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Combined scheme for conditioning circulating cyanide solutions

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Abstract: Due to the specific features of hydrometallurgical processing, where cyanide solutions are used, the composition of the leaching solution undergoes periodic changes referred to in the literature as "fatigue." This adversely affects the rate of gold recovery and cementation, and therefore the overall efficiency of cyanide leaching technology. One of the most important markers determining the "fatigue" of the solution is copper. This study examined the possibility of applying a combined scheme for the purification of circulating cyanide solutions with high concentrations of copper (1196 mg/dm^3), iron (111 mg/dm^3), arsenic (19 mg/dm^3), and sodium cyanide (0.94 g/dm^3). A two-stage technology (reverse osmosis + chemical precipitation) was developed for the reduction of treated solution volumes and the removal of impurities. At the first stage, the solution was separated in a reverse osmosis unit equipped with LP22-8040 membranes, producing permeate and concentrate in a 1 : 1 ratio. The permeate (12 mg/dm^3 Cu and 0.01 mg/dm^3 Fe, pH = 11.13) was returned to the process cycle. At the second stage, the concentrate, which contained 99 % of the initial impurities, was further purified by the stepwise addition of a $CuSO_4$ solution (70 g/dm^3 Cu) in 1–11 cm^3 doses under stirring (500 rpm, 10 min). The results showed that the optimal $CuSO_4$ dose (11 cm^3) provided removal of more than 86 % of Cu from the initial solution, as well as 100 % of Fe and more than 96 % of As. The precipitate obtained in the process consisted of 68.3 % copper, with CuCN and $Cu(OH)_2$ as the main components.

Keywords: gold, cyanide leaching, Merrill-Crowe, cementation, reverse osmosis, copper, arsenic, surface passivation.

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Комбинированная схема кондиционирования оборотных цианистых растворов

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Аннотация: Ввиду специфики работы гидрометаллургических переделов, где применяются цианистые растворы, происходит периодическое изменение состава выщелачивающего раствора, получившее в литературе название «утомляемость». Это в негативном ключе влияет на скорость процесса извлечения и цементации золота, а следовательно, в целом на эффективность технологии цианистого выщелачивания. Одним из наиболее важных маркеров, определяющих «утомляемость» раствора, является медь. В работе исследовалась возможность применения комбинированной схемы очистки оборотных цианистых растворов с высоким содержанием примесей меди (1196 мг/дм^3), железа (111 мг/дм^3), мышьяка (19 мг/дм^3) и цианида натрия (0,94 г/дм^3). Разработана 2-этапная технология (обратный осмос + химическое осаждение) для сокращения объемов обрабатываемых растворов и удаления примесей. На первом этапе раствор разделяли на обратноосмотической установке с мембранами LP22-8040, получая пермеат и концентрат в пропорции 1 : 1. Пермеат (12 мг/дм^3 Cu и 0,01 мг/дм^3 Fe, pH = 11,13) возвращали в технологический цикл. А на втором этапе концентрат, содержащий 99 % исходных примесей, доочищали дозированным введением раствора $CuSO_4$ (70 г/дм^3 Cu) с интервалом доз 1–11 см^3 при перемешивании (500 об/мин, 10 мин). Результаты показали, что оптимальная доза $CuSO_4$ (11 см^3) обеспечивает удаление более 86 % Cu из исходного раствора, а также 100 % Fe и более 96 % As. Полученный в процессе осадок на 68,3 % состоит из меди, а основными компонентами являются CuCN и $Cu(OH)_2$.

Ключевые слова: золото, цианистое выщелачивание, Меррилл-Кроу, цементация, обратный осмос, медь, мышьяк, пассивация поверхности.

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Introduction

In 2010, a cyanide leaching facility for gold extraction from flotation concentrate was established at the concentration plant of LLC "Berezovsky Rudnik", which processes ore from Russia's oldest deposit of the same name. Over the years of operation, continuous improvement of the process and equipment enabled mine specialists to achieve 95—97 % gold recovery into the commercial cement product, while reducing the gold content in the leach tailings of the concentrate to as low as 0.2—0.1 g/t. It is noteworthy that comparable results for direct leaching of gold from pyrite concentrate have not been reported in the literature.

In hydrometallurgical processing, where cyanide solutions are employed, the composition of the leaching solution undergoes periodic changes, described in the literature as "fatigue" [1]. This phenomenon has an adverse effect on the rate of gold recovery and cementation, and thus on the overall efficiency of cyanide leaching technology. One of the most important indicators of solution "fatigue" is the copper concentration in circulating solutions. Previous studies have confirmed the negative impact of "fatigued" solutions on the kinetics of gold leaching [2]. According to the theory proposed by M.D. Ivanovsky, elevated copper concentrations in circulating solutions promote the formation of passivating CuCN films on the gold surface:

$$[Cu(CN)_4]^{3-} = CuCN\downarrow + 3CN^-. \qquad (1)$$

The density of these films depends on the copper concentration in the solution. The formation of cop-

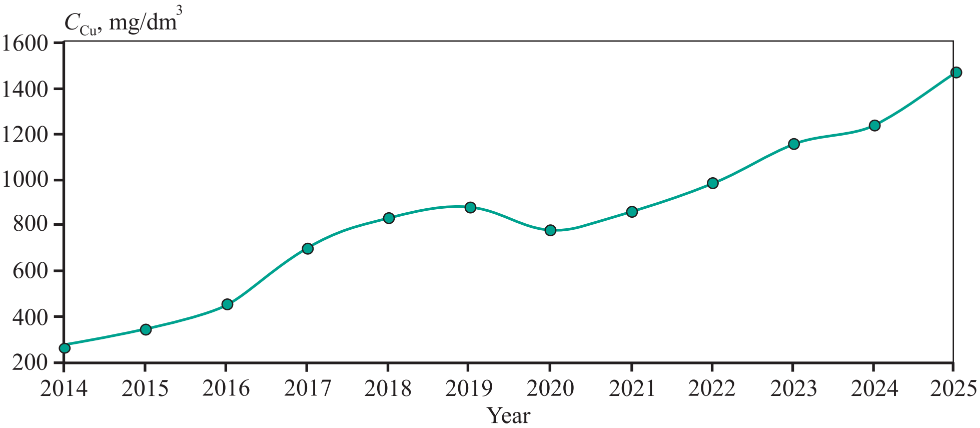

Fig. 1. Average annual copper concentration in the circulating solution

Рис. 1. Среднегодовое содержание меди в оборотном растворе per (I) cyanide (CuCN) becomes possible when the cyanide concentration in the diffusion layer decreases during the dissolution of gold [1; 3—8].

Over the years of hydrometallurgical operation, the copper content in the circulating solution has shown a steady increase (Fig. 1).

In hydrometallurgical practice, short-term periods (1—2 days) are sometimes observed during which leaching efficiency decreases and the gold content in the leach tailings rises to 1.0—2.5 g/t. After this period, with process parameters maintained within the specified limits, leaching performance returns to the optimal level. Prompt laboratory tests ruled out possible causes such as the periodic presence of coarse gold in the concentrate (which requires longer dissolution time) and deviations from process regulations, including low cyanide concentration or insufficient exposure of metal. At the same time, analysis of the leach tailings confirmed the potential for further recovery of gold to previously achieved levels of 0.1—0.2 g/t. Elevated copper concentrations in circulating solutions were found to adversely affect gold precipitation in the Merrill—Crowe plant (Fig. 2). Possible reasons include increased zinc consumption due to copper reduction and passivation of zinc particle surfaces by a dense copper layer. Additional complications occur during filtration of the cementation product: at higher copper concentrations in the pregnant solution, the hydraulic resistance of the cement layer increases sharply. In any case, cementation of gold becomes more difficult, and plant productivity declines.

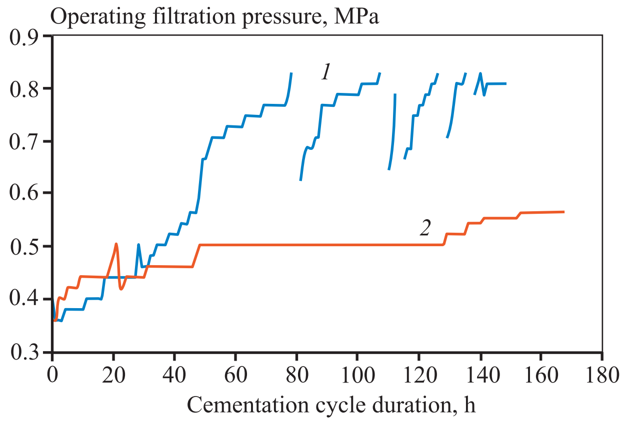

Fig. 2. Change in filter pressure during gold precipitation

1 – cycle with elevated copper content; *2* – standard cycle

Рис. 2. Изменение давления на фильтре осадка в процессе осаждения золота

1 – цикл с повышенным содержанием меди; *2* – стандартный цикл

Table 1. **Characteristics of zinc precipitates**

Таблица 1. Особенности цинковых осадков

Cycle No.	Mass of precipitate, kg	C_{Cu}, % (kg)
1 (abnormal)	171	56.0 (96.1)
2 (normal)	80.4	8.9 (7.15)

The discontinuity of curve 1 reflects shutdowns of the Merrill—Crowe unit caused by a sharp increase in filter-press pressure. When the unit stopped, the cement layer partially detached from the vertical filter surface, and after the feed pump was restarted, the pressure temporarily returned to normal. At the end of the cycle, visual inspection of the cement product revealed dense plates resembling metallic copper. Subsequent analysis showed more than a twofold increase in cement product mass and abnormally high copper content (Table 1).

To partially address this problem, it is recommended to add larger amounts of soluble lead salts to the pregnant solution, which suppress copper precipitation [3]. However, practice shows that at elevated copper concentrations, the effectiveness of lead addition decreases.

Another negative effect of copper in circulating solutions is its interference with the analytical determination of cyanide concentration [9], which may lead to potential violation of process regulations and under-recovery of gold.

The literature describes several methods for removing copper from circulating solutions, which can be classified according to the Cu-containing product formed: CuCN — ARV process [1; 10]; Cu_2S — MNR process [11—16]; $Cu(OH)_2$ — treatment with oxidizing agents [17—20]; Cu^0 — cementation with zinc or electrolysis [3].

In this study, a combined conditioning scheme for circulating solutions was investigated, consisting of two stages.

1. Reverse osmosis for concentrating impurities in a reduced volume. Reverse osmosis (RO) is a technology that removes ions, molecules, and impurities from water through membranes under high pressure [21—24]. The feed solution is separated into two streams: concentrate and permeate. The permeate is an impurity-free solution, while the concentrate contains nearly all components of the original solution. The product volumes and separation coefficients depend on the membrane properties and system pressure.

2. Purification of the concentrate through the formation of sparingly soluble copper (I) cyanide. For impurity precipitation, copper sulfate ($CuSO_4$) was proposed. Prototypes of this technological stage include processes involving treatment with iron salts and oxidation with hydrogen peroxide, where soluble copper salts are used as catalysts for cyanide destruction in wastewater [17—20]. The method considered in this study provides for the precipitation of copper as well as several other impurities accumulated during flotation concentrate leaching.

Thus, the aim of this study was to develop a combined two-stage technology (reverse osmosis + + chemical precipitation with $CuSO_4$) for conditioning circulating cyanide solutions with high concentrations of copper, iron, and arsenic. The technology is designed to reduce treatment volumes and restore the functional properties of the solutions for gold cyanide leaching.

Materials and methods

The circulating cyanide solution from the gold leaching circuit of LLC "Berezovsky Rudnik" served as the object of the study.

Sampling of the circulating solution and reverse osmosis products was carried out on a pilot hydrometallurgical unit with a feed capacity of 60 m^3/h. The unit was equipped with LP22-8040 polyamide thin-film composite membranes with a total active surface area of 2040 m^2. Operating parameters were as follows: solution temperature 20 °C, product volume ratio (permeate : concentrate) 1 : 1, and inlet pressure 1.7 MPa. The feed solution and osmosis products were analyzed for metal and sodium cyanide content, with the results presented in Table 2. Once the permeate was purified to the target impurity level, it was returned to the cyanidation circuit. Further impurity removal from the concentrate was performed under laboratory conditions.

Copper precipitation was carried out using a known process [17; 25] based on the formation of sparingly soluble copper (I) cyanide:

$$Cu(CN)_4^{2-} + CuSO_4 = 2CuCN\downarrow + SO_4^{2-} + (CN)_2. \quad (2)$$

Copper precipitation from the concentrate was studied in a series of tests using a $CuSO_4$ solution (C_{Cu} = = 70 g/dm^3), in order to establish the dosage that yields the lowest copper concentration in solution.

For each test, 100 cm^3 of concentrate was placed in a beaker on a magnetic stirrer. A specified volume of $CuSO_4$ solution (C_{Cu} = 70 g/dm^3) was added at once, followed by stirring for 10 min. The resulting suspension was filtered to obtain clarified solution and precipitate. Solution and precipitate analyses were performed by atomic absorption spectroscopy using a Kvant-2 spectrometer (Russia). Sodium cyanide concentration was determined by titration with nickel nitrate, and pH was measured using an Anion 4100 pH meter (Russia).

Results and discussion

Analysis of the reverse osmosis products (Table 2) confirmed that the solution can be separated into two streams with different component concentrations. Impurities were virtually absent in the permeate. As shown in earlier studies [2], the kinetics of gold recovery using permeate are comparable to those obtained with process water. This allows the permeate to be returned to the circuit after fortification with sodium cyanide, thereby reducing the volume of treated solutions by half. More than 99 % of the impurities from the feed solution remained in the concentrate, which was used for further purification tests. The absence of gold and silver separation in the circulating solution is attributed to their low concentrations and the limitations of the pilot unit.

A series of six tests confirmed the expected dependence of copper precipitation on the $CuSO_4$ dosage

Table 2. **Composition of the circulating solution and reverse osmosis products**

Таблица 2. Составы оборотного раствора и продуктов обратного осмоса

Solution	C, mg/dm^3					C_{NaCN}, g/dm^3	pH
	Au	Ag	Cu	Fe	As		
Circulating	0.01	0.01	1196	111	19	0.94	11.13
Permeate	0.01	0.01	12	0.01	0.6	0.1	11.1
Concentrate	0.01	0.01	2379	222	37	1.78	11.1

Table 3. **Change in concentrate composition**

Таблица 3. Изменение состава концентрата

V_{CuSO_4}, cm^3	C, mg/dm^3					C_{NaCN}, g/dm^3	pH
	Au	Ag	Cu	Fe	As		
1	0.01	0.01	2500	128	7.5	0.06	7.86
3	0.01	0.01	2875	0	6.6	0	6.67
5	0.01	0.01	2240	0	5.5	0	6.56
7	0.01	0.01	1137	0	4.5	0	6.46
9	0.01	0.01	367	0	1.9	0	6.22
11	0.01	0.01	302	0	0.6	0	6.20

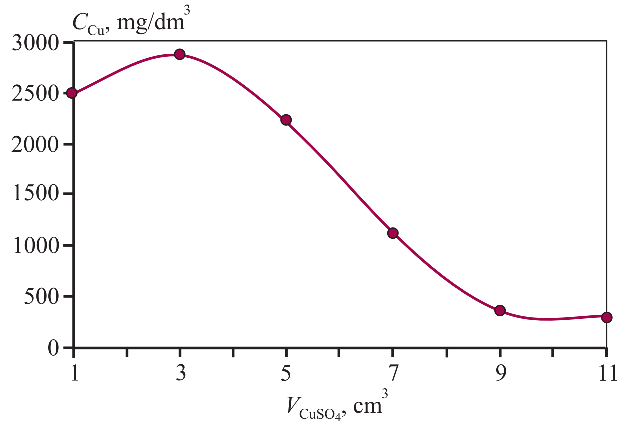

Fig. 3. Change in residual copper concentration in the concentrate with $CuSO_4$ addition

Рис. 3. Изменение остаточного содержания меди в концентрате при добавке $CuSO_4$

(Table 3, Fig. 3). The curve can be divided into two characteristic regions:

1. Increase in copper concentration in the concentrate. At low $CuSO_4$ dosages (1—3 cm^3), Cu^{2+} ions primarily reacted with iron cyanide complexes, free cyanide ions, and hydroxyl ions. This was accompanied by a sharp drop in iron (3) and cyanide ion concentrations (4), a decrease in pH to 6.67, and a rise in copper concentration (4):

$$2Cu^{2+} + [Fe(CN)_6]^{4-} = Cu_2[Fe(CN)_6]\downarrow, \quad (3)$$

$$2Cu^{2+} + 9CN^- + H_3O = \\ = 2[Cu(CN)_4]^{3-} + CNO^- + 2H^+. \quad (4)$$

Partial hydrolysis of copper ions also occurred:

$$Cu^{2+} + 2OH^- = Cu(OH)_2\downarrow. \quad (5)$$

2. Decrease in copper concentration in the concentrate. Further addition of $CuSO_4$ initiated reactions of Cu^{2+} with copper cyanide complexes, forming CuCN (1) and $Cu(OH)_2$ (5). Stabilization of pH at this stage is likely due to the buffering capacity of the sulfate ion:

$$SO_4^{2-} + H_2O = HSO_4^- + OH^-. \quad (6)$$

A reduction in arsenic concentration was also observed; however, the mechanism requires further investigation.

The purified concentrate solution can be returned to the circuit after mixing with permeate and reinforcing with caustic soda and sodium cyanide. A comparison of the purified concentrate—permeate mixture with the original circulating solution is shown in Table 4.

At the maximum $CuSO_4$ dosage, a precipitate with a mass of 0.98 g was obtained. Its composition was as follows (wt. %):

Cu 68.3
Fe 4.2
As 0.37
Other 27.13

Table 4. **Comparative analysis of solutions**

Таблица 4. Сравнительный анализ растворов

Solution	C, mg/dm^3				
	Au	Ag	Cu	Fe	As
Circulating solution	0.01	0.01	1196	111	19
Permeate + purified concentrate	0.01	0.01	157	0.005	0.6

The main components of the precipitate were $Cu(OH)_2$ and CuCN. This copper-rich material, after simple processing such as calcination, may be considered a marketable product. Following further chemical treatment, part of the copper in the form of $CuSO_4$ can be reused to condition fresh batches of circulating "fatigued" solutions.

Conclusions

1. Reverse osmosis technology enables a 2—3-fold concentration of impurities that accumulate in circulating cyanide solutions.

2. The study confirmed that soluble copper salts ($CuSO_4$) can be used to remove impurities from "fatigued" solutions in the form of a copper-rich precipitate.

3. Precipitation with copper sulfate achieved removal rates of 86.8 % for Cu, 100 % for Fe, and 96.8 % for As from the circulating solution.

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