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Collision–attachment law of lepidolite, feldspar and quartz with bubbles in the combined cationic and anionic collector system

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Abstract: The purpose of this study is to explore the collision–attachment law of lepidolite, feldspar and quartz during their interaction with bubbles by particle settlement method and bubble rising method under the action of combined collector. In this study, HQ-330 and dodecylamine were used as combined collector to separate lepidolite, feldspar and quartz by flotation. It also aims to analyse the relationship between collision probability, attachment probability, formation time of three-phase contact line and flotation recovery and the main factors affecting the formation time of three-phase contact line. Experimental results show that when the pH is 7 and the combined collector dosage is 100 mg/L, the separation of lepidolite from feldspar and quartz can be achieved. In the particle settlement experiments, the correlation between collision probability and flotation recovery is low, the correlation between attachment probability and flotation recovery is positive. In the bubble rising experiments, the formation time of three-phase contact line (t_{TPC}) is negatively correlated with flotation recovery, and the combined collector changes t_{TPC} by changing drainage time.

***Keywords*:** combined collector, collision probability, attachment probability, three-phase contact line

1. Introduction

As the lightest metal element, lithium is widely used in lithium battery, ceramics and other industries owing to its very strong metal activity (Li et al. 2020). As one of the main hard rock lithium ores for extracting lithium element, lepidolite is also one of the carrier minerals of rare metals rubidium and cesium (Yang 2020). Its beneficiation products undoubtedly have a great influence on subsequent smelting process and products. Lepidolite is mainly symbiotic and embedded with gangue minerals such as feldspar and quartz. With the development of mineral resources, lepidolite resources are characterised as 'poor, fine and miscellaneous' (Ma and Li 2018, Zhou et al. 2019). To improve the beneficiation index of lepidolite, many researchers found that the combination of cationic collector and anionic collector has stronger selectivity to lepidolite through flotation experiments. In recent years, researchers have used high-speed cameras to film the dynamic process of collision, attachment and detachment between bubbles and mineral particles and the formation of three-phase contact line. Using this method, the selective attachment effect of bubbles on lepidolite particles and the difference in the formation time of three-phase contact line when using combined cationic and anionic collector can be explained. They also have used high-speed cameras to demonstrate the superiority of combined cationic and anionic collector over single collector.

In the process of collision and attachment between ore particles and bubbles, the interaction between them can be divided into three stages: (1) Ore particles come close to bubbles and collide. (2) The hydration film between the ore particle and the bubble thins until it breaks. (3) The ore particles are attached on the surface of the bubble, that is, the ore particles and the bubble form a three-phase contact line (Li et al. 2020). There are many researches of the collision between particles and bubbles, and the collision can be classified into inertial and viscous (Chen, Zhang and Wang 2001). According to the difference of slurry fluid states, several empirical and semi-empirical formulas are used to describe the probability of inertial collision. Examples are Weber's formula (Weber and Paddock 1983), Yoon's formula (Ahmed and Jameson 1985) and Sutherlond's formula (Sutherland 1948). In addition, collision

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probability (P_a) is susceptible to bubble size, particle size and other factors (Zhuo et al. 2019). With the increase of particle density, the influence of particle size on P_a gradually decreases (Zhang 2015).

Compared with collision, there were less studies on attachment. Some researchers conducted detailed investigation of particle–bubble attachment and suggested a new particle–bubble attachment probability (P_a) model (Nguyen, Schulze and Ralston 1997a, Nguyen, Ralston and Schulze 1998). Indirect experiments were adopted and flotation recovery were used to reflect the level of attachment efficiency (Ozdemir et al. 2009, Tarkan, Bayliss and Finch 2009). That is, a certain correlation existed between flotation recovery and P_a. Researchers used high-speed cameras to film the attachment behaviour of plastic particles and bubbles in water, and they obtained the movement law of particle–bubble by studying the sliding angle (Chen and Sun 2019). Researchers studied influence law of the bubble size and surface hydrophobicity in flotation, they found that the t_{TPC} increases when diameter of bubble increases, it decreases when plastic flat hydrophobicity increases, and the TPC spread diameter decreases when the plastic flat hydrophobicity decreases (Nie, Huang and Sun 2019). Researchers used particle settlement method and found that hydrophobic particles slide along the bubble after colliding with the bubble and accumulate at the bottom of it. Thus, hydrophobic particles are more likely to attach the bubble (Wang et al. 2003), that is, the attachment time is shorter. And then there were researchers put forward the interaction between particles and bubbles to explain the formation of three-phase wetting periphery (Nguyen et al. 1997c, Nguyen et al. 1997b, Nguyen et al. 1997a).

With the increasing demand for mineral resources, the content of useful components in silicate minerals in ores is decreasing day by day. As a cheap mineral resource, how to develop a low-cost method to separate lepidolite by flotation has important practical and theoretical significance. Using combined collectors can often achieve better flotation results than single collectors. Most of the previous studies have been done at the macro level, so it is not possible to directly obtain the effect of collectors on the collision and attachment processes between minerals and bubbles. In this article, the combination of anionic collector HQ-330 and cationic collector dodecylamine was used to separate lepidolite, feldspar and quartz by flotation. The collision–attachment law of lepidolite, feldspar quartz during their interaction with bubbles and the relationship between the collision probability (P_c), attachment probability (P_a), formation time of three-phase contact line(t_{TPC}) and flotation recovery were studied by particle settlement method and bubble rising method. In addition, the relationship between collision and rebound time (t_B), drainage time (t_D) and the formation time of three-phase contact line (t_{TPC}) was also deeply explored.

2. Materials and methods

2.1. Experimental materials

The properties of lepidolite, feldspar and quartz used in the experiment are shown in the following Table 1. The purity of the three minerals is more than 95%. The three minerals were prepared by ceramic ball mill, and 0.0374mm-0.075mm minerals were screened out for use.

The reagents used in the experiment were as follows: DDA, HCl and NaOH were prepared with different concentrations of industrial pure reagents with purity ≥99.5%. HQ-330 was an anionic collector independently developed by our laboratory, and deionized water was used as the experimental water.

Table 1. Ore sample properties

Component	Unit	SiO_2	Li_2O	Al_2O_3	Fe_2O_3	K_2O	Na_2O
Lepidolite	%	50.84	4.97	27.24	0.1	9.91	0.43
Feldspar	%	64.99	-	18.8	0.046	11.29	3.66
Quartz	%	97.49		-	-	-	-

2.2. Pure mineral flotation experiment

In the pure mineral flotation experiment, the impeller speed was set at 1650 r/min. The weigh was 2.0 g for the ore sample each time (The mass ratio of the three minerals in the artificial ore mixing experiment was 1:1:1). It was put into the flotation tank. Deionized water amounting to 40 cm^3 was added. The pulp was stirred for 2 min, and the pH of the pulp was adjusted with HCl or NaOH for 3 min. A certain amount of collector was then added, and after waiting, it was stirred for 3 min. Flotation and bubble scraping were performed for 4 min. The froth products and tailings were dried and weighed, the recovery and grade were calculated. The flow of pure mineral flotation test is shown in Fig. 1.

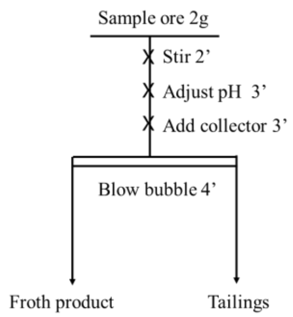

Fig. 1. Flow chart of pure mineral flotation experiment

2.3. Collision-attachment experiments of mineral particles and bubbles

In this part, the particle settlement method (Yang et al. 2020, Subasinghe and Albijanic 2014, Sla et al.) was adopted. A stainless-steel needle tube (inside diameter of 0.3 mm) was used to create single stable bubbles (2mm) in the observation room. A 40 cm^3 solution was used to interact with the 0.2g (0.0374mm-0.075mm) sample, and different conditions required by the experiment were adjusted while stirring with a magnetic agitator (1000r/min, 5min). Then, the same amount of the acted solution was absorbed with a rubber head dropper and put into a funnel with good focusing of bubbles. The rubber head dropper was inserted below the liquid level of the funnel to make the mineral particles settle under the action of gravity, enter the observation chamber uniformly through the funnel and collide with the stable bubbles. Photos were taken and images were recorded at the same time. The schematic diagram of the experimental device is shown in Fig. 2.

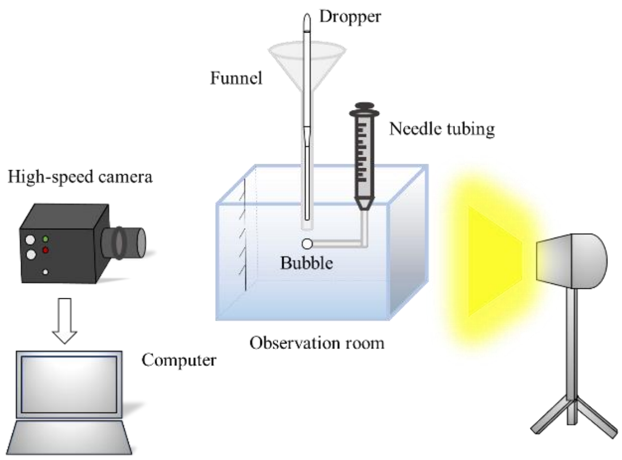

Fig. 2. Schematic diagram of mineral particles and bubbles collision–attachment experimental device

2.4. Method for analysing the P_c and P_a

A high-speed camera was used to record the collision and attachment of particles on the bubble surface in a certain period. When particles collide, the particles 10 s before and after the attachment of three particles were selected for statistics. The P_c was the ratio of the number of particles colliding thrice to the total number of particles. Yoon and Luttrell proposed the use of the ratio of the area A_a capable of attachment to the total area A of bubbles and particles to describe the P_a (Yoon 2000, Sla et al.), which is the same as P_c. The P_a is typically calculated with reference to the closure attachment. As the particle diameter is much smaller than the bubble diameter, it is usually not considered.

2.5. Method for determining the t_{TPC}

In this part, the mineral surface was polished with sandpapers in advance to expose the fresh surface. The mineral surface was then fixed 15mm above the bubble outlet, and it was immersed in the solution for 3 min before the experiment began. During the experiment, bubbles were generated by the peristaltic pump which had a speed of 0.284 cm^3/min. A high-speed camera was used to record the collision and attachment between bubbles and mineral plane. The schematic diagram of the experimental device was shown in Fig. 3:

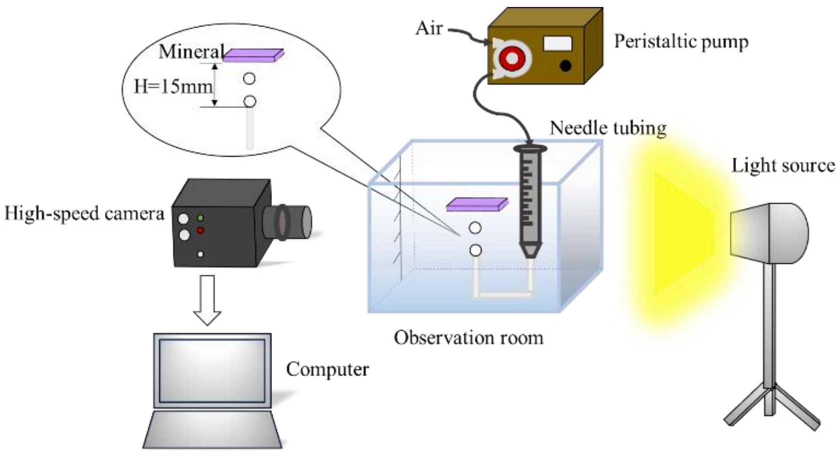

Fig. 3. Schematic diagram of the experimental device for measuring the formation time of three-phase contact line

2.6. Method for analysing the t_{TPC}

In the experiment, the time of the bubble's first collision with the mineral surface was set as 0 ms, in which the high-speed camera was set as 500 frames, the time interval of every two images was 2 ms. The formation time of the three-phase contact line(t_{TPC}) is expressed as $t_{TPC}= t_B +t_D$, where t_B is the total collision time of bubbles from the beginning of the collision to the kinetic energy dissipation of 0, t_D is the time of bubbles from the beginning of the rest on the mineral surface to the liquid film rupture forming stable attachment, that is, the drainage time. Bubble instantaneous rate is the ratio of bubble movement distance and time interval between two adjacent photos at a certain time. The bubble movement distance is measured by image J software.

3. Results and discussion

3.1. Single mineral flotation experiment

3.1.1. Experiments on the ratio of combined cationic and anionic collector

Fig. 4 shows that when pH = 7 and total dosage of collector was 60 mg/L, the recovery of the three minerals increased to varying degrees with the increase of DDA proportion. And when the ratio of HQ-330 to DDA was 4:1, the recoveries of lepidolite, feldspar and quartz were 65.18%, 10.13% and 7.57%. At this time, the recoveries of lepidolite and others had the largest difference, which was expected to achieve separation.

3.1.2. Experiments on the dosage of the combined collector

Fig. 5 shows that when pH = 7 and the ratio of HQ-330 to DDA was 4:1, the recoveries of three minerals increased to varying degrees with the increase of dosage of combined collector. When the dosage of combined collector was 100 mg/L, the recovery of lepidolite was 91.98%, but the recoveries of others were less than 10%.

Therefore, the separation of lepidolite from feldspar and quartz could be realised under this condition. Finally, in subsequent experiments, the mass ratio of anionic and cationic collector was 4:1, and the dosage was 100 mg/L.

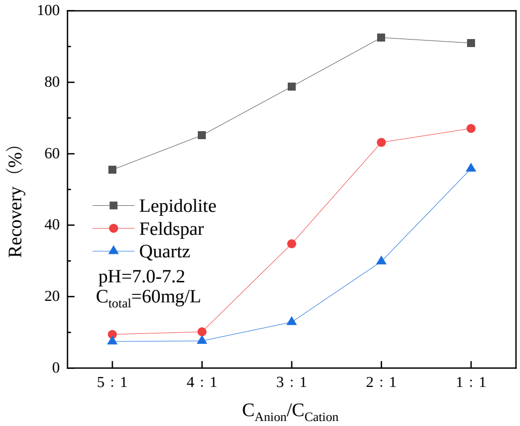

Fig. 4. Flotation behaviours of three minerals under different ratios of anionic and cationic collectors

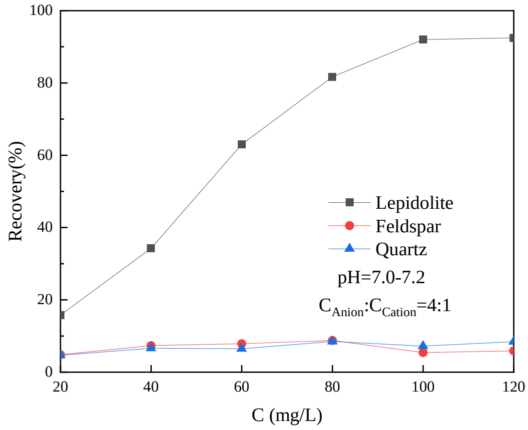

Fig. 5. Flotation behaviours of the three minerals under different dosages of combined collector

3.1.3. Experiments on the pH adjustment

Fig. 6 shows that when the ratio of HQ-330 to DDA was 4:1 and the dosage of combined collector was 100 mg/L, the recovery of lepidolite decreased slightly with the increase of pH. By contrast, the recovery of feldspar increased slightly, whereas the recovery of quartz increased firstly and then decreased.

The result shows that when pH ≤ 7, the recovery of lepidolite differed greatly from others, it can be concluded that lepidolite could be separated.

3.2. Experiments on artificial mixed ores

Fig. 7 shows that the recovery of lepidolite firstly increased and then remained stable at about 90% with the increase of collector. By contrast, the grade of lepidolite decreased slightly but remained above 92%. When the dosage of collector was 40 mg/L, the recovery of lepidolite was 90.5%, and the grade was 93.22%.

This result proves that the combined collector can selectively adsorb on the surface of lepidolite under pH=7 and finally make lepidolite and feldspar and quartz separated.

3.3. Collision and attachment of mineral particles and bubbles

3.3.1 Effects of the ratio of anionic and cationic collector on collision and attachment of minerals with bubbles

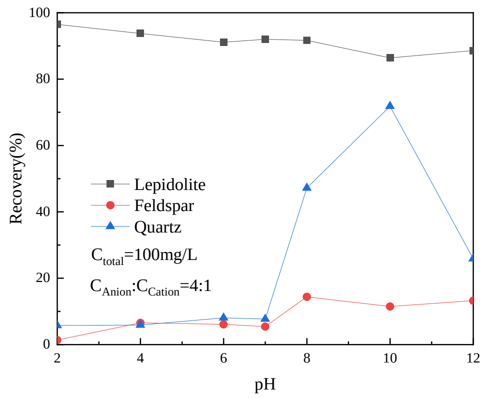

Fig. 6. Flotation behaviours of the three minerals under different pH conditions using combined collector

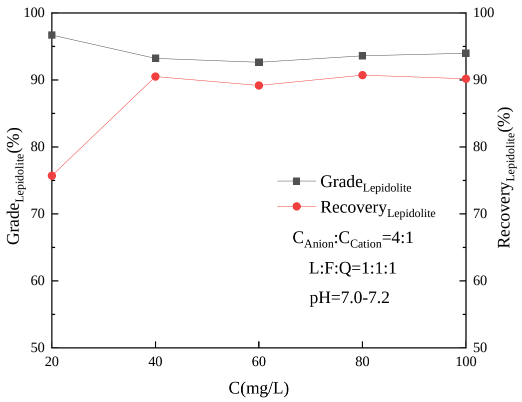

Fig. 7. Experiments on artificial mixed ores

Fig. 8 (a) shows that when pH=7, the P_c between the three minerals and bubbles increased slightly, but the change was not significant, and all of them were more than 80%.

Fig. 8 (b) shows that the P_a was inversely proportional to the ratio with dosage of anionic collector. Besides, the P_a of lepidolite was always greater than that of feldspar and quartz. And when the ratio of anionic and cationic collector was 4:1, the P_a of lepidolite was the most different from that of others.

The result shows that the P_c is basically unchanged when the collector composition changes, and the change trend of P_a is consistent with the flotation recovery.

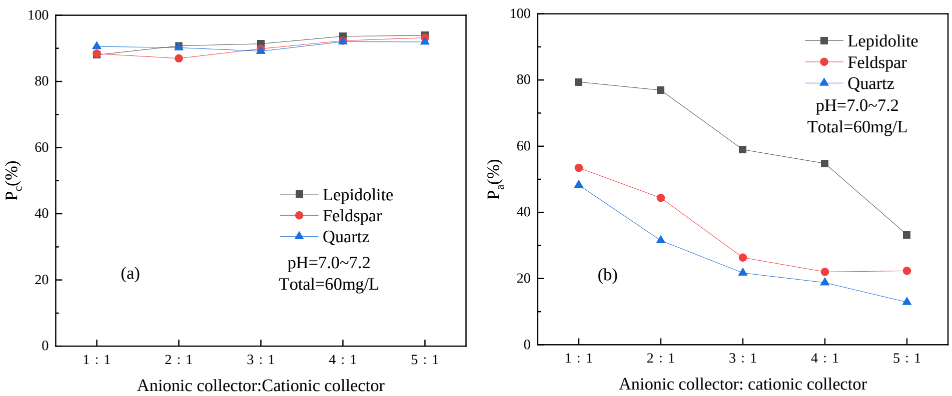

Fig. 8. P_c (a), P_a (b) of lepidolite, feldspar and quartz with bubbles at different ratios of anionic and cationic collectors

3.3.2. Effects of dosages of combined collector on collision and attachment of minerals with bubbles

Fig. 9 (a) shows that under pH=7, when the dosage of combined collector increased, the P_c of minerals and bubbles rise slightly but didn't change much, all of them were more than 80%.

Fig. 9 (b) shows that when the dosage of combined collector increased, the P_a of lepidolite changed greatly, whereas that of feldspar and quartz didn't change basically. With the increase of combined collector, the P_a of lepidolite increased and reached the maximum when the dosage was 100 mg/L (>90%), while that of feldspar and quartz changed slightly and were lower than 20%.

The result shows that the Pc is basically unchanged when the dosage of collector changes, and the change trend of P_a is consistent with the flotation recovery.

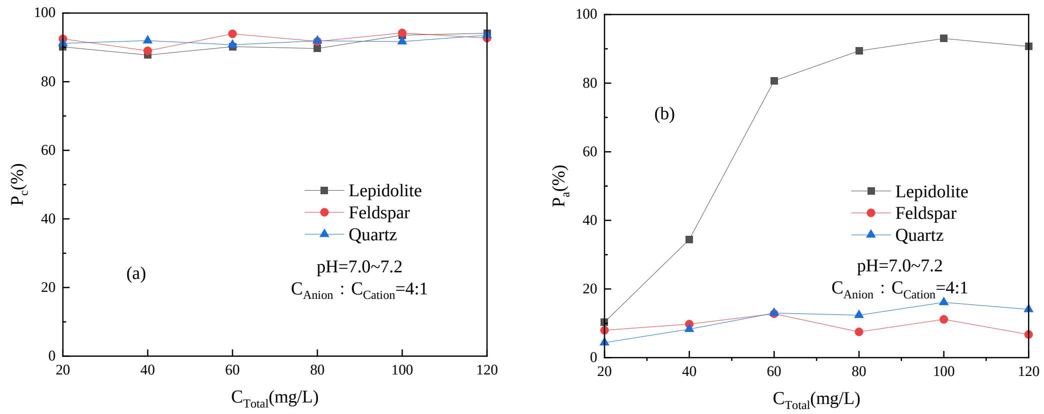

Fig. 9. P_c (a), P_a (b) of lepidolite, feldspar and quartz with bubbles at different dosages of combined collector

3.3.3 Effect of pH on collision and attachment of minerals with bubbles

Fig. 10 (a) shows that when pH changed, the P_c between minerals and bubbles rise slightly but not significant, and all of them were more than 80%.

Fig. 10 (b) shows that the P_a of lepidolite decreased slightly with the increase of pH, but all of them were more than 80%, while that of feldspar were always lower than 20%. And the P_a of quartz increased gradually with the increase of pH within the range of pH ≤ 10, and it reaches the maximum (>60%) when pH = 10. In the range of pH = 10–12, it gradually decreased. The results also indicated that in the range of pH=2-8, lepidolite could be separated from feldspar and quartz.

The result shows that the P_c is basically unchanged when the pH changes, and the change trend of P_a is consistent with the flotation recovery.

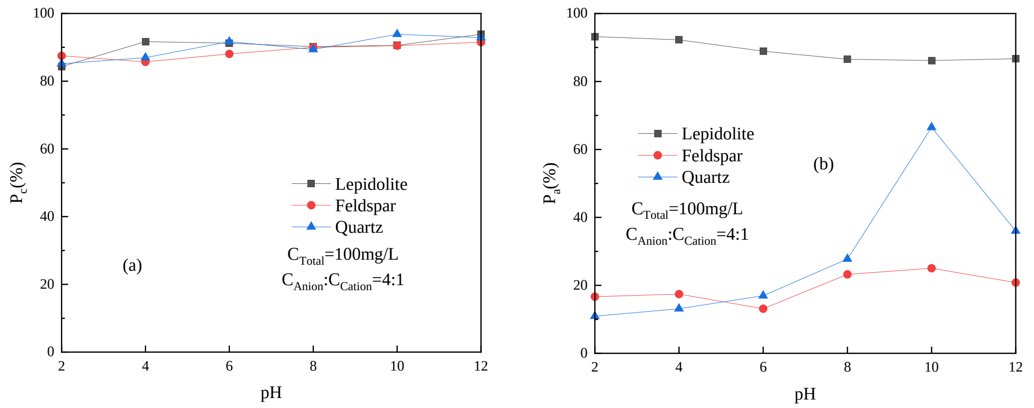

Fig. 10. P_c (a), P_a (b) of lepidolite, feldspar and quartz with bubbles at different pH using combined collector

3.4. Formation experiment of TPC between bubbles and mineral surface

3.4.1. t_{TPC} between bubbles and mineral surface using combined collector

Fig. 11(a) shows that in the solution of 100 mg/L combined collector, the velocity of the bubble on lepidolite surface changed slightly again after being stationary for a certain period, indicating that the bubble was attached on the surface and TPC was generated. In this process, the t_{TPC} of lepidolite was 114 ms, the t_B and t_D were 78ms and 36ms. This means that the surface of lepidolite was very hydrophobic at this time.

Fig. 11(b and c) shows that after collision and rebound, bubbles didn't form TPC on feldspar and quartz surfaces. This means that the surfaces of feldspar and quartz were more hydrophilic.

The result shows that the combined collector has an effect on the attachment process between bubbles and lepidolite, and promotes the formation of TPC, but has no effect on feldspar and quartz. This result also proves that the combined collector has selective adsorption effect on lepidolite.

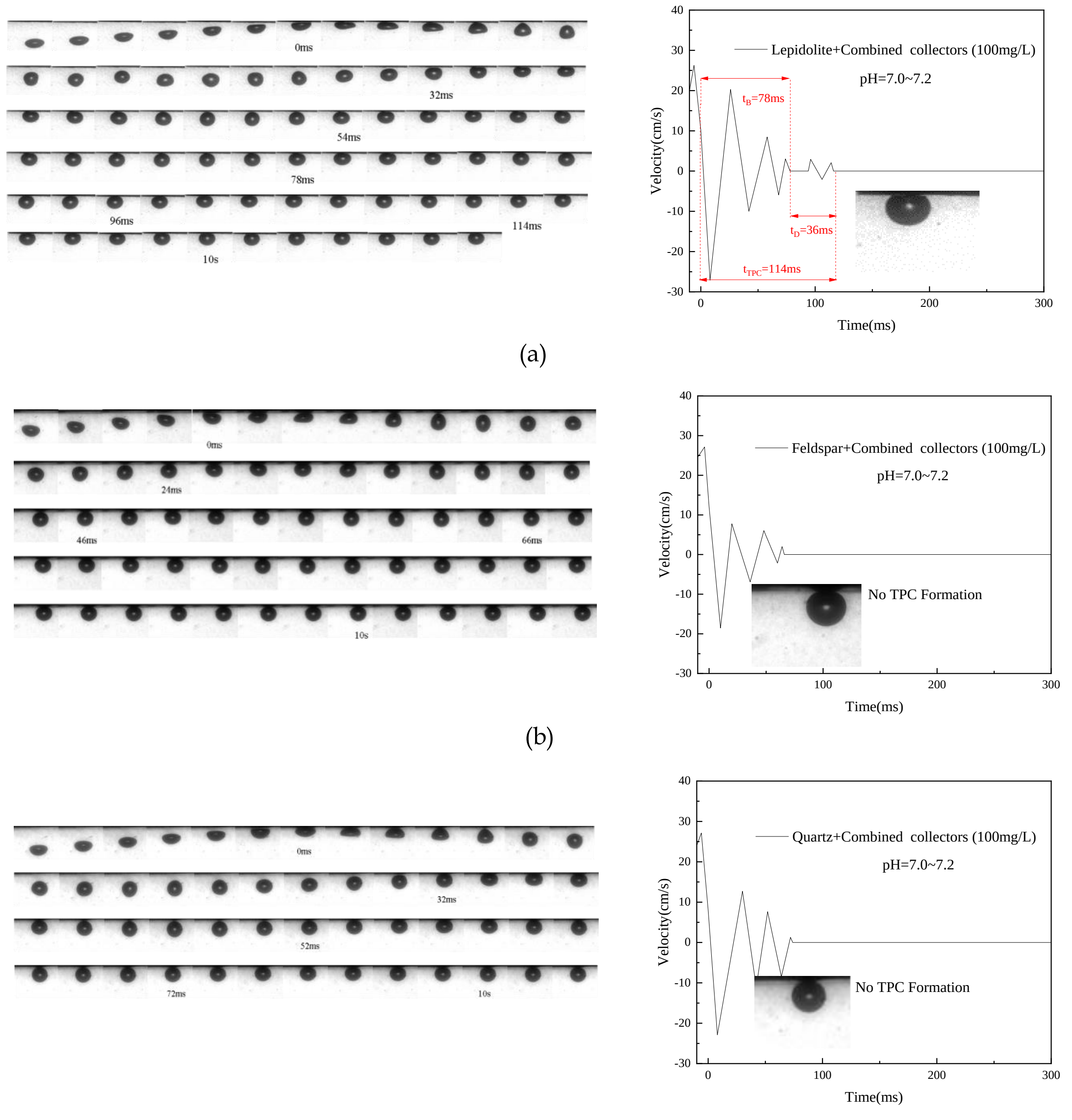

Fig. 11. Collision-rebound and velocity-time diagrams of bubbles and mineral surface

3.4.2. Effects on the t_{TPC}、t_B、t_D of lepidolite surface under different conditions

Fig. 12 (a)shows that when combined collector increased, the t_{TPC}、 t_B and t_D decreased in different degrees.

Fig. 12 (b)shows that when pH increased, the t_{TPC}、 t_B and t_D rise in different degrees. Especially, t_D was mainly affected by the surface properties of minerals and bubbles, which were easily changed by solution properties.

On the one hand, this result illustrated t_{TPC} depended on t_B and t_D in which t_D was dominant. On the other hand, this result illustrated the t_{TPC} was negatively correlated to the flotation recovery. The smaller t_D was, the smaller t_{TPC} was, indicating that the liquid film was more likely to break and form TPC. In flotation, minerals were more likely to be attached to bubbles to form mineralized foam, that was, the higher recovery was.

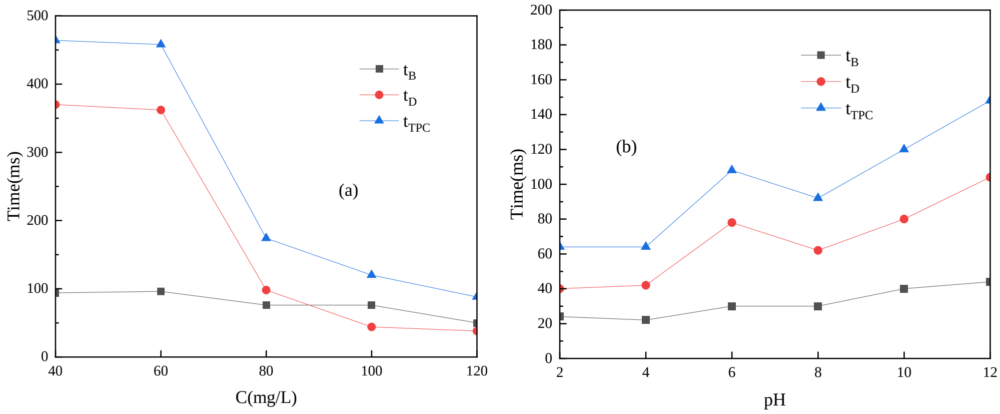

Fig. 12. Effects on combined collector dosage (a) and pH (b) on the t_{TPC}、t_B、t_D between bubbles and lepidolite surface

4. Conclusions

1. According to the flotation results of pure minerals, it was feasible to use HQ-330 and DDA as the combined collector for separation of lepidolite, feldspar and quartz. When pH=7, the ratio of HQ-330 to DDA was 4:1, the dosage of combined collector was 100 mg/L, the separation of lepidolite from feldspar and quartz could be realized. The results of artificial mixed ore experiments also confirmed this conclusion.
2. When the particle settlement method was used to study the interaction between bubbles and minerals, the collision probabilities (P_c) of lepidolite, feldspar and quartz were not affected by the properties of the solution, and they had a low correlation with the flotation recoveries. The attachment probabilities (P_a) of lepidolite, feldspar and quartz were greatly affected by solution properties, and they were positively correlated with the flotation recovery. Firstly, it was proved that the combined collector adsorbed on the surface of lepidolite selectively and thus improved its hydrophobicity. Secondly, the addition of combined collector greatly changed the attachment probability and according to the correlation between the attachment probability and flotation recovery, the attachment probability can be used as the theoretical basis for the flotation separation of lepidolite from feldspar and quartz.
3. When bubble rising method was used to study the interaction between bubbles and minerals, the TPC was formed in a short time between bubbles and lepidolite, while it was not formed between bubbles and feldspar and quartz. This result again confirmed that the combined collector can realize the separation of lepidolite, feldspar and quartz. Secondly, the t_{TPC} was negatively correlated with flotation recovery in which t_D was the main factor affecting it. And the combined collector could reduce t_{TPC} by reducing t_D. Therefore, t_{TPC} can also be used as a criterion for flotation separation.

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