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## Study on gas holdup characteristics of micro-bubble countercurrent contacting flotation column

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**Abstract:** In order to explore the gas holdup distribution of the microbubble countercurrent contact flotation column in the hematite column cationic reverse flotation process, respectively using conductance method and pressure drop method for air and water as experimental medium characteristics research of flotation column, the research group carries on the gas holdup, examines the aeration quantity, the fill medium and cation collector alkyl polyamine ether (An amine collector—GE-609 which consists of four elements: carbon, hydrogen, oxygen and nitrogen) within the column on the influence of the axial and radial gas holdup. The results show that the axial gas holdup from the bottom of the column to the top of the column increases with the increase of height in the range of 0.05-0.07dm<sup>3</sup>/s. The radial distribution of gas holdup generally shows the distribution law of “intermediate high, low on both sides”. After the filling medium is added into the column, the radial gas holdup is evenly distributed compared with the empty column, and the gas holdup is increased. Due to the high foaming performance of GE-609, the gas holdup in the column can be increased by changing the amount of aeration and the concentration of the reagent, which can exceed 60% at the maximum. Like other alcohol foaming agents, as time passes, the effect of GE-609 weakens and the gas holdup in the column gradually decreases. The addition of quantitative HCl can improve the foaming performance of GE-609, and the gas holdup in the column is significantly improved and the stability is enhanced.

**Keywords:** column flotation, conductance method, pressure drop method, gas holdup, GE-609

### 1. Introduction

In order to solve the problems such as complex reagent system, long process and high cost of high-temperature flotation in hematite anionic collector reverse flotation process (Filippov et al., 2014), the research group develops hematite cationic reverse flotation process which has simple reagent system and can process at normal temperature flotation (Wang et al., 2017). The innovation of this process is that, firstly, a new type of flotation column characterized by high-efficiency recovery of fine mineral particles is introduced. Compared with traditional flotation machine and conventional flotation column, the structure and principle of the flotation column are more effective, which not only has the characteristics of high selectivity and high enrichment ratio of column flotation, but also has the advantages of fine and uniform bubbles in the column, large surface area and more contact opportunities with ore particles under countercurrent conditions due to the introduction of a spray gun micro-bubble generator in the column. Under the premise of ensuring concentrate grade, a higher metal recovery rate can be obtained (Wang and Han, 2020). Second, the cationic collector GE-609 used in the process has the advantages of low temperature resistance and good selectivity compared with the conventional cationic collector. More importantly, GE-609 has high foaming performance that eliminates the need for foaming agents, simplifies floatation agent scheme and saves the costs in industrial applications.

Flotation columns have been widely used in mineral flotation field (Song et al., 2019; Harbort and Clarke, 2017). The gas holdup is an important parameter that determines the efficiency of column sorting, and directly determines the amount of floating minerals that bubbles can load. The high gas holdup in the flotation column means that the bubbles are more stable and resistant to break, have stronger mechanical properties and can carry more minerals, which is beneficial to flotation. However, when the gas holdup is too low or too high during mineral flotation, it will affect the back mixing and mass transfer in the column, which will affect the yield and recovery rate of the concentrate (Prakash et al., 2018; Sarhan et al., 2017). Different minerals have their own optimal range of gas holdup, and they can be affected by flotation device, surfactant (foaming agents, collectors, pH regulators, etc.), and the hydrophobicity and dissemination particle sizes of useful minerals. In practical production, the most appropriate gas holdup is determined by the floatability of each mineral and reagent conditions required for the separation, so as to adjust the mineral flotation index.

The gas holdup is mostly affected by the surfactant. Adding foaming agent is the main and most direct and effective method to change the gas holdup of the flotation column. The foaming agent disperses the air in the column into bubbles of smaller size, at the same time enhances the resistance of bubbles to deformation, improves the stability of bubbles, and effectively suppresses the merger between the bubbles (Zhu et al., 2018; Corona-arroyo et al., 2015; Eskinlou et al., 2018). Stable bubbles have a direct impact on the flotation concentrate grade and recovery rate. Both excessively stable and unstable bubbles have disadvantages. A suitable foaming agent can enhance the stability of bubbles and increase the gas holdup (Saeed., 2011). However, too stable bubbles can carry useful minerals during flotation, and at the same time, more gangue minerals are recovered due to entrainment, which directly affects the recovery rate (Rollbusch et al., 2015). Azgomi et al (2009) proposes a method that the performance of foaming agents can be evaluated by their persistence which refers to the duration of small bubbles generated by agents in the column. The evaluation method is to monitor the change of gas holdup with time online. As the reagent becomes thinner, the bubbles get less and larger and they rise even more fast.

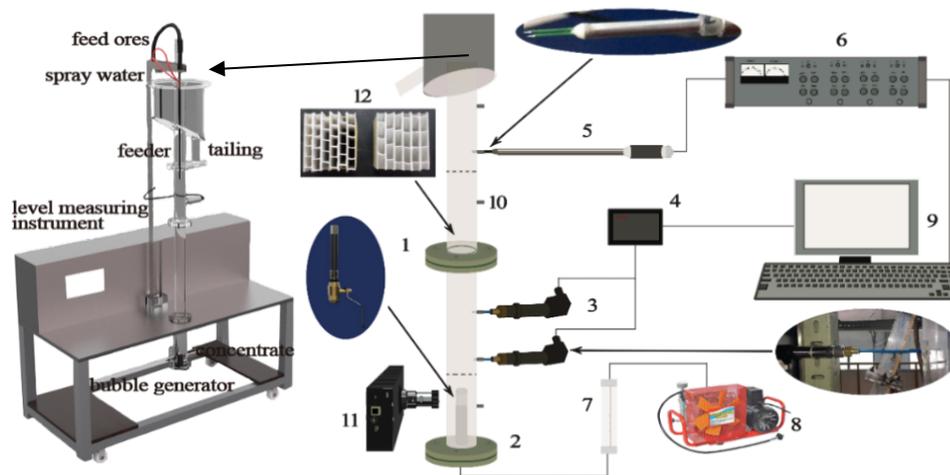
Based upon the studies of recent years, amine collectors and many oxygen collectors have the properties similar with that of foaming agents (Zhou et al., 2016). GE-609, a new type of amine cationic collector, performs well in collecting and selecting in the reverse flotation of hematite. But at present, there is little research on the foaming performance of GE-609, that is, the effect on bubble performance in flotation, and less research on the effect of the agent on gas holdup in column sorting. Therefore, carrying out research in this area is of great significance both in optimizing flotation reagents and in studying the mechanism of agents and bubbles. In this paper, namely on the basis of preliminary experiment research, the research group measures the column type flotation column in the cationic reverse flotation technology of hematite, investigates the influence of aeration condition and filling medium on local gas holdup in axial and radial direction of column. At the same time, the effect of GE-609, a new cationic collector, on gas holdup is studied. The research group compares GE-609 with other common foaming agents, investigates its foaming performance and persistence. The results can provide theoretical basis for optimizing the operating conditions of the flotation column.

## 2. Materials and methods

### 2.1. Experimental design

In this paper, the microbubble counter-current contact flotation column in the hematite column cationic reverse flotation process is taken as the research object, and the water is used as the experimental medium in the column. The experimental device mainly includes five parts: flotation column (1, 2), bubble parameter measuring instrument (5, 6), pressure sensor (3, 4), pneumatic system (7, 8) and data acquisition (9, 10). See Fig. 1 for details. The inner diameter of the flotation column is 50 mm and the height is 1700 mm. The bubble generator is installed at the bottom. The cylinder has a measuring hole every 200 mm from the column bottom. Filling medium can be added in the column. The camera is used to take pictures of bubble groups at the bottom of the column. Filling medium can be added in the position which is marked with a dotted line on the column in Fig. 1, and the adding height can be adjusted according to the experimental requirements. The filling medium is inclined hole grid plate made of polyethylene obtained through optimization design in the previous experimental study. The

aperture specification is 15×5 mm and the Angle of inclined hole is 45° (Wang et al., 2017), as shown in Fig. 1.



1- flotation column; 2- bubble generator; 3- pressure sensor; 4- data acquisition card; 5- conductivity probe; 6 - bubble parameter measuring instrument; 7- air flowmeter; 8- air compressor; 9- computer; 10 - test points; 11- camera; 12- filling medium

Fig. 1. Equipment flow chart

The cationic collector used in the experiment is an alkyl polyamine ether (Zhu et al., 2018) which is also called GE-609 commercially, and its molecular formula is  $C_{16}H_{38}N_2O$  (Ge et al., 2014). It is provided by Wuhan University of Technology. Fig. 2 shows the structural model of the molecule. The concentration of hydrochloric acid used in the experiment is 5%, and the three foaming agents used are 2- Octanol, Polyethylene glycol(F150) and Terpeneol. The detailed information is shown in Table 1.

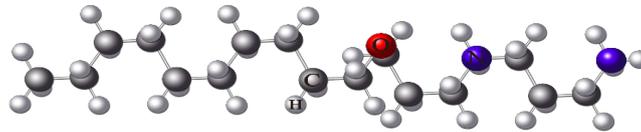


Fig. 2. Molecular model of cationic collector alkyl polyamine ether

Table 1. Basic properties of flotation reagents used in the laboratory

Flotation reagent	Manufacturer	Molecular mass(g/mol)	Molecular formula
2-Octanol	Tianjin yongda	130.23	$C_8H_{18}O$
F150	chemical reagent co.	425	$C_{21}H_{44}O_8$
Terpineol	LTD	154.25	$C_{10}H_{18}O$

## 2.2. Measurement and principle of gas holdup

At present, there are two main methods for measuring local gas holdup in the laboratory and industry, the conductance method and pressure drop method (Corona-Arroyo et al., 2015; Prakash et al., 2016). In this paper, the two methods are used to measure the local gas holdup of the flotation column using a double-headed conductivity probe and a pressure sensor. The specific measurement process is as follows: feed water from the top of the column, feed gas from the air compressor to the air inlet at the bottom of the flotation column, and adjust the filling amount through the gas mass flowmeter. According to the previous research results (Wang et al., 2017), the aeration quantity range in the test is 0.033-0.083dm<sup>3</sup>/s. When the aeration is stable, open the corresponding measuring equipment to read the data.

The conductance probe equipment used in this paper, developed by the institute of process research, Chinese Academy of Sciences, can respectively measure the gas holdup of the flotation column at the axial position (measuring the axial height H/ diameter D from the flotation column) and the radial

position (measuring the distance  $r/$  the radius  $R$  of the column). The measurement principle is to measure the local electrical conductivity in the flotation column with two conductance probes arranged at a certain distance. According to the significant difference of gas-liquid two-phase electrical conductivity, the conductance probe can accurately reflect the change process of the gas phase at its position and convert it into the corresponding electrical signal, and then calculate the local gas holdup. Place the conductivity probe in gas-liquid two phase flow, two of the same measurement channel conductance probe  $P_A, P_B$  arranges along the direction of bubble motion. Probe signal drives source to produce ac voltage signal. In the process of bubble floatation, the same bubble is punctured by  $P_A$  and  $P_B$  probes successively. The conductance value of the tip position of the probes changes, and the voltage  $V_a$  and  $V_b$  as shown in Fig. 3 are formed after the circuit of rectification, amplification, level adjustment and transformation. The original data are collected and recorded by computer. The local gas holdup is calculated according to formula (1).

$$\varepsilon_g = \frac{\sum W_{ti}}{t} \quad (1)$$

where  $\varepsilon_g$  is the local gas holdup,  $t$  is the sampling time,  $W_{ti}$  is the time of the probe in the  $i$ -th bubble.

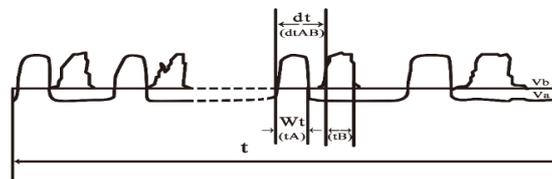


Fig. 3. Bubble waveform

When using pressure drop method to measure local gas holdup, it is assumed that static pressure of the liquid phase is only considered and dynamic pressure is ignored. The measuring principle is to use pressure sensor (provided by Beijing Star sensor technology co., LTD.) to read the pressure values  $P_a$  and  $P_b$  of any two adjacent test points at different heights in the longitudinal direction of the column (point  $a$  is above point  $b$ ).

$$P_a = \rho_l g h_a (1 - \varepsilon_a) \quad (2)$$

$$P_b = \rho_l g h_b (1 - \varepsilon_b) \quad (3)$$

where  $\rho_l$  is liquid phase density;  $h_a$  and  $h_b$  are the heights of  $a$  and  $b$  to the liquid level respectively;  $\varepsilon_a$  and  $\varepsilon_b$  are respectively the gas holdup of point  $a$  and point  $b$ .  $h(1 - \varepsilon)$  is the height of water in the gas-liquid mixture. Then the pressure difference at point  $a$  and point  $b$   $\Delta P$  is:

$$\Delta P = \rho_l \Delta h (1 - \varepsilon_g) \quad (4)$$

where  $\varepsilon_g$  is the average gas holdup between points  $a$  and  $b$ ;  $\Delta h$  is the difference in height between points  $a$  and  $b$ , then the gas holdup between  $a$  and  $b$  can be expressed as:

$$\varepsilon_g = 1 - \frac{\Delta P}{\rho_l g \Delta h} \quad (5)$$

### 3. Results and discussion

#### 3.1. Effect of aeration amount on local gas holdup

##### 3.1.1. The local gas holdup is distributed radially along the flotation column

The foaming method of the microbubble countercurrent contact flotation column adopts the micropore foaming mode, and the quantity of the aeration directly changes the bubble size, so the adjustment of the aeration amount is a major factor affecting the gas holdup in the column. Under different aeration conditions, the local gas holdup at different positions (height  $H/$  diameter  $D$ ) in the axial direction of the flotation column is measured with a double-ended conductivity probe. The distribution of local gas holdup at different positions in the axial direction of the flotation column is obtained under different aeration conditions, as shown in Fig. 4. It can be seen intuitively from the figure that the gas holdup of

each point in the axial direction of the flotation column increases with the increase of the aeration quantity, and there is a certain linear relationship between the local gas holdup and the aeration quantity; Under the same aeration condition, the bottom of the flotation column has the lowest gas holding rate and the top gas holdup is higher. This is similar with the measurement of axial gas holdup of Cyclonic-Static micro-bubble flotation column (FCSMC) by Li et al (2008). This phenomenon may result from that bubbles are apt to merger and break since clear water is used in the system during the experiment and no surfactant is added. From the change of gas holdup, the aeration quantity is in the range of 0.05-0.07 dm<sup>3</sup>/s, and the distribution law of gas holdup from the bottom of the column to the top of the column is similar, that is, the local gas holdup increases with the increase of the height; When the aeration quantity is 0.033 dm<sup>3</sup>/s and 0.083 dm<sup>3</sup>/s, the distribution law is not obvious.

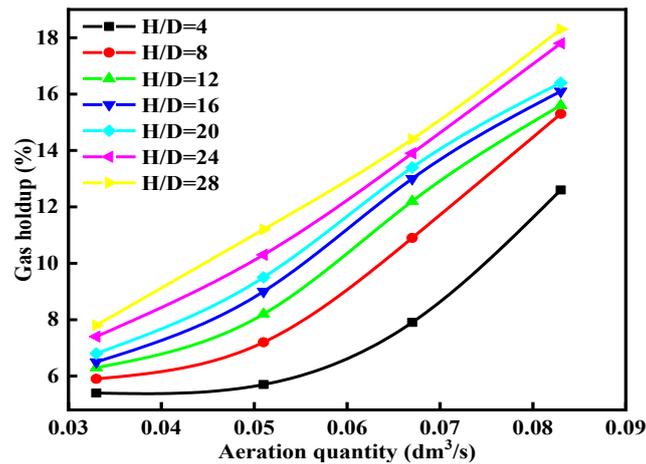


Fig. 4. Influence of aeration quantity on axial gas holdup ( $r/R=0$ )

At the same time, it can be seen from the camera that there is a part of pure liquid phase at the bottom of the column, as shown in Fig. 5. This is because the bubble generator is installed at the bottom of the flotation column. Under a small aeration quantity ( $Q=0.033\text{dm}^3/\text{s}$ ), the initial bubble velocity is small, and when it is not running to the bottom of the column, it starts to move upward in the opposite direction under the action of buoyancy. As a result, the gas holdup at the bottom of the flotation column is low, only 4.6%. As the amount of aeration increases, the bubble generated has a large initial velocity, and the pure liquid phase region at the bottom of the flotation column is gradually reduces, and correspondingly, the bottom gas holdup value is increases. When  $Q$  is increased to  $0.083\text{dm}^3/\text{s}$ , the gas holdup increases to 12.3%. The increase of the gas holdup in the middle of the flotation column may be due to the decrease of the upper liquid pressure, bubble growth and merger. In the upper part of the flotation column, the gas holdup is large due to the reduction of the static pressure in the column and the gas-liquid phase separation.

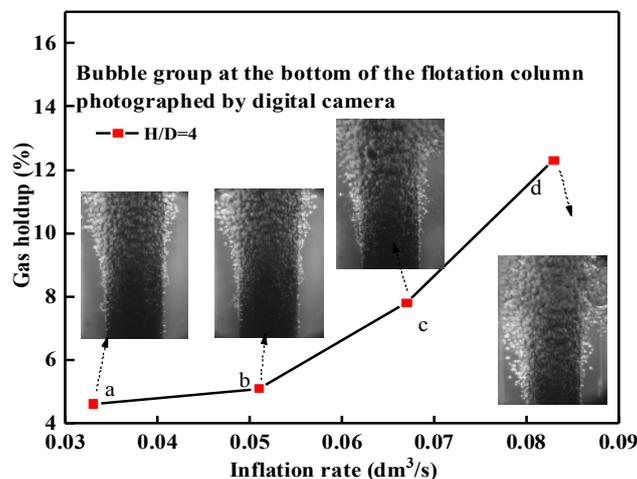


Fig. 5. Bubbles generated under different inflation conditions captured by high-speed video recorder

### 3.1.2. Radial distribution of local gas holdup along flotation column

Under different aeration quantity, the radial gas holdup at different positions ( $H/D=4, 8, 12$ ) of the empty column and the flotation column (filling height 200 mm, 400 mm and 600 mm) added the filling medium is carried out by using a double-headed conductivity probe. The radial distribution of local gas holdup along the flotation column under different aeration quantity is obtained as shown in Fig. 6, in which (a, c, e are empty columns, b, d, f are filled with filling medium). It can be seen from 6(a) in the figure that at the measurement position  $H/D=4$ , the radial distribution of the gas holdup generally exhibits a “cap” shape with “high in the middle and low on both sides”, and as the aeration quantity increases, the regular distribution of the “cap” shape becomes more prominent. In the middle of the flotation column ( $H/D=8, 12$ ), the gas holdup distribution is similar to that of radial distribution at the bottom of the flotation column, and shapes like a “cap” with “high in the middle and low on both sides”. But its distribution shape is not obvious with that when  $H/D = 4$ . It can be seen from Fig. 6(d) that after the filling medium (filling height is 200 mm) is added, the gas holdup is substantially symmetrically distributed along the radial direction of the flotation column. Moreover, the gas holdup in the middle and edge are not quite different, and it increases compared with that in the empty column. After the filling medium is higher (filling height reaches 400 mm and 600 mm respectively), the gas holdup distribution pattern in the radial direction is similar to that in Fig. 6(d), and the gas holdup ratio further increases.

When the bubble group rises, it is affected by the superficial gas velocity, and the bubble tends to gather toward the center, resulting in a phenomenon that the radial gas holdup is low on both sides. The filling medium changes the cylinder into a number of narrow spaces, and these spaces ensure that the liquid phase and the bubbles can move up and down stably, which significantly improves the uneven distribution of the bubbles in the flotation column. Thus, the filling medium plays the role of dispersing gas. The filling medium can cut large bubbles into small and uniform ones, and distribute them evenly throughout the cross section, effectively improving the local gas holdup in the flotation column.

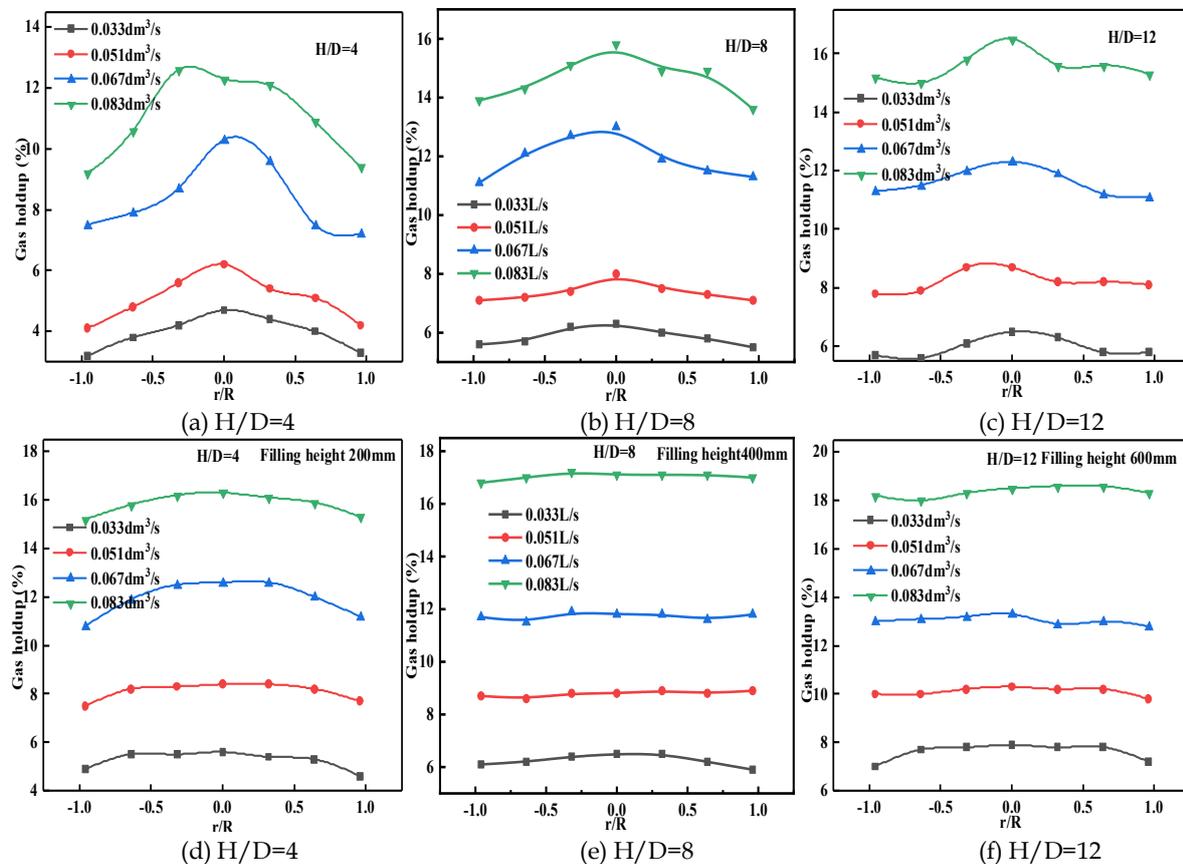


Fig. 6. Radial gas holdup distribution of flotation column

### 3.2. Effect of cationic collector GE-609 on gas holdup

The foaming agent can reduce the surface tension of gas and liquid, effectively suppress the diameter of the bubble, and has an important influence on the gas holdup (Rafiei et al., 2011). Since the number of bubbles in the column is doubled after the addition of the foaming agent, the bubble diameter is significantly reduced (Gulden et al., 2018). However, the measurement of small bubbles by the double-headed conductivity probe loses accuracy, so the GE 609 with foaming performance is added and the local gas holdup is measured by pressure drop method. Two pressure sensors is fixed in the middle of the flotation column (when  $H/D = 8$  and  $H/D = 12$ ). When the flotation column is stable, the research group turn on the equipment to collect and record the change of gas holdup in this fixed area in real time.

#### 3.2.1. Effect of GE-609 concentration on gas holdup

The increase in the aeration quantity increases both the number of bubbles in the flotation column and the diameter of the bubbles, and the gas holdup is proportional to the number of bubbles and inversely proportional to the diameter of the bubble. Therefore, it is of great significance to study the effect of the aeration amount and the concentration of the foaming agent on the gas holdup to optimize the performance of the flotation column. Fig. 7 shows the effect of GE-609 concentration on the gas holdup of the flotation column under different aeration conditions. It is known from the figure that with the same GE-609 concentration, the gas holdup gradually increases with the increase of the aeration quantity. When the amount increases to a certain value, the increase degree decreases. Under the same aeration condition, with the increase of GE-609 concentration, the gas holdup increases significantly at first. When the concentration of GE-609 exceeds  $12.8 \text{ mg/dm}^3$ , the increase degree of gas holdup decreases. Thereafter, the concentration of GE-609 is continuously increased. When the aeration quantity exceeds  $0.067 \text{ dm}^3/\text{s}$ , the gas holdup trend is similar to that of GE-609 at  $12.8 \text{ mg/dm}^3$ ,  $16 \text{ mg/dm}^3$ , and  $22.4 \text{ mg/dm}^3$ . When the concentration of GE-609 is  $22.4 \text{ mg/dm}^3$  and the aeration quantity is  $0.083 \text{ m}^3/\text{s}$ , the gas holdup value is the highest, which is 61.89%.

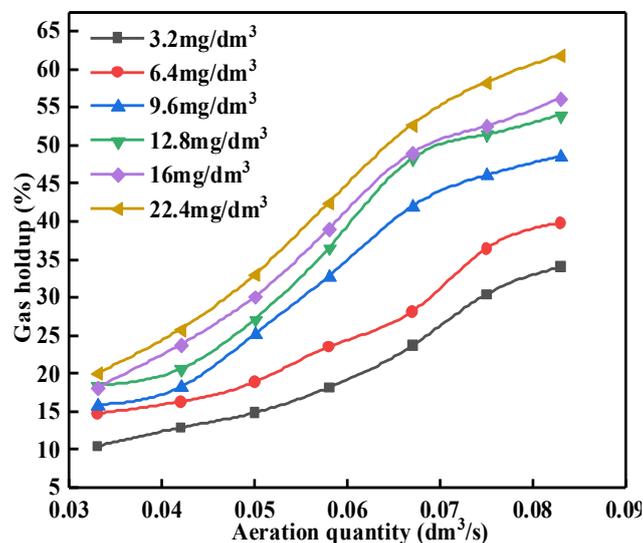


Fig. 7. Relationship between gas holdup and aeration quantity under different GE-609 concentrations

#### 3.2.2. Comparison between GE-609 and alcohol foaming agent

In order to further explore the effect of the cationic collector GE-609 on the gas holdup, three high-efficiency alcohol foaming agents, 2-Octanol, F150 and terpineol, which are commonly used in metal ore flotation, are used as the comparison. Under the same aeration condition, their effects on gas holdup are investigated. Table 2 shows the measured gas holdup in the flotation column with different concentrations of four kinds of reagents when the aeration quantity  $Q=0.067 \text{ dm}^3/\text{s}$ .

Table 2. A summary of the gas holdup (%) using different reagents at a concentration range of 3.2-22.4 mg/dm<sup>3</sup>

Concentration (mg/dm <sup>3</sup> )	Terpineol	F150	GE-609	2-Octanol
3.2	10.03	19.18	13.09	26.26
6.4	18.27	24.09	23.68	39.90
9.6	24.77	38.72	25.16	48.38
12.8	30.15	43.84	36.99	64.58
16	37.47	47.44	48.291	66.25
19.2	37.93	54.69	49.03	67.25
22.4	39.07	56.09	49.74	67.78

The data in Table 2 is plotted as a curve as shown in Fig. 8. It is known from the figure that as the concentration of the four reagent increases, the gas holdup increases. Due to the different types of reagents, the increase rate of gas holdup is different. When the concentration increases to a certain degree, the increase degree of gas holdup decreases, and the upward trend gradually stabilizes. Considering bubbles seriously, the gas holdup is inversely proportional to the size of bubbles, and size decreases with the increase of the foaming agent concentration. When the concentration reaches the critical coalescence concentration (CCC), the size of bubbles tends to be stable (Kracht and Rebolledo, 2013), The merger no longer occurs, so the bubble size does not change any more, resulting in an increase in the gas holdup first and the increase degree decreases then until the gas holdup gradually stabilizes. Because the foaming performance and the gas holdup of the agent are positively correlated (Tan et al., 2013), it can be seen from the figure that when the concentration is same, the foaming performance of the four reagents is ranked as terpeneol <GE-609<F150 <2-Octanol. That is, 2-Octanol has the strongest foaming performance, followed by F150, and GE-609 is between terpeneol and polyethylene glycol.

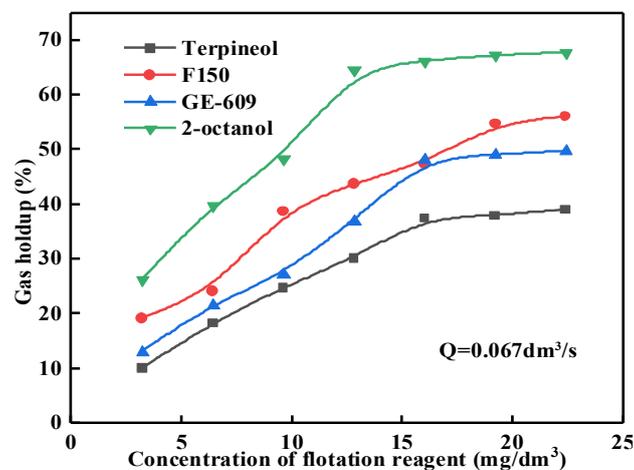


Fig. 8. Effect of flotation reagent concentration on gas holdup

The main reason for the foaming properties of the four reagents being different is the influence of the molecular structure of the reagents themselves. The polar group determines their solubility while the non-polar group, after being squeezed by the cohesion of water molecules which makes the agents rapidly accumulate to the gas-liquid interface, makes the reagents foam. For chain-like structures, the surface activity can be increased by 3.14 times for each additional carbon atom in a non-polar group (Nikolai et al., 2014). The 2-Octanol has a long chain length and exhibits a strong foaming ability. The gas holdup exceeds 60% when the concentration of 2-Octanol reaches 12.8mg/dm<sup>3</sup>. Although terpeneol, GE-609 and F150 all have higher carbon content than 2-Octanol, their foaming ability cannot compare with that of 2-Octanol since terpeneol contains six-membered rings in its molecule, GE-609 molecule contains a carbon-oxygen double bond, F150 molecule contains a bifunctional group, and 2-Octanol is with a single chain structure. Besides, because the above three reagents contain different molecular carbon

content, their different foaming capability is different. The foaming properties of the four reagents can be ranked as terpineol < GE-609 < F150 < 2-Octanol.

### 3.2.3. Comparison between GE-609 and alcohol foaming agent

Fig. 9 is a graph showing the relationship between the gas holdup and the time at the same aeration quantity and the same position in the column under the same concentration conditions of GE-609 and two alcohol foaming agents. On-line monitoring for 1 hour then the research group finds that the gas holdup in the column decreases significantly within 1 hour after 2-Octanol, terpineol and GE-609 being added, while after F150 being added, the gas holdup almost does not decrease, but to be stable. The persistence of 2-Octanol is the worst. After 0.25 hour, the gas holdup begins to decrease, and it decreases by about 10% during the whole measurement period. Terpineol followed, with a decrease of about 7 percent over the period. The gas holdup of GE-609 begins to decrease after half an hour, and it decreases about 5 percent over the period.

Since GE-609 is an ether amine collector, it is not easily soluble in water. In the actual flotation operation, hydrochloric acid is usually added to form an amine salt to increase the concentration of amine ions of GE-609 dissolved in water. The real-time change of gas holdup after adding different amounts of hydrochloric acid to GE-609 is measured, the measurement results are shown in Fig. 10. After adding quantitative GE-609 for agitation for 0.5 hour, HCl is fed into the column. When HCl:GE-609 (molar ratio) = 1:1, the gas holdup significantly improves. After GE-609 and HCl are fully effective, the gas holdup increases from 19% to about 26% and remains stable within 1 hour. The research group continues to add HCl, when the column HCl: GE-609 = 1:2 (molar ratio), the gas holdup continues to

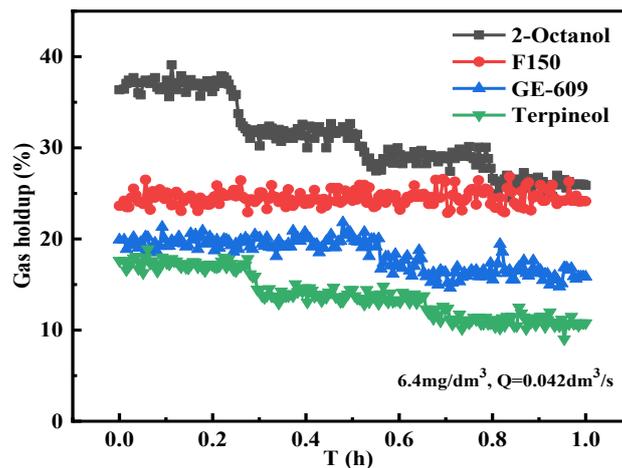


Fig. 9. Gas holdup with time diagram

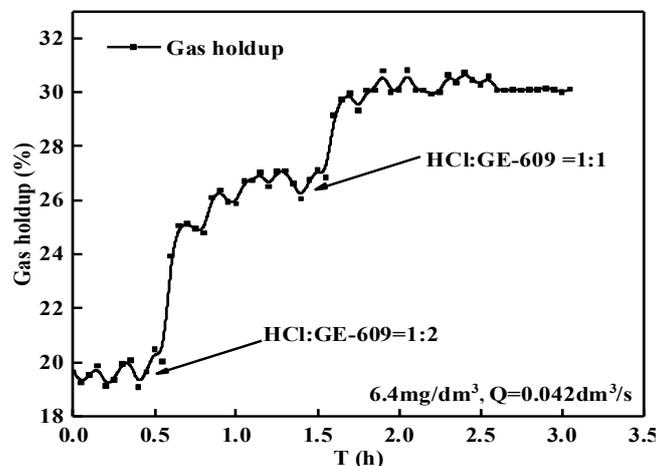


Fig. 10 Change of gas holdup under the action of HCl and GE-609

increase, reaching about 31%; After the 1-hour monitoring, it is found that the gas holdup remains stable and no significant change occurs.

It should be pointed out that the research in this paper is aimed at the foaming performance of the cation, and its main function is the collector. In the actual flotation operation, the collection performance of GE-609 should be fully considered. Excessive HCl increases the foaming of the reagent, but it also reduces its harvesting performance, affecting concentrate grade and recovery. Therefore, the amount of HCl should be added according to actual needs.

#### 4. Conclusions

- (1) From the bottom to the top of the micro-bubble countercurrent contacting flotation column, the local gas holdup gradually increases along the axial direction of the column, and there is a linear relationship between the aeration amount and the axial gas holdup. The local gas holdup along the radial distribution of the flotation column presents a "cap" shape with "high in the middle and low on both sides". The closer to the bottom of the column, the more obvious this distribution is. After the filling medium is added into the column, the radial gas holdup distribution is relatively uniform, and the gas holdup of the empty column increases.
- (2) GE-609 has efficient foaming properties. Compared with the three alcohol foaming agents, the foaming properties of the four reagents when the concentration of the reagents is the same is: terpineol <GE-609<F150<2-Octanol, GE-609 is between terpineol and polyethylene glycol.
- (3) The foaming property of GE-609 is not stable, and the durability of the reagents decreases with time passes, and the gas holdup drops significantly. After the addition of quantitative HCl, its durability is significantly improved and the gas holdup in the column largely increases.

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