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Study of Reagent Scheme, Entrainment and Their Relationship in Chalcopyrite Flotation in the Presence of Bentonite and Kaolinite

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Abstract: Entrainment has been considered as an important factor affecting clayey ore flotation. In this study, the effect of reagent dosage on chalcopyrite flotation in the presence of bentonite and kaolinite was investigated through entrainment. It was found that increasing the collector and frother dosage had little influence on copper recovery in the presence of bentonite, but decreased the copper grade substantially, owing to the increase in entrainment. With regard to kaolinite, increasing the reagent dosage increased the copper grade prominently, due to the decrease in entrainment. The substantial variation was related to the different interactions between the reagent and different clay minerals. The smaller surface area and hydration property of bentonite made most of the reagent remain in the solution, facilitating high entrainment, while kaolinite, with its larger surface area, adsorbed most of the reagent, which decreased the entrainment. The results of this study suggest a guideline of controlling reagent scheme in clayey ore flotation, based on the specific structure and properties of different clay minerals.



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Keywords: flotation; reagent dosage; bentonite; kaolinite

1. Introduction

With the depletion of high-quality ores, an increasing volume of low-grade ore containing different types and amounts of clay minerals as gangue is currently being processed. Most clay minerals are phyllosilicate minerals comprising silica tetrahedral (T) sheets and alumina octahedral (O) sheets joining together in proportions of 1:1 (T-O) and 2:1 (T-O-T) [1]. Kaolinite belongs to the 1:1 structural group, and bentonite is a typical 2:1 clay mineral with montmorillonite as the main component. The basal surface of clay minerals carries permanent negative charges due to isomorphous substitution, in which part of Si^{4+} ions in tetrahedral positions are replaced by Al^{3+} ions, or Al^{3+} ions in octahedral sites are replaced by Mg^{2+} ions [2]. The terminal OH groups on the edge surface carry either positive or negative charges depending on the type of metal ions and the pH of the aqueous solution. To balance the negative charges on the kaolinite surface, the exchangeable cations are located at the external basal surfaces or near the edges. For montmorillonite, the negative charge on basal faces is neutralized by an interlayer of exchangeable cations, such as Na^+ , K^+ , Ca^{2+} and Mg^{2+} , to make it chemically stable [3]. According to the relative magnitude of the basal and edge surface potentials and their relative positions, clay particles may associate by face-to-face (FF), edge-to-face (EF) and edge-to-edge (EE) interactions in relatively concentrated suspensions [4].

In the mineral processing industry, clay minerals have deleterious effects on the flotation of valuable minerals. The relationship between clay content, pulp rheology and flotation has been established. Merve Genc et al. [5] found that nickel sulphide ores

containing chrysotile as gangue led to an increase in Casson yield stress of the pulp, which correlated with a dramatic decrease in nickel grade. Patra et al. [6] observed that the addition of fibrous minerals in copper flotation increased pulp viscosity significantly, which contributed to a poor flotation result. Zhang and Peng [7] established a strong relationship between pulp viscosity and copper recovery in the presence of bentonite and kaolinite with low and high crystallinity: the higher the pulp viscosity, the lower the copper recovery. A prior study further found that increasing the concentration of bentonite reduced the amount of froth due to the increase in pulp viscosity and thus decreased copper recovery, while increasing the kaolinite content mainly decreased the copper grade owing to entrainment, with little effect on copper recovery [8]. Farrokhpay et al. studied the effect of different clay minerals on a copper ore from Australia, and also found that swelling clay bentonite adversely affected copper recovery and grade due to the significant change of rheology and froth stability, while non-swelling clay kaolinite reduced copper grade, but illite had the least effect on flotation performance [9]. The previous studies indicate that substantial investigations on pulp rheology and flotation recovery in the presence of clay minerals have been conducted, and entrainment has also been considered as an important factor.

In mineral flotation, entrainment is a mechanical mass transfer process, in which particles suspended in water between bubbles enter from the top of the pulp region into the froth region and then are transferred to the concentrate, which is not chemically selective [10]. The factors affecting entrainment have been investigated extensively, of which particle size and froth stability are vital [11]. Changes in air rate, froth depth and frother concentration result in variation in flotation performance that can be attributed to changes in froth stability [12]. A very stable froth facilitates the incorporation of fine particles into the froth's interstitial film. In turn, clay minerals with fine particle size have more chances to be recovered by entrainment [13]. The prior study and the investigation from Farrokhpay et al. both found that the froth stability varied in the presence of different clay minerals, bentonite and kaolinite [8,9]. Liu and Peng used hydrophilic kaolinite Q38 in flotation and found there was a nearly linear relationship between mass recovery and water recovery both in tap water and saline water, which indicated a high degree of kaolinite entrainment [14]. However, entrainment in the presence of different clay minerals in flotation has not been investigated thoroughly.

Clay particles are characterized by small size and high specific surface area, which increase their adsorbing reagent capacities in flotation. The reagent consumption in flotation has been considered as an important aspect of the flotation strategy [15,16]. In the prior study, it was also recorded that the collector and frother dosage affected the flotation performance of a practical ore differently in the presence of clay minerals [17]. To further verify the possible relationship between reagent scheme, entrainment and types and content of clay minerals, an artificial clayey ore mixed by chalcopyrite, quartz and clay was used in this study. The objective was to investigate the effect of reagent dosage on chalcopyrite flotation in the presence of bentonite and kaolinite and to understand the relative mechanism thereof.

2. Experimental Section

2.1. Materials

Chalcopyrite with a purity of 98% mixed with quartz with a purity of 97% was used as an artificial clean ore, both of which were purchased from Mingfa Mineral Specimen Company in Guangzhou, China [18]. Sodium bentonite was purchased from Sibelco Group, Australia, and was composed of 63% sodium montmorillonite, 12% quartz and 25% albite, as described elsewhere [8]. Kaolinite with 98% purity was purchased from Mingfa Mineral Specimen Company in Guangzhou, China. Particle size distributions of these clay mineral samples were similar, with 80% of particles being smaller than 33 μm . Butyl xanthate with a purity of 85% was obtained from Zhuzhou Reagents Factory in Hunan province, China, and was used as the collector. Methyl isobutyl carbinol (MIBC) was used as the frother in

the laboratory tests. Deionized water was used throughout the experiments. Lime was used to control the pH value of the slurry in flotation.

2.2. Grinding and Flotation

The chalcopyrite and quartz ore were crushed to -1 mm, separately, followed by grinding in a laboratory planetary ball mill with zirconia as the grinding media, to obtain an 80% passing size of $106\ \mu\text{m}$ ($P_{80} = 106\ \mu\text{m}$). In the clean ore mixture, 10 g of chalcopyrite was used as the valuable mineral, with 90 g of quartz as gangue mineral, which was then transferred to a 0.5 L plexiglass flotation cell. The agitation speed was kept at 1700 rpm throughout the flotation experiments. For each test, a calculated amount of quartz, 10 g, 20 g and 30 g, was replaced by the same amount of a well-mixed clay mineral, respectively, to make up 10 wt.%, 20 wt.% and 30 wt.% artificial clayey ore samples at the same pulp density. The different particle sizes were not found to contribute to rheology and flotation results [7]. The slurry was conditioned with collector for 3 min and with frother for 1 min. The flotation froth was scraped every 5 s. Four flotation concentrates were collected after cumulative times of 1, 2, 4 and 6 min with an air flow rate of $0.4\ \text{m}^3/\text{h}$. The pH value was maintained constant at 9.0 for all of the flotation tests. The key elemental composition of the concentrate and tailing were analyzed using X-ray fluorescence (XRF, Axios Max, PANalytical B.V.)

2.3. Reagent Adsorption

The adsorption amount of the reagent on the surface of the mineral was determined by the residual concentration method using a TOC-L analyzer (Shimadzu, Japan). First, the method of stepwise dilution was used to dilute the pharmaceutical solution into a standard solution of different concentrations, based on which, the concentration of the reagent was determined and the standard curve of the reagent was drawn. Second, the flotation sample with reagent addition after the conditioning stage was centrifuged, and the upper layer of clear liquid was taken. Then, the upper layer of the supernatant was measured, and the measurement results were converted to obtain the residual concentration of the reagent.

2.4. Clay Mineral Characterization

The surface area, pore volume and pore diameter of bentonite and kaolinite were measured using a QuadasorbSI-3 (Quantachrome Instruments). The outgas temperature was maintained at $30\ ^\circ\text{C}$ and the adsorption–desorption isotherms of N_2 were measured at $-196\ ^\circ\text{C}$. The Brunauer–Emmett–Teller (BET) method was used to calculate the surface area. The pore volume of clay minerals was calculated based on the adsorption amount at a relative pressure of (P/P_0) 0.99. The Barrett–Joyner–Halenda (BJH) method of adsorption analysis was used to calculate the pore size of clay minerals.

3. Results and discussion

3.1. Effect of Reagent Dosage on the Flotation of Clayey Ores

Figures 1 and 2 show the flotation results of artificial clean ore doped with different amounts of bentonite or kaolinite when using different reagent schemes. As illustrated in Figure 1a, generally, copper recovery decreased from about 96% to 61%, as the proportion of bentonite increased from 0 to 30 wt.%. When reagent dosage was doubled, the change in copper recovery was not prominent in the presence of bentonite. Figure 1b shows that, at low reagent dosage, the copper grade was constant at about 28%, as the bentonite content increased from 0 to 20 wt.%. Upon further increasing the bentonite concentration to 30 wt.%, the copper grade decreased slightly. Increasing the reagent dosage decreased the copper grade significantly within the same bentonite concentration.

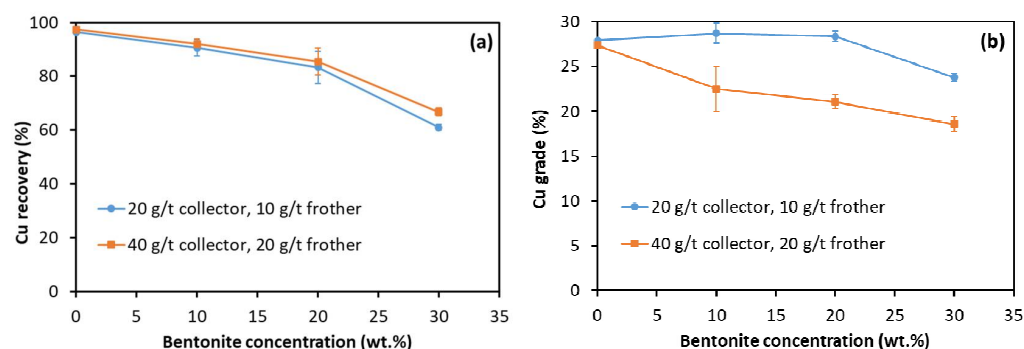


Figure 1. Effect of reagent dosage on chalcopyrite flotation in the presence of bentonite with different concentrations: (a) copper recovery; (b) copper grade.

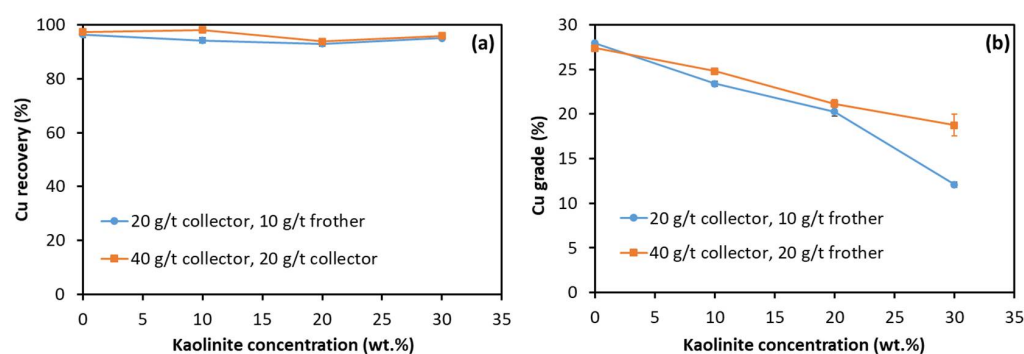


Figure 2. Effect of reagent dosage on chalcopyrite flotation in the presence of kaolinite with different concentrations: (a) copper recovery; (b) copper grade.

For kaolinite, the general trends in Figure 2 indicate that with increasing the kaolinite proportion from 0 to 30 wt.%, the copper recovery remained constant but the copper grade decreased. When the reagent dosage was doubled, the copper recovery was not affected, as shown in Figure 2a, while the copper grade increased slightly, as shown in Figure 2b.

The results in Figures 1 and 2 were generally in accordance with the prior study. At a low reagent dosage, increasing the proportion of bentonite reduced copper recovery with little effect on copper grade, while increasing the kaolinite content mainly decreased the copper grade with little effect on copper recovery. As illustrated in previous studies, increasing the clay mineral concentration increases the viscosity of the pulp, and decreases valuable mineral recovery [7–9]. Compared with kaolinite, bentonite had a more prominent influence on pulp rheology [19–21]. On the other hand, at high reagent dosage and high bentonite concentration, both copper recovery and copper grade decreased, which was in agreement with the finding from Farrokhpay et al. that both the substantial change of pulp rheology and entrainment played an important role [9]. Hence, it is also indicated that the reagent scheme played an important role in chalcopyrite flotation in the presence of bentonite and kaolinite.

3.2. Effect of Reagent Dosage on Mass and Water Recovery

Figures 3 and 4 show the corresponding mass and water recovery in chalcopyrite flotation in the presence of bentonite and kaolinite with different reagent dosages. As can be seen in Figure 3, both mass recovery and water recovery in the presence of bentonite were relatively low at a lower reagent dosage. Increasing the concentration of bentonite did not affect mass recovery, which was maintained between 7% and 8%, but decreased water recovery from about 15% to 10%. Increasing the reagent dosage enhanced mass recovery and water recovery significantly, which was more prominent at a high bentonite concentration. The prominent increase in water recovery shown in Figure 3b might have been responsible for the increase in mass recovery shown in Figure 3a, when the reagent

dosage was doubled. With the same bentonite concentration, copper recovery did not change significantly, as shown in Figure 1a. Thus, the substantial decrease in copper grade shown in Figure 1b should be ascribed to the increase in the recovery of gangue minerals at a high reagent dosage.

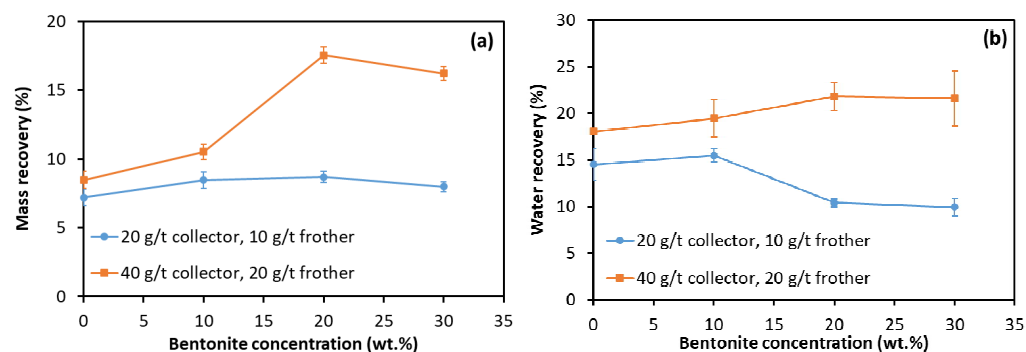


Figure 3. Effect of reagent dosage on mass and water recovery in the presence of bentonite: (a) mass recovery; (b) water recovery.

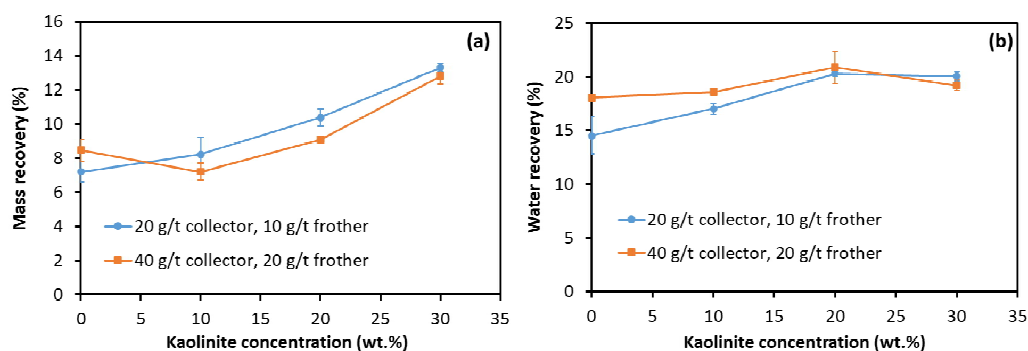


Figure 4. Effect of reagent dosage on mass and water recovery in the presence of kaolinite: (a) mass recovery; (b) water recovery.

For kaolinite, increasing the reagent dosage decreased mass recovery, as shown in Figure 4a, which was different in the case of bentonite, as shown in Figure 3a. Meanwhile, Figure 4b shows that, within the same kaolinite concentration range, water recovery increased slightly as the reagent dosage was increased. As copper recovery was maintained, as shown in Figure 2a, the slight increase in copper grade at a higher reagent dosage, shown in Figure 2b, should be owing to the reduction of mass recovery shown in Figure 4a. As illustrated in the prior study, increasing the kaolinite concentration could facilitate the formation of network structures that usually have a low density and tend to move upwards into froth [8]. Thus, the combination of Figures 2b and 4a indicates that the reduction of mass recovery at a higher reagent dosage, as shown in Figure 4a, might be due to the decrease of kaolinite content in the concentrate, resulting in the increase of copper grade shown in Figure 2b.

3.3. Effect of Reagent Dosage on Entrainment

The above results indicate that increasing the reagent dosage contributed to a substantial increase in mass and water recovery for bentonite-ore, and to a decrease in mass recovery for kaolinite-ore. The variation in mass recovery and water recovery of clayey ore with different reagent dosages shown in Figures 3 and 4 might be related to entrainment, as entrainment is a strong function of mass recovery and water recovery [22,23]. The degree

of entrainment (ENT) of bentonite-ore and kaolinite-ore under different reagent schemes was calculated based on an unsized equation, as following [23]:

$$ENT^{con:tail} = \frac{\left(\frac{M_{gangue}}{M_{water}}\right)_{con}}{\left(\frac{M_{gangue}}{M_{water}}\right)_{tail}}$$

where M is the mass (g).

As can be seen in Figures 5 and 6, generally, the degree of entrainment (ENT) increased with increasing the concentration of bentonite or kaolinite in chalcopyrite flotation, which illustrates the decrease of copper grade reported in the previous studies and the study from Farrokhpay et al. [8,9,17]. Moreover, it is indicated that increasing the reagent dosage increased ENT significantly for bentonite-ore but decreased the ENT for kaolinite-ore. Hence, the reduction of copper grade in the presence of bentonite in Figure 1b at high reagent dosage was due to the increase in the ENT, while the increase in copper grade in the presence of kaolinite in Figure 2b was related to the decrease in ENT at a higher reagent dosage. It should be noted that in the absence of clay minerals, although increasing the reagent dosage increased both mass recovery and water recovery, as shown in Figures 3 and 4, the ENT was maintained constant in Figures 5 and 6. Thus, the reagent scheme may have a more direct relationship with the entrainment of clay minerals.

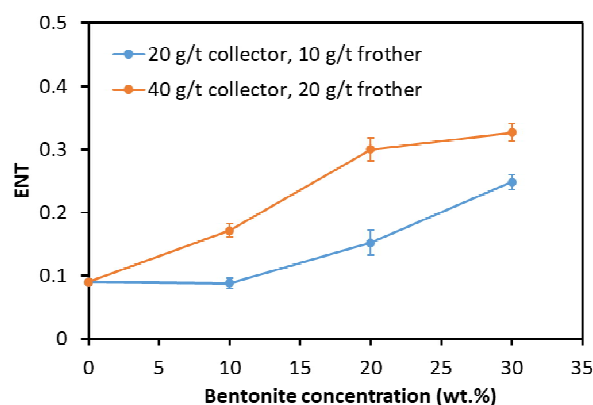


Figure 5. Effect of reagent dosage on the degree of entrainment (ENT) in the presence of bentonite.

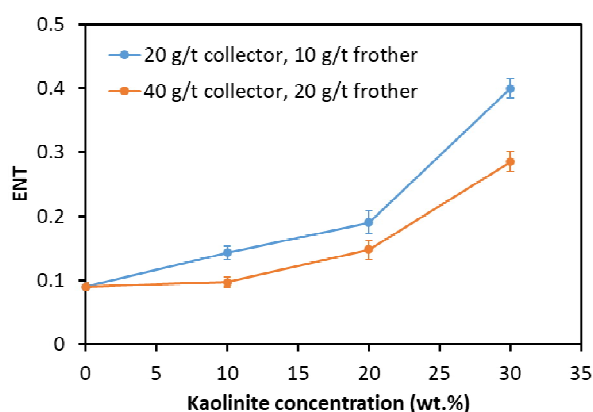


Figure 6. Effect of reagent dosage on the degree of entrainment (ENT) in the presence of kaolinite.

To further investigate the composition of the entrainment gangue, Table 1 shows the content of Al_2O_3 and SiO_2 in a concentrate of chalcopyrite flotation in the presence of 20 wt.% bentonite and 20 wt.% kaolinite, respectively. The composition of kaolinite and sodium bentonite in this study was $Al_2Si_2O_5(OH)_4$ and $Na_{0.33}[(Al_{1.67}Mg_{0.33})(O(OH))_2(SiO_2)_4]$, respectively, while

SiO₂ was the main component of quartz. The results shown in Table 1 generally indicate that both clay minerals and quartz were found in the concentrate, which is similar to the finding from Farrokhpay [9]. Moreover, in this study, as the reagent dosage increased, more SiO₂ was reported in the concentrate for bentonite-ore compared to Al₂O₃, while the content of Al₂O₃ and SiO₂ was reduced proportionally for kaolinite-ore. It can therefore be concluded that increasing the reagent dosage might significantly increase both bentonite and quartz in the concentrate, while kaolinite might be mainly depressed. XRD analysis of the specific component of gangue in the concentrate and the mineralogy transformation between them will be investigated in a future study.

Table 1. The average content of Al₂O₃ and SiO₂ in the concentrate (%).

Ore Sample	Reagent Scheme	Al ₂ O ₃	SiO ₂
Bentonite-ore	Low reagent dosage	7.28	38.96
	High reagent dosage	9.62	50.33
Kaolinite-ore	Low reagent dosage	16.37	41.34
	High reagent dosage	14.48	38.04

3.4. Effect of Reagent Consumption

As the reagent scheme affected the ENT of clayey ore differently, it is assumed that bentonite and kaolinite may have different interactions with the reagent. Figures 7 and 8 show the reagent consumption in the conditioning stage in copper flotation with 20 wt.% clay minerals. As can be seen in Figure 7, after the conditioning stage, most of the collector generally remained in the pulp solution in the presence of 20 wt.% bentonite, while collector consumption was almost 100% in the presence of 20 wt.% kaolinite. In Figure 8, the residual concentration of frother was higher for bentonite-ore than for kaolinite-ore, regardless the froth dosage, which was different from the report by Farrokhpay et al., where no frother was consumed either for clean ore or clayey ore with different clay minerals [9]. Based on the results shown in Figures 7 and 8, it is clear that reagent consumption affected the mass and water recovery differently in the presence of bentonite and kaolinite, as shown in Figures 3 and 4, which then resulted in the different effects on copper recovery and grade shown in Figures 1 and 2. The larger amount of residual reagent in the pulp facilitated the increase in non-selective entrainment, as illustrated in Figure 5 and Table 1, resulting in a lower copper grade in the presence of bentonite. On the other hand, the high consumption of reagent in the presence of kaolinite reduced the amount of reagent in flotation solution. Upon further increasing the reagent dosage, an increasing amount of reagent was available for selective collection in flotation, leading to an increase in copper grade.

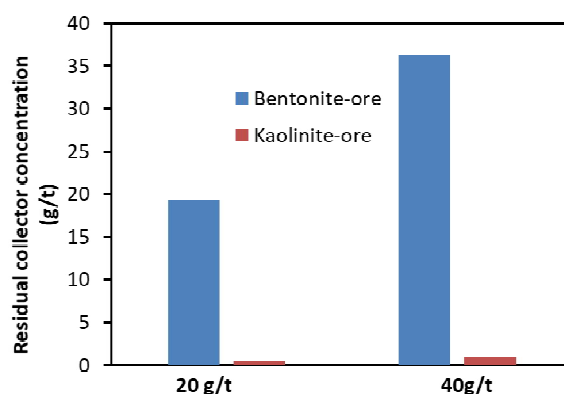


Figure 7. Residual collector concentration in the conditioning stage of chalcopyrite flotation in the presence of 20 wt.% bentonite or kaolinite.

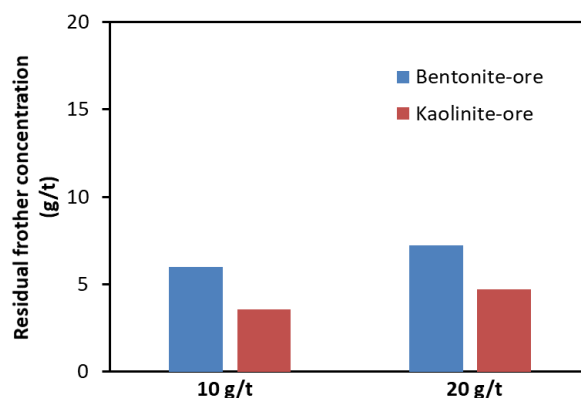


Figure 8. Residual frother concentration in the conditioning stage of chalcopyrite flotation in the presence of 20 wt.% bentonite or kaolinite.

3.5. Mechanism of Reagent Adsorption on Different Clay Minerals

The variation of reagent consumption with different types of clay minerals might be associated with their unique physicochemical properties. As shown in Table 2, at room temperature the bentonite used in this study had a smaller pore diameter, surface area and pore volume than those of kaolinite, which illustrates the different capacities for reagent adsorption.

Table 2. The surface area, pore volume and pore diameter of bentonite and kaolinite.

	Bentonite	Kaolinite
Surface Area, m ² /g	11.958	21.935
Pore volume, cc/g	0.054	0.097
Pore diameter, nm	2.716	4.303

The difference in adsorption capacity for bentonite and kaolinite could be continuously exaggerated in aqueous solution. In clay science, kaolinite exhibits no interlayer expansion swelling in aqueous solution, due to the Al-OH . . . O-Si- hydrogen bonding supplemented by dipole-dipole and van der Waals interactions which could hold the layers together within a particle [24]. Bentonite with a 2:1 structure is more reactive than kaolinite with a 1:1 structure. In deionized water or aqueous solution with low salt concentration, water molecules are able to penetrate between bentonite platelets, which increases the interlayer distance significantly, resulting in intercrystalline and osmotic swelling [19,25]. Miller et al. suggested that a pore size smaller than 1 nm would be inaccessible to a MIBC molecule [26]. Hence, it is possible that the extensive hydration of bentonite in aqueous solution further made the pore size inaccessible to a collector or frother molecule, with most of the reagent remaining in the pulp solution, as shown in Figures 7 and 8.

Based on the results of this study, the mechanism of reagent adsorption on different clay minerals is proposed, as illustrated in Figure 9. The smaller surface area together with the hydration property of bentonite makes it difficult for reagent adsorbing onto the mineral surface. Increasing the reagent dosage increased the amount of reagent in the pulp solution (Figures 7 and 8), resulting in the increase of ENT (Figure 5), which reduced the copper grade, as shown in Figure 1b. On the other hand, due to the high specific surface area (Table 2) and the unlikely hydration property of kaolinite, the reagent is more likely to adsorb on the kaolinite surface, resulting in the high reagent consumption shown in Figures 7 and 8. Upon further increasing the reagent dosage, more of the reagent would be available for selective chalcopyrite flotation. According to Sis and Chander [27], collector and frother molecules could penetrate at the air-liquid interface into the diffuse monolayer of the solid, which would increase the attachment of chalcopyrite to air bubbles, resulting in the higher copper grade shown in Figure 2b. Thus, the surface area, pore size and the

extent of clay mineral hydration affected the adsorption of reagent differently, leading to the variation in flotation results.

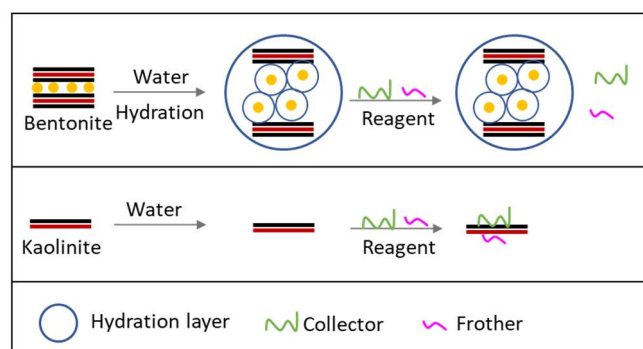


Figure 9. A schematic view of the mechanism of reagent adsorption for different clay minerals.

4. Conclusions

The reagent scheme played an important role in chalcopyrite flotation doped with different clay minerals. Generally, increasing the collector and frother dosage had little influence on copper recovery in the presence of either bentonite or kaolinite, but affected copper grade differently, which was correlated with reagent consumption in the conditioning stage. As a larger amount of reagent left in the pulp in the presence of bentonite contributed to a significant increase in entrainment, the copper grade decreased when the reagent dosage was doubled. On the other hand, only a small amount of reagent was detected after the conditioning stage in the presence of kaolinite. Upon further increasing the reagent dosage, an increasing amount of reagent would be available for selective collection in flotation, leading to an increase in copper grade in the presence of kaolinite. The different effects of reagent consumption in flotation were associated with the physicochemical property and different hydration nature of bentonite and kaolinite. The findings in this study offer support for controlling reagent schemes in flotation based on the presence of relevant clay minerals.

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Conflicts of Interest: The authors declare no conflict of interest.

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