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# APPLICATION OF NEW COLLECTORS IN FLOTATION OF COPPER-LEAD-ZINC ORE FROM THE TISHINSKY DEPOSIT

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**ABSTRACT**

The work is devoted to improving the technology of flotation enrichment of copper-lead-zinc ore of the Tishinsky deposit based on the development and research of new flotation reagents. It has been shown that increasing the efficiency of separation of sulfide minerals is an important factor in the rational use of mineral resources and improving the technical and economic performance of processing polymetallic raw materials. The aim of this study was to synthesize and evaluate the potential of new surfactants as collectors in the flotation of copper-lead-zinc ore. Alkylated diethanolamines and their dithiocarbamate derivatives were used as the subjects of the study. N-nonyl-N,N-di(2-hydroxyethyl)amine and disodium salt of N-nonyl-N,N-bis(2-xanthateethyl)amine were synthesized. The resulting reagents were found to be useful in copper-lead-zinc ore flotation processes, offering potential for increased selectivity and recovery of valuable components.

## INTRODUCTION

At the present stage, the field of mineral processing is developing rapidly, with widespread implementation of new technological solutions aimed at the comprehensive and efficient utilization of mineral raw materials. This trend is directly associated with the decreasing content of valuable components in ores, the increasing complexity of their mineralogical composition, and the tightening of industrial requirements. In this context, rational use of mineral resources, improvement of product quality, reduction of technogenic environmental impact, and minimization of energy consumption represent the key strategic objectives of the modern mining and metallurgical industry (Li et al., 2023).

The effective solution of these challenges requires further advancement of the theoretical and practical foundations of mineral beneficiation processes. In particular, in-depth investigation of the laws governing selective mineral liberation, their physicochemical properties in aqueous media, and the mechanisms of interfacial interactions is of great importance. In the processing of polymetallic ores, achieving high selectivity in mineral separation is one of the main factors determining the efficiency of beneficiation technologies (Zhao et al., 2022).

Flotation is one of the most widely applied and technologically advanced methods for the beneficiation of polymetallic ores. The efficiency of this process largely depends on the type of flotation reagents, their chemical nature, and their interaction with mineral surfaces. When added to the ore pulp, flotation reagents act as essential technological components that ensure or significantly facilitate mineral separation. Without reagents, the flotation process would be limited

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to only a few naturally floatable minerals. Among flotation reagents, collectors play a particularly important role. They adsorb onto the surfaces of naturally hydrophilic minerals, imparting hydrophobic properties to them. As a result, mineral particles effectively attach to air bubbles, enhancing both the intensity and selectivity of the flotation process. Therefore, the development of new, highly efficient, and selective collector reagents remains one of the most relevant directions in flotation theory and practice (Otaqulov et al., 2024).

The copper–lead–zinc ores of the Tishin deposit are characterized by a complex mineralogical composition and strong intergrowth of sulfide minerals, which leads to significant technological difficulties during beneficiation. Therefore, the application of next-generation reagents in the flotation processing of these ores can improve beneficiation performance, increase the recovery of valuable components, and enhance the overall economic efficiency of the process (Abramov, 2005).

In this regard, the investigation of novel surfactants based on alkylated diethanolamines and their dithiocarbamate derivatives as flotation collectors is of considerable scientific and practical interest. The use of such reagents can ensure selective flotation of sulfide minerals and contribute to the further improvement of polymetallic ore beneficiation technologies.

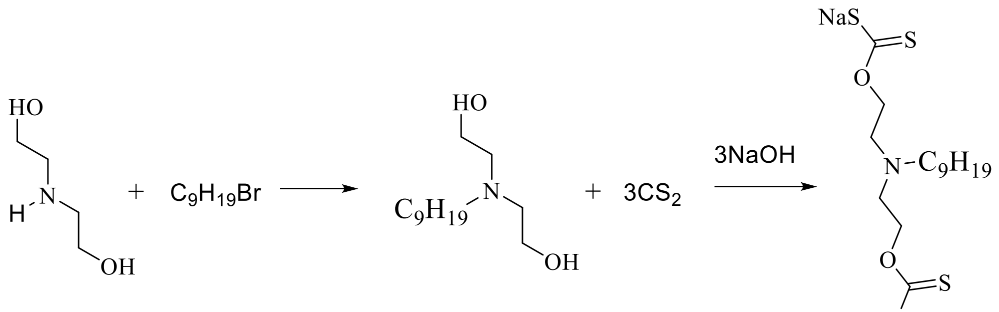
The aim of this study is to synthesize new surfactants and evaluate their potential as collectors in the flotation of copper–lead–zinc ores.

## MATERIALS AND METHODS

In order to obtain novel surfactants, alkylated diethanolamines and their dithiocarbamate derivatives were synthesized. Their flotation activity was investigated, and as a result, sample **A-3** was found to exhibit high collecting performance in the flotation of sulfide ores.

N-nonyl-N,N-di(2-hydroxyethyl)amine (**A-1**) was synthesized via the reaction of diethanolamine with bromooctane.

Disodium N-nonyl-N,N-bis(2-xanthogenatoethyl)amine (**A-3**) was synthesized by the reaction of N-nonyl-N,N-di(2-hydroxyethyl)amine (**A-1**) with carbon disulfide ($CS_2$) in an alcoholic medium in the presence of sodium hydroxide (Figure 1).



A-1

A-3

**Figure 1.** Chemical reaction scheme for the synthesis of the organic compound
*Note – Compiled by the authors*

The structures of the synthesized compounds were characterized using Fourier-transform infrared (FTIR) spectroscopy and nuclear magnetic resonance (NMR) spectroscopy, while their chemical composition was determined by elemental microanalysis.

In the ^1H NMR spectrum of compound A-1, a singlet characteristic of the $CH_3$ group protons appears at $\delta = 0.88$ ppm, while multiplets in the range of $\delta = 1.27$ ppm correspond to the protons of the nonyl group $-(CH_2)_9-$. The protons of the $NCH_2$ group resonate at $\delta = 4.17$ ppm (m). In addition, the hydroxyl proton appears as a broadened singlet at $\delta = 3.65$ ppm.

In the ^13C NMR spectrum of compound A-1, absorption signals corresponding to the carbon atoms of the $CH_3$ and $–(CH_2)_7–$ groups are observed in the regions of 14.10 and 22.47–22.65 ppm, respectively. The signal at 59.30 ppm is attributed to the carbon atom of the $CH_2OH$ group, whereas the signals at 29.48 and 31.38 ppm correspond to the carbon atoms of the $C_9H_{19}CH_2N$ and $–N(CH_2)–$ groups, respectively.

In the FTIR spectrum of compound A-3, characteristic stretching vibrations of the C=S and C–S bonds are observed at 1262 and 623 $cm^{-1}$, respectively. Based on the obtained results, a technological scheme for the synthesis of the A-3 collector was developed (Figure 2).

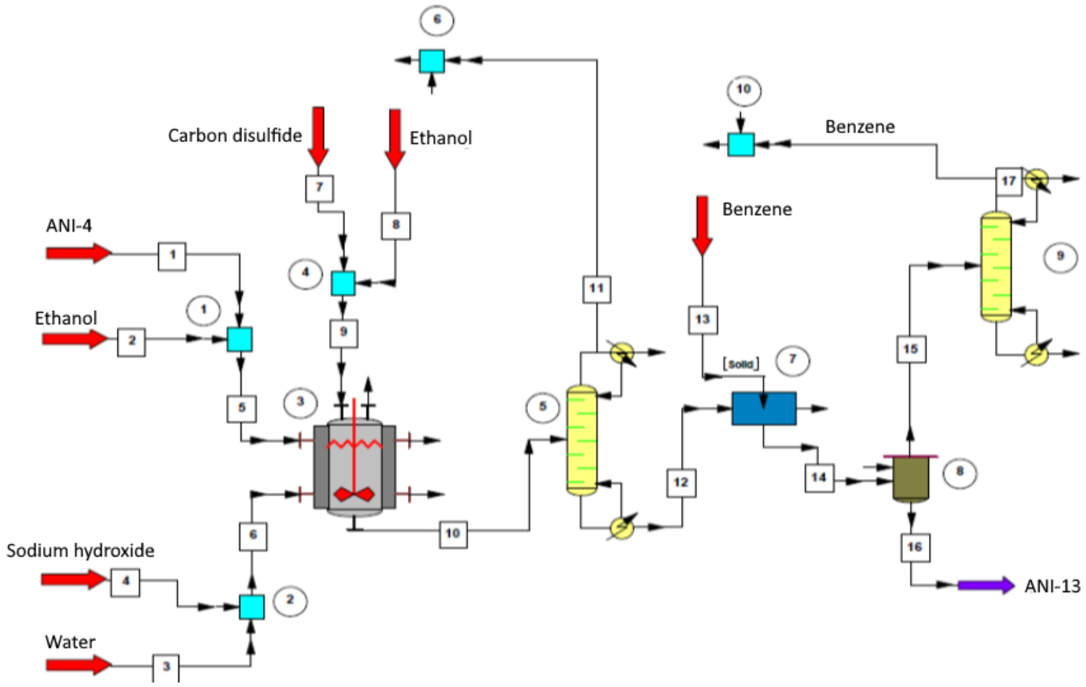


**Figure 2.** Technological scheme for obtaining the A-3 collector

*Note – Compiled by the authors*

Description of the technological process (Figure 2):

Compound A-1 was dissolved in ethanol, while sodium hydroxide was dissolved in water, after which the reaction mixture was mixed. A mixture of ethanol and carbon disulfide was introduced at room temperature for one hour, followed by distillation of the solvent (ethanol). Ethanol was then reintroduced into the reaction mixture, the crystalline precipitate was washed with benzene, dried, and the solvent was distilled again, with benzene being reused for washing the precipitate.

The aim of the conducted studies was to increase the flotation activity of the synthesized reagents compared with the base reagent. Reagents A-3 and A-9 containing xanthogenate groups are generally collectors. The lower their activity at the water/air interface, the better they adsorb on the mineral surface. In this case, since reagent A-3 exhibits lower surface activity, it is expected to have higher collecting ability (Figure 3).

In this study, the following reagents were used as collectors: sodium butyl xanthate, which is widely applied at all concentration plants in Kazakhstan, as well as the new collector reagents – sodium N-(nonyl)-N-bis(ethyl xanthogenate) (A-3), N-(heptyl)-N-(2-hydroxyethyl)amino-2-ethyl xanthogenate (A-9), and HET.

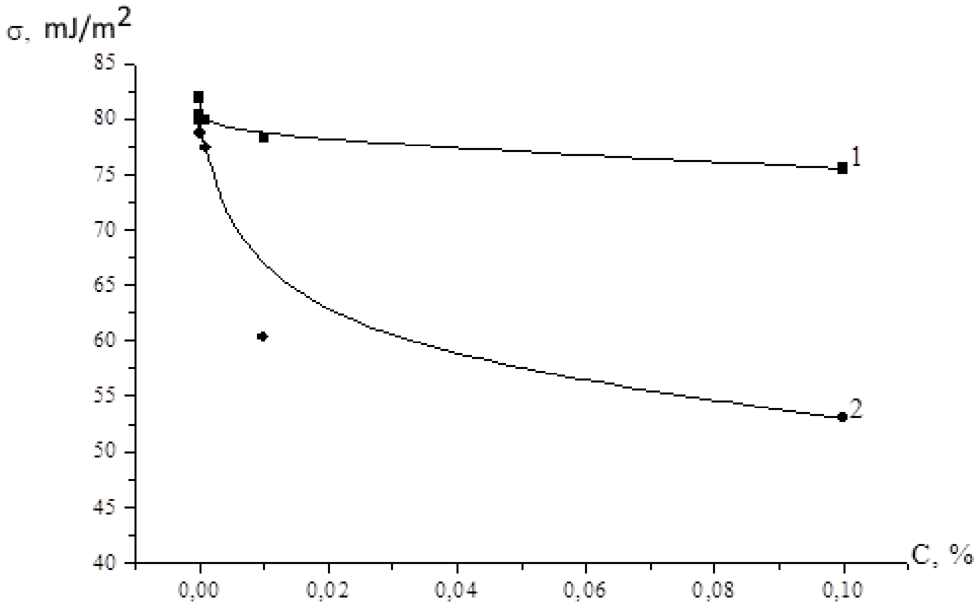


**Figure 3.** Dependence of surface tension on concentration for solutions of A-3 (1) and A-9 (2)
*Note – Compiled by the authors*

**RESULTS**

The results of the studies showed that the use of A-3 and HET as flotation reagents is effective in the processing of zinc–pyrite raw materials. Under the basic reagent regime, the copper content in the collective copper–lead concentrate was 12.8% with a recovery of 86.06%, while the lead content was 10% with a recovery of 78.6%.

The results of flotation experiments demonstrated that when reagent A-3 is used as a collector at a dosage of 23 g/t of ore, the lead content in the collective copper–lead concentrate reaches 11.1%, although the recoveries of copper and lead decrease. When HET is used, the contents of copper and lead in the collective copper–lead concentrate increase: copper rises from 12.88% to 13.6%, and lead from 10% to 18.3%. Consequently, reagents A-3 and HET exhibit selective action toward copper and lead, respectively (Table 1).

**Table 1.** Results of copper–lead flotation under different reagent regimes

| Product name | Yield, % | Composition, % | | | | Recovery, % | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Cu | Pb | Zn | Fe | Cu | Pb | Zn | Fe | |
| Basic reagent mode 500 gr | | | | | | | | | | |
| Cu–Pb concentrate | 3.56 | 12.8 | 10 | 8.4 | 23.9 | 86.06 | 78.6 | 10.94 | 25.0 | BX – 23 g/t (butyl xanthate)<br>T-80 – 17 g/t |
| Industrial process 2 | 2.08 | 1.76 | 2.3 | 7.5 | 22.9 | 3.77 | 5.67 | 3.11 | 7.65 | |
| Industrial process 1 | 6.52 | 0.3 | 0.4 | 4.6 | 16.7 | 1.10 | 1.71 | 3.27 | 9.55 | |
| Froth control flotation | 9.3 | 0.1 | 0.4 | 4.8 | 16.7 | 0.95 | 4.48 | 8.91 | 24.95 | |
| Tailings | 78.54 | 0.1 | 0.1 | 4.7 | 2.6 | 8.09 | 9.47 | 73.75 | 32.80 | |
| Feed (initial ore) | 100 | 0.67 | 0.54 | 5.2 | 7.6 | 100 | 100 | 100 | 100 | |
| Cu–Pb concentrate | 1.5 | 11.3 | 21.1 | 6.6 | 13.8 | 33.0 | 58 | 2.0 | 3.2 | A-3 – 23 g/t<br>T-80 – 17 g/t |
| Industrial process 2 | 0.82 | 3.1 | 4.5 | 6.2 | 7.3 | 4.9 | 6.8 | 1.0 | 0.9 | |
| Industrial process 1 | 5.48 | 0.98 | 1.15 | 4.9 | 5.5 | 10.4 | 11.6 | 5.4 | 4.6 | |
| Froth control flotation | 4.56 | 2.0 | 0.9 | 4.9 | 7.0 | 17.7 | 7.5 | 4.5 | 4.9 | |
| Tailings | 87.6 | 0.2 | 0.1 | 4.9 | 6.4 | 34.0 | 16.1 | 87.0 | 86.3 | |
| Feed (initial ore) | 100 | 0.52 | 0.55 | 4.94 | 6.5 | 100 | 100 | 100 | 100 | |

end of table 1

| Product name | Yield, % | Composition, % | | | | Recovery, % | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Cu | Pb | Zn | Fe | Cu | Pb | Zn | Fe | |
| Basic reagent mode 500 gr | | | | | | | | | | |
| Cu–Pb concentrate | 2.46 | 13.6 | 18.3 | 6.75 | 23.2 | 57.1 | 62.4 | 3.3 | 9.0 | HET – 30 g/t<br>BX – 23 g/t (butyl xanthate)<br>T-80 – 17 g/t |
| Industrial process 2 | 1.2 | 1.4 | 2.1 | 5.7 | 19.75 | 2.9 | 3.5 | 1.4 | 3.7 | |
| Industrial process 1 | 7.08 | 0.4 | 1.5 | 4.6 | 11.0 | 4.8 | 14.7 | 6.5 | 12.3 | |
| Froth control flotation | 3.92 | 0.9 | 1.4 | 4.6 | 8.1 | 6.0 | 7.6 | 3.6 | 5.0 | |
| Tailings | 85.34 | 0,2 | 0.1 | 5.0 | 5.2 | 29.1 | 11.8 | 85.2 | 70.0 | |
| Feed (initial ore) | 100 | 0.59 | 0.72 | 5.01 | 6.34 | 100 | 100 | 100 | 100 | |
| *Note – Compiled by the authors* | | | | | | | | | | |

Further studies examined the effect of the new collector on the flotation of the collective copper–lead concentrate. The dosage of the new collector A-3 was varied in the range of 38–76 g/t. When this reagent was used together with butyl xanthate, the copper content and its recovery increased by 3.9%.

The analysis of the obtained results showed that when reagent A-3 was used as a collector in the copper–lead flotation cycle, the contents of copper, lead, and zinc increased from 3.8% to 6.0%; however, the recoveries of copper and lead decreased. At the same time, the recovery of zinc and pyrite in the tailings increased to 84%. As can be seen from the results presented in the table, when reagent A-3 was used as a collector at a dosage of 23 g/t of ore, the lead content in the collective copper–lead concentrate reached 21.1%, although the recoveries of copper and lead decreased. Meanwhile, the recoveries of zinc and pyrite in the tailings increased to 87% and 86.3%, respectively.

Thus, it was established that reagent A-3 exhibits a selective effect toward copper and lead.

**Table 2.** Flotation results when using new collector reagents.

| Product name | Yield, % | Composition, % | | | | Recovery, % | | | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Cu | Pb | Zn | Fe | Cu | Pb | Zn | Fe | |
| Basic reagent mode 1000 gr | | | | | | | | | | |
| Cu–Pb concentrate | 9.3 | 4.9 | 5.8 | 7.2 | 33.4 | 79.8 | 75.5 | 13.5 | 53.7 | BX – 76 g/t (butyl xanthate)<br>T-80 – 54 g/t |
| Industrial process 2 | 1.8 | 0.6 | 1.25 | 10.4 | 23.2 | 1.9 | 3.2 | 3.8 | 7.2 | |
| Industrial process 1 | 6.9 | 0.3 | 0.9 | 8.7 | 11.0 | 3.6 | 8.7 | 12.1 | 13.1 | |
| Froth control flotation | 2.0 | 0.2 | 0.5 | 8.6 | 6.97 | 0.7 | 1.4 | 3.5 | 2.4 | |
| Tailings | 80.0 | 0.1 | 0.1 | 4.15 | 1.7 | 14.0 | 11.2 | 67.1 | 23.5 | |
| Feed (initial ore) | 100 | 0.57 | 0.71 | 4.95 | 5.87 | 100 | 100 | 100 | 100 | |
| Cu–Pb concentrate | 7.8 | 5.05 | 1.2 | 8.2 | 33.4 | 77.0 | 40.7 | 12.7 | 44.8 | BX – 38 g/t (butyl xanthate)<br>A-3 – 38 g/t<br>T-80 – 54 g/t |
| Industrial process 2 | 1.17 | 0.6 | 1.0 | 8.2 | 24.7 | 1.4 | 5.1 | 1.9 | 5.0 | |
| Industrial process 1 | 7.9 | 0.3 | 0.4 | 7.2 | 13.9 | 4.6 | 13.8 | 11.3 | 18.9 | |
| Froth control flotation | 1.95 | 0.3 | 0.6 | 8.1 | 10.4 | 1.1 | 5.1 | 3.1 | 3.5 | |
| Tailings | 81.18 | 0.1 | 0.1 | 4.4 | 2.0 | 15.9 | 35.3 | 71.0 | 27.9 | |
| Feed (initial ore) | 100 | 0.51 | 0.23 | 5.03 | 5.82 | 100 | 100 | 100 | 100 | |
| Cu–Pb concentrate | 3.66 | 8.7 | 11.8 | 9.6 | 13.1 | 64.6 | 71.8 | 7.1 | 8.2 | A-3 – 76 g/t<br>T-80 – 54 g/t |
| Industrial process 2 | 1.5 | 2.0 | 2.5 | 7.95 | 5.8 | 6.1 | 6.2 | 2.4 | 1.5 | |
| Industrial process 1 | 3.82 | 0.86 | 0.8 | 5.2 | 5.8 | 6.7 | 5.1 | 4.0 | 3.8 | |
| Froth control flotation | 2.1 | 1.1 | 0.6 | 5.5 | 6.4 | 4.7 | 2.1 | 2.3 | 2.3 | |
| Tailings | 88.92 | 0.1 | 0.1 | 4.7 | 5.5 | 18.0 | 14.8 | 84.2 | 84.1 | |
| Feed (initial ore) | 100 | 0.49 | 0.60 | 4.96 | 5.81 | 100 | 100 | 100 | 100 | |
| *Note – Compiled by the authors* | | | | | | | | | | |

## DISCUSSION

The conducted studies confirmed that the synthesized flotation reagents, particularly A-3 and A-9 containing xanthogenate functional groups, demonstrate pronounced flotation activity in the flotation of copper–lead–zinc ore. The mechanism of action of these reagents is closely related to their molecular structure and the specific features of their interaction with mineral surfaces.

It is known that reagents A-3 and A-9 belong to the group of collectors, and for such reagents a lower surface activity at the water/air interface ensures a higher adsorption capacity on the mineral surface. The research results showed that reagent A-3 has low surface activity, which indicates its ability to form a stable adsorption layer on the mineral surface and enhance the hydrophobization of sulfide minerals. In this regard, reagent A-3 exhibits high collecting ability and has a positive effect on the selectivity of the flotation process.

The results of flotation experiments demonstrated the effectiveness of using reagents A-3 and HET in the processing of zinc–pyrite raw materials. Compared with the basic reagent regime, the use of the new reagents altered the content of valuable components in the concentrate and ensured their more selective separation. In particular, the use of reagent A-3 at a dosage of 23 g/t led to an increase in the lead content in the collective copper–lead concentrate, although a decrease in the recovery of copper and lead was observed. This phenomenon can be explained both by the high selectivity of the reagent and by possible re-flotation or partial transfer of minerals to tailings during the flotation process (Abramov, 2010).

When using reagent HET, a significant increase in the content of copper and lead in the collective copper–lead concentrate was observed. This indicates the high frothing ability of HET and its capacity to ensure effective attachment of mineral particles to air bubbles during flotation. As a result, the quality of the concentrate improved and the accumulation of valuable components increased.

Additional studies in which the dosage of reagent A-3 was varied in the range of 38–76 g/t showed that, when used together with butyl xanthate, it allows increasing the copper content and its recovery by up to 3.9%. This synergistic effect is explained by the combination of the selective adsorption of A-3 and the high flotation activity of the basic collector. However, when A-3 was used alone, a decrease in the recovery of copper and lead and an increase in the transfer of zinc and pyrite to tailings were observed, which confirms the selective action of this reagent toward copper and lead (Abramov, 2010).

In general, the obtained results showed that the newly synthesized reagents possess high flotation activity and are characterized by an enhanced ability to hydrophobize mineral surfaces and form stable froth. These properties make it possible to consider reagents A-3 and HET as promising collectors and frothers for the flotation beneficiation of copper–lead–zinc ores.

## CONCLUSIONS

The conducted studies demonstrated the effectiveness of newly synthesized flotation reagents for improving the flotation beneficiation technology of copper–lead–zinc ore from the Tishinsky deposit. It was established that under the base reagent regime the copper content in the concentrate was 12.8% (recovery 86.06%) and the lead content was 10% (recovery 78.6%).

When reagent A-3 was used at a dosage of 23 g/t of ore, the lead content increased to 11.1%, while the recoveries of copper and lead slightly decreased; the recovery of zinc and pyrite in the tailings amounted to 84%. When reagent HET was applied, copper increased to 13.6% and lead to 18.3%. When A-3 was used together with butyl xanthate, the contents of copper, lead and zinc increased by 3.8–6.0%, while the recovery of zinc and pyrite in the tailings was 84–87%.

The results show that the synthesized reagents A-3 and HET possess high flotation activity and selective action. This is explained by their ability to enhance the hydrophobization of mineral surfaces and ensure their interaction with air bubbles. Thus, the use of the new reagents makes it possible to improve concentrate quality, increase the recovery of valuable components, and enhance the overall efficiency of the flotation process.

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