Research of hydrometallurgical method of leaching gold from flotation tails with using bio-oxidation

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Abstract. This paper shows the results of the comparative study of the efficiency of gold extraction methods from technogenic flotation tails by agitation cyanidation and biooxidation followed by leaching. A representative sample of flotation tails was taken at gold extraction plant of Altyntau Kokshetau LLP. It was established that in the test sample contains 0.47 g / t Au and 0.62 g / t Ag. The gold extraction degree from flotation tailings with 80% content of 0.071 mm fraction was 50%, and that with 90% content of 0.071 mm fraction was 60%. Gold extraction plant's flotation tailings grinded to 0-0.044 mm fraction with sodium peroxide were subjected to pre-oxidation. The gold extraction degree from flotation tailings with 90% content of 0.044mm fraction at a solid-liquid ratio of 1:4 and a cyanide concentration of 1 g/dm³ is 65.2%. By using an active strain of thionic cultures the gold extraction degree was 72.1%.

Keywords: gold hydrometallurgy, bio-leaching, flotation tails, bio-oxidation.

Introduction

The development and mastering of the technology for extracting gold from accumulated technogenic mineral objects, primarily from dumps, tailings, the water of gold recovery plants and industries are particular importance for many gold mines in which the raw material reserves of conditioned ores are close to depletion. In this regard, the relevance of this area of research is due to the presence in our country's a large number of poor gold ores and their production waste, which is a potential reserve for increasing gold production.

One of the most important areas of overcoming the technical and economic problems of the gold mining industry is the abandonment of outdated technology, designed for the practically disappeared category of large gold. It is necessary to move to new high-tech processes and devices, a new environmentally friendly technology that ensures...
the extraction of very thin classes of precious metals [1–9].

In recent years, researchers have paid attention to the development of new methods that can be competitive and effective in terms of mining precious metals. Currently, the use of achievements in the field of biotechnology, methods of bioleaching and bio-opening, in the process of industrially valuable strains of microorganisms are used for bio-extraction of gold and associated metals from technogenic raw materials, it is the most acceptable and less expensive [10–19].

Therefore, the research aimed at improving the efficiency of gold extraction from technogenic mineral raw materials is not only of scientific and practical but also social and environmental importance.

In concerning with this depletion of gold ore deposits in Kazakhstan, the extraction of gold from the tailings and dumps of gold recovery factories becomes relevant. The technology for processing ore raw materials with a low content of precious metals should be inexpensive, to ensure a sufficiently high degree and complexity of their extraction, and provide for effective measures to protect the environment.

A characteristic feature of the sample under study, as gold-bearing raw materials, is the low content in the main valuable component. The extraction of gold in the industry is carried out in the ore preparation cycle (crushing, grinding), during the enrichment processes (gravity, flotation). Often, even ultra-fine grinding of the material does not allow to achieve the necessary degree of opening, since the gold-bearing raw materials are very diverse in their material composition and gold content forms, it is needed an individual technology for each specific case in their processing.

Methods of conducting technological experiments

The phase and chemical composition of the content of the main components of the sample was studied by using a complex of modern physico-chemical methods of analysis, which including the following types: atomic absorption spectrophotometric analysis, X-ray phase, X-ray fluorescence (elemental, semi-quantitative), electron microscopic. To perform the assay, chemical, X-ray fluorescence, mineralogical, X-ray spectral analyzes, private samples with a particle size of 90% of -0.074 mm were selected.

The estimated gold content in the sample according to chemical analysis is given in Table 1. It has been established that the sample of flotation tailings that was get averaged and crushed to a particle size of 0.074 mm contains Au - 0.47 g/t and Ag - 0.62 g/t. The content of the main and impurity components in the samples are characterized by the spectral analysis data shown in Figure 1.

As a result of IR spectrophotometry, it was revealed that α-SiO2 quartz is present in the spectrum - 1086, 797, 777, 694, 464, 397, 373 cm⁻¹, muscovite KAl2[(OH, F)2] AlSi3O10] – KAl2[(OH, F)2] AlSi3O10] – 3618, 1031, 534, 476, 429 cm⁻¹, oligoclase – (Ca0.1–0.3, Na0.9–0.7) (Al, Si) Si2O8 – 1008, 760, 728, 646, 534, 464, 429 cm⁻¹, microcline – K[AlSi3O8] – 1141, 1086, 777, 728, 646, 608, 584, 534, 464, 429 cm⁻¹, carbonates – 1418, 874 cm⁻¹. The valent vibrations of Fe³⁺ – O (in silicates, carbonates) manifest themselves in the range of 400-300 cm⁻¹; Fe²⁺ – O (silicates, carbonates) – 373, 330 cm⁻¹. Cu⁺ – O bonds (in salts) – 266 cm⁻¹.

Currently, the study of the mineral composition of gold-bearing ores, as well as the forms in which are found precious metals, is not without mineralogical analysis based on modern research methods. Associated ore components are presented (Table 2):

- quartz (SiO2) – 46%;
- sulfides: marcasite (FeS2) - 10%, pyrite (FeS2) - 9%, (FeS2) and bismuthin (Bi2S3) are very rare.
- iron oxides and hydroxides: magnetite (FeFe2O4) – 35%, hematite (αFe2O3), goethite/hydroheterite (HFeO/HFeO - 2H2O) are present in insignificant amounts.

### Table 1 – The chemical composition of samples flotation tailings

| Components | Content |
|------------|---------|
| Gold (Au) fire assay (tigel smelting) | 0.47 g/t |
| Gold (Au) atomic absorption analysis | 0.43 g/t |
| Silver (Ag) atomic absorption analysis | 0.62 g/t |
| Iron (Fe) atomic absorption analysis | 1.86 % |
| Zinc (Zn) atomic absorption analysis | 0.003 % |
| Lead (Pb) atomic absorption analysis | 0.003 % |
Copper (Cu) atomic absorption analysis | 0.002 %  
Arsenic (As) atomic absorption analysis | 0.145 %

Figure 1 Infrared spectrum of the initial sample of flotation tailings

Results and discussion

Micrographs of related components and gold particles are detected which shown in Figures 2-5.

As a result of mineralogical studies, it was established that the particle size of fine-dispersed gold particles in free form is Au 2.5×3.9 microns, in aggregations the particle size of Au particles is 1.2-8.2 microns, and it is also in fine-dispersed form. The shape of the grains is irregular, isometric. The color of gold is bright yellow. Gold in the flotation tailings is in associations with aggregates of quartz (a hydrophobic mineral).

The elemental composition and presence of gold have been studied using SEM-RSMA. It is known that scanning electron microscopy allows to study directly the surface of materials and to obtain, with comparatively high resolution, both qualitative and quantitative information on the chemical composition of the object in conjunction with the surface topography. An electron probe microscope with energy dispersive (EDX) and wave dispersive (WDX) analyzers are used to analyze the elemental composition. The results of topographic images of the scanning microscope are shown in Figure 6.

Table 2 - Characteristics of the dimensions and prevalence of the main components

| List of main components | Chemical formula of mineral | % of the studied minerals | Component sizes, mkm/ mkm² |
|------------------------|-----------------------------|---------------------------|-----------------------------|
| Quartz                 | SiO₂                        | 46                        | 5-150                       |
| Magnetite Mgt          | FeFe₂O₄                     | 35                        | 8-10                        |
| Marcasite              | FeS₂                        | 10                        | 10-30                       |
| Pyrite Py              | FeS₂                        | 9                         | 5-20                        |
| Arsenopyrite Ars       | FeAsS                       | rare                      | 20-27                       |
| Hematite Hem           | αFe₂O₃                      | rare                      | 18-27                       |
| Goethite/ Hydroheterite/Fe | HFeO₄/ HFeO₂·ag | rare                      | 30-40                       |
| Bismuthin              | Bi₂S₃                       | rare                      | 2.5                         |
| Gold Au                | Au                          | rare                      | 1.2-8.2                     |
| Total:                 |                             | 100                       |                             |
Figure 2 Finely dispersed gold in association with waste rock and bismuthin

Figure 3 - Finely Dispersed Gold in Association with Waste Rock and Bismuthin
In the sample under study, elements such as silicon, oxygen are largely present, and when focused on individual fragments, iron, sulfur, and gold are noted. Thus, with an increase in resolution of 1,500 times in the residual fragments of sulfide minerals, the spectra of gold associated with pyrite were recorded. Next, to obtain results on the phase composition of the sample under study, modern spectroscopic methods of investigation were used - X-ray fluorescence and X-ray phase analyzes (Table 3). Studies of X-ray fluorescence analysis showed that samples of flotation tailings have the following composition, %: Si - 14.2; Fe - 12.7; Ca - 12.9; S - 4.8; Al - 2.1; Na - 0.7; Zn-0.3; Pb - 0.3; Mg - 0.2; Cu - 0.1; As - 0.02. As a result of X-ray phase analysis, it was found that the composition of the sample contains the following mineral compounds: quartz - 35.7%, clinohlore - 11.1%, muscovite - 5.8%, calcite - 10.0%, dolomite - 15.2%, albite - 14.3%, pyrite - 7.9%.

Based on data from analytical studies, it can be seen that gold in the sample under study is in associations with both quartz and pyrite, and is present in a generally finely dispersed form in intergrowths, which requires a careful approach when choosing a method of extracting gold. The presence of sulphide minerals of marcasite and pyrite is because these tailings were obtained in the process of selective flotation, with the use of depressant reagents that reduce the transfer of these minerals to concentrate. In several scientific works, the authors considered similar types of raw materials and methods for their processing [20-23].

Selection of nutrient media and optimal growing conditions for the cultivation of microorganisms. Determination of the technological parameters of the opening of flotation tailings by the biochemical method. Waste of flotation beneficiation differs from ores and concentrates both in the content of the main components and in the content of mineral components and nutrient substrates necessary for the full development of the natural microbiocenosis. In this regard, the next stage of research was the study of flotation tails microflora, identifying the main groups of microorganisms among native cultures, isolating individual bacterial cells, isolating active strains for further use in the process of bioleaching gold. The conducting microbiological studies one of the most important tasks is the selection and optimization of nutrient media. The selecting media is not only the necessary to take into account not only the components (sources of nitrogen, carbohydrate, protein, amino acids, vitamins) necessary for the growth and accumulation of biomass, but also include the compounds necessary for the optimum growth of some difficultly identifiable groups of microorganisms. Thus, for the isolation of chemolithotrophic and heterotrophic microorganisms, nutrient media were selected from the sample under study, which positively influenced the optimal growth and development of the native microbiota.

Table 3 Phase composition of flotation tailings

| Component         | Formula                                      | Mass % |
|-------------------|----------------------------------------------|--------|
| Quartz, syn       | SiO₂                                         | 35.7   |
| Clinohlore        | Mg₄.₈₈₂Fe₀.₂₂₂Al₁₈₈₁Si₂₉₆O₁₃₆(OH)₈            | 11.1   |
| Muscovite         | K₀.₉₂₂Al₅(Al₀.₉₂₂Si₃.₀₆₆O₁₀₄)(OH)₁.₇₄₄Fe₂.₂₅₆ | 5.8    |
| Calcite           | CaCO₃                                        | 10.0   |
| Dolomite          | CaMg(CO₃)₂                                   | 15.2   |
| Albite, low       | Na(AlSi₂O₆)                                 | 14.3   |
| Pyrite, syn       | FeS₂                                         | 7.9    |
It is known that various microorganisms take part in the microbiological processes occurring in the tailing dumps. Therefore, various selective media were used to detect the above groups of bacteria, respectively. To identify acidophilic thionic bacteria in the microflora of the object under study, the elective nutrient media were prepared - Silverman and Lyundgren's 9K media with different concentrations of iron (5 and 44.2 g/l) pH ~ 2.5-3.0; media 9KS pH ~ 1.5; Leten's media pH ~ 4.0; To count for thionic bacteria, Waxman's media pH is ~ 4.0. For the detection of saprophytic heterotrophic groups of microorganisms, the FPB media (fish peptone broth) and FPA (fish peptone agar) pH ~ 7.0-7.4 was used, as well as to account for microscopic fungi Chapek’s media pH ~ 4.0 and yeast-like fungi Saburo’s media pH ~ 6.5.

In the process of cultivation, in addition to the main components that make up the prescription basis of differential media, other parameters were also taken into account, such as temperature, aeration, redox potential and pH, purity of reagents and sterility of the used nutrient media.

Based on this, and scientific achievements of other researchers, optimal conditions were selected for the cultivation of aboriginal strains and the laboratory strain of acidophilic bacteria: temperature conditions of 18 – 20°С under the conditions of a thermoshaker or thermostat depending on the allocated groups of microorganisms, mixing speed 200-250 t.p.m., the amplitude of rotation 5.

The analysis showed that after 7 days, an active growth of heterotrophic groups of microorganisms was observed, the color of the nutrient medium changes from light yellow to brown, the surface of the medium was covered with a thin film, a precipitate forms on the bottom of the flask, and a specific smell was observed. To establish the morphological features of the bacteria, present in the sample, a microslip was prepared with further Gram differential staining. The results are shown in Fig. 7.

As can be seen from the figures, the microflora is mainly represented by rod-shaped forms of gram-negative cells, which are arranged in pairs or singly. Before the start of the main experiments, the regularity of the influence of such parameters as the
size of pulp, the concentration of cyanide and the ratio Solid: Liquid was analyzed. Indicators of gold extraction at different ratios of these parameters are shown in Table 4.

As shown from Table 4, in the process of bioleaching there is an increase in gold extraction, also such grinding is difficult under production conditions. However, it was decided to test one version of a sample of this size in combination with an oxidizing reagent.

The next stage of the work was tasked with comparative testing a biochemical method for extracting gold from flotation tailings. For comparison, the following leaching variants were carried out:

- **Variant No. 1** - agitation leaching using NaCN 1 g/dm³, Solid: Liquid ratio = 1:4, without prior acid washing and bacterial dissection (control variant);

- **Variant No. 2** - agitation leaching - cyanidation with preliminary acid washing for 3 hours and bacterial dissection with a duration of 120 hours, Solid: Liquid ratio = 1:4; temperature 25°C; concentration of cyanide solution 1 g/dm³; concentration of sulfuric acid wash solution 20 g/dm³;

- **Variant No. 3** - agitation leaching with using sodium peroxide 10 g/dm³ and solution sodium cyanide with a concentration of 1 g/dm³, Solid: Liquid ratio = 1:4, with a preliminary acidic washing without bacterial opening;

- **Variant No. 4** - agitation leaching using NaCN 1 g/dm³, Solid: Liquid ratio = 1:4, with prior acid washing.

After filtration, the resulting solution was submitted for chemical analysis to determine the gold content, the cake was subjected to drying and further bacterial dissection (variants No. 1 and No. 2). Next, the second stage is a bacterial dissection for 5 days with a bacterial solution of A. Ferrooxidans containing Fe**3+** 5 g/dm³; the number of bacteria is 10⁶ cells/ml. For the accumulation of A. Ferrooxidans biomass for the purpose of their subsequent use of bacterial dissection, a nutrient media was prepared for bacteria A. Ferrooxidans: FeSO₄ - 5 g/dm³; (NH₄)₂SO₄ - 1.5 g/dm³; MgSO₄ - 0.5 g/dm³; K₂HPO₄ - 0.5 g/dm³; H₂SO₄ - 1 ml/dm³. The ratio of Solid: Liquid with bacterial dissection was also set in a ratio at 1:4.

**Table 4** Indicators of gold extraction at different ratios solid: liquid

| Size class | NaCN, % | Solid : Liquid ratio |
|------------|---------|---------------------|
| 0.1 mm     | 0.05    | 1:1                 |
| 0.1 mm     | 0.1     | 1.2                 |
| 0.1 mm     | 0.2     | 1.3                 |
| 0.071 mm   | 0.05    | 1.4                 |
| 0.071 mm   | 0.1     | 1.5                 |
| 0.071 mm   | 0.2     | 1.6                 |
| 0.044 mm   | 0.05    | 1.7                 |
| 0.044 mm   | 0.1     | 1.8                 |
| 0.044 mm   | 0.2     | 1.9                 |

During the entire period of bacterial dissection, daily sampling of the solution was carried out in small quantities (1-2 ml), in order to assess the activity and viability of microorganisms, and also determined the change in pH and ferric concentration (Table 4). Upon completion of the bacterial dissection, the solution obtained after filtration was analyzed for the content of gold and related elements, and the cake after drying was used for the next stage, cyanidation.

The third stage - cyanidation was carried out with a solution of NaCN 1 g/dm³ (variants No. 1 and 2) and cyanidation with solutions of hypochlorite and sodium cyanide with a concentration of 1 g/dm³ (variants No. 3 and No. 4) for 24 hours. After the cyanidation process, filtration was carried out, the resulting solution and the cake were sent for chemical analysis to determine the content of gold and accompanying metals to determine the degree of the extracted noble metal, which passed into the cyanide solution after the agitation leaching. Results in the extraction of gold, as well as the concentration of accompanying metals in the solution, are presented in Tables 5-6.

As shown from table 4, in the process of bioleaching there is an intensive growth of bacterial cells, the number of which on the 5th day of reaches
5.9 × 10^7. At the same time, the pH of the pulp decreases to 1.8 and the concentration of ferric iron is 7.6 g/dm^3, whereas in the initial solution was 2.8 g/dm^3, which indicates that the ferrous iron gradually becomes trivalent due to the active oxidation of iron by acidophilic bacteria.

Table 5 Characterization of the growth and cultivation of bacteria in the leaching solution

| Parameters | Initial indicator | Indicator after 5 days |
|------------|-------------------|-----------------------|
| pH         | 2.2               | 1.8                   |
| Eh, mV     | 897               | 821                   |
| Fe^{3+}, g/dm^3 | 2.8           | 7.6                   |
| Fe_{total}, g/dm^3 | 2.8              | 7.6                   |

From the data, it can be seen that during normal cyanidation without prior acid washing and bacterial dissection, part of the cyanide is spent on the extraction of accompanying elements, such as copper, iron, arsenic, zinc. It also leads to a decrease in the extraction of the main valuable component - gold. Preliminary acid washing of the sample with subsequent neutralization allows transferring a significant part of the interfering impurities in the acid solution, which increases the efficiency of further cyanidation. Thus, with conventional cyanidation, gold recovery amounted to 57.41%, and during cyanidation with preliminary acid washing, it was 62.1%.

The use of sodium peroxide as an additional oxidizing agent, in combination with a preliminary acid washing, made it possible to extract 65.2%. The use of the bacterial culture of A. Ferrooxidans in the preliminary oxidation of the raw material under study, later on during cyanidation, made it possible to achieve the maximum, compared to other options, gold recovery rate - 72.0%. As a result of the experiments, a method was developed for the extraction of gold from the flotation tailings, including pretreatment of flotation tailings with sulfuric acid and A. Ferrooxidans bacterial solution for 120 hours, followed by neutralization of the acidic medium and cyanidation. On the basis of the obtained data, a technological scheme was developed for processing this type of raw material, shown in Figure 8.

Table 6 - Extraction of gold and impurities of other elements into solutions with different variants

| Options               | Extraction of elements into solution, % |
|-----------------------|-----------------------------------------|
|                       | Cu | Zn | Fe | As | Au |
| Solute after acid washing | 78.02 | 5.53 | 18.43 | 2.16 | 0.0 |
| Solute after bacterial dissection | 67.39 | 5.70 | 92.49 | 4.68 | 0.0 |
| Variant 1 (Final productive solute) | 82.94 | 1.73 | 66.70 | 14.04 | 57.41 |
| Variant 2 (Final productive solute) | 3.11 | 0.23 | 8.84 | 1.44 | 72.0 |
| Variant 3 (Final productive solute) | 1.06 | 0.16 | 6.26 | 1.33 | 65.2 |
| Variant 4 (Final productive solute) | 1.61 | 0.28 | 13.63 | 0.25 | 62.1 |

Figure 8 - Scheme of the biochemical method of gold recovery from the flotation tailings of the gold production plant
Conclusions

Thus, from the obtained data on the study of gold-bearing raw materials - dump flotation tailings, the following main conclusions are formulated:

Physical and chemical properties, mineralogical composition of the technological sample of flotation tailings were studied using modern methods and analytical instruments of research. Electron microscopic and mineralogical studies have established the presence of gold by irregular, isometric morphology in fine form in free form and intergrowths, with a particle size of 2.5×3.9 microns and 1.2-8.2 microns, respectively, gold grain, it is also found as a residual sulfide mineral and quartz.

The main studied element in the composition of the samples studied is gold, and to the chemical method of analysis it was revealed that the gold content in the initial sample is in the range of 0.43-0.47 g / t, the accompanying element of silver is 0.62 g / t. As a result of X-ray phase analysis, it was found that the main rock-forming minerals are quartz - 35.7%, dolomite - 15.2%, albite - 14.3%, and the presence of residual sulfides in the form of pyrite - 7.9%;

The expediency of preliminary removal of non-ferrous metal and iron impurities by sulfuric acid washing was confirmed, which makes it possible to increase the efficiency of cyanidation;

The positive effect of preliminary oxidation of the technological sample, contributing to the additional extraction of gold from residual sulphides;

The optimal concentration of 9K nutrient medium for Acidithiobacillus Ferroxoxidans strains used for bio-opening of the flotation tailings was selected, the adaptation of the bacterial culture to this raw material was analyzed.

The most accepted biochemical method of processing flotation tailings has been established - including preliminary acid washing, subsequent bacterial oxidation, neutralization and cyanation. The degree of extraction of gold from the flotation tailings during biochemical leaching reached 72%, which is 6.8% more than with the use of a chemical oxidant sodium peroxide. In addition, the bacterial solution is less expensive and non-toxic in comparison with the oxidizing agent - sodium peroxide. Spent biochemical solutions, after measuring the residual living cells of bacteria and their nutrients, can be regenerated and reused. Also, with the accumulation of sufficient concentrations of non-ferrous metals in waste solutions after acid washing, their further processing by hydrometallurgical technologies for producing copper and zinc is possible.

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Биототықтыруды қолдана отырып алтынды флотациялық қалдықтардан ерітінділеудің гидрометаллургиялық әдісін зерттеу

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Түйіндеме
Макала деп алынды техногендік флотация қалдықтарынан агитациялық шығарылу ведисемен алуың және алдына ала биототықтыру ерітіндіге алуың салыстырылып зерттеу нәтижелері келтірілген. Флотациялық қалдықтарының үлгісі «Алтынтау Көкшетау» ЖШС алтын өндіру фабриканың алыны, Тексерілген сынамада 0,47 г / т Au және 0,62 г / т Ag бар екендігі анықталды. 80% 0,071 мм ірілік класындағы (фракциясындағы) флотация қалдықтарының алының алыну денгейі 50% құрайды, ал 90% 0,071 мм фракциясында 60% құрайды. 0 – 0,044 мм фракциясына дейін ұшытқалған алтын өндіретін фабриканың флотациялық қалдықтары натрий пероксидімен алыны ала тотықтырылады. Қатты заттың сұйыққа қатынасы 1:4 және шығару
Исследование гидрометаллургического метода выщелачивания золота из хвостов флотации с использованием биоокисления

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Аннотация. В статье приведены результаты сравнительного исследования эффективности методов извлечения золота из техногенных хвостов флотации методом агитационного цианирования и с предварительным биоокислением. Представительный образец хвостов флотации был взят на золотонизвлекательной фабрике TOO «Алтынтау Кошетау». Установлено, что в исследуемом образце содержится 0,47 г / т Au и 0,62 г / т Ag. Степень извлечения золота из хвостов флотации с содержанием 80% класса крупности (фракции) 0,071 мм составляла 50%, а с содержанием 90% класса крупности (фракции) 0,071 мм составляла 60%. Флотационные хвосты золотоизвлекательной фабрики, измельченные до фракции 0-0,044 мм, подвергались предварительному окислению перекисью натрия. Степень извлечения золота из хвостов флотации с содержанием 90% фракции 0,044 мм при соотношении твердого вещества и жидкости 1: 4 и концентрации цианида 1 г / дм³ составляет 65,2%. При использовании активного штамма тионных культур степень извлечения золота составила 72,1%.

Ключевые слова: гидрометаллургия золота, биологическое выщелачивание, хвосты флотации, биоокисление.

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