

One Novel two-step Bio-Oxidation Pretreatment of Arsenic-Containing Gold-Bearing Concentrate

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A novel process was developed to markedly increase gold cyanidation performance from an arsenic-containing gold-bearing concentrate. Before cyanidation, a two-step bio-oxidation pretreatment was employed to decrease the arsenic content in the residue and obtain a high cyanidation efficiency for gold. Bio-oxidation results indicated that most of the arsenic was rapidly removed from the mineral into the solution in the first bio-oxidation step, and a high redox potential benefited the bio-oxidation process. With an increase in the redox potential of the solution, the As(III) concentration in the solution was decreased. After the first step of the bio-oxidation process, only 4.38% of the arsenic remained in the residue when the slurry density was 7%. After the second step of the bio-oxidation process, the arsenic content in the residue was decreased to 1.81% when the slurry density for the second step of the bio-oxidation process was 14%. Cyanidation tests indicate that the leaching rate of gold could reach 95.66% after sequential two-step bio-oxidation; this leaching rate was much higher than that of the direct cyanidation and one-step bio-oxidation pretreatment.

Keywords: Redox potential; Bio-oxidation; High arsenic gold concentrate; Cyanidation

1. INTRODUCTION

Cyanidation is the most common method used to extract gold from its ore or concentrate. However, direct cyanidation of refractory ores results in inadequate gold recoveries due to the entrapping of fine gold within the sulphide minerals or carbonaceous materials [1-3]. Thus, one environmentally friendly biological treatment method is regarded as one of the most effective processes to pretreat refractory concentrates because it is much cheaper compared with conventional technology [4, 5]. In the past two decades, the bio-oxidation pretreatment of refractory gold concentrate before the cyanidation process has been widely used in industry around the world [6-9].

The bio-oxidation of sulphide minerals plays an essential part in the process of bio-hydrometallurgy, and a two-step process for gold concentrate treatment was proposed to achieve intensification [10, 11]. In this process, a microbial produced ferric sulphate was used to leach the metals during the first step. In this step, the high temperature would increase the process rate significantly. In addition, during the second step, elemental sulphur was oxidized by the regenerated ferric iron, and sulphides are oxidized terminally. For the high temperature during the first step, microbe regeneration was carried out by different clones of acidophilic, chemolithotrophic microorganisms [12-14]. The BRISA technology, which is based on two stages of treatment for ores, including chemical leaching and regeneration of the ferric iron by microbes, has been applied on secondary copper sulphides [15] and chalcopyrite [16] successfully. Fomchenko [17] proposed one new concept of the two-stage bacterial-chemical process for sulphidic material leaching. This concept implies consecutive stages of biological and chemical oxidation for refractory gold-bearing sulphidic concentrates. Sun [18] investigated the impact of dissolved oxygen (DO) concentration on bio-oxidation process of refractory sulphide gold ores. Zhang [19] studied the enhanced effect of pyrolusite in the bio-oxidation process of refractory arsenopyritic gold ore.

Currently, with the increasing industrial demand for gold-bearing ores, the utilization of high arsenic-containing concentrates has become inevitable [20]. However, the bioleaching efficiency of this type of concentrate is still a big problem for the bioactivity of microbes could be inhibited under high As(III) concentration [21]. Several studies have indicated the transformation of higher toxicity As(III) to less toxicity As(V) in the high redox potential condition is beneficial for its bio-oxidation process [12, 22]. Therefore, mesophiles and moderate thermophiles have been limited to use in the bio-oxidation process of arsenic-pyrite refractory gold material prior to cyanidation [21, 23]. To date, there are few reports on bio-oxidation pretreatment regarding arsenic-containing concentrate. Therefore, in this work, the pretreatment of one high arsenic gold-bearing concentrate by the two-step bio-oxidation process was proposed using acclimated mixed microbes. The pulp redox potential and the arsenic ion concentration during the bioleaching process were also investigated.

2. EXPERIMENTAL

2.1. Arsenic-containing gold-bearing concentrate

The high arsenic-containing gold-bearing concentrate was obtained from Hunan Province, China. The samples were finely ground to -0.074 mm with a ball mill in the laboratory before bioleaching. The chemical component analysis results are displayed in Table 1. These results indicate that the sample mainly contained 60.16 g/t Au, 50.27 g/t Ag, 32.33% Fe, 13.84% As, and 23.62% S (wt%). The sample contained trace amounts of elements such as Cu, Pb and Zn. Mineralogical analysis results indicated that the main sulphide minerals in the sample were pyrite (47.43%) and arsenopyrite (18.07%). The phase analysis regarding gold and arsenic of the sample is shown in Table 2 and Table 3. The results explained that more than half of the gold was deposited in sulphide, and most of the arsenic was deposited in pyrite.

Table 1. Chemical element analysis of the sample

Elements	Au(g·t ⁻¹)	Ag(g·t ⁻¹)	Cu	Pb	Zn	Fe	Sb	S	As
Contents (Wt.%)	60.16	50.27	0.097	0.042	0.16	32.33	0.16	23.62	13.84

Table 2. Phase analysis of gold

Occurrence	Exposed	Wrapped in sulphide	Wrapped in other minerals	Total
Occupancy (%)	35.69	60.25	4.06	100

Table 3. Phase analysis of arsenic

Occurrence	Wrapped in pyrite	Wrapped in other minerals	Total
Occupancy (%)	91.58	8.42	100

2.2. Microorganisms and culture conditions

Mixed microbes obtained from Tibet Province in China were used for the bio-oxidation experiment. The amplified 16S rDNA cloning and sequencing result indicated the presence of *Leptospirillum ferriphilum*, *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans* in the mixed microbes. The microbes were cultured in shake flasks with a capacity of 200 mL and stirred with a speed of 200 rpm at 40 °C using an orbital incubator. The 9K basal medium was used for cell cultivation. This medium was composited with the following components: (NH₄)₂SO₄ (0.4 g/L), FeSO₄·7H₂O (25 g/L), K₂HPO₄ (0.15 g/L), MgSO₄·7H₂O (0.5 g/L), and Ca(NO₃)₂ (0.01 g/L). These chemicals were dissolved with distilled water to create a solution. The initial pH for the solution was adjusted to 2.0 with sulphuric acid. All of the reagents used in this experiment were of analytical purity and purchased from Tianjin Kermel Reagent Co., Ltd. All of the microorganisms were sub-cultured into basal salts medium, the energy was supplemented with 1% arsenic-containing gold-bearing concentrate. The resulting culture was used as an inoculum for the bioleaching experiments.

2.3. Bioleaching process

The first step of the bio-oxidation process was conducted in a 2 L mechanical agitated reactor containing 600 mL of microbe cultures (40%) and 900 mL of 9K medium (60%). The concentrate weight as a variable value was added into the reactor to form pulp with a mass density of 3%, 5%, 7%, 9% and 11%. The pulp was stirred at a rate of 550 r/min and aerated at a speed of 0.15 m³/h. The temperature during the bio-leaching process was maintained at 40 °C with a water bath. This step lasted for 6d with an initial pH of 1.4. Then, the pulp was filtered, and the residual solids were used for the second step of bio-oxidation. The pulp densities during the second-step of bio-oxidation were changed to 14%, 16% and 18%. The stirring rate was enhanced to 720 r/min. The other experimental

conditions were the same as the first-step bio-oxidation process. During the two-step bio-oxidation process, solution pH, redox potential and the concentration of Fe(II), Fe(III), As(III) and total As were measured every day. When the second step lasted for 6 d, the filtration was used to separate the solid and the leachate.

2.4. Cyanidation tests

The gold extraction of the residual solid after two-step bio-oxidation was determined by the cyanide leaching method. A quantity of water was added into the reactor and mixed with the solid to generate a slurry with a 20% (w/w) solid concentration. The slurry pH was maintained at approximately 11 with CaO. Then, the slurry was stirred for 6 h after the CaO treatment, and 15 kg/m³ of active carbon accompanied with 20 kg/t sodium cyanide was added into the slurry. After leaching for 24 h, the residual solid was filtered, washed three times, dried, and weighed. Then, the gold concentrate in the leachate was measured.

2.5. Analytical methods

The ferrous ion concentration was determined by titration with potassium dichromate and N-phenylanthranilic acid was used as an indicator. The ferric iron concentration was determined by titration with chelating agent EDTA [23]. The As(III) and total As concentration were determined by iodometric titration [24]. The concentration of As(V) was calculated by the subtraction of total As and As(III) concentration. Inductively coupled plasma-atomic emission spectrometer (ICP-AES) (America Baird Co. PS-6) were used to determine the gold concentration. The Au leaching rates were calculated by Eq. 1. The slurry pH and redox potential was monitored by a pH meter (PHS-3C).

$$\mu = (M\beta - m\alpha) / M\beta \quad (1)$$

Where, β and α were the content (mass fraction) of Au before and after cyanidation; M and m were the masses of the initial feed and undissolved residues, respectively.

3. RESULTS AND DISCUSSION

3.1. Microbe acclimation

Pyrite and arsenopyrite were confirmed as the main minerals in this concentrate. The pyrite and arsenopyrite were oxidized by a synergetic effect of the strong oxidizing agent (Fe³⁺ and H₂SO₄), which was produced by mixed bacteria [25-27]. As shown in Eq. 2, the arsenic concentration in the leachate will increase with oxidation time, and previous studies have reported that the bio-activity of microbes could be inhibited by the high As(III) concentration in the leachate [28-30]. A high redox potential is a benefit to transform higher toxicity As(III) to less toxic As(V) (Eq. 3 and 6) [22, 31]. Thus, it is critical to improve the high arsenic tolerance of microbes to maintain a high redox potential even in the high arsenic environment. In this work, the high arsenic-containing gold-bearing concentrate (Table 1) was continuously added to the basal medium to improve the adaptability of

microbes to the high arsenic concentration. The process was maintained at approximately 40 °C and at a pH range of 1.0-2.0. When the redox potential of the slurry reached 600 mV, the 1% arsenic-containing gold-bearing concentrate was added to decrease the redox potential to less than 500 mV. When repeating this process for 60 days, the redox potential of the slurry would recover to 600 mV from 500 mV in just one day. These acclimated microbes were used in the following bio-leaching experiment. However, to reduce the toxicity of As(III) to microbes, solid-liquid separation processes, such as filtration, were also conducted to reduce the As(III) concentration in the solution.



$$\varphi = \varphi_0' + \frac{RT}{zF} \ln \frac{[\text{Fe}^{3+}]}{[\text{Fe}^{2+}]} \quad (6)$$

3.2. Bio-oxidation of the arsenic-containing concentrate

3.2.1 First-step bio-oxidation process

During the first step of bio-oxidation process, arsenic was supposed to rapidly dissolve into the slurry for the oxidation of arsenopyrite. Thus, the slurry density and oxidation time were the main factors that influenced this process. The redox potential and As(III) concentration of the solution as a function of slurry density and oxidation time was monitored to determine the optimum slurry density and oxidation time. The results are shown in Figure 1 and Figure 2, respectively.

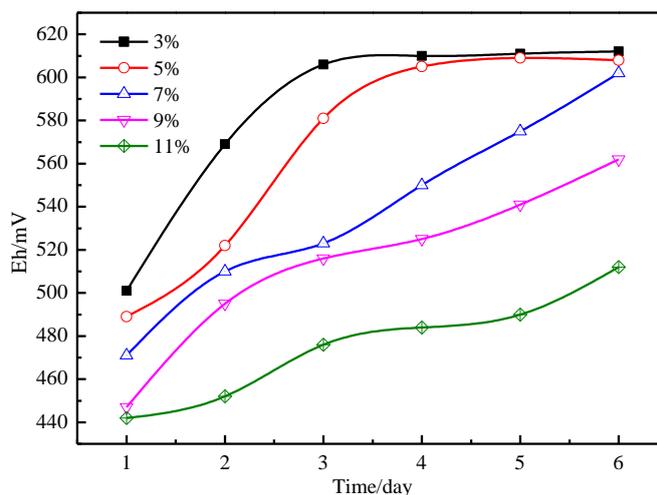


Figure 1. The redox potential of the solution at a different slurry density in the first-step bio-oxidation process.

As shown in Figure 1, the redox potential of the solution was increased slowly with the oxidation time when the slurry density was 9% and 11%. The maximum redox potential was 512 mV and 562 mV when the slurry density was 9% and 11%, respectively. However, when the slurry density

was 7%, the redox potential rose sharply with the oxidation time. In addition, the redox potential reached 602 mV after being oxidized with 6 d in the case where the slurry density was 7%. When the slurry density was 3% and 5%, the redox potential first increased rapidly and then held steady. The maximum redox potential was 608 mV and 612 mV after being oxidized with 6 d, respectively, and when the slurry density was 5% and 3%. We concluded from the results that the redox potential of the solution was decreased with an increase of the slurry density, and a higher redox potential was usually obtained at a low slurry density, such as 7%, 5% and 3%.

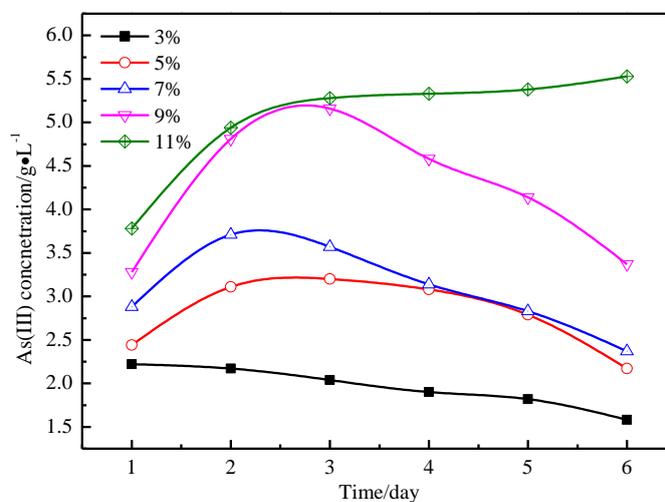


Figure 2. The As(III) concentration in the solution at different slurry densities in the first bio-oxidation process.

Figure 2 indicates the effects of slurry density and oxidation time on the As(III) concentration in the solution. The results displayed in the figure revealed that the As(III) concentration in the solution was gradually increased with the oxidation time when the slurry density was 11%. However, the As(III) concentration in the solution was increased and then decreased with oxidation time when the slurry densities were 9%, 7% and 5%. When the slurry density was 3%, the As(III) concentration in the solution was speedily reduced with the oxidation time. We concluded from the figure that the higher As(III) concentration was usually related to the higher slurry density.

Compared with the results shown in Figure 1 and Figure 2, some relationships between the As(III) concentration and the redox potential of the solution could be concluded. A higher As(III) concentration was consistent with a higher slurry density. During the initial bioleaching process, arsenic in the concentrate was oxidized to H_3AsO_3 (Eq. 2), and then, the H_3AsO_3 in the solution was oxidized to H_3AsO_4 (Eq. 3) under a high redox potential environment. When the slurry density was 3%, the redox potential of the solution was high enough to quickly oxidize the As(III) in the solution to As(V). Thus, the As(III) concentration in the solution was relatively low and decreased with oxidation time. With further increasing of the slurry densities to 5%, 7% and 9%, the concentrate was bioleached, and arsenic was rapidly dissolved in the leachate in the form of As(III), and therefore, the As(III) concentration was first increased with the oxidation time. Then, when the redox potential of the solution was gradually increased and the oxidation speed was higher than the dissolved speed, the

As(III) concentration in the solution was decreased. When the slurry density was 11%, the redox potential of the solution was too low to oxidize As(III), and the activity of the microbes was inhibited by accumulated As(III) [12, 20]; therefore, the oxidation process was almost terminated and the As(III) in the solution was gradually increased with oxidation time. The results displayed in Figure 1 and Figure 2 revealed that the high redox potential benefited the bio-oxidation process, which was consistent with previous studies [5]. Thus, considering the redox potential, the concentration of As(III) and the efficiency, a slurry density of 7% with 6 d bio-oxidation was designated the optimized slurry density and the oxidation time for first-step bio-oxidation process.

After the first-step bio-oxidation, most arsenic in the concentrate had been removed into the solution. To avoid the inhibition of As(III) in the leachate to microbe bioactivity, filtration was adapted to separate the leachate and residue. The arsenic content in residue was analysed and the results are shown in Figure 3.

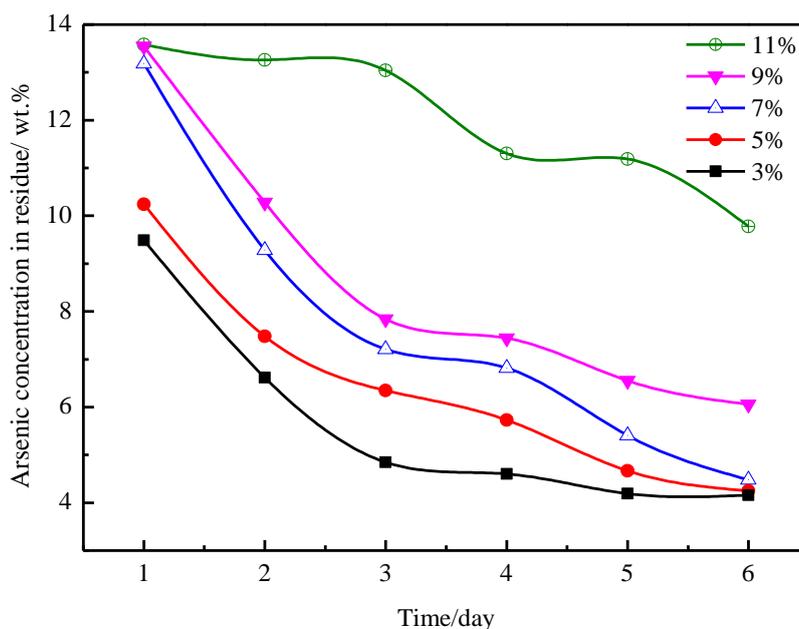


Figure 3. Arsenic content in the bioleaching residue after one-step bio-oxidation.

The results displayed in Figure 3 indicate that the arsenic in the residue was reduced with the bio-oxidation process. The higher the slurry density, the more slowly the arsenic concentrations in the residue were decreased. When the slurry density was 3%, 5% and 7%, almost 70% of the arsenic in the concentrate was removed into the solution, and the arsenic content in the residue decreased from 13.84% to lower than 4.48% after 6 d of bio-oxidation. When the slurry density increased to 9% and 11%, the removal rate of the arsenic decreased rapidly, especially when the slurry density was 11%, and only 29.34% of the arsenic was removed from the concentrate; additionally, the arsenic content was still 9.78% after 6 d of bio-oxidation. The first step bio-oxidation exhibited a high arsenic removal rate even when the arsenic content in the concentrate was as high as 13.84%, and the bio-oxidation process was much more convenient compared to other pretreatment methods such as chemical leaching [11, 12, 32].

3.2.2 Second-step bio-oxidation process

After the first step oxidation, the content of As in the residual decreased from 13.84% to 4.48% when the slurry density was 7% with 6 d of oxidation. Thus, the slurry density for the second-step bio-oxidation process could be increased. The pulp densities of 14%, 16% and 18% were investigated with 6 d of oxidation in the second-step bio-oxidation process. The redox potential and As(III) concentration of the solution were measured, and the results are shown in Figure 4 and Figure 5.

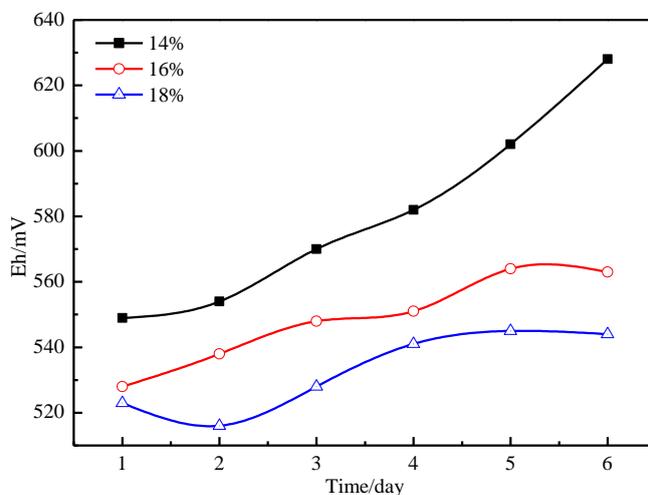


Figure 4. The redox potential of the solution at different slurry density in second-step bio-oxidation process.

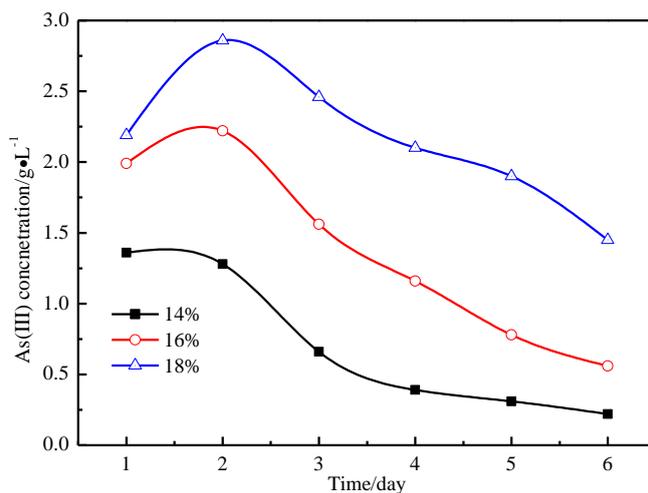


Figure 5. The As(III) concentration in the solution at different slurry densities in the second-step bio-oxidation process.

As shown in Figure 4, the redox potential of the solution slowly increased with the oxidation time when the slurry densities were 16% and 18%. The maximum redox potential was no more than 560 mV. However, when the slurry density was 14%, the redox potential rose sharply with the

oxidation time. The redox potential reached 628 mV after 6 d of oxidation. We concluded from the results that the redox potential of the solution was decreased with an increase of the slurry density, and a higher redox potential was usually obtained at a relatively lower slurry density, which showed the same tendency as the first-step bio-oxidation process.

Figure 5 indicates the effects of slurry density on the As(III) concentration in the solution during the second-step bio-oxidation process. The results revealed that the As(III) concentration in the solution was increased and then decreased with the oxidation time when the slurry densities were 16% and 18% for the oxidation of microbes for the residue and arsenic. In addition, the small increase of the As(III) concentration was because the oxidation speed was lower than the dissolution speed of the concentrate in the initial oxidation process. However, when the slurry density was 14%, the concentration of As(III) in the solution was relatively low, and the redox potential was high enough to oxidize As(III); thus, the As(III) concentration in the solution was gradually reduced with the oxidation time.

The results displayed in Figure 4 and Figure 5 also indicate that a higher As(III) concentration was usually related to a higher slurry density. With the oxidation of the residue, arsenic was first bioleached and dissolved in the leachate in the form of As(III) and then it was oxidized to As(V) with a high redox potential of the solution. After the first-step of bio-oxidation, most of the arsenic was removed from the concentrate, and thus, the second-step of bio-oxidation obtained a higher redox potential and lower As(III) concentration in the solution after 6 d of oxidation despite the high slurry density, which indicated that the first-step of bio-oxidation of the high arsenic concentration was critical and important.

After two-step oxidation, the residue and leachate were also separated with filtration. The As, Fe and S content in the residue was also detected and the results are shown in Table 4.

Table 4. The As, Fe and S content in residue after bio-oxidation.

Slurry density	Content/%			Bio-oxidation rate/%		
	As	Fe	S	As	Fe	S
7%	4.84	16.81	18.89	65.03	41.57	20.03
7%, 14%	1.81	6.62	4.28	86.92	79.52	81.88
7%, 16%	1.83	9.33	6.88	86.78	71.14	70.87
7%, 18%	1.85	11.61	10.47	86.63	64.09	55.67

The results displayed in Table 4 indicate that the As, Fe and S content in the residue was gradually reduced and the slurry density decreased. When the slurry density was 14%, the arsenic content in the residue was as low as 1.81% after 6 d of bio-oxidation. In addition, there was little difference of arsenic content in the residue when the slurry densities were 14%, 16% and 18%, respectively. In summary, the two-step bio-oxidation process exhibited a significant advantage over one-step bio-oxidation. A previous study [17] decreased the As content from 8.21% to 0.41% by adapting a two-stage bacterial-chemical oxidation; however, considering the high As content (13.84%) in this work and the complicity and expenses of chemical leaching, the two-step bio-oxidation of the

high arsenic concentrate was also a suitable method to pretreat the high arsenic-containing gold-bearing concentrates.

3.3. Cyanidation of pretreated concentrate

To clarify the pretreatment efficiency on cyanidation of this high arsenic-containing gold-bearing concentrate, the oxidized residues under various conditions were subjected to cyanidation leaching; the results are shown in Table 5. Notably, only 34.86% of the Au could be extracted through direct cyanidation without any pretreatment methods, and the content of Au in the cyanidation tailings was still $39.19 \text{ g}\cdot\text{t}^{-1}$, which did not meet the requirements for industrial production. The gold recovery was dramatically increased to 94.00% when the first-step of the bio-oxidation process was used to pretreat the concentrate. When the two-step bio-oxidation pretreatment process was applied, the leaching rate of Au (95.66%) was increased and the content of Au (8.63%) in the residue concentrate was relatively low. In addition, the processing capacity was significantly improved because the slurry density in the second-step bio-oxidation was high compared with the one-step bio-oxidation pretreatment process.

Various pretreatment methods of refractory gold ores and concentrates have been proposed by previous studies and obtained satisfactory gold recovery. Liu [12] obtained a 93.51% leaching rate of Au after the concentrate was pretreated by a two-step chemical-bio oxidation. Muravyov [11] reached 77.9% Au recovery by using two-step bio-oxidation pretreatment. Guo [6] applied bio-oxidation and a two-step thiourea leaching method to dispose of the refractory gold concentrate and the leaching rate was approximately 95%. Compared with these methods, a novel two-step bio-oxidation pretreatment of high arsenic-containing gold-bearing concentrate was cheaper and more convenient with higher Au leaching rate.

Table 5. The content of gold before and after different oxidation conditions.

Samples		Bio-oxidation time/d	As content /%	Au content before cyanidation/ $\text{g}\cdot\text{t}^{-1}$	Au content after cyanidation/ $\text{g}\cdot\text{t}^{-1}$	Leaching rate/%
Pretreatment	Slurry density					
-	-	-	13.84	60.16	39.19	34.86
One step	7%	6	4.48	187.32	11.24	94.00
Two-step	7%, 14%	6, 6	1.81	212.00	10.93	94.84
Two-step	7%, 16%	6, 6	1.83	199.00	8.63	95.66
Two-step	7%, 18%	6, 6	1.85	179.50	11.69	93.49

4. CONCLUSION

In this work, the acclimated microbes, which could generate a high redox potential even in a high arsenic environment, were used to bio-oxidize the high arsenic gold concentrates prior to cyanidation tests. Therefore, one two-step bio-oxidation pretreatment process was conducted to

improve the cyanidation efficiency of a high arsenic-containing gold-bearing concentrate. After the first-step bio-oxidation process, the arsenic content in the concentrate was decreased from 13.84% to 4.38% after 6 d of bio-oxidation when the slurry density was 7%. The redox potential of the solution, which was a benefit to the bio-oxidation process for oxidizing As(III) in solution to As(V) was increased with oxidation time. In addition, the As(III) concentration in the solution was descending after a small increase. After the second step of the bio-oxidation process, only 1.81% of arsenic was retained in the residue when the slurry density for the second step bio-oxidation process was 14%. Cyanidation tests showed that a very low leaching rate of 34.86% for gold was obtained for direct cyanidation of this concentrate. This result did not meet the acceptable level of Au recovery because Au that was too high was disposed of in the residue. However, bio-oxidation pretreatment could dramatically improve the leaching rate of Au during the cyanidation process. After two-step bio-oxidation pretreatment, a maximum Au leaching rate of 95.66% was achieved when the slurry density was 7% for the first step and 16% for the second step. After cyanidation, the Au content in the residue could be as low as $8.63 \text{ g}\cdot\text{t}^{-1}$.

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