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Effect of the foaming performance of ammonium dibutyl dithiophosphate on the flotation of slime-containing copper sulfide ore

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Abstract: The use of ammonium dibutyl dithiophosphate (ADD) as a collector in the flotation of slimecontaining copper sulfide ore typically produces a sticky froth, which results in poor flotation. The mechanism and effects of copper sulfide ore flotation in synergistic systems comprising ADD and terpenic oil reagents have been systematically investigated to solve this problem. A high ratio of ADD to terpenic oil is not conducive to the flotation of fine-grained copper sulfide ores; however, adjusting this ratio may improve floatation by reducing the effect of the slime. Lowering the ratio from 5:1 to 1:1 increased the copper grade from 17.7% to 20.8%, while the recovery was largely unchanged. Notably, adjusting this ratio also reduced the cost of the flotation reagent. To study the mechanism by which the ADD-to-terpenic oil ratio affects the foam performance, the froth stability tests of the gas-liquid twophase and gas-liquid-solid three-phase systems were performed. Reducing the proportion of ADD reduced the froth water content and weakened the ability of the froth to collect gangue by adsorbtion with copper ions; this reduced gangue entrainment and maximized recovery and product quality.

Keywords: ammonium dibutyl dithiophosphate, terpenic oil, foaming property, froth flotation, slime

1. Introduction

Ammonium dibutyl dithiophosphate (ADD, $(C_4H_9O)_2PSSNH_4$) is the most common collector used in the industry of sulfide mineral flotation for the separation of pyrite and copper-bearing sulfide minerals. ADD offers adequate selectivity and foaming performance. Since the addition of ADD has an impact on the flotation froth, Chinese sulfide flotation plants typically add very low quantities of frothers, while some concentrators do not add frothers at all (Luo et al., 2018). However, with a higher slime content in the pulp, ADD exhibits a poor effect on slime entrainment in sulfide mineral flotation, thereby reducing the concentrate grade. An effective method is therefore required to reduce the effect of slime on copper sulfide flotation.

The weathering and erosion of minerals in the deposit is partly responsible for the formation of slime; however, some fine-grained minerals are produced by various mineral processing activities, such as mining, transportation, crushing, grinding, and stirring (Wei and Sandenbergh, 2007). When the slime content in the pulp reaches a critical value, flotation undergoes a series of adverse effects: (1) the slime is readily mixed through the froth product, causing a reduction in selectivity; (2) the slime covers the surface of the coarse particles, which reduces the flotation of the coarse-grained minerals; (3) reagent consumption is increased owing to the higher surface area of the slime; and (4) the slime makes the mineral pulp sticky, which worsens the inflatable condition. One of the most common methods to address muddy ores involves de-sliming materials before flotation; however, this process complicates the flotation process and causes the loss of metal in the tailings.

Foam characteristics are known to affect slime entrainment during flotation, thus, these characteristics should be optimized to achieve highly efficient froth flotation. Ata (2011) found that the liquid content and drainage of the solids of flotation mineralized foam affect both the recovery and grade of the product. Cheng et al. (2018) found that the flotation recovery of fine serpentine is depends

strongly on numerous factors, including the liquid content and stability of the froth, and the hydrophobicity of the particles at the air-liquid interface. The addition of a frother can significantly reduce the surface tension of the liquid phase, which affects the aggregation, bubble size, and liquid content of the froth and plays a critical role in the froth drainage process. The frother type also has a significant effect on the gangue drainage behavior (Ekmekçi et al., 2003; Akdemir et al., 2010; Xing et al., 2017). Ekmekci et al. (2003) optimized the flotation froth stability and the drainage process of gangue particles by selecting an appropriate frother; thereby enabling the grade of the impurity Cr_2O_3 in the fine-grained concentrate to be reduced from 8% to less than 3%. Mcfadzean et al. (2016) modified the froth structure using frother blends of PPGs and MIBC in a 4:1 ratio. This resulted in a more stable froth in comparison to single component frothers; thereby improving the recovery and grade of valuable minerals and reducing the entrainment of hydrophilic gangue. Therefore, modifying the ratio of ADD to frother can improve the flotation of slime-containing copper sulfide ores.

To date, researchers have mainly focused on the practical application of ADD and the mechanism of its interaction with minerals. Zhang et al. (2015) was able to increase the floatability of mineral particles by increasing the amount of ADD; however, when the amount of ADD is increased beyond a certain limit, the hydrophobic interaction between the particles is greater than the electric double layer repulsion that occurs from the adsorption of ADD. Although the method of using ADD in combination with regulators or other collectors in the flotation of sulfide ores has been optimized (Pang et al., 2013; Zhang and Qin, 2015; Wang et al., 2019; Yin et al., 2019; Wu et al., 2021), the effects of the combined ADD and frother on the floatation froth are not well understood. In particular, the effect of this combination on the flotation of muddy sulfide minerals has not been reported.

The flotation plant at the Zijinshan Copper Sulfide Mine in Fujian Province, China, uses ADD and terpenic oil as the collector and frother, respectively. Recently, the proportion of mineral particles finer than -10 µm in the beneficiation plants has increased, reaching almost 20% in the flotation feed ore. This increase results in a sticky froth phase which is responsible for the reduced flotation of slime-containing copper sulfide ore. In this study, effect of varying the ratio of ADD to terpenic oil on the flotation of slime-containing copper sulfide ore was investigated. The foaming ability and foam performance of different ADD:terpenic oil ratios in a gas-liquid two-phase system and a gas-liquid-solid three-phase system were obtained using a foam scanning analyzer and a pulp foam analysis device, respectively. The flotation mechanism of copper sulfide ore and the cause of entrainment in synergistic systems comprising ADD and terpenic oil was comprehensively investigated. This study provides a basis for the use of reagents in the flotation of slime-containing sulfide ores.

2. Materials and methods

2.1. Materials

Samples of copper sulfide, with a copper grade of 0.36%, was procured from the Zijinshan Copper Sulfide Mine in Fujian Province, China. The chemical compositions of the ore samples are listed in Table 1. The main copper minerals present in the ore samples were digenite, covellite, and enargite, while and the main gangue minerals were pyrite, quartz, dickite, and alunite (Table 2). All samples were crushed to a particle size of less than 2 mm using a jaw crusher followed by a roller crusher.

Component	Cu	Au	S	K ₂ O	Fe	SiO ₂	Al_2O_3	SO ₃
Content	0.33	0.20	3.10	0.91	3.56	70.48	8.52	3.55

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Table 1. The main components of the ore sample (wt.%)

Table 2. The main minerals found in the ore sample (wt.%)									
Products	Digenite	Covellite	Enargite	Pyrite	Quartz	Dickite	Alunite	Other	Total
Content	0.5	0.10	0.10	7.28	71.85	10.25	8.73	1.0	100

2.2. Methods

2.2.1. Flotation tests

2.2.1.1. Reagent ratio flotation test

An ore sample (500 g) was ground in a Φ 200 mm × 400 mm XMQ-type ball mill to a particle size of -0.074 mm, accounting for 62% with a pulp density of 55.6%. The obtained pulp was then transferred into an XFG-type flotation machine with a 1.5 L cell. The flotation process was first conducted using lime as a regulators, with a consumption of 3 kg/t, which was agitated for 5 min. ADD was then added as the collector and the mixture was agitated for 2 min. Terpenic oil was thereafter added as the frother and the mixture was agitated for a further 1 min. The total dosage of ADD and terpenic oil was set to 60 g/t. The scraped froth was manually collected for 4 min. All reagents used in the flotation tests were of industrial grade and obtained from Zijinshan Copper Sulfide Concentration Plant.

2.2.1.2. Particle size flotation test

The grounded samples were sieved into three size ranges: +0.074, -0.074 +0.037 and -0.037 mm. A sample of each size range (approximately 500 g) was prepared for the particle size flotation test. The dosage of the reagent was consistent in the reagent ratio flotation test. Pulp conditioning was conducted for 1 min, after which an appropriate concentration of the reagent was added to the pulp and agitated. The agitation time and flotation scraping times was the same as those used in the reagent ratio flotation test.

The flotation products were dried in an oven at 80 °C for 24 h, filtered, and weighed. The flotation test was performed in triplicate under varying conditions. The flotation recoveries and the grade of concentrate obtained from the mineral samples were calculated, and the result was presented as the mean value.

2.2.2. Foam stability tests of the gas-liquid two-phase system

The foaming performance of the frother in the gas-liquid two-phase system was investigated using a Foamscan instrument (Teclis Scientific, France). Five groups were prepared with ADD-to-terpenic oil mass ratios of 3:1, 2:1, 1:1, 1:2, and 1:3 (the total amount of ADD and terpenic oil was 0.24 g in all groups). These groups were then diluted to a total volume of 50 mL with deionized water, and the diluted group was injected into the sample pool using a syringe. The inflation flow rate and inflation time of the test were 150 mL/min and 40 s, respectively. The foaming ability of the frother and the foam stability were evaluated using the foam formation volume and the foam collapse rate, respectively, the latter of which is derived from the slope of the plot of the liquid volume as a function of time.

2.2.3. Foam stability tests of the gas-liquid-solid three-phase system

The foam stability test of the gas-liquid-solid three-phase system was conducted on a custom-made pulp foam analysis device consisting of a plexiglass cylinder with an air intake nozzle, porous plug, and scale for observing the foam height (Fig. 1). The diameter and height of the plexiglass cylinder were 6 and 25 cm, respectively. The pore diameter of the porous plug ranged from 20–30 µm, while the inflation rate and time were 3 L/min and 5 min, respectively. The ADD:terpenic oil mass ratios in the five groups were 5:1, 2:1, 1:1, 1:2, and 1:5. The pulp was prepared in the same manner as in the flotation test. Here, the pulp (300 mL) was extracted from the stirring flotation tank using the siphon method and quickly transferred to the aerated glass cylinder to determine the maximum height of the foam until the froth became stable.

2.2.4. Surface tension test

The surface tension of the pulps containing different ADD:terpenic oil ratios of ADD was measured using a PAT-1M tensiometer (Sinterface Technology, Germany). Each measurement was conducted using the same volume of solution, which was stirred with a magnetic stirrer for 3 minutes before the

combined drug solution was added to the test tank of the instrument. Each group was tested in triplicate and the surface tension was taken as the average value.



Fig. 1. Schematic of the pulp foam analysis device: (a) schematic representation of the foam measuring system; (b) photographs of the foam analysis device: 1-the graduated scale for observing the foam height, 2-the porous plug, 3-the gas flowmeter, and 4-the air pump

2.2.5. Zeta potential test

Zeta potential measurements were conducted using a ZEN-3700 (Malvern Instruments, UK). A mineral sample (0.05 g) with a particle size of less than 5 μ m was dispersed into 20 mL of deionized water (20 mL). The pH of the suspension was then adjusted with sodium carbonate solution. The zeta potential of each sample was measured in triplicate and the average value was reported.

3. Results and discussion

3.1. Flotation tests

The ore samples were screened and classified prior to the flotation test. The size distribution of the ore is illustrated in Fig. 2. The content of the $-10 \mu m$ ore in the flotation feed accounted for 19.03% of the total feed , while a high the slime content was observed.



Fig. 2. Particle size analysis diagram of the raw ore

During the flotation process, the floating of slime was significantly affected by the froth, which is affected by changes in the ADD:terpenic oil dosage ratio. Additionally, the recovery and product grade are significantly affected by slime entrainment. The floatation test was conducted to explore the effect the dosage ratio on the floatability of slime-containing copper sulfide ore from Zijinshan (Fig. 3). The

dosages of ADD and terpenic oil in the production site of the Zijinshan beneficiation plants were 50 and 10 g/t, respectively. A rough copper concentrate with a concentrate grade and recovery of 3.40% and 72.4%, respectively, was obtained through a rougher. Reducing the dosage of ADD from 50 to 30 g/t and increasing that of terpenic oil from 10 to 30 g/t increased the concentrate grade and recovery to 4.71% and 80.47%, respectively. The flotation recovery steadily decreased as the proportion of ADD was continuously increased, as expected. The improved grade of the concentrate indicated a reduction in the mechanical entrainment of slime in the froth. According to the existing reagent system of the Zijinshan Copper Mine, the effect of the slime on floatation can be reduced by reducing the dosage of ADD and increasing that of terpenic oil. Additionally, in China, the cost of ADD is almost double that of terpenic oil; thus reducing the dosage of ADD can reduce the overall reagent cost of the concentrator, even while increasing the dosage of terpenic oil.



Fig. 3. Flotation test results under different reagent ratios

Closed-circuit tests were conducted confirm that the effect of slime on flotation can be reduced by varying the ADD:terpenic oil ratio. These tests compared the flotation of copper sulfide ore using two reagents (Fig. 5); the first dosages of ADD and terpenic oil were each 30 g/t, while the second dosages were 50 and 10 g/t, respectively. Using the same dosage of ADD as that employed in rough flotation at the production site, the grade and recovery of copper concentrate were 17.7% and 89.1%, respectively. Reducing the proportion of ADD improved grade and recovery to 20.8% and 89.4%, respectively; thus, reagent adjustment can effectively improve the grade of copper concentrate.



Fig. 4. Flow sheets of closed-circuit tests



Fig. 5. Closed-circuit test results under different ratios of two agents

Floatability is known to vary based on mineral particle size (Ni et al., 2016; Li et al., 2016). Accordingly, we conducted particle size floatation tests to explore the effect of different ADD:terpenic oil dosage ratios on the floatability of various particles size in the Zijinshan copper sulfide ore (Fig. 6). Regardless of the collector-to-frother ratio, the floatation of -0.037 mm fine particles was lower than coarse particles, while the grade of rough copper concentrate and recovery were less than 3% and 70%, respectively. The change in the ADD-to-terpenic oil ratio had essentially no effect on the floatation of coarse particles (+0.074 mm). With particle sizes between -0.074 and +0.037 mm, reducing the proportion of ADD slightly improved the grade of copper concentrate, while the recovery was largely unchanged. Notably, the separation of fine particles was significantly affected. With high dosages ADD, the grade and recovery of copper concentrate were low at 1.36% and 44.49%, respectively. With 30 g/t ADD and an equal amount of terpenic oil, both the grade and recovery of the copper concentrate improved to 2.46% and 66.07%, respectively. As expected, the ADD:terpenic oil ratio had the most significant effect on the floatation of fine ore, which shows that changes in this ratio reduce the influence of slime on floatation and optimize the floatation of slime-bearing ore.



Fig. 6. Flotation of particles of varying size under different reagent ratios

3.2. Foam stability tests of gas-liquid two-phase system

Testing the performance of the frother with the Foamscan instrument involves measuring the electrical conductivity using the instrument's electrode and monitoring the relevant parameters using the optical probe. The instrument displays real-time changes in froth volume, froth drainage, and electrical conductivity, at different froth heights. The content of liquid encased in the froth is measured using the

volume of residual liquid and electrical conductivity. Changes in the ADD:terpenic oil ratio causes various froth properties to also change.

This experiment analyzes the foaming ability, foam stability, and foam water content at different reagent ratios. Fig. 7 shows the changes in froth volume over time, wherein the first 40 s represent the inflation stage and the subsequent time reflects the stability of the foam. As the ADD:terpenic oil ratio is reduced from 3:1 to 1:3, the maximum volume of the froth decreases from 155 to 97 mL, indicating that the foaming ability of the reagent weakens as the proportion of ADD decreases. Considering the foam stability, a lower proportion of ADD decreased froth stability, with an ADD:terpenic oil ratio of 1:3 resulting in a sharp reduction in foam stability.



Fig. 7. Comparison of the changes in foam volume over time a various ADD:terpenic oil ratios



Fig. 8. Comparison of the changes in the foam water content at various ADD: terpenic oil ratios

The longitudinal coordinates in Fig. 8 represent the water content the foam, which is proportional to the foam volume. To facilitate comparison, the moisture content ratio (MCR) of the bubble is defined as the ratio of the foam water content (Fig. 8) to the foam volume (Fig. 7) of the maximum volume with different reagent ratios, as shown in Table 3. Higher proportions of ADD result in higher MCRs. At a dosage ratio of 3:1, the MCR reached a maximum of 0.26. A dosage ratio of 1:1 resulted in the lowest MCR; however, further increasing the proportion of terpenic oil gradually increased the MCR. Related studies have shown that an increase in moisture content typically increases the number of undesired mineral particles in the concentrates, which lowers the concentrate grade (Ekmekçi et al., 2003; Wang et al., 2017). As such, the highest copper concentrate grade is achieved using the reagent combination with a dosage ratio of 1:1, which is consistent with the flotation test results.

Reagent ratio	3:1	2:1	1:1	1:2	1:3
Moisture content ratio	0.26	0.24	0.21	0.22	0.23

Table 3. Foam MCR of ADD/terpenic oil mixtures at different ratios

3.3. Foam stability tests of the gas-liquid-solid three-phase system

To more accurately reflect the effect of changes in the ADD:terpenic oil ratio on froth in a three-phase system, a foam analysis device was designed. Fig. 9 shows the change height of the pulp and froth at various reagent ratios. The experimental results showed that as the initial dosage of ADD of 50 g/t decreased to 40g/t, the total height of the pulp and froth increased, while further reducing the proportion of ADD, the total height first decreased and then increased, and reaching a minimum with a reagent dosage of 20 + 40 g/t. The height of the froth phase followed the same trend.



Fig. 9. Total height of froth and pulp at different reagent ratios

This behavior is largely consistent with that of the two-phase system; however, in contrast to the two-phase system, a high proportion of terpenic oil in the three-phase system improves the foaming ability. This can be explained by the fact that the concentration of the frother used in experiments on the two-phase system were much higher than that in the actual flotation. Additionally, a larger proportion of ADD resulted in a larger average bubble diameter (Fig. 10); resulting in a lower foam drainage rate (Ruby and Majumder, 2018) and higher froth viscosity. The bubble diameter has a significant impact on the collision and attachment efficiency, the former of which is known to decrease with increasing bubble diameter (Xing et al., 2017; Pienaar et al., 2019). Table 4 shows the recovery of concentrate water obtained from the flotation tests with various proportions of regents. The recovery of concentrate water is lowest when the reagent ratio is 1:1. This is consistent with the MCR of the foam in the two-phase system. Zheng et al. (2006) observed a strong correlation between foam entrainment and water recovery. With find-grained ores, this relationship is essentially linear; thus, the phenomenon of foam entrainment can be inhibited by adjusting the proportion of reagents so as to reduce the recovery of concentrate water.

The surface tension of the pulp was measured at various ADD: terpenic oil ratios (Table 5). The minimum surface tension occurs at an ADD: terpenic oil ratio of 1:1, which may be explained by synergistic effects between the ADD and frother in the pulp. Such synergistic effects can be optimized by adjusting the ADD: terpenic oil ratio (the optimum ratio being 1:1 in this case) to reduce the surface tension and produce a more stable bubble foam (Hadler and Cilliers, 2019). The surface tension of the solution is lowest with a reagent ratio of 1:1, which is expected to make the pulp more prone to foaming; however, the froth/pulp height is similar to the minimum value (Fig. 9), indicating that the surface tension is not the main factor affecting the froth height. The MCR of the bubbles increases with an

increasing amount of ADD, while the froth drainage rate decreases owing to the presence of slime. The sticky, slime-containing foam adheres to the wall of the container, resulting in a in a higher froth/pulp height.



Fig. 10. The foaming properties of the three-phase system with various reagent ratios

Reagent ratio	5:1	2:1	1:1	1:2	1:5		
Water recovery, %	29	26	23	24	_		
Table 5. Surface tension of ADD/terpenic oil mixtures with different ratios							
Reagent ratio	5:1	2:1	1:1	1:2	1:5		
Surface tension, mN/m	59	54	51	55	62		

Table 4. Recovery of concentrate water during the flotation test with various reagent ratios

3.4. Foam entrainment analysis

The phenomenon of foam entrainment of gangue minerals commonly occurs during the flotation process, particularly in the flotation of fine-grained minerals, in which it reduces the selectivity of the flotation. In the process of mineral flotation, two different regions of the flotation tank can be defined: one comprising the pulp phase and the other comprising the froth phase. During flotation, bubbles carry a large amount of water from the pulp phase to the froth phase. Simultaneously, a portion of the hydrophilic fine gangue enters the froth phase via water on the surface of the bubbles, after which it is scraped into the concentrate cell (Wang and Liu, 2021). A schematic of foam entrainment in the flotation process is shown in Fig. 11.

The drainage of the flotation froth in the three-phase system determines the water content of the concentrate froth, which, in turn, determines the water recovery of the concentrate and thereby affects the gangue entrainment by the froth. The water content of the produced froth was low, which reduced slime entrainment. In both the two-phase and three-phase systems, the water content of the froth is lowest at a dosage ratio of 1:1, with the least gangue entrainment and the highest concentrate grade. This was attributed to synergistic effects between ADD and terpenic oil, which enhanced the hydrophobicity of the foam and minimized the surface tension of the pulp. These synergistic effects

were affected by the ADD:terpenic oil ratio, with optimal affects being observed at a ratio of 1:1. Froth drainage is a complex phenomenon involving physical, chemical, hydrodynamic processes and is simultaneously affected by various factors (Kruglyakov et al., 2008). The average diameter of the bubble affects the liquid drainage rate, while high proportions of ADD result in large bubbles and thereby decrease the drainage rate. Cho et al. (2002) showed that the interaction of different frothers with water, through hydrophilic groups, resulted in the combination of different amounts of water with the bubble liquid membrane. This resulted in different foam water contents contributing to varying gangue entrainment. The polar group of ADD has a higher polarity than that of terpenic oil and thus ADD is more strongly hydrophilic (Yu et al., 2000). Both slime entrainment and the water content of the froth can be reduced by reducing the dosage of ADD (Fig. 12).



Fig. 11. Schematic of foam entrainment in flotation



Fig. 12. Mechanism diagram of the entrainment with foam produced by ADD

The process of gangue discharge during flotation in the three-phase system determines both the content of fine-grained gangue in the concentrate froth and the rate of entrainment. Since minerals are collected by the bubble produced by ADD, this bubble captures both metal ions and fine gangue minerals adsorbed onto metal ions, which further increases the difficulty of gangue desorption. Additionally, the adsorbed minerals act as a barrier on the froth surface, further increasing the foam stability and resulting in the adhesion of froth to the flotation cell.

In the flotation process used at the Zijinshan Copper Mine, some copper ions were dissolved from the pulp, while secondary copper sulfide minerals were readily oxidized to produce copper ions. The concentration of copper ions in the pulp, which act as activators and significantly influence the flotation of gangue minerals, was approximately 1.2×10^{-4} mol/L. In this study, the flotation of pure quartz, which is the primary gangue mineral in the ore, was explored using ADD and copper ions as the collector and activator, respectively. The copper ions effectively activated the quartz and improved its flotation recovery with various dosages of ADD and terpenic oil (Fig. 13(a)). The Zeta potential of the quartz surface increased significantly upon addition of copper ions, demonstrating that the copper ions were adsorbed on the quartz surface (Fig. 13(b)). Absorption between find-grained copper minerals and gangue minerals can also occur, resulting in an interaction phenomenon that aggravated the entrainment and floating of slime. The likely causes of froth entrainment and stickiness as a result of ADD are shown in Fig. 14.



Fig. 13. (a) Quartz flotation with different reagent dosages and (b) Zeta potentials of quartz in the absence and presence of copper ions with a reagent dosage of 30+30 g/t

The effect of the combined reagent varies as the dosages of ADD and terpenic oil change. Reducing the amount of collector and increasing the amount of frother to achieve an optimal collector:frother ratio may lead minimize the surface tension, thereby improving the foam stability. This likely arises from the hydrophilicity of the polar group of ADD; therefore, increasing the amount of terpenic oil can offset the hydrophilic nature of ADD, which can reduce the moisture content of the froth. This reduces the collecting ability of the froth, which limits the amount of fine gangue minerals absorbed with metal ions or target minerals.



Fig. 14. Schematic of the effect of ADD dosage on foam entrainment

4. Conclusions

- (1) Provided that the total amount of ADD and terpenic oil remains constant, reducing the ADD:terpenic oil mass ratio can reduce the effect of slime on flotation while maintaining an essentially constant recovery value. This approach simultaneously improved the concentrate grade and the reagent costs of the flotation procedure.
- (2) Foam stability tests of the gas-liquid two-phase system showed that a high ADD dosage increases the moisture content of the froth.
- (3) Foam stability tests of the gas-liquid-solid three-phase system showed that a smaller proportion of ADD reduces the average bubble diameter and thereby increases the foam drainage rate, which lowered the water recovery of the concentrate. Furthermore, the foaming rate of the pulp increases with the proportion of terpenic oil.
- (4) The collecting ability of the formed bubbles is increases with the proportion of ADD. This reduces the probability of the desorption of fine-grained gangue minerals from the metal ions, which enhances the entrainment of the slime.

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