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Effect of Gangue Minerals on Pulp Rheology and Flotation Behavior of Smithsonite

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Abstract: The pulp rheology is an important factor affecting flotation performance. Gangue minerals have a significant impact on the rheological property of smithsonite ($ZnCO_3$). The complex pulp rheology (high viscosity, poor dispersion) will adversely affect the grade and recovery of concentrate. To address this problem, the effect of gangue minerals (kaolinite, calcite, and quartz) on the rheological property and flotation behavior of smithsonite was investigated with the pulp rheology measurement and a flotation test. The results showed that the flotation recovery is closely associated with the pulp rheology and high apparent viscosity and that yield stress resulted in a low recovery. The detrimental influence of gangue minerals on the rheology and yield of smithsonite was kaolinite > calcite > quartz. The coarse mineral particles (150–74 μm, 74–38 μm) could reduce the apparent viscosity and yield stress and present good floatability, while fine particles (38–23 μm, −23 μm) could greatly increase the values and show poor floatability. Therefore, to control the pulp rheology of smithsonite, it is necessary to remove fine gangue minerals (−38 μm) before flotation, especially the fine kaolinite.

Keywords: gangue mineral; smithsonite; pulp rheology; apparent viscosity; yield stress; flotation

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1. Introduction

Zinc is an important nonferrous metal that mainly exists in the form of sulfide and oxide deposits. Zinc oxide minerals are characterized by complex composition, fine dissemination size, serious slimming phenomenon, and high soluble salt content [1–3]. Various inevitable ions such as Zn, Ca, and Mg ions have great influence on zinc floatability, resulting in the unsatisfactory recovery index in production practice [4]. Zinc oxide minerals with industrial value include zinc carbonate smithsonite, heteropolar ore, silicon zinc ore, zinc blende, red zinc ore, etc.; among them, smithsonite is the most common zinc mineral. Common gangue minerals include calcite, dolomite, quartz, clay minerals, and limonite [5]. Flotation is the most commonly used method for beneficiation of oxidized zinc minerals. The cationic collector such as dodecylamine (DDA); anionic collectors such as oleic acid (OA), potassium amyl xanthate (KAX), hexyl mercaptan (HM); and mixed collector (cationic/anionic) are often used [6,7]. Depressants of gangue include sodium silicate, sodium hexamethaphosphate, starch [8]. Among the dispersants tested, using sodium hexametaphosphate could obtain a higher zinc grade and recovery [9]. Compared with sulfide ores with a large difference in floatability of gangue minerals, the main problems in zinc oxide ore flotation are the presence of slimes. The mineral surface is always covered by the slimes, which hinders the interaction between reagents and minerals, thus reducing the collection efficiency [10]. Moreover, the flotation conditions of various minerals are different, and the interference between flotation reagents is serious.

Pulp rheology is a discipline to study the flow and deformation properties of slurry fluid under the action of external shear stress during mineral processing [11,12]. Pulp rheology (yield stress and apparent viscosity) is a quantitative indicator to characterize the

degree of particle aggregation or dispersion, which is an important factor affecting mineral flotation recovery [13–15]. Rheology is an important property to understand and maintain the economic viability of the flotation process especially of low-grade and complex ores [16]. The flotation pulp belongs to a complex multiphase suspension system. The rheological property of the pulp is affected by the mineral composition, especially the clay minerals. Different types of clay minerals usually exist in the ore. They have several potential harmful effects on flotation. First, when clay minerals attach to the mineral surface, they will reduce the floatability of the valuable minerals. Second, clay minerals form a network structure in the pulp, which may lead to the increase of pulp viscosity or gangue entrainment, and then decrease the flotation recovery and the grade of flotation products [17].

Basnayaka et al. [18] studied the effects of two clay minerals, kaolinite and bentonite, on the rheology and flotation of pyrite pulp. The results showed that both the type and the concentration of clays had an impact on the flotation of gold ore with pyrite as the carrier mineral. More specifically, bentonite reduced the recovery of pyrite in the ore and had a significantly greater impact on the rheological properties of the pulp than kaolinite. Cruz et al. [19,20] also found that kaolinite had no significant impact on the apparent viscosity of pulp under the action of corresponding flotation reagents, but it significantly increased the yield stress of the foam layer, indicating that kaolinite entered the concentrate in the form of entrainment and worsened the flotation. Zhang and Peng [21] studied the influence of clay minerals on the rheology and flotation of copper and gold mineral slurry. It was found that the interaction of clay mineral particles was the main factor affecting the rheological property of the pulp, especially the influence of bentonite on the pulp viscosity which increased with the increase of concentration. The higher the pulp viscosity is, the lower the copper recovery will appear. Under the same concentration conditions, when the content of bentonite in the pulp increased, the apparent viscosity and yield stress of the flotation pulp increased sharply.

The regulation of pulp rheology to improve flotation indexes has attracted more attention. Chen et al. [22] utilized garnet to control the rheology of flotation pulp in fine scheelite flotation and found that the pulp viscosity decreases and the flotation rate increases when garnet was added in the cleaning flotation. Liu et al. [23] also found that the addition of coarse garnet particles (−104 + 44 μ m) could decrease the pulp viscosity and yield stress remarkably, the hetero-coagulation between sulfide and serpentine was limited, and the synthetic actions improved Cu-Ni sulfide flotation.

This research aims to investigate the influence of different gangue minerals on the rheological properties and flotation performance of smithsonite under different conditions. Effects of gangue mineral content, pulp concentration, and particle size on the apparent viscosity and yield stress of smithsonite were systematically studied and are presented. It is then followed by a discussion on the effect of different gangue content and sodium hexametaphosphate on flotation behavior of smithsonite.

2. Materials and Methods

2.1. Materials

The smithsonite was taken from Liaoning Province, China, while calcite was obtained from Beijing, China, and kaolinite and quartz were collected from Hebei Province, China. The purity of smithsonite, kaolinite, calcite, and quartz were all above 95%. The zirconia ceramic ball mill with rubber lining was used for grinding. The grinding products were dry sieved to obtain particles in different size ranges (150–74 μm, 74–38 μm, 38–23 μm, −23 μm). The pH regulator sodium hydroxide (NaOH) was analytically pure; the octadecylamine used as collector was chemically pure; the sodium hexametaphosphate used as depressant was analytical pure; and all waters in the whole experiments were deionized.

2.2. Pulp Rheology Measurement

Pulp rheology was measured with the rotary rheometer (MCR720, Anton Paar, Graz, Austria). It is necessary to improve the rheometer partially because the flotation pulp

is an unstable solid–liquid–gas three-phase suspension system that is easy to settle; the rheological properties are very complex and difficult to measure. In order to weaken the influence of particle sedimentation on the measurement results, the traditional cylindrical inner cup was replaced with a paddle-type stirring rotor (Figure 1). Rheological measurements mainly include the shear rate vs shear stress test (controlled shear rate mode) for measuring the apparent viscosity of the pulp (equation 1) and the shear stress vs. shear deformation test (controlled shear stress mode) for the yield stress of the pulp. The samples for rheology tests were prepared by mixing required mineral with water to meet desired pulp concentration. The pulp pH was adjusted to 9.5−10.0 using NaOH solution, and a pH meter was used to measure pH. The tests were conducted at room temperature (about 20 °C).

$$\eta = \tau / D \quad (1)$$

where η is the apparent viscosity (Pa · s), D is the shear rate (s^{-1}), and τ is the shear stress (Pa).



Figure 1. The paddle-type stirring rotor.

2.3. Flotation Test

The flotation test was conducted in a flotation machine (XFG, Jilin Exploration Machinery Plant, Changchun, China) with a 40 mL cell; the stirring speed was 1800 rpm. First, the mineral powder was added to the cell with 40 mL deionized water. The pulp pH was regulated to 9.5–10.0 using NaOH solution. Then the inhibitor sodium hexametaphosphate and the collector octadecylamine were added into the pulp in turn. The conditioning time of each regent was 3 min. The flotation time was maintained at 6 min, and forth was manually scrapped every 15 s during flotation. The concentrates (foam product) and tailings (tank product) were collected with watch glass. After flotation, the products were filtered, dried and weighted. The flotation yield was calculated based on the weight of the dry products.

2.4. Zeta Potential Measurements

The zeta potentials of smithsonite, kaolinite, calcite, and quartz in the absence and presence of octadecylamine at pH 9.5 were measured with a zeta potential analyzer (Delsa™ Nano C, Beckman Coulter, Brea, CA, USA). A suspension containing 0.1 wt% of the solid content was agitated for 5 min with a magnetic stirrer so that the mineral particles could be fully dispersed. The zeta potential was measured at least twice for each sample, and the average was reported as the final value.

3. Results and Discussions

3.1. Effect of Gangue Mineral Content on Apparent Viscosity and Yield Stress of Smithsonite Pulp

With octadecylamine as the collector, the pulp pH and solid concentration were set as 9.5−10.0 and 28.57%, respectively. The particle size range of the minerals was −23 μm. The pulp apparent viscosity and yield stress for mixed mineral of smithsonite-kaolinite, smithsonite-calcite, and smithsonite-quartz under different gangue mineral contents were investigated, and the results are shown in Figure 2. Figure 2 shows that the pulp apparent viscosity and yield stress increased gradually with the increase of kaolinite and calcite content in the binary-mixed minerals of smithsonite-kaolinite and smithsonite-calcite. This became significant when the gangue mineral content exceeded 20%; the values increased rapidly. Moreover, the apparent viscosity and yield stress of smithsonite in the presence of kaolinite was higher than that of calcite. Wang et al. [24] studied the effects of fine kaolinite, illite, and pyrophyllite on the rheology and flotation of pyrite and diaspore-mixed ore. The addition of kaolinite and illite could increase the apparent viscosity of the pulp and reduce the flotation recovery of pyrite.

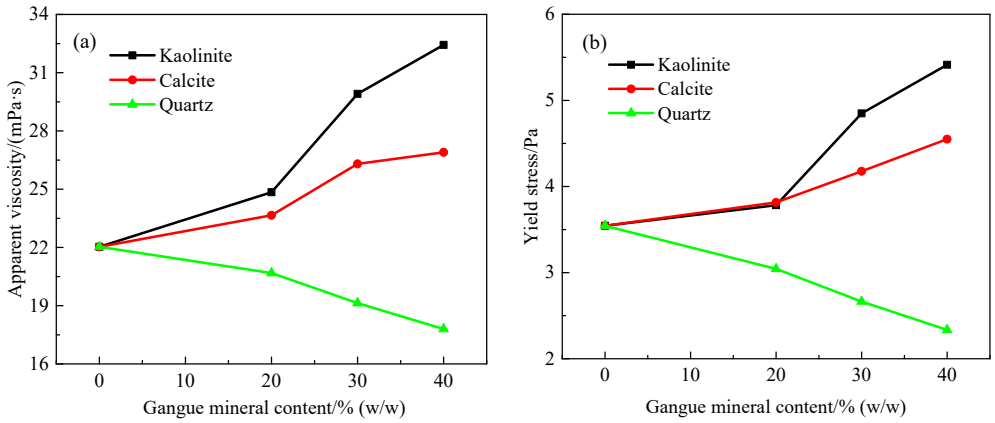

Figure 2. Effect of gangue mineral content on (**a**) apparent viscosity and (**b**) yield stress of smithsonite pulp using octadecylamine concentration: 150 mg/L at pH 9.5−10 and 28.57% solid concentration.

However, the apparent viscosity and yield stress of the mixed minerals of smithsonite-quartz decreased with the increase of quartz content, indicating that quartz could disperse the mixed mineral pulp. The zeta potential of minerals was tested to explain this reason, and the results are shown in Figure 3. Zeta potential can characterize the stability of the colloidal dispersion system: the higher the absolute value (positive or negative) of zeta potential, the more stable of the dispersion system. As can be seen from Figure 3, the surface of kaolinite, calcite, and quartz was negatively charged at pH 9.5, but the absolute value of zeta potential of quartz was obviously higher than that of kaolinite and calcite. This shows that quartz particles have better dispersion due to the strong electrostatic repulsion, so the apparent viscosity and yield stress of the mixed minerals of smithsonite-quartz decreased.

The shape of particles is an important factor affecting the apparent viscosity. Generally, the higher the sphericity of particles is, the lower the apparent viscosity will become. Figure 4 showed the shape characteristics of smithsonite, calcite, kaolinite, and quartz particles analyzed using Scanning Electron Microscopy (SEM). It can be seen that the sphericity of smithsonite is relatively high, and there are no particularly sharp edges, while calcite has sharp edges, and the overall evenness is poor. This is also one of the factors that the viscosity of calcite pulp was higher than that of smithsonite. The particles of kaolinite are loose and irregular. It can be seen that the particles are actually layered silicate structure, which can easily form a network structure in the pulp and, thus, seriously affect the rheological properties of the pulp. Among the pulp viscosities of various single minerals, the apparent viscosity and yield stress of kaolinite pulp have significantly increased compared with the other three kinds of pulps at the same concentration. The sphericity of quartz

particles is low, but the apparent viscosity of quartz pulp is lower than those of the other three minerals. Such findings turned out to be contrary to the conclusion that the higher the sphericity of particles is, the lower the apparent viscosity will become. It shows that the particle shape is not the main factor affecting the viscosity of quartz pulp.

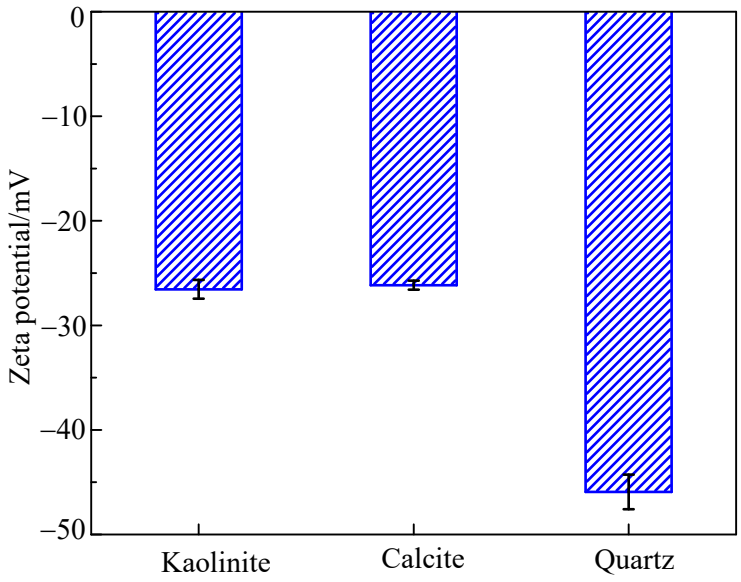

Figure 3. Zeta potential of kaolinite, calcite, and quartz at pH 9.5.

3.2. Effect of Concentration on Pulp Apparent Viscosity and Yield Stress of Smithsonite

The pulp pH was 9.5–10.0, the particle size range of the minerals was −23 μm, and the gangue mineral content was fixed at 30% (*w*/*w*). The apparent viscosity and yield stress of the mixed mineral pulp of smithsonite-kaolinite, smithsonite-calcite, and smithsonite-quartz at different pulp concentrations are shown in Figure 5. The pulp concentration has a great impact on the rheological property. With the increase of the pulp concentration, both the apparent viscosity and yield stress of the mixed mineral pulp increased, especially the apparent viscosity and yield stress of the smithsonite-kaolin-mixed mineral which changed significantly. But the apparent viscosity and yield stress of the smithsonite-quartz-mixed mineral increased slowly. When the pulp concentration was 28.57%, the apparent viscosity of the smithsonite-kaolin-mixed mineral was 29.91 mPa·s, and the yield stress was 4.84 Pa; meanwhile, the apparent viscosity of the smithsonite-quartz-mixed mineral was 23.62 mPa·s, and the yield stress was only 1.55 Pa. When the pulp concentration increased to 40%, the apparent viscosity of the smithsonite-kaolin-mixed mineral reached up to 78.63 mPa·s, and the yield stress increased to 15.20 Pa, while the apparent viscosity of the smithsonite-quartz-mixed mineral was 34.21 mPa·s, and the yield stress was 2.43 Pa at the same pulp concentration. It can be concluded that when the mineral content in the mixed mineral pulp increases, the internal friction of mineral particles in the shear field increases, and the interaction between particles is enhanced. The fine particles in a pulp will affect the effective dispersion of mineral particles. The increase of the apparent viscosity of the pulp will cause the reduction of the selectivity of flotation reagents and, thus, result in entrainment in flotation operation and affect the concentrate grade. Relevant research [25–27] showed that a low concentration pulp displays a Newtonian behavior. As the amount of solid in a pulp increases, the rheological behavior of the slurries shifts from Newtonian to non-Newtonian, with the progressive appearance of a yield stress and an exponential increase in the pulp viscosity. This could cause a deleterious rheological effect on flotation. Cruz et al. [28] studied the rheology of kaolinite pulp and found that kaolinite concentration affected the rheogram and apparent viscosity of pulp.

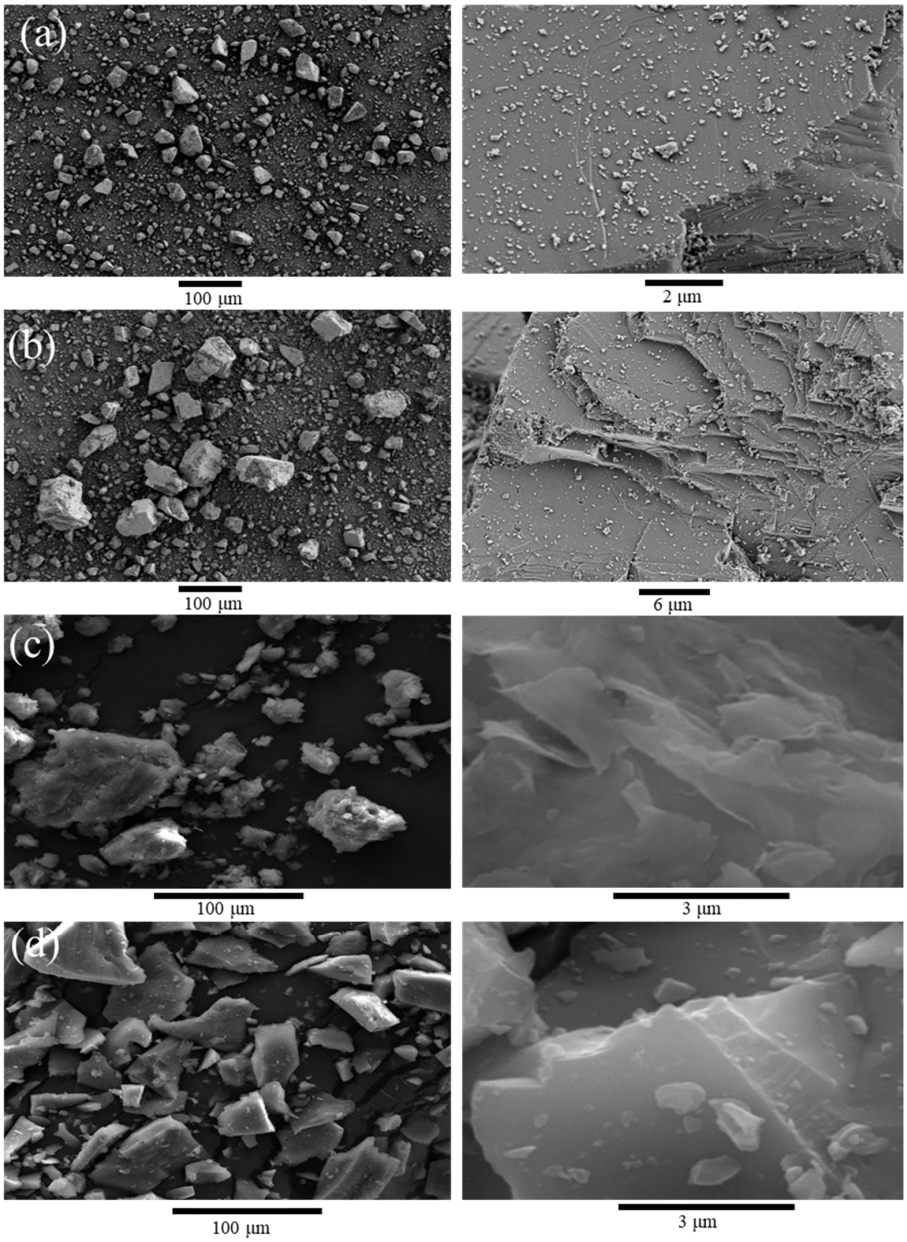

Figure 4. SEM images of (**a**) smithsonite, (**b**) calcite, (**c**) kaolinite, and (**d**) quartz.

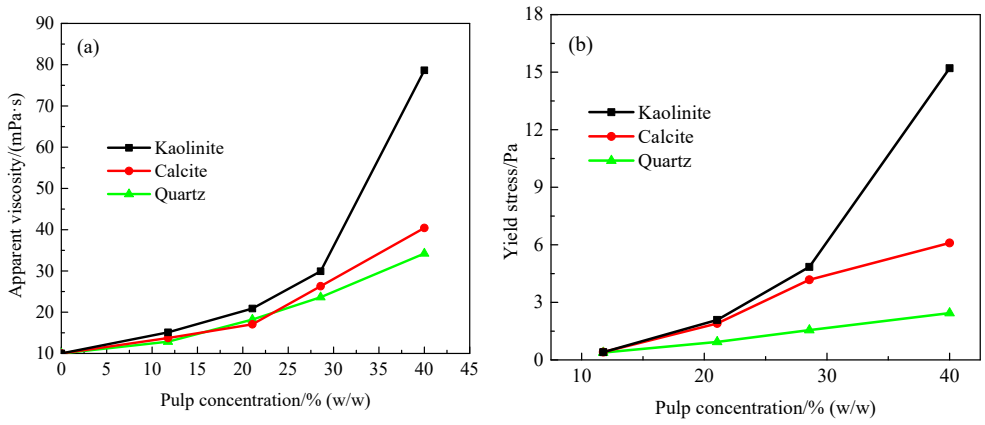

Figure 5. Effect of pulp concentration on (**a**) apparent viscosity and (**b**) yield stress of mixed ore pulp (octadecylamine concentration: 150 mg/L).

3.3. *Effect of Particle Sizes on Apparent Viscosity and Yield Stress of Smithsonite Slurry*

The pulp pH and concentration were 9.5–10.0 and 28.57%, respectively, and the effects of particle size on apparent viscosity and yield stress of mixed minerals of smithsonite-kaolinite, smithsonite-calcite, and smithsonite-quartz are shown in Figure 6, Figure 7, and Figure 8, respectively.

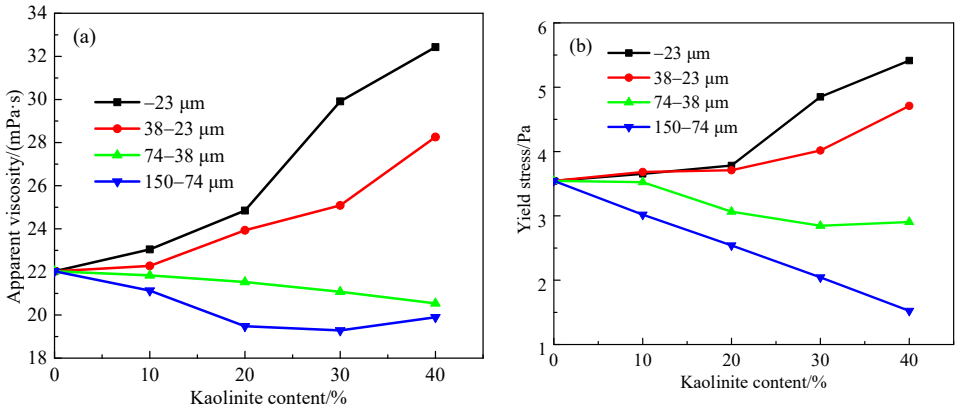

Figure 6. Effects of particle size on pulp's (**a**) apparent viscosity and (**b**) yield stress of the mixed mineral of smithsonite- kaolinite using octadecylamine concentration: 150 mg/L at pH 9.5−10 and 28.57% solid concentration.

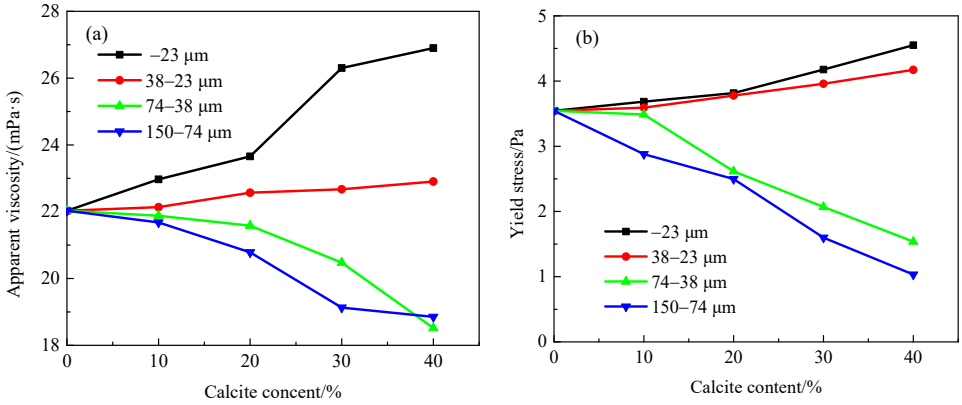

Figure 7. Effects of particle size on pulp's (**a**) apparent viscosity and (**b**) yield stress of the mixed mineral of smithsonite-calcite using octadecylamine concentration: 150 mg/L at pH 9.5–10 and 28.57% solid concentration.

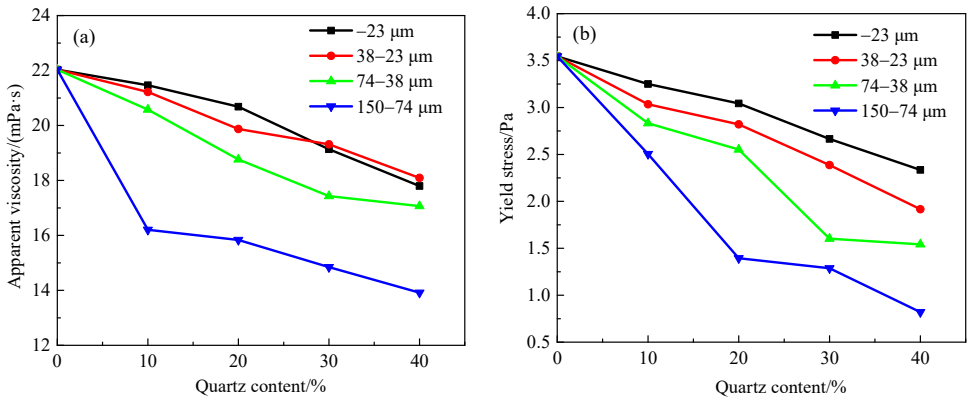

Figure 8. Effects of particle size on pulp's (**a**) apparent viscosity and (**b**) yield stress of the mixed mineral of smithsonite-quartz using octadecylamine concentration: 150 mg/L at pH 9.5–10 and 28.57% solid concentration.

As shown in Figure 6, with the increase of the content of kaolinite in the mixed mineral of smithsonite-kaolinite, the apparent viscosity and yield stress of different particle sizes presented different trends. With the increase of the content of coarse kaolin (150–74 μm, 74–38 μm), the apparent viscosity and yield stress of the pulp slightly decreased. Nevertheless, with the increase of the content of fine kaolin (38–23 μm, −23 μm), the apparent viscosity and yield stress showed an increasing trend. The higher the kaolinite content was, the greater the apparent viscosity and yield stress would appear. Das et al. [29] evaluated the influence of mineralogy and particle size on the viscosity of high concentration pulp and found that the pulp containing fine particles presented a higher viscosity and poor rheology and that mixing montmorillonite with some pure minerals can improve the rheological properties of pulp.

The mixed mineral of smithsonite-calcite showed a similar trend with smithsonite-kaolinite. However, compared with the mixed mineral of smithsonite-kaolinite, the influence of fine fraction on the apparent viscosity and yield stress of smithsonite-calcite pulp was relatively small (see Figure 7). For different particle sizes of quartz, it is noteworthy that the apparent viscosity and yield stress of the smithsonite-quartz decreased with the increase of quartz content in the mixed mineral. In addition, the apparent viscosity and yield stress decreased greatly with the increase of particle size of quartz, especially the 150–74 μm. Thus, the presence of quartz in the pulp could improve the rheological properties.

3.4. Basic Floatability of Minerals

In pulp, the basic behavior of the flotation process is that the mineral particles with hydrophobic surface adhere to the bubbles and then float up. In this research, the floatability of smithsonite, quartz, kaolinite, and calcite with different particle sizes under the action of octadecylamine were investigated through flotation experiments. The pulp pH and concentration were 9.5–10.0 and 28.57%, respectively. The flotation results are shown in Figure 9.

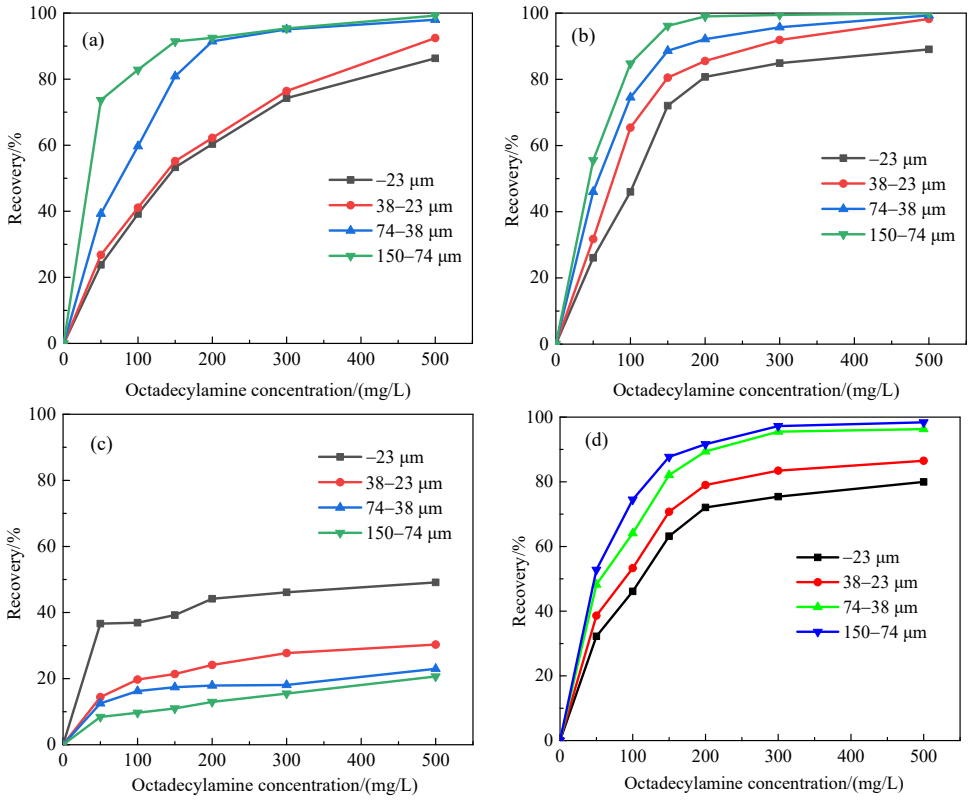

Figure 9. The floatability of (**a**) smithsonite, (**b**) quartz, (**c**) kaolinite, (**d**) calcite using octadecylamine concentration: 150 mg/L at pH 9.5−10 and 28.57% solid concentration.

Figure 9 showed that smithsonite had good floatability under the action of octadecylamine, but its floatability decreased significantly with the decrease of its particle size. Similarly, quartz and calcite with different particle sizes showed a similar change trend with smithsonite. That is, the fine particle size presented a poor floatability. The floatability of quartz is slightly better than that of smithsonite. The floatability of kaolinite is poor. Contrary to smithsonite, calcite, and quartz, the flotation recovery of kaolinite was improved with the decrease of particle size. It was floated up largely through foam entrainment.

The adsorption of octadecylamine on a mineral surface can be explained through the zeta potential. Figure 10 showed the zeta potential of minerals in the presence of 150 mg/L octadecylamine at pH 9.5. The zeta potentials of smithsonite, quartz kaolinite, and calcite are positive, indicating that the cationic collector octadecylamine was adsorbed on the surface of the four minerals. It is noteworthy that the floatability of smithsonite, quartz, kaolinite, and calcite are well consistent with the zeta potential values. Namely, quartz has the good floatability with the highest zeta potential, followed by smithsonite. Kaolinite showed poor floatability with a low zeta potential.

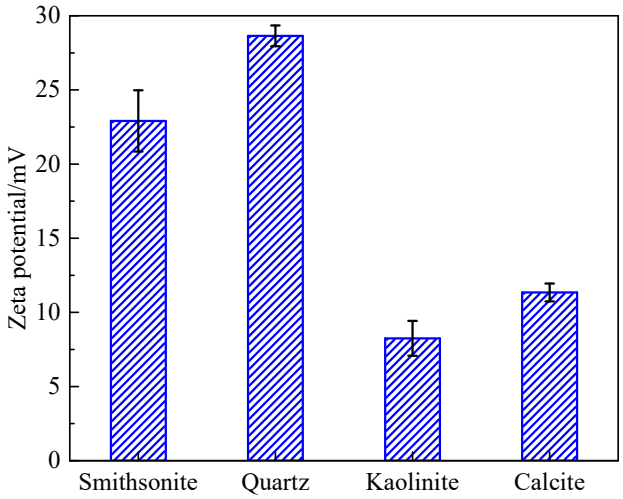

Figure 10. Zeta potential of smithsonite, quartz, kaolinite, and calcite in the presence of 150 mg/L octadecylamine at pH 9.5.

3.5. Flotation Behavior of Gangue Minerals under the Action of Sodium Hexametaphosphate as a Depressant

With octadecylamine as the collector, the dosage was 150 mg/L, and the pH was 9.5–10.0; the flotation behaviors of fine smithsonite, quartz, kaolinite, and calcite with sodium hexametaphosphate as the inhibitor for gangue minerals are shown in Figure 11.

Comparing the trends of the flotation recoveries of four minerals in the presence of sodium hexametaphosphate, it can be seen that with the increase of sodium hexametaphosphate concentration, the flotation of calcite and quartz was significantly inhibited, while the flotation recovery of smithsonite had only a little effect at low concentration (less than 50 mg/L). The recovery decreased significantly with the increase of sodium hexametaphosphate concentration. In the absence of sodium hexametaphosphate, the recoveries of smithsonite, quartz, kaolinite, and calcite were 63.17%, 72.05%, 35.21%, and 53.30%, respectively. When the concentration of sodium hexametaphosphate was 50 mg/L, the recovery of smithsonite decreased slightly to 59.73%, while the recoveries of calcite, kaolin, and quartz were reduced to 38.06%, 14.10%, and 12.01%, respectively. This shows that the addition of sodium hexametaphosphate could cause the floatability difference among smithsonite, quartz, kaolinite, and calcite. Adding sodium hexametaphosphate into the flotation system of smithsonite, the floatability of gangue minerals was reduced. On the other hand, sodium hexametaphosphate as a dispersant could effectively reduce the agglomeration between gangue mineral particles, thus, making the particles more

dispersed and effectively reducing the inclusion of fine minerals during flotation. Relevant research [30,31] showed that the flow characteristics and apparent viscosity of pulp are very sensitive to some flotation agents, and the flotation agents added in the flotation process can change the rheology and flotation performance of pulp. For example, added dispersants (such as sodium silicate, etc.) help to disperse the slurry and reduce the viscosity of the slurry.

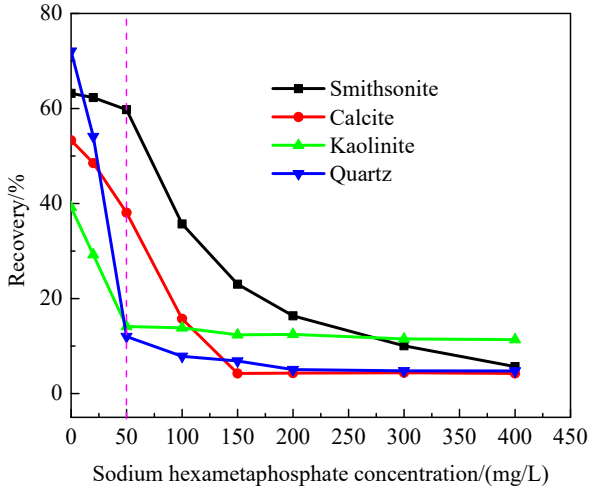

Figure 11. Effect of sodium hexametaphosphate on flotation recoveries of smithsonite and gangue minerals using octadecylamine concentration: 150 mg/L at pH 9.5–10 and 28.57% solid concentration.

In order to explore the influence of gangue content on the flotation behavior of smithsonite, octadecylamine was used as the collector, pH was controlled at 9.5–10.0, the particle size range of the minerals was −23 μm. The test results are shown in Figure 12. Different quartz contents had a slight influence on the yield of smithsonite. Calcite showed increased yield when mixed in over 20% *w*/*w*. However, the presence of kaolinite had an adverse effect on the yield of smithsonite. With the increase of kaolinite content, the yield of smithsonite decreased rapidly.

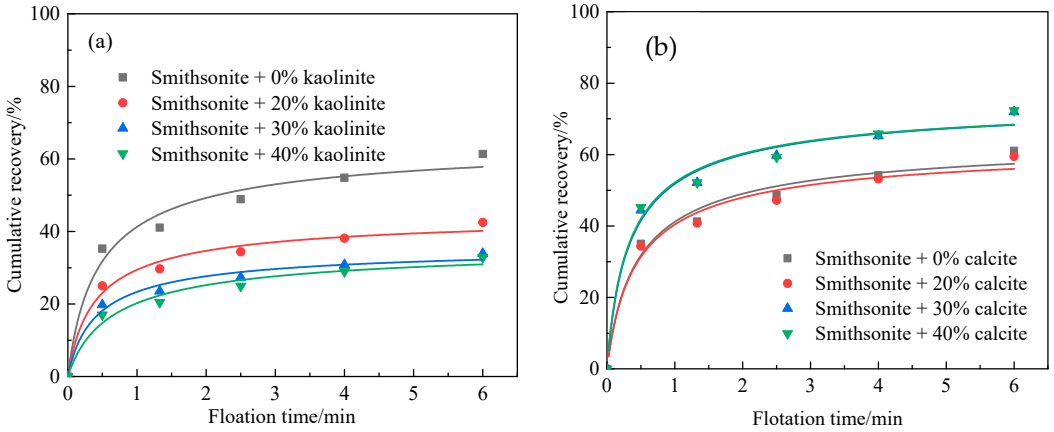

Figure 12. *Cont.*

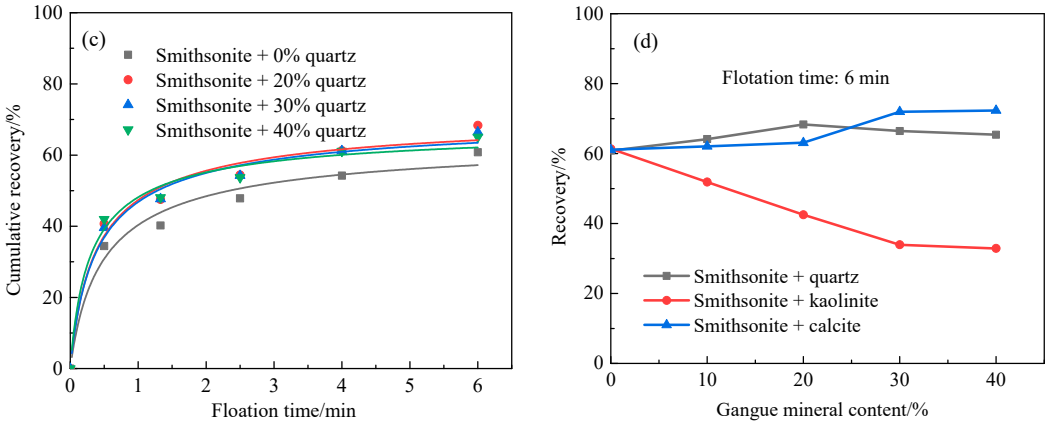

Figure 12. Effects of different gangue contents on flotation behavior of smithsonite with (**a**) kaolinite (**b**) calcite (**c**) quartz (**d**) gangue mineral content using octadecylamine concentration: 150 mg/L at pH 9.5–10 and 28.57% solid concentration. In order to better understand the mechanism of different gangue minerals affecting the flotation of smithsonite, SEM-EDS was used to analyze the flotation concentrates of the mixed minerals. The results are shown in Figures 13–15. It can be seen that kaolinite, as a clay mineral that is relatively loose in shape, showed an adherent state on the surface of smithsonite, producing an obvious aggregation. Therefore, it can be concluded that kaolinite has a great impact on the pulp environment. Quartz and smithsonite, calcite, and smithsonite existed in a relatively dispersed form and did not produce obvious aggregation. Therefore, it can be concluded that quartz and calcite have little impact on the pulp environment.

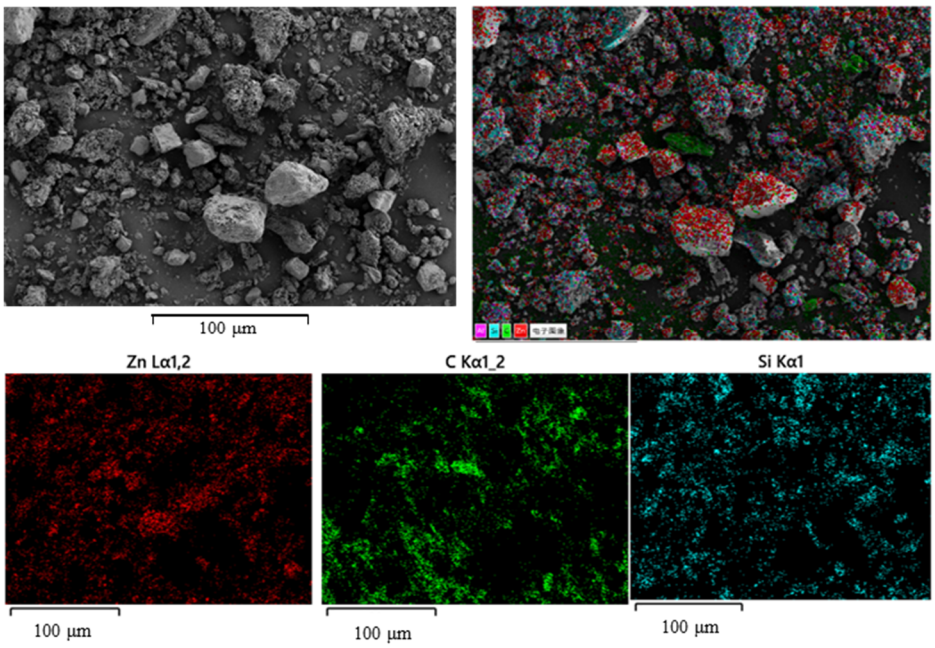

Figure 13. SEM-EDS pictures of flotation concentrate of the mixed mineral of smithsonite-kaolinite.

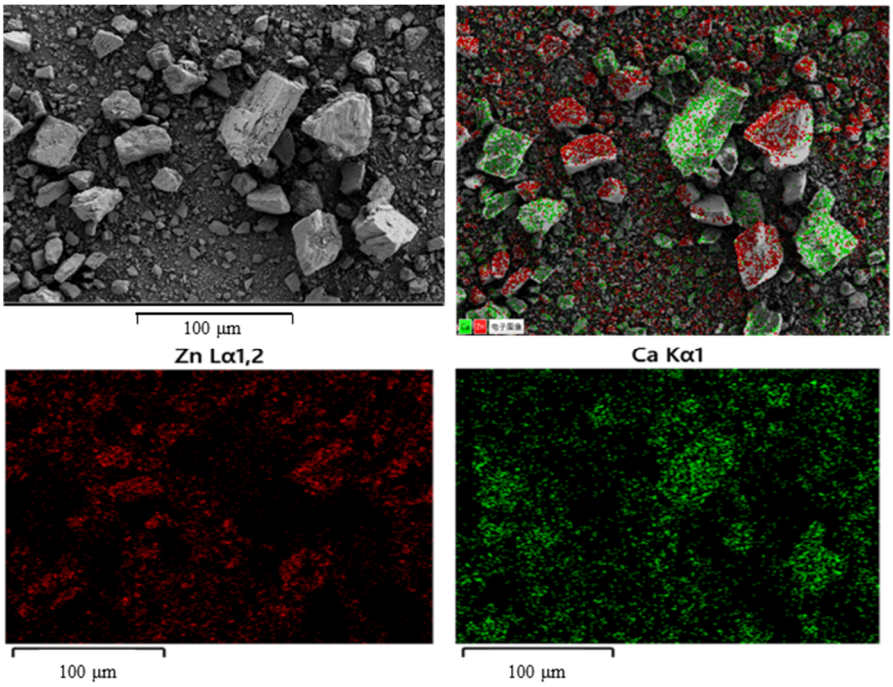

Figure 14. SEM-EDS pictures of flotation concentrate of the mixed mineral of smithsonite-calcite.

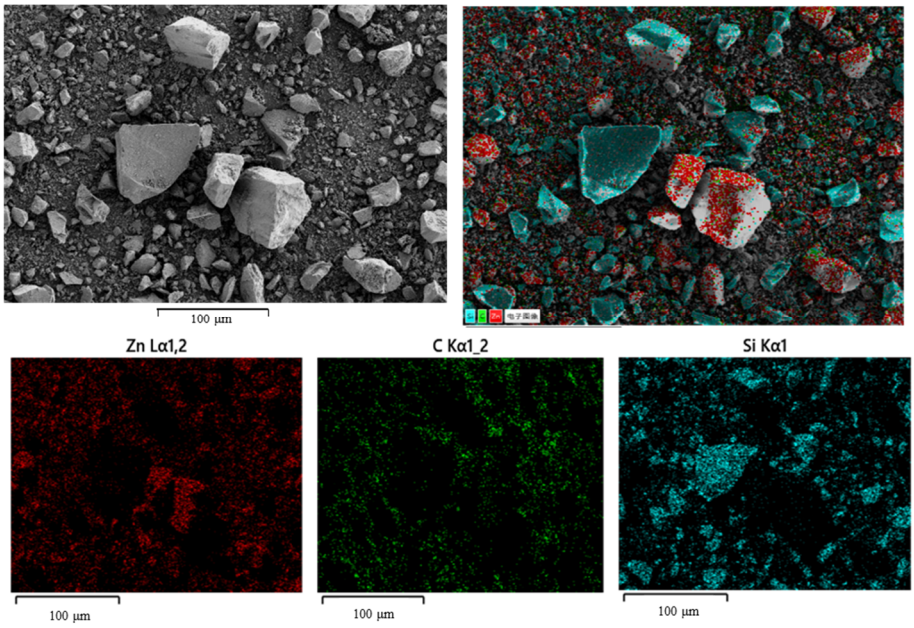

Figure 15. SEM-EDS pictures of flotation concentrate of the mixed mineral of smithsonite-quartz.

4. Conclusions

There is a close relationship between pulp rheology and flotation recovery. The adverse influence of gangue minerals on the pulp rheology and the flotation of smithsonite was kaolinite > calcite > quartz. Especially, the loose and irregular layered silicate structure of kaolinite could significantly increase the apparent viscosity and yield stress of smithsonite, resulting in a low yield of smithsonite. Meanwhile, the zeta potential measurement further demonstrated that a small amount of octadecylaminec was adsorbed on kaolinite. The coarse kaolinite and calcite (150–74 μm, 74–38 μm) could reduce the apparent viscosity and yield stress and present good floatability. However, the fine kaolinite and calcite (38–23 μm, −23 μm) could increase the apparent viscosity and yield stress and show poor floatability.

Different particle sizes of quartz could greatly decrease the apparent viscosity and yield stress due to the electrostatic repulsion with highest zeta potential. In the flotation of smithsonite, the fine gangue particles (−38 μm) need to be removed in advance so as to control the pulp rheology.

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