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Effects of physical and physico-chemical factors on pulp rheology of smithsonite

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Abstract: Pulp rheology is an important factor affecting flotation. The effects of particle size (150-74 μm, 74-38 μm, 38-23 μm, -23 μm), pulp density (11.76%-34.78%), pH (5.3-12.4), collector concentration (25-500 mg/dm3), and stirring intensity (400-900 rpm) on the rheology of smithsonite, kaolinite, quartz, and calcite minerals were investigated in detail. Additionally, the agglomerate morphology of particles was observed by a polarizing microscope. The results showed that as the mineral particle size decreased and pulp density increased, the apparent viscosity and yield stress of the pulp increased. Especially the fine mineral particles (-23 μm) presented a higher apparent viscosity and yield stress. The order of apparent viscosity and yield stress for the minerals from large to small was: kaolinite>calcite>smithsonite>quartz under different pH values, the collector concentrations, and stirring intensities. In the presence of collector of octadecylamine, smithsonite, kaolinite, and calcite particles could form aggregates, especially smithsonite particles presented obvious agglomeration with large particle size and compact network structure. The agglomeration effect of calcite and kaolinite particles were weaker than that of smithsonite. The particle agglomeration resulted in the increase of the apparent viscosity and yield stress of the pulp. Quartz particles did not form clusters, hence the pulp's apparent viscosity and yield stress were the lowest. The research on the changes in rheological properties of the pulp will hopefully provide some guidance for future flotation.

Keywords: smithsonite, pulp rheology, apparent viscosity, yield stress, particle agglomerate

1. Introduction

Rheology is a science that studies the flow and deformation of materials. It is used to characterize the law between shear stress and strain produced by fluid in the process of flow. Rheology reveals the interaction between particles in the fluid and the internal microstructure of the fluid by studying the relationship among stress, deformation, deformation rate, and viscosity of fluid (Barnes et al., 1989). Pulp rheology plays an important role in tailings transportation, grinding efficiency, filtration, and reagent dispersion of in flotation pulp (Boger, 2000; Shi and Napier-Munn, 2002).In the flotation process, the flotation dynamic process of mineral particles can be divided into four stages: collision, adhesion, rise and floating of particles and bubbles (Zhou et al., 2021). When the apparent viscosity and yield stress of the flotation pulp increase, the turbulence intensity of the pulp will be significantly reduced, and the movement resistance of bubbles and mineral particles will increase. It is difficult to achieve the uniform distribution of pulp density by a mechanical stirring of general strength, which will lead to the decrease of the dispersion of mineral particles and bubbles in the flotation system. There appears little chance of collision and adhesion in the particles and bubbles so that the bubbles are difficult to mineralize, and the bubbles loaded with minerals are also difficult to float. This indicates that pulp rheology has a significant impact on flotation dynamics.

Pulp rheology (yield stress and apparent viscosity) is a quantitative index to characterize the particle aggregation or dispersion degree, which is an important factor affecting mineral flotation recovery (Farrokhpay et al., 2005; Richmond et al., 1998; Wang and Li, 2020). Generally, the pulp of high-grade ore is Newtonian fluid or similar Newtonian fluid with low viscosity, but the pulp of low-grade ore is

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typical non-Newtonian fluid, showing pseudoplastic fluid or Bingham fluid behavior with high pulp viscosity, which will deteriorate the flotation index (Farrokhpay, 2012). The change of pulp rheology has a direct impact on the flotation processes, such as the collision and aggregation between particles. By studying the flow and deformation properties of flotation pulp under the action of external shear stress and various factors, it will provide some guidance for the flotation separation of minerals. The study of pulp rheology mainly involved the sulfide ore (Muster and Prestidge, 1995; Prestidge, 1997), oxidized ore (Zhou et al., 2001), clay mineral (Papo et al., 2002), coal slime (Boylu et al., 2004).

During the flotation, many factors mainly including mineral types, pulp density, particle size, temperature, mixing strength, pulp pH, flotation reagents, etc can affect the pulp rheology, (He et al., 2006). Das et al. (2011) 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 mixing montmorillonite with some pure minerals can improve the rheological properties of pulp. Wang et al. (2020) 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. Pyrophyllite little effect on the rheology, but it could also reduce the recovery of pyrite due to the formation of heterogeneous flocculation between pyrophyllite and pyrite. Hu et al. (2020) studied the effect of serpentine on chalcopyrite pulp rheology and flotation. The results showed that increasing the serpentine concentration can increase the apparent viscosity of mixed ore pulp to increase the entrainment of serpentine and the MgO content in the concentrate.

Zinc ores can be divided into zinc sulfide ore and zinc oxide ore according to the oxidation degree of zinc minerals. The mineral composition of zinc oxide ore is complex. The main valuable minerals are smithsonite and heteropolar ore. The common gangue minerals include calcite, dolomite, quartz, clay minerals, and limonite (Duarte and Lima, 2022). Zinc oxide ore mainly adopts the flotation process (Zhao, 2007). However, because zinc oxide ore is highly hydrophilic, and the surface properties of silicate or carbonate gangue coexisting in the flotation system have little difference, resulting in poor selectivity of flotation agents (Zhao et al., 2021). In particular, the serious sliming of zinc oxide contains a large number of primary slime and secondary slime, this results in high pulp viscosity and difficult flotation separation.

In this study, the influence of mineral types, particle size, pulp density, pulp pH, collector concentration, and stirring intensity on the rheological properties of smithsonite, kaolinite, quartz, and calcite minerals were investigated. The relationship between the particle aggregation/dispersion and pulp rheology was studied by agglomeration test.

2. Materials and methods

2.1. Materials

The smithsonite sample was collected from Liaoning Province, the calcite sample was obtained from Beijing, and the kaolinite and quartz samples were taken from Hebei Province, China. The purity of smithsonite, kaolinite, calcite and quartz were analyzed by X-ray diffraction (as shown in Fig. 1), and the values were all above 95%. The zirconia ceramic ball mill with rubber lining was used for the grinding of the samples. The grinding product was dry sieved into different particle sizes (150-74 μm, 74-38 μm, 38-23μm, -23 μm). Analytical pure hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used as the pH regulators for the pulp, which were purchased from Chongqing Chuandong Chemical (Group) Co., Ltd., China, and chemical pure octadecylamine was used as a collector, which was provided by Tianjin Kemiou Chemical Reagent Co., Ltd., China. The experimental water is deionized water(AAKHL-III, Chengdu Aike Water Treatment Equipment Co., Ltd., China), the resistivity was 18.25 MΩ·cm.

2.2. Methods

2.2.1. Pulp rheology measurements

The MCR72 modified rotary rheometer (Anton Paar, Austria) was used to measure the rheological properties of the pulp. Because the flotation pulp is an unstable solid-liquid-gas three-phase system that is easy to settle, the rheological properties are very complex and difficult to detect, it is necessary to improve the rheometer partially. In order to weaken the influence of particle sedimentation in pulp on the measurement results, the traditional cylindrical inner cup was replaced by a paddle-type stirring rotor. Rheological measurements mainly include the shear rate vs shear stress test (controlled shear rate mode) for measuring the apparent viscosity of the pulp and the shear stress vs shear deformation test (controlled shear stress mode) for the yield stress of the pulp. The test sample came from the prepared pulp after mixing. The experiment equipment is shown in Fig. 2.

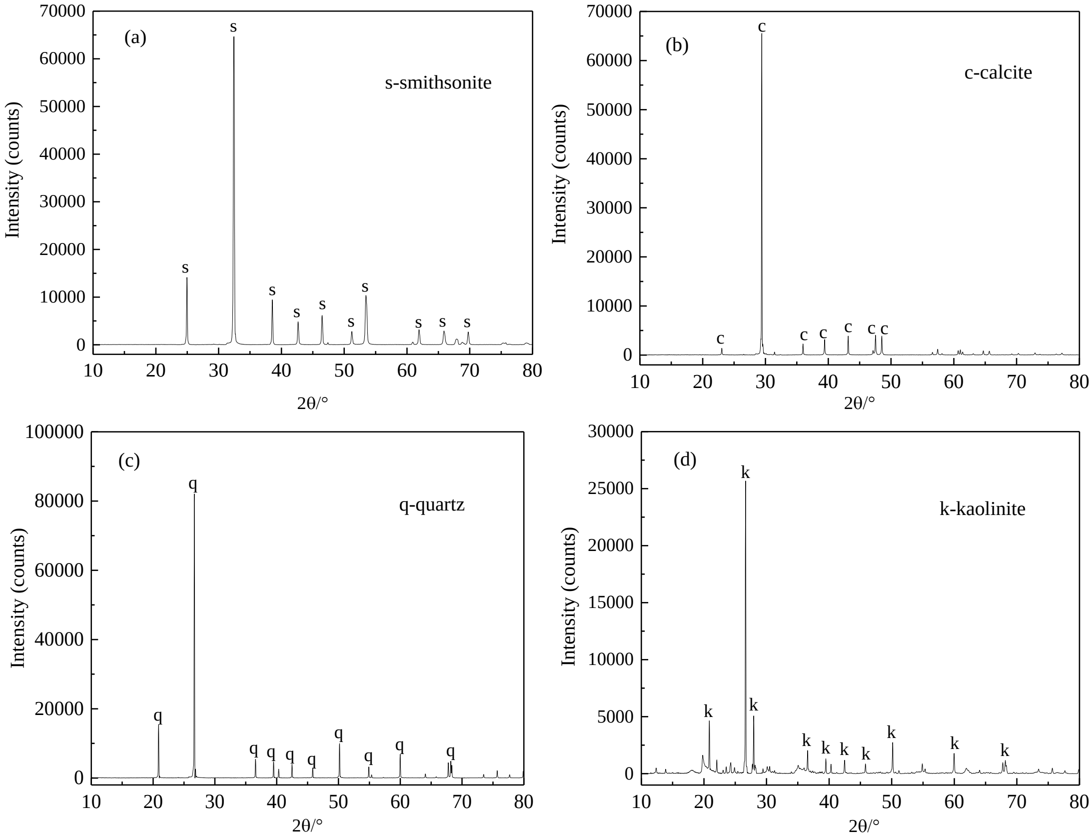

Fig. 1. XRD patterns of smithsonite (a), calcite (b), quartz (c) and kaolinite (d)

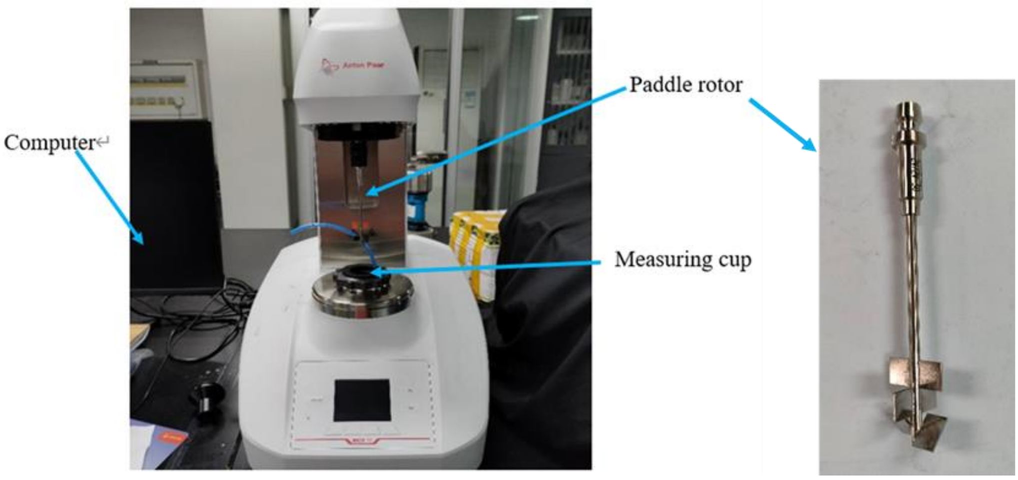

Fig. 2. MCR72 modified rotary rheometer

2.2.2. Particle agglomeration

The agglomeration of fine minerals with different octadecylamine concentrations carried out in a 250 cm3 beaker. The minerals were prepared into a pulp with a mass concentration of 12.5 g/dm^3. The pulp was dispersed by ultrasonic wave for 2 min, and then different concentrations of octadecylamine were added into the slurry. Next, the pH of the slurry was adjusted to 9.5-10.0, and the slurry was stirred for 5 min with a four-blade straight paddle top mixer with a diameter of 40 mm. After the agglomeration test, the particle size distribution of aggregates was determined by a laser particle size analyzer (LS13320, Beckman Coulter, USA). Meanwhile, a small amount of pulp was transferred to the glass slide to observe the morphology of the aggregate by the polarizing microscope (CX21-P, Olympus, Japan).

3. Results and discussions

3.1. Effects of mineral types, pulp density, and particle size on rheological properties

The different pulp density was prepared from smithsonite, kaolinite, calcite, and quartz suspensions were prepared at different particle sizes (150-74 μm, 74-38 μm, 38-23μm, -23 μm). Comparison was made on the variation trend of the pulp apparent viscosity and yield stress of smithsonite, kaolinite, calcite, and quartz pulp density and particle size in the absence of collector, and the results are shown in Fig. 3 to Fig. 6, respectively.

As can be seen from Fig. 3 to Fig. 5, when the pulp density was low, the apparent viscosity and yield stress were low. With the increase of the pulp density of smithsonite, kaolinite, and calcite, the apparent viscosity and yield stress of the pulp increased rapidly. Relevant research showed that a low concentration pulp displays a Newtonian behaviour, as the amount of solid in pulp increases, the rheological behaviour 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 (Becker et al., 2013; Farrokhpay et al., 2010; He et al., 2004). On the other hand, with the decrease of the particle size of

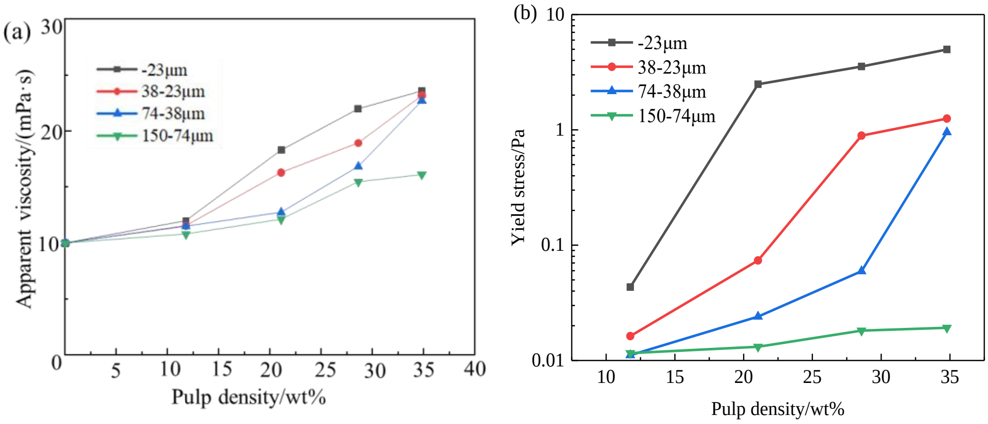

Fig. 3. Relationship between apparent viscosity (a), yield stress (b) and the pulp density of smithsonite with different particle sizes (natural pH 8.10)

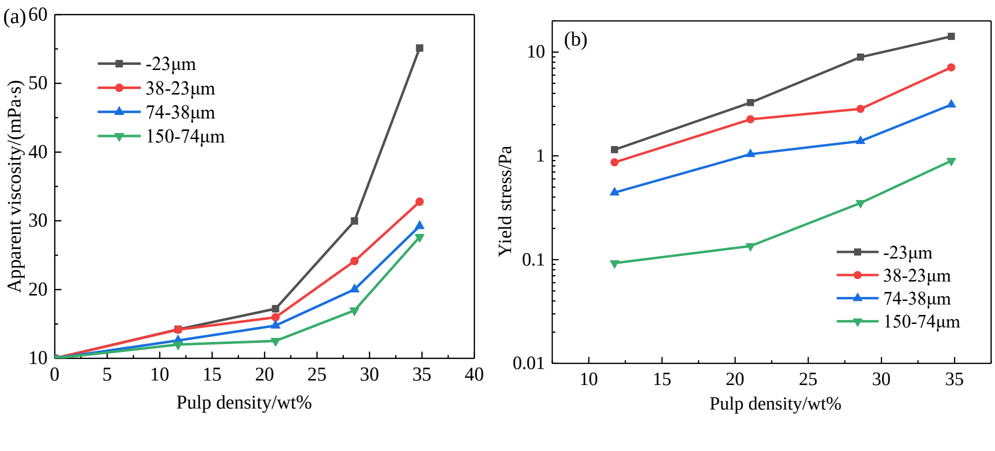

Fig. 4. Relationship between apparent viscosity (a), yield stress (b) and the pulp concentration of kaolinite with different particle sizes (natural pH 8.66)

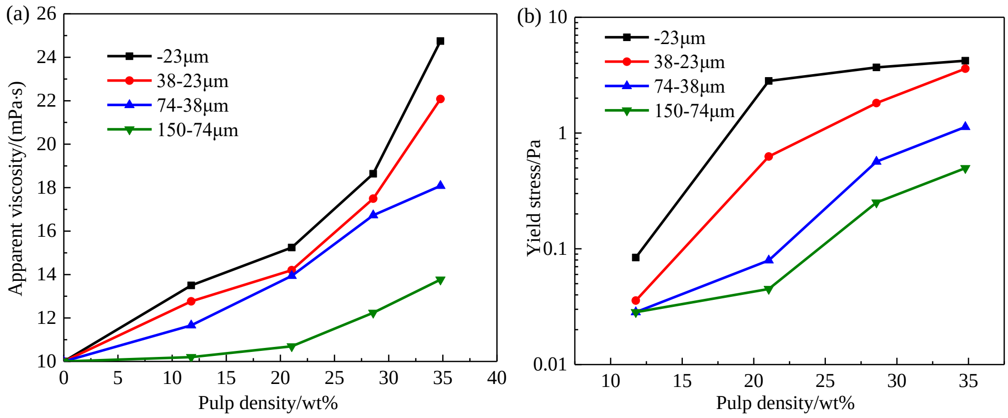

Fig. 5. Relationship between apparent viscosity (a), yield stress (b) and the pulp density of calcite with different particle sizes (natural pH 9.27)

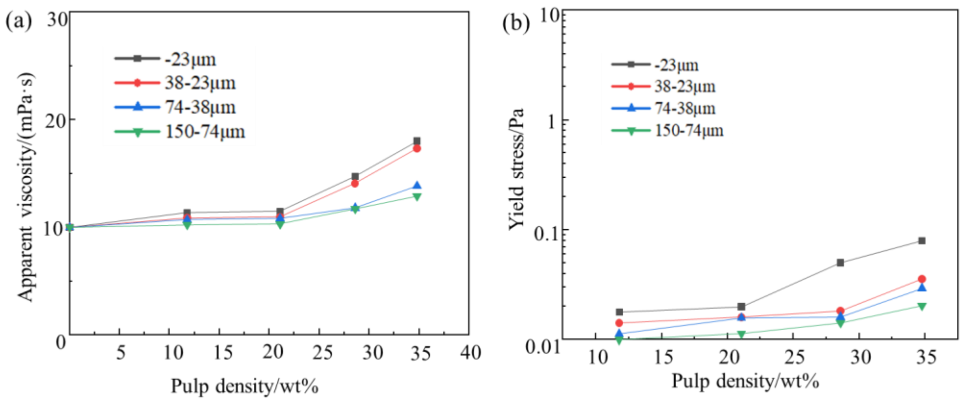

Fig. 6. Relationship between apparent viscosity (a), yield stress (b) and the pulp density of quartz with different particle sizes (natural pH 6.72)

smithsonite, kaolinite, and calcite, the apparent viscosity and yield stress of the pulp increased. When the pulp density was 28%, the pulp apparent viscosity and yield stress of the coarse-grained smithsonite (-150+74 μ m) were 15.47 mPa·s and 0.018 Pa, which was close to the rheological properties of pure water (the apparent viscosity is mPa·s, and the yield stress is 0). For fine-grained smithsonite (-23 μm), the apparent viscosity and yield stress of the pulp increased to 22.02 mPa·s and 3.54 Pa, respectively, at the same concentration. The reason is that when the particle size in the pulp finer and the content of fine particles increased, the internal friction of the pulp in the shear field increased, the interaction between particles strengthened, and so did the structure. This would result in the increase of the apparent viscosity of the pulp during flotation operation. The fine smithsonite in the pulp would affect the effective dispersion of mineral particles. The increase of the apparent viscosity of the pulp would lead to the decrease of the selectivity of flotation agents, and thus resulting in the inclusion or entrainment in flotation, and the concentrate grade would be affected.

The apparent viscosity and yield stress of quartz pulp changed little at low pulp density is shown in Fig. 6. At high pulp density, the apparent viscosity and yield stress increased slightly. But with the decrease of quartz particle size in the pulp, the apparent viscosity and yield stress of the pulp did not change significantly. Compared with kaolinite, smithsonite, and calcite, the overall apparent viscosity and yield stress of quartz pulp lower, the quartz particles showed a good dispersion state.

For the same particle size, the apparent viscosity and yield stress of the pulp presented the same order: kaolinite>calcite>smithsonite>quartz. Zhang et al. (2015) also found that the poorly crystallized kaolinite increased the pulp viscosity. In all particle sizes, the coarse particle size generally showed very low apparent viscosity and yield stress, but the fine-grained (-23 μm) showed high apparent viscosity and yield stress. The presence of yield stress indicates that complex structures are formed in the pulp

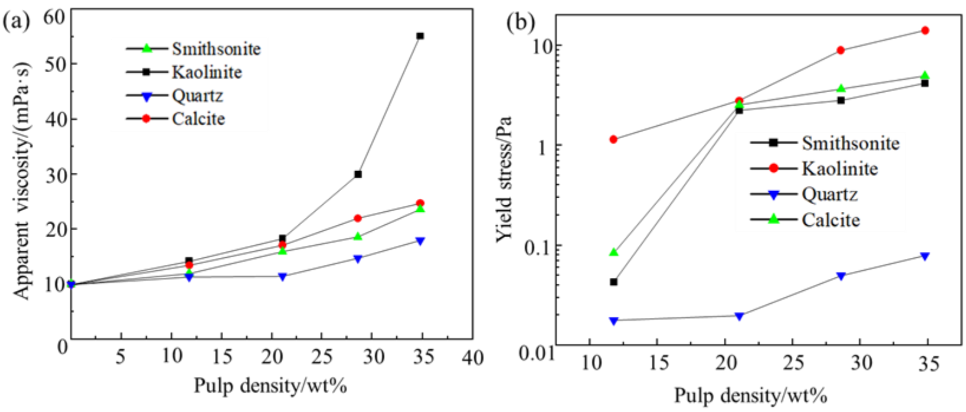

Fig. 7. Relationship between apparent viscosity (a), yield stress (b) and the pulp density of fine mineral -23μm (natural pH 6.72)

(Zhang and Peng, 2015). Therefore, the investigation of the rheology of the four fine particles (-23μm) will help us better understand the pulp structure formed between particles and the selective aggregation and dispersion of minerals in fine flotation pulp.

The relationship between apparent viscosity, yield stress and the pulp density of fine-grained (-23 μm) smithsonite, kaolinite, calcite, and quartz suspensions are shown in Fig. 6, a low pulp density showed a low apparent viscosity and yield stress. But with the increase pulp density, the apparent viscosity and yield stress of fine kaolinite increased sharply. Cruz et al. (2013) studied the rheology of kaolinite pulp and found that kaolinite concentration affected the rheogram and apparent viscosity of pulp. The particle interactions of clay minerals are the driving forces for changes in pulp rheology. The apparent viscosity and yield stress of calcite and smithsonite presented a similar change trend, both increased with the increase of pulp concentration, and show a higher change degree than that of quartz, but lower than that of kaolinite. The following shows the order of apparent viscosity and yield stress of the four kinds of single mineral pulp from large to small, kaolinite > calcite > smithsonite > quartz. Kaolinite is a typical aluminosilicate clay mineral with a 1:1 alumina to silica layered structure, which will expand after encountering water, and the viscosity will increase. Therefore, kaolinite showed a high apparent viscosity and yield stress. Quartz is a typical hydrophilic mineral, which was well dispersed in solution, so it shows a low apparent viscosity and yield stress. Ndlovu et al, (2014) also found that the rheological behaviour of pulp is closely associated with mineral type, and phyllosilicate minerals, particularly swelling clays minerals, can result in higher viscosities compared with nonphyllosilicate minerals such as quartz. Both smithsonite and calcite are carbonate minerals with similar surface properties, little difference in rheological properties, this leaded to little difference in the pulp rheology.

3.2. Effect of pulp pH on rheological properties

The valuable mineral smithsonite and gangue minerals calcite, kaolinite, and quartz have different surface charges due to the dissolution, adsorption, and ionization in aqueous solutions under different pH values. This will lead to different structures of mineral pulp. Using HCl and NaOH as the pH regulators, the influence of pH on the apparent viscosity and yield stress of fine-grained smithsonite, calcite, kaolinite, and quartz is shown in Fig. 8.

It can be seen from Fig. 8 that the apparent viscosity and yield stress of quartz pulp were the lowest and the dispersion was the best under different pH values. The apparent viscosity and yield stress of kaolinite pulp were the largest, and the values decreased with the increase of pH, especially the yield stress. The correlation between the yield stress and the charges in the clay particles can be explained by the dependence of the basal surface charges on pH (Gupta and Miller, 2010). It is reported that a silica face-alumina face interaction is the main mode of aggregation at low pH, and this type of association increases the staking of kaolinite layers promoting the edge–face and face–face associations with increasing pH. A further increase in pH decreases face–face and edge–face associations. When pH is high, all the surfaces of the kaolinite particles become negative and therefore dispersed in the suspension, resulting in a reduction in yield stress (Gupta et al., 2011). The yield stress of smithsonite and calcite pulp was similar, and the overall change was small. But the apparent viscosity of smithsonite decreased slightly under strong alkaline conditions, while the apparent viscosity of calcite increased. The apparent viscosity and yield stress of smithsonite pulp were both lower in the pH range of 9.5-10.0.

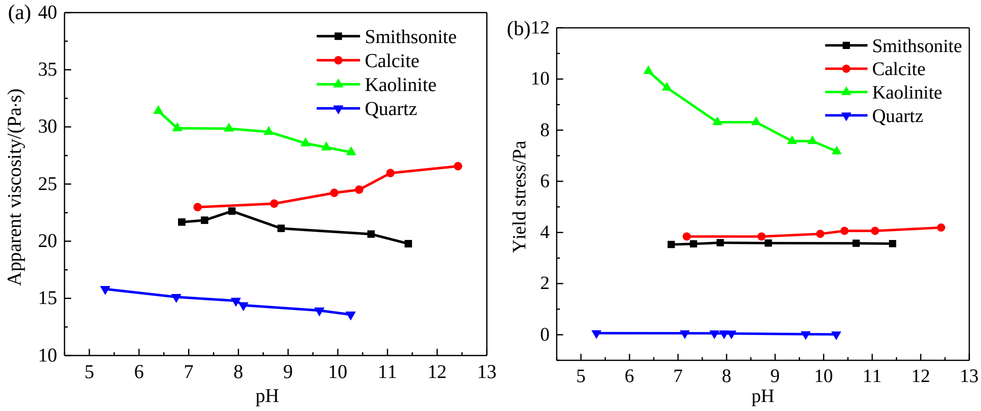

Fig. 8. Effect of pH on the apparent viscosity and yield stress of fine-grained smithsonite, calcite, kaolinite, and quartz

3.3. Effect of collector octadecylamine concentration on pulp rheological properties

The apparent viscosity and yield stress of the collector octadecylamine solution at different concentrations are shown in Fig. 9. The results showed that the apparent viscosity of octadecylamine solution at different concentrations didn't change, and the yield stress was 0. In general, the rheological properties of octadecylamine solution were close to that of water.

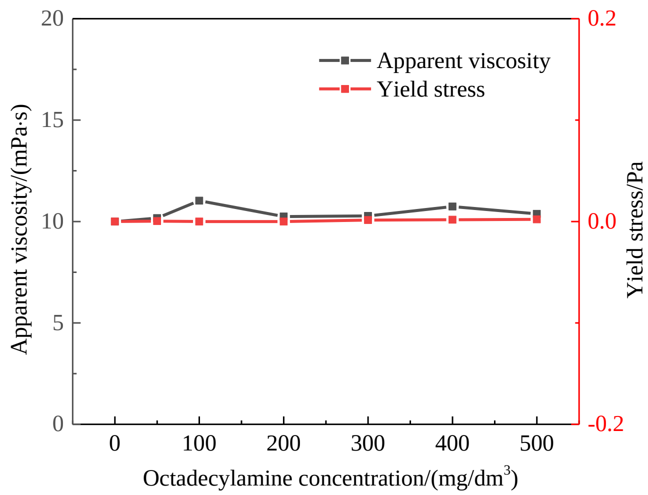

Fig. 9. Apparent viscosity and yield stress of octadecylamine solution at different concentrations (pH 9.5-10.0)

The apparent viscosity and yield stress of the pulp under different octadecylamine concentrations were measured, and the results are shown in Fig. 10. With the increase of octadecylamine concentration, the interaction between mineral particles was enhanced, which resulted in the increase of the apparent viscosity and yield stress of the pulp. However, the apparent viscosity of fine smithsonite, kaolinite, quartz, and calcite pulps presented different change trends. With the increase of octadecylamine concentration, while the apparent viscosity of fine kaolinite pulp presented an obvious increase, that of fine smithsonite, calcite, and quartz pulp showed a mild increase. In the absence of octadecylamine, the apparent viscosities of fine smithsonite, kaolinite, quartz, and calcite pulps were 22.02 mPa·s, 29.97 mPa·s, 14.76 mPa·s, and 26.19 mPa·s, respectively. When the concentration of octadecylamine was 150 mg/dm^3, the apparent viscosities of fine smithsonite, kaolinite, quartz and calcite pulps were 26.23 mPa·s, 46.77 mPa·s, 19.28 mPa·s, and 28.81 mPa·s, respectively.

Similarly, with the increase of octadecylamine concentration, while the yield stress of fine kaolinite pulp increased significantly, the yield stress of fine smithsonite and calcite pulp showed a mild increase. The yield stress of fine smithsonite, kaolinite, quartz, and calcite in the absence of octadecylamine were 3.54 Pa, 8.95 Pa, 0.05 Pa, and 3.21 Pa, respectively. When the concentration of octadecylamine was 150 mg/dm^3, the pulp yield stress of fine smithsonite, kaolinite, quartz, and calcite increased to 5.52 Pa, 13.68 Pa, 2.15 Pa and, 5.76 Pa, respectively. The increased pulp yield stress presented the strength change of the network pulp structure formed by hydrophobic association of minerals after the action of collectors.

The effect of octadecylamine concentrations on the agglomeration of smithsonite, kaolinite, quartz, and calcite particles are shown in Fig. 11. In the absence of octadecylamine, the four mineral particles were uniformly dispersed. In the presence of octadecylamine in the pulp, octadecylamine was adsorbed on the surface of mineral particles to form clusters through hydrophobic association, enhancing the attraction between particles so that the pulp showed a large apparent viscosity and yield stress. With the increase of octadecylamine concentration, the agglomeration of smithsonite, kaolinite, and calcite

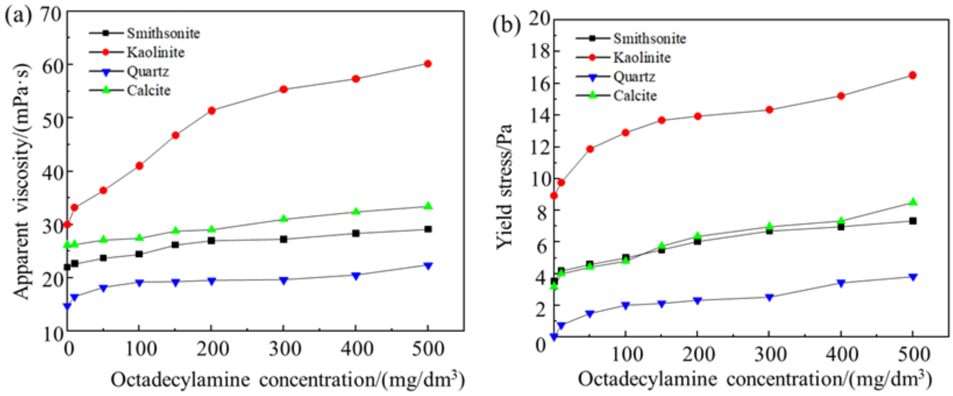

Fig. 10. Effect of octadecylamine concentration on pulp apparent viscosity (a) and yield stress (b) of fine mineral (pulp concentration: 28.57wt%, pH 9.5-10.0)

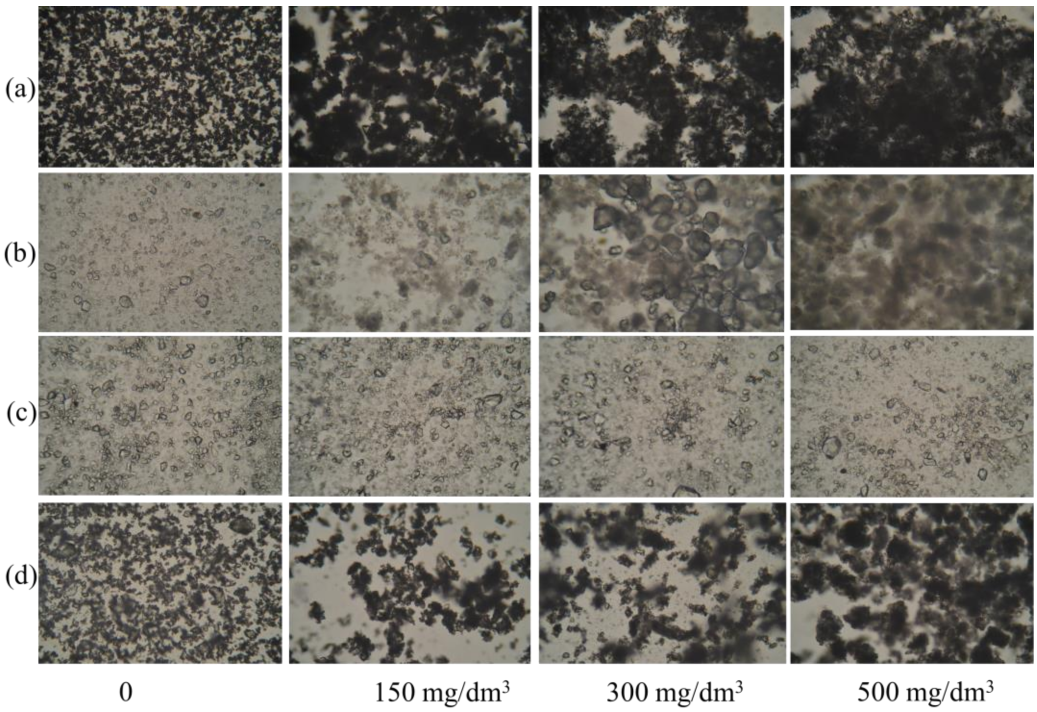

Fig. 11. Effect of octadecylamine concentration on agglomeration of smithsonite (a), kaolinite (b), quartz (c), and calcite (d) particles

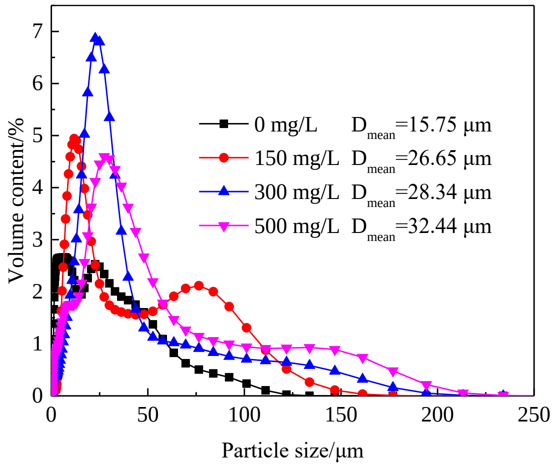

Fig. 12. Particle size distribution of smithsonite aggregates under different octadecylamine concentrations

particles gradually increased, thus resulting in the increase of pulp apparent viscosity and yield stress. Among them, smithsonite presented the strongest agglomeration effect. A large number of closely structured aggregates could be observed. The average particle size (D_{mean}) of smithsonite was 15.75 μm in the absence of octadecylamine. When the concentration of octadecylamine was 150 mg/dm^3 and 500 mg/dm^3, the D_{mean} of the aggregates increased to 26.65 μm and 32.44 μm, respectively (Fig. 12). The agglomeration of calcite and kaolinite was weaker than that of smithsonite. However, kaolinite is a typical clay mineral with large surface area and surface energy, which usually shows high viscosity. Therefore, although the agglomeration effect of smithsonite is better than that of kaolinite, the apparent viscosity and yield stress of kaolinite are higher than that of smithsonite. Both smithsonite and calcite are carbonate minerals, showing a similar agglomeration phenomenon. Under different octadecylamine concentrations, quartz particles presented a good dispersion state and didn't form clusters, hence the pulp apparent viscosity and yield stress were the lowest.

3.4. Effect of stirring intensity on pulp rheological properties

When the concentration of octadecylamine was 150 mg/dm^3, the influence of stirring intensity on the rheological properties of the pulp is shown in Fig. 13. With the increase of stirring intensity, the pulp apparent viscosity and yield stress of fine smithsonite and calcite first decreased and then increased, while the pulp apparent viscosity and yield stress of kaolinite gradually decreased, showing a shear thinning behavior. The pulp had initial yield stress at a low shear rate, and later presented a shear-thinning type behaviour at higher shear rates. This can be interpreted by overcoming the force that holds the layers together to initiate flow. Under shearing, the large aggregates of clay minerals were separated into smaller units. As the shear rate increased, the further breakdown of the aggregates resulted in shear thinning (Zhang and Peng, 2015).

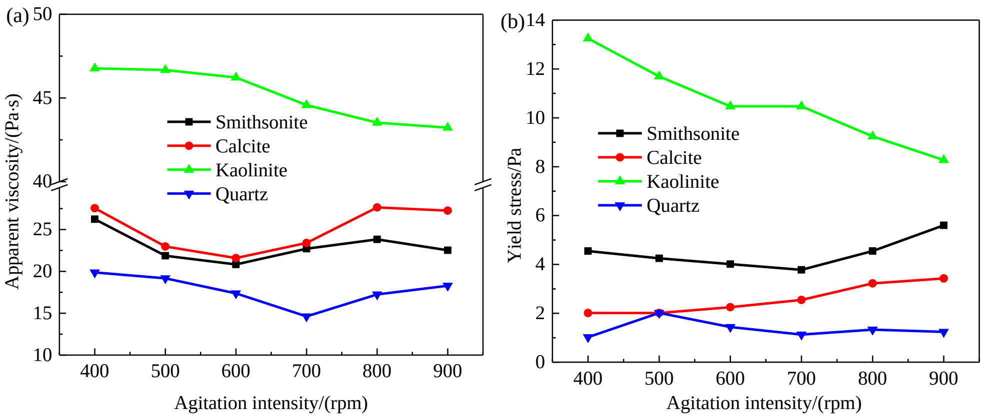

Fig. 13. Effect of stirring intensity on apparent viscosity and yield stress of fine mineral pulp (pulp density: 28.57wt%, pH 9.5-10.0)

The influence of stirring intensity on the aggregate morphology of fine smithsonite, kaolinite, quartz, and calcite is shown in Fig. 14. Under the induction of octadecylamine, smithsonite particles presented obvious agglomeration with large particle size and compact network structure. However, the agglomerate particle size was relatively small at 600 rpm, resulting in the reduction of its apparent viscosity. The agglomeration effect of kaolinite particles under the action of octadecylamine was weak. The dispersion was improved with the increase of stirring strength, resulting in the decrease of apparent viscosity and yield stress. Quartz particles were dispersed under different stirring intensities, and the apparent viscosity and yield stresses were the lowest. Under different stirring intensities, the calcite particles had certain agglomeration, but the effect was weaker than that of smithsonite. Therefore, the particle agglomeration phenomenon is consistent with the results of pulp rheology measurement.

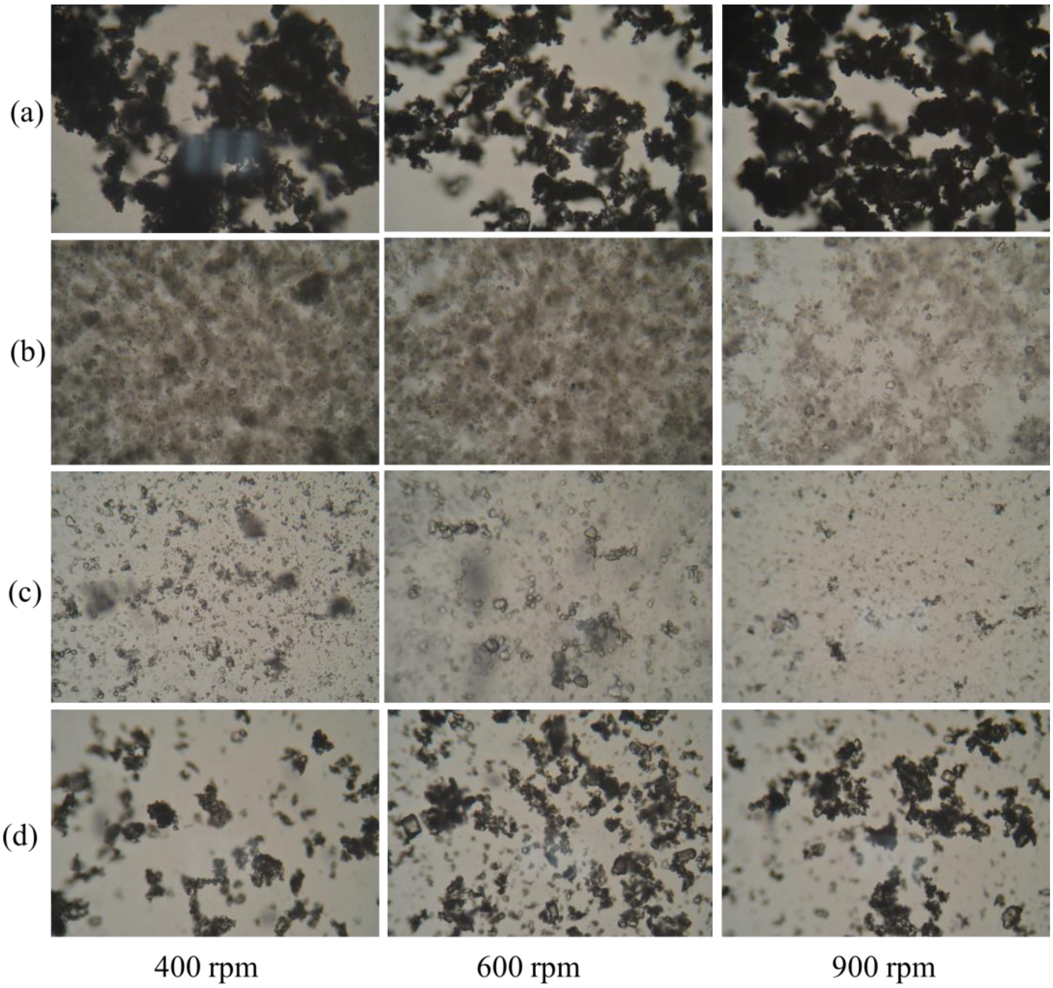

Fig. 14. Aggregates morphology of smithsonite (a), kaolinite (b), quartz (c) and calcite (d) under different stirring intensities (octadecylamine concentration: 150 mg/dm^3)

4. Conclusions

The effects of particle size, pulp density, pH value, collector concentration, and stirring strength on the pulp apparent viscosity and yield stress of smithsonite, kaolinite, quartz, and calcite were studied by the rheology measurements. The particle agglomerate morphology under different collector concentrations and stirring intensity was observed by agglomeration measurements and the conclusions were summarized as:

(1) In the absence of flotation reagents, the pulp apparent viscosity and yield stress of smithsonite, kaolinite, quartz, and calcite increased with the decrease of mineral particle size. the coarse particles (150-74 μm) generally showed a low apparent viscosity and yield stress, while the fine particles (-23 μm) presented a high apparent viscosity and yield stress.

(2) The apparent viscosity and yield stress of fine kaolinite increased sharply with the pulp density. The variation trend of the apparent viscosity and yield stress of calcite and smithsonite was similar, and both increased with the pulp density. The pulp apparent viscosity and yield stress of quartz were low, showing a good dispersion state. The order of apparent viscosity and yield stress of the four kinds of single mineral pulp was kaolinite>calcite>smithsonite>quartz at different pulp densities and pH values.

(3) The rheology of the collector octadecylamine solution was close to that of pure water. Under different octadecylamine concentrations and stirring intensities, smithsonite, kaolinite, and calcite particles could form aggregates. Especially, smithsonite particles presented obvious agglomeration with large particle size and compact network structure. The agglomerations of calcite and kaolinite were weaker than that of smithsonite. The particle agglomeration would increase the apparent viscosity and yield stress of the pulp. Quartz particles didn't form clusters in the presence of octadecylamine, hence the pulp apparent viscosity and yield stress were the lowest.

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