-oxidation process has relatively mild reaction conditions, which are not only easier to achieve in actual production processes but provide a safer production environment, demonstrating strong potential for application.

Table 1 summarizes the characteristics, advantages, and disadvantages of the two gold ore pretreatment technologies mentioned above. It can be seen that both pretreatment technologies have their place in the processing of refractory gold ores.

Table 1. Comparison of the acidic pretreatment for refractory gold ores.

Pretreatment	Advantages	Disadvantages
Pressure oxidation	high sulfide oxidation degree fast kinetics wide applicability less pollution	complex process high CapEx and OpEx high corrosion resistance from equipment
Bio-oxidation	wide applicability less pollution low cost simple process	High construction costs demanding requirements for processing conditions.

3. Gold Extraction in Acidic Condition

In the current field of gold extraction, cyanidation remains the predominant technique [55–57]. However, cyanide is prone to hydrolysis in the pulp, producing highly toxic hydrogen cyanide (HCN), which can volatilize from the solution, leading to the loss of cyanide and environmental pollution [9,58–63]. To maintain the chemical stability of cyanide, it is essential to maintain an appropriate alkalinity in the solution during the cyanidation process to inhibit the hydrolysis of cyanide [64–66]. With the growing awareness of environmental protection, coupled with the potential for serious environmental issues arising from improper handling of cyanide tailings, researchers continue to seek non-cyanide gold leaching technologies. Figure 5 and Table 2 lists some of the alternative leaching agents that may replace cyanide. The subsequent sections of this paper will focus on gold leaching methods under acidic conditions, involving alternative leaching agents such as thiourea, halogens, and thiocyanates.

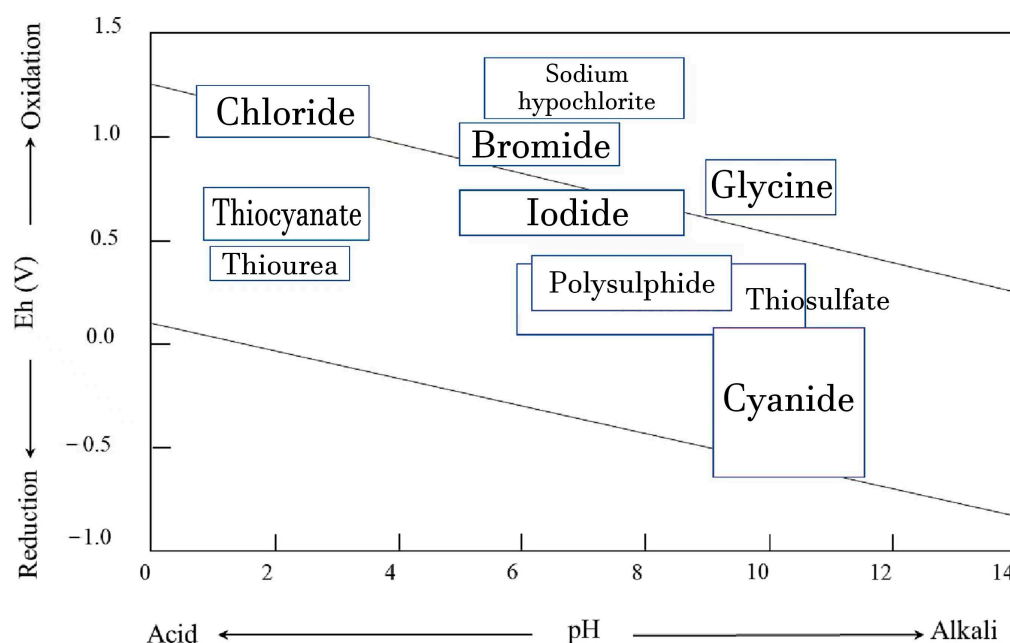


Figure 5. Eh-pH range of each complex forms [67].

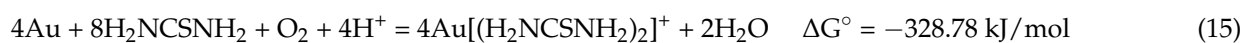
Table 2. Suggested alternatives to cyanide [13].

Reagent Type	Concentration Range	pH Range	Basic Chemistry	Research Level	Extent of Commercialization
Ammonia	High	8–10	Simple	Low	Pilot tests, +100 °C
Ammonia/cyanide	Low	9–11	Simple	Extensive	Applied to Cu/Au ores
Ammonium thiosulfate	High	8.5–9.5	Complex	Extensive	Semi-commercial
Slurry CN-electrolysis	Low	9–11	Simple	Historical	Limited historical
Sodium sulfide	High	8–10	Simple	Low	Geological interest only
Alpha-hydroxynitriles	Moderate	7–8	Fairly simple	Fairly popular	None
Malononitrile	Moderate	8–9	Fairly complex	Low	None
Alkali cyanoform	Poorly defined	~9	Poorly defined	Low	None
Calcium cyanide	Poorly defined	~9	Poorly defined	Low	None
Alkaline polysulfides	High	8–9	Poorly defined	Low	None
Hypochlorite/chloride	High chloride	6–6.5	Well defined	Extensive	Historical and modern
Bromocyanide	High	6–7	Poorly defined	Historical	Historical
Iodine	High	3–10	Poorly defined	Low	None
Bisulfate/sulfur dioxide	High	4–5	Fairly simple	Low	None
Bacteria	High	7–10	Fairly complex	Low, growing	None
Natural organic acids	High	5–6	Fairly complex	Low	None
DMSO, DMF	Poorly defined	7	Poorly defined	Very low	None
Bromine/bromide	High	1–3	Well defined	Low	Historical
Thiourea	High	1–2	Well defined	Fairly popular	Some concentrates
Thiocyanate	Low	1–3	Well defined	Low	None
Aqua regia	High	Below 1	Well defined	Low	Analytical and refining
Acid ferric chloride	High	Below 1	Well defined	Low	Electrolytic Cu slimes
Ethylene thiourea	High	1–2	Poorly defined	Very low	None
Haber process	Poorly defined		Proprietary	One entity	None
Bio-D leachant	Poorly defined		Proprietary	One entity	None
High temperature chlorination	High	6–7	Simple	Historical	Historical

3.1. Thiourea Leaching Process

Thiourea, with the chemical formula H_2NCSNH_2 , is an organic complexing agent that possesses strong reducing properties and a strong complexing ability with metal ions. Thiourea leaching can be divided into acidic thiourea leaching and alkaline thiourea leaching, based on the acidity or alkalinity of the medium, with the acidic thiourea leaching being the more maturely developed process at present [68]. Compared to the widely used cyanidation process for gold leaching, this process demonstrates higher gold dissolution

efficiency and has limited impact on the environment [69]. The leaching mechanism of thiourea can be described as follows: gold is oxidized by the oxidizing agent (O_2 or Fe^{3+}) and then forms complexes with thiourea, thereby entering the leaching solution. The main reaction is as follows:



Acidic thiourea leaching typically uses sulfuric acid (0.5 to 1.5 mol/L) as the medium. Under acidic conditions, thiourea is easily oxidized by strong oxidizing agents to form dimethyl disulfide, which is a more reactive oxidizing agent and has a certain auxiliary effect on gold leaching. However, under certain conditions, it can decompose into thiourea, aminocyanide, and elemental sulfur. The decomposition products can cover the mineral surface, causing passivation of gold and hindering subsequent leaching. Therefore, the thiourea process generally requires an acidic environment and uses Fe^{3+} as the oxidizing agent [70,71]. In the thiourea leaching process, ferric sulfate is often chosen as the oxidizing agent, and its presence can significantly enhance the stability of thiourea during the gold leaching [72–74]. Adding sulfide ions to the solution can effectively promote the dissolution of gold [75]. Under acidic conditions, gold is oxidized by the oxidizing agent and then undergoes a complexation reaction with thiourea to form $Au[(H_2NCSNH_2)_2]^{2+}$, thereby achieving selective dissolution of gold. The main reaction is as follows:



In the acidic thiourea gold leaching process, thiourea is oxidized by excess Fe^{3+} to form sulfur. The generated sulfur not only adheres to the mineral surface, preventing the leaching agents from contacting the gold, but adsorbs the complexes formed by the chelation of thiourea with gold, thus affecting the leaching efficiency. This situation can also occur when the leaching temperature is too high or the concentration of thiourea is too high, leading to the decomposition of thiourea and the formation of sulfur. These conditions impose relatively strict requirements on the production environment [76]. To maintain the stability of thiourea, the pH during the leaching process should be kept around 1.0, which has high requirements for the equipment as it can cause significant corrosion. This undoubtedly increases the initial investment costs and subsequent maintenance costs. Under acidic conditions, thiourea has poor selectivity and can effectively dissolve other metals as well, which means that acidic thiourea gold leaching may not perform well when dealing with some complex and refractory gold ores. Li observed that the presence of foreign ions, including Cu^{2+} , Ag^+ , Pb^{2+} , Zn^{2+} , AsO_3^{3-} , and AsO_4^{3-} , exerts a detrimental effect on the gold leaching process in acidic thiourea solutions. Under the influence of these ions, there is a significant increase in the consumption of thiourea, which consequently leads to a marked deterioration in the efficiency of gold dissolution by thiourea [77].

Guo et al. [78] conducted a bio-oxidation two-stage thiosulfate leaching experiment on a refractory gold ore from Xinjiang, achieving a gold recovery rate of 95%. With similar thiosulfate consumption, this method outperforms the one-stage thiosulfate leaching, which has a recovery rate of 92.2%, and does not require the addition of oxidizing or reducing agents. While the efficiency of this method is not as high as that of the one-stage leaching, it does not incur additional costs and is environmentally friendly, making it highly practical for gold extraction. Guo et al. [79] used sodium lignosulfonate, urea, and Fe^{3+} as additives and applied the thiosulfate method to conduct a two-stage leaching study on gold and silver for a refractory gold ore, followed by pilot testing. The results indicated that with a thiosulfate dosage of 8 g/L, direct thiosulfate leaching could achieve a gold recovery rate of 84.42% and a silver recovery rate of 44.15%. When using 0.9 g/L of sodium lignosulfonate, 2 g/L of urea, and 3 g/L of Fe^{3+} , only 6 g/L of thiosulfate was needed to achieve a gold

recovery rate of 88.71% and a silver recovery rate of 52.65%. After mechanical activation, the recovery rate of gold and silver reached 96.51% and 70.43%, respectively. In the pilot tests, the recovery rate of gold and silver reached 96% and 68%, respectively.

Olyaei et al. [80] conducted comparative leaching experiments on the same samples using cyanidation and thiourea leaching. The results showed that, under optimal conditions, the cyanidation process achieved a gold recovery rate of 95.21%, with the specific conditions being d_{80} of 63 μm , a pH of 10.5, cyanide consumption of 1.23 kg/t, and a leaching time of 12 h. By contrast, the acidic thiourea leaching process required more chemical reagents, with a maximum gold recovery rate of 90.48%, and thiourea consumption, leaching time, and a pH of 13.32 kg/t, 5 h, and 1.7, respectively. Additionally, the experiment found an interaction between the concentrations of ferric sulfate and thiourea, with the highest gold recovery rate occurring at a molar ratio of 1:1 between ferric sulfate and thiourea.

Lin et al. [81] employed a gold leaching process that integrates thiosulfate with thiourea. Under the conditions of 0.2 mol/L thiosulfate, 0.12 mol/L thiourea, 5 mmol/L copper ions, 0.6 mol/L ammonia, liquid-to-solid ratio of 3, and temperature of 308 K, the gold extraction efficiency within 2 h was comparable to that of the 24-h cyanidation process. Gold in the leachate primarily existed in the form of gold (I) thiourea complex and gold (I) thiosulfate complex. The addition of thiourea effectively inhibited the formation of a passivation layer and promoted the leaching of gold through synergistic effects, with the rapid leaching rate potentially attributed to this synergism.

Hou et al. [82] proposed a clean gold leaching process using a Fenton oxidation-assisted thiourea system. Figure 6 shows the proposed gold dissolution process in the Fenton/TU system. The optimal conditions are 0.1 mol/L H_2O_2 , 0.05 mol/L Fe^{2+} , 20 g/L thiourea, and a pH 1.5, achieving a 100% leaching recovery rate within 30 min. The addition of nitrilotriacetic acid (NTA) reduced thiourea consumption from 45.6% to 11.84%, lowered the solution potential, and minimized side reactions between Fe^{3+} and thiourea. The introduction of NTA regulated the generation of $\bullet\text{O}_2$, decreased thiourea consumption, and still achieved 100% gold extraction in 60 min.

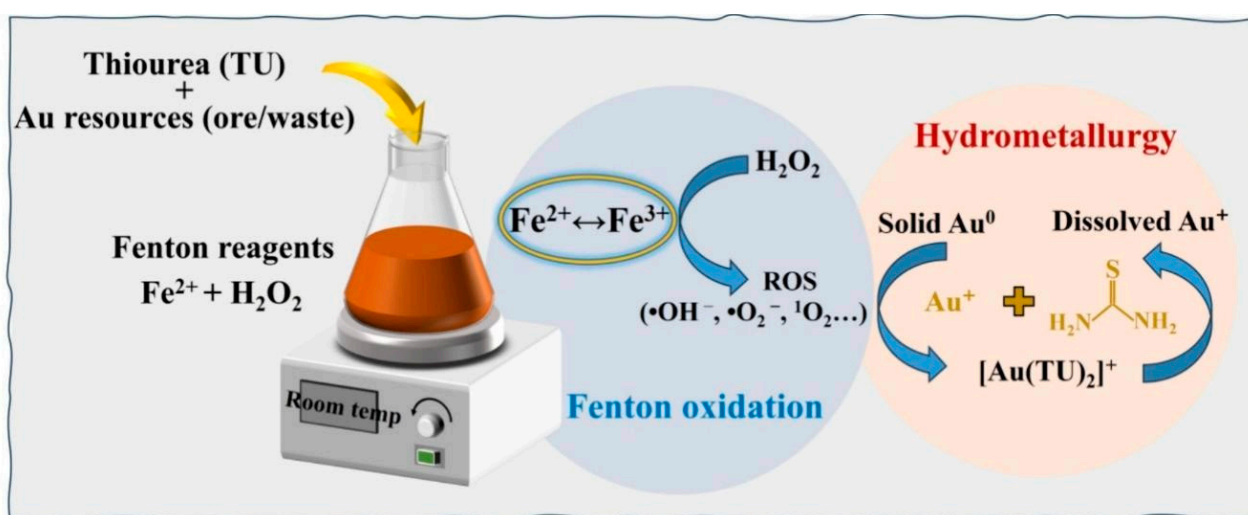


Figure 6. The proposed gold dissolution process in the Fenton/TU system [82].

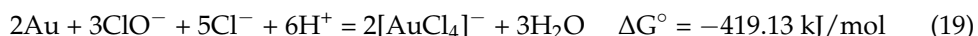
In summary, the acidic thiourea leaching process has the advantages of fast leaching speed and low pollution, but its drawbacks include high costs, high consumption of reagents, and corrosion-resistant equipment [78,83–85]. In the future, a more environmentally friendly and efficient thiourea gold leaching process should be developed, and suitable stabilizers should be screened and optimized.

3.2. Halogen Leaching Process

The halogen leaching process has strong oxidizing properties, and the principle of gold extraction involves the oxidation of gold by halogens, while halogen ions complex with gold in the solution to form soluble complexes, such as $[\text{AuX}_4]^-$ ($\text{X} = \text{Cl}/\text{Br}$), gold migrates from the solid phase to the liquid phase, entering the leaching solution, thereby achieving the separation of gold from minerals, such as sulfate, arsenate, and silicate gels. In acidic environments, the relatively mature process of halogen gold extraction includes chlorination and bromination. Both processes utilize the strong oxidizing nature of halogens to effectively leach gold from gold ores under acidic conditions, forming stable gold halogen complexes.

3.2.1. Chlorination Leaching Process

Gold extraction by the chlorination leaching process is primarily conducted under acidic conditions. In an acidic environment, $[\text{AuCl}_4]^-$ is more stable, which favors the dissolution and leaching of gold. Additionally, the consumption of chlorine is reduced, helping to lower the operating costs associated with chlorine production and ensuring the economic viability of the chlorination process. Therefore, gold extraction by chlorination is typically carried out under acidic conditions to enhance the leaching efficiency and selectivity of gold. In the chlorination process, the oxidizing agents are Cl_2 and hypochlorous acid (HClO) formed by the reaction of Cl_2 with water. Cl^- then complexes with the oxidized gold to form the soluble $[\text{AuCl}_4]^-$, allowing gold to enter the solution. The main reactions are as follows:



The chlorination leaching process for gold extraction has greater reaction kinetics compared to the traditional cyanidation process [86]. Chloride leaching is regarded as a highly promising non-cyanide gold leaching technology, which has shown positive results in the treatment of primary gold ores, gold concentrates, and flotation tailings [87–89].

During the chlorination leaching process (30 °C to 50 °C), it has been found that the gold leaching increases with the rise in the initial hydrochloric acid concentration and temperature, and the gold leaching also increases as the diffusion capacity of various substances is enhanced. Researchers have successfully attempted to strengthen the diffusion capacity of substances using ultrasound [90–92].

Karppinen et al. [93] pioneered the application of the Carbon-in-chloride-leach (CICL) process to old cyanidation residues for the recovery of residual gold. Under the conditions of $\text{Cl}^- = 1 \text{ mol/L}$, $\text{Cu}^{2+} = 0.5 \text{ mol/L}$, 14 g/L activated carbon, an 8-h leaching duration, and solid-to-liquid ratio of 100 g/L at 90 °C, it was demonstrated that 40% of the gold could be recovered from the cyanidation residues. At reduced concentrations of copper ions and chloride, the gold recovery rate was only marginally lower, at 36.4%.

Pak et al. [94] conducted a thermodynamic study on the chloride leaching of high-sulfur gold ores, and the results indicated that maintaining a redox potential above 1.0 V for more than 2 h, increasing the chloride concentration, and decreasing the concentration of $[\text{AuCl}_4]^-$ were beneficial to the chloride leaching process. Under the optimum conditions, namely a pH = 4, $[\text{NaCl}] = 75 \text{ g/L}$, liquid–solid ratio = 3:1, a redox potential above 1.0 V, a reaction temperature of 40 °C, and a leaching time of 2 h, the gold extraction rate reaches 96.54%.

The Kell process was proposed by Adams et al. [95,96], which is a low-energy, low-pollution, and high-recovery process applicable to a wide range of refractory gold ores; a patent has been obtained for the process. Figure 7 shows the flowsheet of the Kell process. The flotation concentrate is subjected to POX. HCl preleach can yield Ag product, while chlorination leaching produces Au product, with the tailings containing PGM can be further separated and refined. If necessary, heat treatment can be used for further impurity removal.

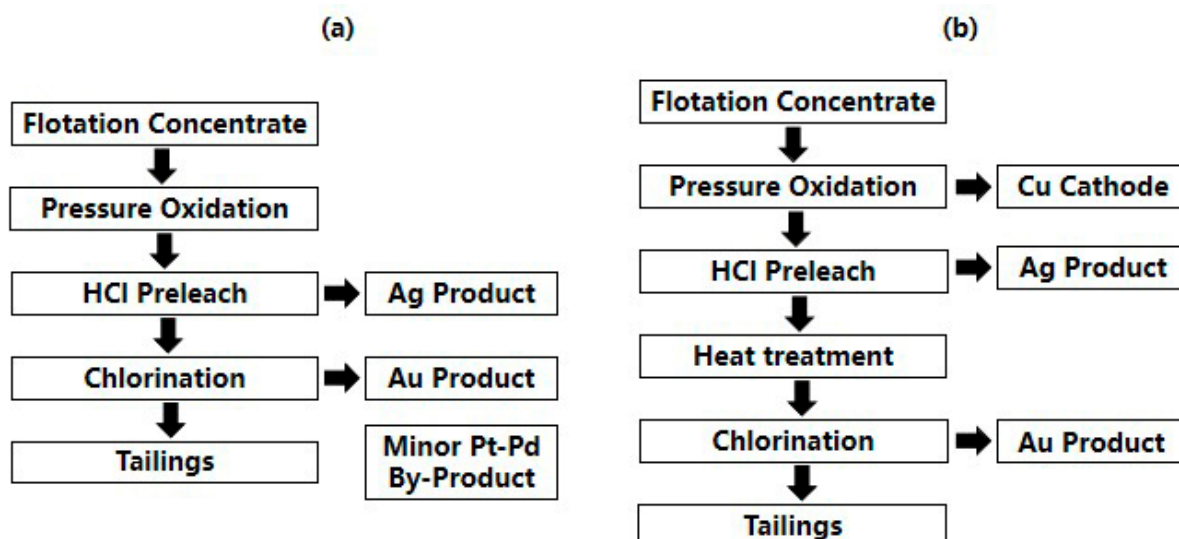
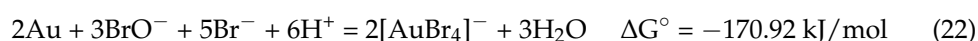


Figure 7. Kell Gold flowsheets for (a) Au–Ag concentrates with minor Pt–Pd; (b) Au–Ag–Cu–C concentrates [95].

3.2.2. Bromination Leaching Process

Similar to the chlorination leaching, in the bromination process, Au is first oxidized to Au^{3+} , which then complexes with Br^- in the solution to form the $[\text{AuBr}_4]^-$ complex and enters the solution. The concentration of bromine and bromide ions are the most significant factors affecting bromide leaching; the higher their concentrations, the faster the kinetics and the higher the gold leaching rate. The addition of hydrochloric acid can reduce the use of bromide ions [97,98]. Although the bromination leaching process is very similar to the chlorination leaching process, there are some differences. Compared to the Cl_2 used in chlorination leaching, the Br_2 used in bromination is a deep red, fumigating liquid, which is relatively easier to transport and store. It also does not require the specialized liquefaction equipment that is needed for chlorination. Elemental bromine can also be recycled and reused. The reaction mechanism for gold leaching in the bromination process is as follows:



Qiang et al. [99] investigated the feasibility of bromate–thiourea synergistic leaching of gold sulfide ore. Under the optimal experimental conditions, with a potassium bromate concentration of 0.1 mol/L, thiourea concentration of 0.6 g/L, hydrochloric acid concentration of 0.2 mol/L, agitation speed of 250 rpm, liquid-to-solid ratio of 4, and leaching time of 10 min, the leaching can reach 92.3%. The leaching time is reduced by approximately 98.61% compared with the conventional leaching time. Although this process has a high consumption of reagents, the leaching is high.

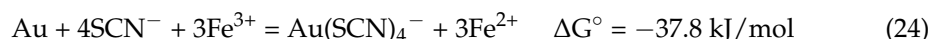
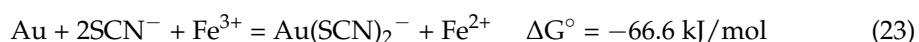
Due to the difficulty in preserving bromine, NaBr, NaBrO, and hydrochloric acid are used as substitutes for bromine. Sousa et al. [100], in the study of the Castromil gold mine, investigated the impact of factors such as temperature, liquid-to-solid ratio, and oxidants on the bromide leaching process for gold extraction. The experimental results indicated that bromide leaching was more effective under high-temperature and low liquid-to-solid ratio conditions, with the gold leaching reaching up to 73% under optimal conditions. The addition of oxidants, such as H_2O_2 , Fe^{3+} , and chlorides, increased the gold leaching to 80%. Furthermore, after a roasting pre-treatment, the gold leaching was further increased to 89%.

Ahtiainen et al. [101] conducted experimental studies on the synergistic chlorobromide leaching of gold from refractory gold concentrates (sulfidic type) and double refractory gold concentrates (sulfidic and preg-robbing types). The experimental results confirmed for the first time that without any pretreatment, it is possible to extract up to 88% of gold from double refractory gold concentrates by combining chlorobromide leaching with activated carbon recovery and the addition of lead nitrate. In the residues of refractory gold concentrates treated with POX, when the degree of sulfide oxidation reached 97%, the recovery rate of gold by chlorobromide leaching could reach 99%. The study demonstrates that, in a chlorobromide medium, high-efficiency gold extraction can be achieved from both refractory and double refractory gold ores, even in a single-stage process.

Halogen leaching processes have the advantages of lower toxicity, shorter processing times, and better leaching outcomes, but they also have drawbacks, such as higher production costs and stringent equipment requirements, which necessitate further improvements in the future. Low-toxicity and high-stability halogen alternatives should be investigated, and milder reaction conditions should be explored to improve leaching efficiency.

3.3. Thiocyanate Leaching Process

The leaching of gold with thiocyanate primarily takes place under acidic conditions. This is because an acidic environment favors the formation of complexes between thiocyanate ions and gold, which promotes the dissolution of gold entering the leaching solution. Additionally, thiocyanate ions are more stable under acidic conditions, which is beneficial for the leaching of gold [102,103]. As an emerging non-cyanide leaching agent, thiocyanate has a leaching mechanism where it can form stable complexes with gold ions, specifically $\text{Au}(\text{SCN})_4^{2+}$, thereby achieving the selective leaching of gold. Thiocyanate often uses Fe^{3+} as an oxidizing agent, and the main reactions for gold leaching under acidic conditions are as follows:



Thiocyanate has a lower toxicity, being 1000 times less toxic than cyanide, and is considered a very strong gold leaching agent. Additionally, the entire leaching process operates at a lower pH, reducing the corrosive impact on the environment and equipment; the preparation and transportation costs of thiocyanate are also lower; thiocyanate has higher stability and a higher gold recovery rate, and it also performs well in gold leaching at higher temperatures [104].

Thiocyanate can be removed through biodegradation or other harmless treatments. For instance, after leaching, the gold-containing thiocyanate solution can be treated to recover gold through processes such as electrolysis, precipitation, or adsorption, while simultaneously regenerating thiocyanate, forming a closed-loop system. This approach reduces operational costs and environmental impact [105].

Azizitorghabeh et al. [106] tested various leaching parameters using the response surface methodology to optimize the gold recovery rate from oxidized ore, and ultimately found that the maximum gold recovery rate of 96% was achieved under conditions of 25 °C, pH = 2, with 0.5 mol/L thiocyanate, 0.1 mol/L Fe^{3+} , and a pulp density of 50% over a period of 24 h. Wu et al. [107] employed a mixed gold leaching system composed of ammonium thiocyanate and glycine. Within this system, glycine functions to suppress the oxidative decomposition of thiocyanate, while also enhancing the leaching efficiency of gold due to its dissolution properties. In the experiments, the concentrations of thiocyanate, glycine, and the oxidant Fe^{3+} were set at 0.6 mol/L, 5 g/L, and 0.05 mol/L, respectively. The stirring speed was controlled at 600 rpm, the pH was maintained at 2, the temperature was kept at 298 K, and the leaching process lasted for 3 h. Under the condition of a liquid-to-solid ratio of 4:1, this mixed gold leaching system achieved a gold extraction rate as high as 93.15%.

Yang et al. [108,109] used ferric sulfate as an oxidant to investigate the leaching behavior of gold in thiourea–thiocyanate solutions. The study found that the introduction of trace amounts of thiourea to the ferric thiocyanate solution resulted in a synergistic effect on the dissolution of gold, leading to a leaching that exceeded the results obtained when using either ferric thiocyanate or ferric thiourea solutions separately. This synergistic effect was attributed to the formation of the mixed ligand complex $\text{Au}(\text{Tu})_2\text{SCN}$.

One of the main reasons why the thiocyanate gold leaching process has not yet been commercialized is that the process requires a higher redox potential than that needed for cyanide gold leaching [110]. However, the chemical reactions of thiocyanate gold leaching not only allow for more effective gold extraction under more lenient leaching conditions, but the reactions demonstrate the potential for sustainable development. Efforts should be focused on exploring the optimization of reaction conditions to enhance the leaching rate, and on developing recycling technologies.

Table 3 presents a quantitative analysis of the leaching reagent costs based on laboratory standards. It should be noted that, in actual industrial production, the production costs are influenced by a variety of factors, not just the prices of the reagents.

Table 3. Economic evaluation of acidic gold leaching process.

Leaching Process	Reagent Dosage (g/t)	Reagent Unit Price (USD/kg)	The Cost of Reagents for Processing 1 Ton of Ore (USD/t)
Thiourea leaching process	200–300	13.82	2.76–4.14
Halogen leaching process	100–200	69.1	4.23–8.34
Thiocyanate leaching process	100–200	51.85	5.18–10.35

Acidic leaching processes are all non-cyanide, which generally have issues, such as high costs, poor stability, complex leaching systems, and high operational difficulty, that still need to be resolved. However, due to their high leaching efficiency and lower environmental pollution, as our country moves towards a resource-saving and environmentally friendly society, the use of non-cyanide leaching for gold will be the development direction for future gold leaching processes. On the whole, in the hydrometallurgical processing of gold, the cyanidation method still dominates. Table 4 lists the advantages and disadvantages of thiosulfate, halogen, and thiocyanate leaching processes, and provides a comparison of several gold extraction methods we have discussed.

Table 4. Advantages and disadvantages of acidic gold leaching process.

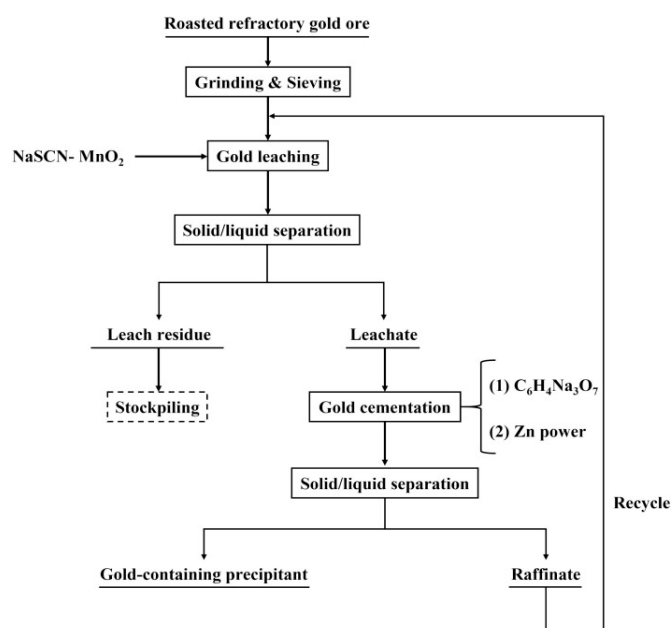
Leaching Process	Advantages	Disadvantages
Thiourea leaching process	High recovery rate environmentally friendly low pollution	Low acidity poor stability high reagent cost need corrosion-resistant equipment
Halogen leaching process	Fast leaching kinetics high recovery rate environmentally friendly low pollution low investment	High reagents consumption Highly corrosive immature technology
Thiocyanate leaching process	Environmentally friendly; low pollution; highly efficient and stable; good selectivity; low toxicity	High reagent consumption requires acidic conditions need corrosion-resistant equipment

3.4. Research Progress on Thiocyanate Leaching

Our research team is dedicated to addressing the issue of high reagent consumption in the thiocyanate gold leaching process. We have made promising progress and achieved notable research outcomes. Li et al. [111] developed a novel manganese dioxide-assisted thiocyanate leaching system and implemented a cyclic leaching scheme. Under the thiocyanate leaching system for gold, the main leaching reactions are as follows:



Figure 8 illustrates the process flow of this leaching system. The refractory gold concentrate, after roasting pretreatment, is subjected to NaSCN-MnO₂ leaching to obtain a gold-bearing leachate. Then zinc powder and citric acid are added to the leachate for gold recovery, followed by solid–liquid separation. The thiocyanate in the filtrate is returned to the leaching process.

**Figure 8.** Cyclic extraction and recovery scheme in the MnO₂-assisted thiocyanate system [111].

Under the leaching conditions of 1.20 mol/L NaSCN, 4.00 mmol/L MnO₂, a pH of 1.00, a liquid-to-solid ratio of 4 mL/g, and a leaching duration of 24 h, the maximum gold extraction rate reaches 94.8%. This not only ensures a high gold extraction but reduces thiocyanate consumption by 75%. This acidic leaching system achieves a satisfactory gold recovery rate while minimizing reagent consumption, making it particularly suitable for refractory gold ores that have undergone acidic pretreatment.

4. Conclusions

Pressure oxidation and bio-oxidation methods have been widely applied industrially worldwide. These pretreatment technologies have unique areas of advantage, and they should be chosen reasonably based on the properties of the materials to be treated. Therefore, in the future, it is necessary to improve the existing process technologies according to the characteristics of various refractory gold ores, expand the advantages of various pretreatment technologies, and achieve progress in both economic and environmental aspects.

From the perspective of gold recovery technology, pyrometallurgical enrichment has high energy consumption and conflicts with energy saving and environmental protection. In wet leaching, cleaner non-cyanide leaching methods are limited in large-scale application due to issues such as high reagent consumption and poor stability, and the mainstream technology for gold recovery is still cyanidation. In the future, it will be necessary to address the economic viability, stability, and applicability of non-cyanide leaching, and reduce the pollution from cyanide leaching. Given the recyclability of thiocyanate, the authors believe that the thiocyanate cyclic leaching system for gold has strong development potential. Developing and improving non-cyanide leaching process equipment and reducing pollution from the cyanide method will be hot topics for future research.

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