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PREPARATION OF PYRITE-ARSENOPYRITE CONCENTRATE FOR GOLD LEACHING THROUGH THE USE OF POLYREAGENT COMPLEXES

The article focuses on the causes of refractoriness of sulfide ore stock with superdispersed gold inclusions. In order to increase gold recovery, a hypothesis about effective (peroxide) preparation of refractory raw materials in the process of leaching has been described. The author focuses on theoretical justification of the physical-chemical model and the two-stage mechanism of oxidation (photoelectrochemical and bacterial) of material using microorganisms *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans*. A scheme of two-staged (physicochemical and bacterial) oxidation was developed. It is based on photoelectrochemical effects for the leaching of gold from refractory materials. It is shown that photoelectrochemical pre-oxidation makes it possible to increase development of the mineral matrix with its further bacterial oxidation. It also helps to increase the efficiency of invisible gold extraction from sulfide ores. Laboratory tests have been performed on the combined oxidation scheme on electroactivation carbon-in-leach of invisible gold from the Kokpatas deposit's sulfide ores. The authors present the results of the laboratory tests of the activation methods for impacting upon the refractory ores before metal leaching, which prove the efficiency of such non-traditional methods for developing mineral matrix. Gold extraction using the combined technology of sulfide and arsenopyrite oxidation increases by 18 %.

Keywords: refractory ores, pyrite, arsenopyrite, invisible gold, physical and chemical oxidation and bacterial oxidation, polyreagent schemes, reactive oxygen, electrochemical and photochemical processes.

The widespread natural occurrence of gold ores with invisible valuable components conditions the need for their large-scale involvement in ore processing. One of the ways to enhance the efficiency of refractory ore gold leaching is to intensify the process of refractory sulfide matrix destruction. In this regard, a new scientific approach to the selection of effective recovery methods for refractory gold minerals is required [1–10].

The research subject is poor bulk pyrite-arsenopyrite float concentrate with a gold content of 25 g/t, obtained from refractory sulfide ore from Kokpatas deposit ($\beta^{\text{Au}} = 2.9 \text{ g/t}$, $S_{\text{tot}} = 5.8 \%$, $S_{\text{sul}} = 5.6 \%$, $S_{\text{oxid}} = 0.13 \%$, $\text{Fe}_{\text{tot}} = 7.4 \%$, $\text{Fe}_{\text{sul}} = 4.2 \%$, $\text{As}_{\text{tot}} = 0.52 \%$, $C_{\text{tot}} = 1.2 \%$, $C_{\text{tot}} = 0.44 \%$).

The research objective is to substantiate and develop combined methods for the processing of refractory gold-bearing raw materials through targeted photoelectrochemical effects using strong oxidizers.

The research task is to conduct experimental investigations of the influence of two-stage oxidation on mineral matrix

development in the course of refractory mineral processing.

Research techniques: multifactorial experiment planning techniques, granulometric, mineralogical, spectral, chemical (including phase), X-ray phase, optical and electron microscopic, microscopic, bacterioscopic, atomic absorption, assay, X-ray diffraction analysis and other analysis techniques, plus aggregate laboratory tests on minerals. Experimental data were subjected to mathematical treatment.

Intensification of the refractory mineral oxidation process and enhancement of the efficiency of subsequent gold recovery in the course of sorption cyanidation is achieved through two-stage oxidation: physicochemical (through targeted photoelectrochemical effects) and bio-oxidation [11, 12].

The essence of the process is that prior to bacterial oxidation, sulfide materials are subjected to a preliminary oxidizing (peroxide) preparation by treating the mineral matter with reagents, containing reactive oxygen, which were obtained through a photochemical and electrochemical synthesis from primary gases, chemical compounds and water.



As is known, the Eh difference between the environment and electrical potential of sulfide minerals during oxidation shall lie within 100 to 200 mV; if they are equal, no oxidation will take place, while a lower difference value testifies to low oxidation efficiency, so

$$Eh_{\substack{\text{react. medium} \\ \text{(oxidizer gas} \\ \text{mixture)}}} - Eh_{\text{sulph. matrix}} = 100 \text{ to } 200 \text{ mV. (1)}$$

The predominant propagation of Acidithiobacillus ferrooxidans and Acidithiobacillus thiooxidans is limited by a rather narrow interval of Eh (400 to 750 mV). Bacteria do not develop in the reducing environment and die after long-term exposure to such conditions. In this case, consideration should be given to the fact that at Eh 400 mV leaching of iron take place, and at Eh 750 mV that of sulfur. The required Eh values:

$$Eh_{\substack{\text{opt} \\ \text{oxidizer gas} \\ \text{mixture}}} = Eh_{\text{sulph. matrix}} + 200 \text{ mV} =$$

= 1400 + 200 = 1600 mV – for peroxide preparation, where $Eh_{\text{sulph. matrix}} = 1400 \text{ mV}$ is sulfide matrix Eh.

After gas phase, UV irradiation is complete, decomposition of reactive oxygen and, primarily, O_3 takes place. Upon ozone introduction into the pulp, its greatest part degrades into O_2 and O^* . Reactive atomic oxygen, in its turn, transforms into singlet oxygen with a redox potential being 3-4 times lower than that of ozone. Other reactive forms of oxygen are more stable. As the ozone concentration experiences a 3 to 4-fold decrease, Eh decreases in direct proportion.

The two-stage oxidation duration (T_{treat}) is composed of the time of photoelectrochemical oxidation (t_1), bio-oxidation (t_2), and the reaction period of polyreagent complexes of reactive oxygen (chlorine-containing compounds) with the pulp mineral phase (Δt). The duration of photoelectrochemical oxidation depends on the constituent phases. The first is the phase of photoelectrochemical effects

determined by the dissolution rate of reactive oxygen in the acid solution for obtaining the required reactive oxygen content:

$$t_1^I = \frac{C_{O_2}^{\text{final}} - C_{O_2}^0}{v_1} = \frac{15 - 10}{2} = 2.5 \text{ h, where the}$$

initial reactive oxygen concentration $C_{O_2}^0 = 10 \text{ mg/l}$, the final reactive oxygen concentration $C_{O_2}^{\text{final}} = 15 \text{ mg/l}$, the oxygen dissolution rate in the solution $v_1 = 2 \text{ mg/(l}\cdot\text{h)}$.

The second phase of photoelectrochemical oxidation is determined by the reaction rate of hydrogen (low-soluble) and reactive oxygen with the production (synthesis) of reactive ion-radical complexes and the rate of their interaction with the mineral matrix:

$$t_1^{II} = \frac{C_{H_2O_2}^{\text{final}} - C_{H_2O_2}^0}{v_2} = \frac{20 - 2}{1.8} = 10 \text{ h,}$$

where the initial concentration $C_{H_2O_2}^0 = 2 \text{ mg/l}$; the final concentration $C_{H_2O_2}^{\text{final}} = 20 \text{ mg/l}$, H_2O_2 formation rate during hydrogen and ozone interaction $v_2 = 1.8 \text{ mg/l}\cdot\text{h}$, $\Delta t = 2 \text{ h}$.

Thus, the duration of photoelectrochemical oxidation is 14.5 h (the parameter values were assumed according to the experimental data obtained on the Yellow Jacket deposit ores [17]).

The duration of the bio-oxidation stage depends on the duration of four phases:

phase 1 being a lag phase, i.e. the initial development phase characterized by the bacteria adaptation to the ambient environment:

$$\frac{t_2^{\text{bio}}}{t_2^{\text{physchem+bio}}} = \frac{40 \text{ h}}{10 \text{ h}};$$

phase 2 being an exponential phase comprising an acceleration phase, an exponential growth phase and a growth deceleration phase:

$$\frac{t_2^{\text{bio}}}{t_2^{\text{physchem+bio}}} = \frac{50 \text{ h}}{20 \text{ h}};$$

phase 3 being a stationary phase:

$$\frac{t_2^{\text{bio}}}{t_2^{\text{physchem+bio}}} = \frac{15 \text{ h}}{15 \text{ h}};$$



phase 4 being the bacteria dying stage $\frac{t_2^{\text{bio}}}{t_2^{\text{physchem+bio}}} = \frac{20 \text{ h}}{20 \text{ h}}$, where t_2^{bio} is the duration of the bio-oxidation phases for the classic (tank) option, h; $t_2^{\text{physchem+bio}}$ is the duration of the bio-oxidation phases with preliminary two-stage oxidation, h.

$$\frac{\sum t_2^{\text{bio}}}{\sum t_2^{\text{physchem+bio}}} = \frac{125 \text{ h}}{65 \text{ h}} = 1.93 \quad (2)$$

Consequently, the duration of bio-oxidation features a 2-fold decrease on average if preliminary photoelectrochemical oxidation is applied before leaching.

The experimental verification (aggregate laboratory tests) of the two-stage oxidation theory was conducted using a process scheme simulated on the pulp of leach plant GMZ-3 (ref. Fig. 1). The priority of the proposed process is confirmed by the following patents: 2350665, 2361937 [13, 14].

For experiments, use was made of an integrated sample containing 20 individual samples, weighing 10 kg each. The granulometric characterization of gold in the flotation pyrite-arsenopyrite concentrate showed that the major part of the valuable component (99.9%) falls under the 0.074-mm grain-size category, i.e. there is practically no gold in the ore (except for the South 1 site with visible gold).

The sulfide-arsenide (pyrite-arsenopyrite) concentrate is prepared using a combination of peroxide-hydroxide complexes produced by ozonized air bubbling of the electrolytic cell interelectrode space in sulfuric environment followed by additional oxidation using heterotrophic bacteria [15]. The pyrite-arsenopyrite ratio in pyrite-arsenopyrite ores is subject to continuous changes, so the bio-oxidation efficiency is reduced. Combined oxidation is proposed to eliminate this deficiency.

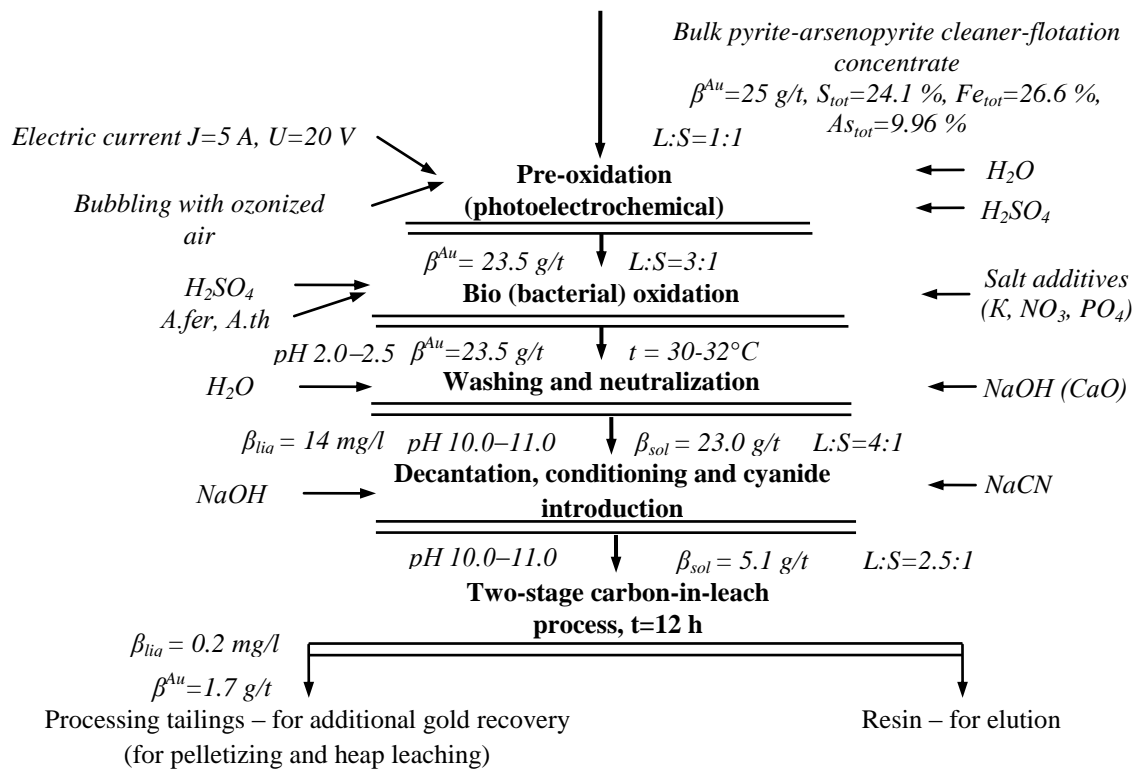


Fig. 1. Process scheme of activation leaching of poor pyrite-arsenopyrite gold-bearing concentrate through combined oxidation techniques



The laboratory setup for the process research of ores included the following equipment: a 30-liter laboratory agitator with tubular electrodes used as air tubes; a quartz flask with a fluoroplastic (chemically inert) plug having inlet and outlet glass tubes passed through it; a compressor and an electrical cabinet with a rectifier and a regulated unit; DRT-230 UV lamp mounted above the flask at a distance of about 15 cm (for direct pulp irradiation, it is secured by means of two stands); a photoelectrochemical reactor; a bacterial oxidation column; a bacteria propagator; a vessel for bacteria decontamination.

The proposed process included the following sequence of operations: preparation of a water-based leaching solution; electrolysis of the bubbled solution (≈ 1.5 h, $U = 20$ V), H_2SO_4 introduction; UV irradiation (DRT-230 UV lamp, 5 min with ongoing electrolysis); mineral matter treatment with a polyreagent solution, oxidation; introduction of salt additives, supply of an additional portion of sulfuric acid, introduction of *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans*; bacterial oxidation with controlled arsenate ion concentration; washing and neutralization with the addition of NaOH or CaO; conditioning up to pH 10–11, introduction of NaCN (up to 1 g/l) and precyanidation; two-stage sorption leaching.

Initially, two-stage oxidation of the pyrite-arsenopyrite float concentrate was performed using a sulfuric acid solution (3%) treated in an electrolyzer and bubbled for 1 hour with air supplied through the quartz flask arranged within the DRT-230 UV lamp radiation zone. The reactor capacity is 30 L. The obtained polyreagent solution representing a combination of a reactive oxidizer and a complexing substance was used to treat the mineral matter [16]. Exposure to the polyreagent complex caused intensive physical and chemical processes in the liquid and solid phases, which resulted in the primary oxidation of the sulfide matrix, transformation of its surface layers,

mainly in the area of active centers, into a sulfate form and partially into a sulfide form, and creation of further favorable conditions for bacterial oxidation. The matrix of the invisible goal containing minerals demonstrated at least a 60-65% oxidation.

After subjecting ore to the photoelectric activation treatment, the second oxidation stage, bio-oxidation, was performed. Bacteria were introduced on a slurry carrier, formed by grinding. In this case, development of the bacteria on the slurry carrier is relatively fast and at the same time allows for concentrating the bacteria before introducing them into the basic matter for leaching, which results in the fast development of bacteria growth centers in the mineral matter.

Precyanidation was performed to obtain the maximum gold concentration into the solid phase solution. In the course of sorption leaching, full dissolution of gold takes place as well as its activated carbon sorption. The optimal physicochemical and process parameters (L:S = 5:1, duration of leaching (36 h) and precyanidation (5 h), gold content in liquid and solid phases versus the preliminary cyanidation cycle time) were determined experimentally. The precyanidation time was reduced by 1 h for the pulp air bubbling with a view to improve the recovery during sorption leaching. The experimental research results are presented in Fig. 2 to 7 and in Table 1. Arsenic contained in the arsenopyrite is an inhibiting factor for the bacterial leaching process, so the culture was adapted to the extremely high arsenic content. In order to reduce the dissolved arsenic concentration and preserve the residual reactive oxygen that would intensify subsequent bacterial oxidation, the solid content in the activated pulp was brought to L:S = 5:1. The pulp solid phase experienced a significant increase in the elemental sulfur (over 3 times) and sulfates (almost by an order).



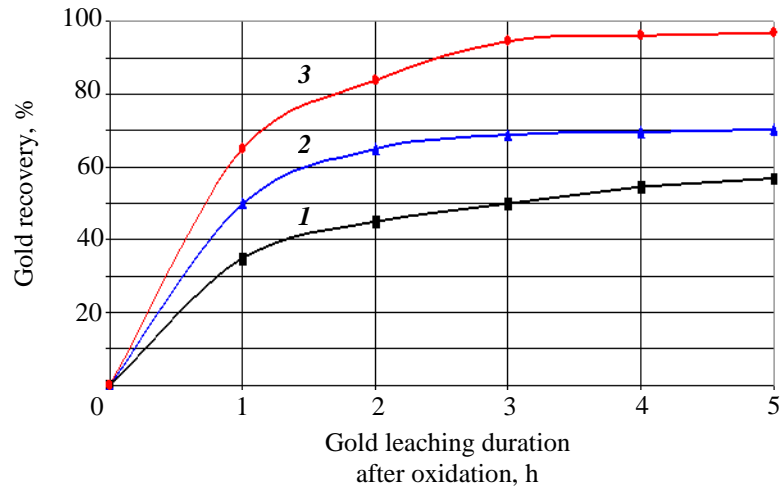


Fig. 2. Gold recovery vs pulp density during cyanidation:
 1 – L:S = 1.5:1; 2 – L:S = 3:1; 3 – L:S = 5:1

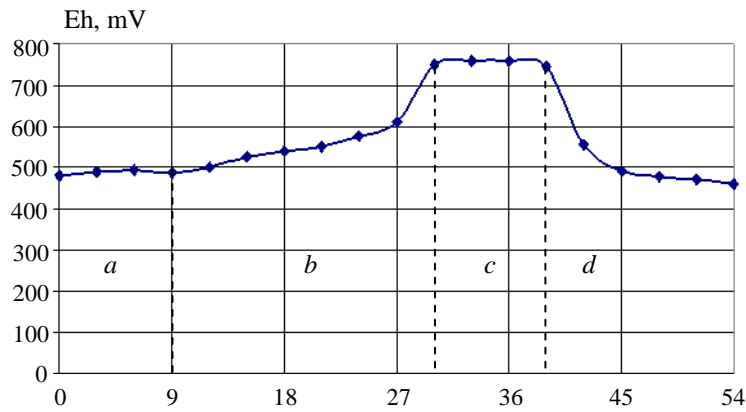


Fig. 3. Eh vs iron leaching duration:
 a – lag phase;
 b – exponential phase;
 c – stationary phase;
 d – dying phase

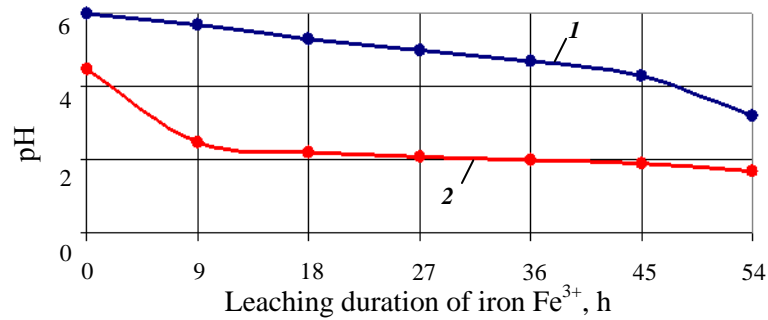


Fig.4. Acid production vs leaching duration:
 1 – bio-oxidation (reference option)
 2 – physicochemical and bio-oxidation (experimental option)



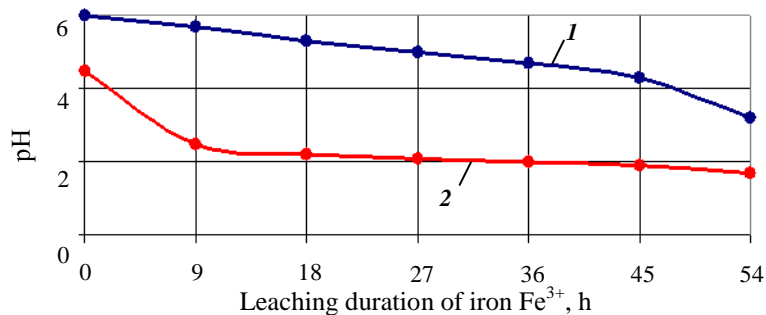


Fig. 4. Acid production vs leaching duration:
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 2 – physicochemical and bio-oxidation (experimental option)

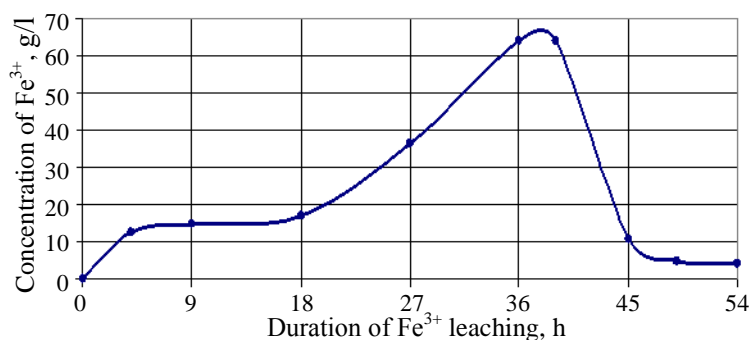


Fig. 5. Fe³⁺ concentration in solution vs leaching duration

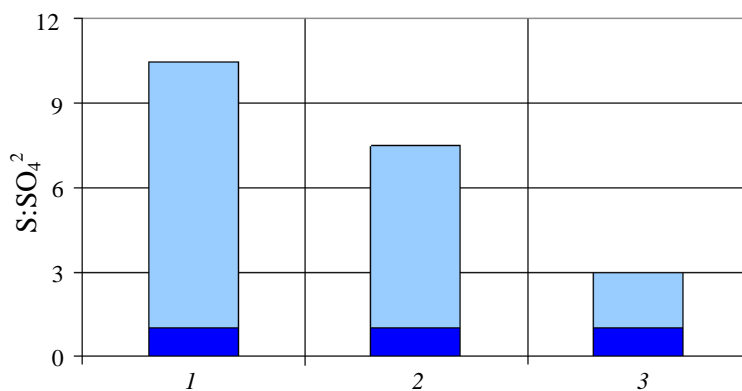


Fig. 6. Ratio of unoxidized (light) and oxidized (dark) sulfur species (S:SO₄²⁻):
 1 – in base ore (9.5:1);
 2 – after physicochemical oxidation (6.5:1);
 3 – after physicochemical and bio-oxidation (2.0:1)

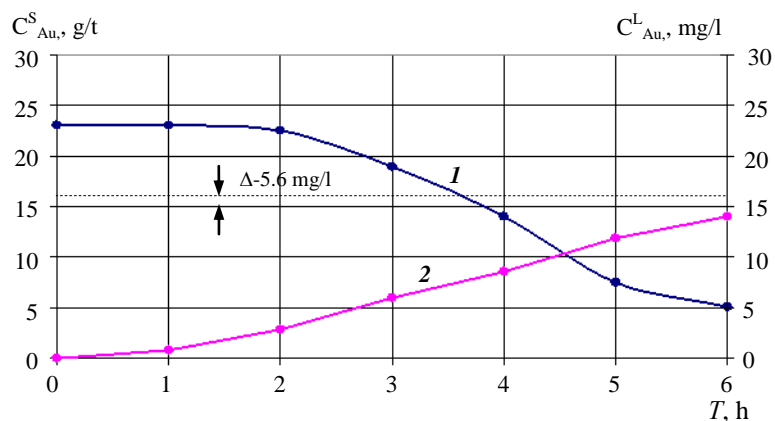


Fig. 7. Gold content in liquid and solid phases vs sorption cyanidation time:
 1 – gold content in solid phase, g/t;
 2 – gold content in liquid phase, mg/l.

Table 1

Sulfide concentrate leaching behavior

Time of leaching, h	Eh, mV	Period of leaching	Concentration in solution, g/l		Number of bacteria cells/ml
			Fe ²⁺	Fe ³⁺	
0	490	Initial	5.1–5.6	12.4–18.7	4 × 10 ⁶
3	490				
6	493				
9	487				
12	500				
15	525	Active	0	33.2–63.9	10 × 10 ⁷
18	540				
21	550				
24	577				
27	610				
30	750				
33	758				
36	760				
39	745				
42	555				
45	492	Decline	0	20.7–26.3	4 × 10 ⁷
48	477	Final	0	5.5–5.1	3 × 10 ⁶
51	470				
54	460				

Durations of the lag phase (12 h) and exponential phase during which the microbial population growth and activity are described by the exponential law (30 h) are essential for the bacterial oxidation process. The total leaching duration was determined by the duration of the said two phases while the actual time, by the exponential phase duration.

The bio-oxidation (BO) process was accompanied by a decrease in pH and an increase in the redox potential of the product

solution. In order to ensure active activation of iron (II) – Fe²⁺, the pH value was maintained at an optimal level of 2.0 to 2.5. In the course of 24-hour arsenopyrite and pyrite leaching, the solution's pH varied from 4.5 to 2.1 and reached a value of 1.7 in 72 hours. In so doing, active bio-oxidation of arsenopyrite and pyrite took place along with reactive iron hydrolysis with sulfuric acid generation. The Eh value was characteristic of the oxidation-reduction reaction progressing in the pulp. The sulfide-sulfosalt



The distinguishing feature of the Darasun field ores (Darasun, Teremkinskoye, Talatui gold ore deposits) is the high content (15 to 60%) of sulfides (pyrite, arsenopyrite, chalcopyrite, galenite, sphalerite, pyrrhotine, etc.) and sulfosalts.

Further development of the Transbaikal raw material base is planned through the introduction of new competitive high-performance ore processing technologies requiring no high capital or operational costs and providing a quick return on investments as compared to the traditional valuable component recovery techniques.

Thus, use in manufacturing processes of highly reactive non-toxic oxygen-hydrogen ion-radical compounds, obtained through targeted photoelectrochemical effects, provides for a significant increase in the invisible gold recovery from the poor bulk pyrite-arsenopyrite float concentrate and involvement into processing of mineral stock sites with potentially large gold reserves that serve as sources for the national gold reserves.

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