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Influence of measuring process properties on phosphate rock slurry rheology based on Brookfield method

Aoao Chen [1], Qin Zhang [234]

[1] Mining College, Guizhou University, Guizhou, Guiyang 550025, China
[2] Guizhou Academy of Sciences, Guizhou, Guiyang 550001, China
[3] National & Local Joint Laboratory of Engineering for Effective Utilization of Regional Mineral Resources from Karst Areas, Guizhou, Guiyang 550025, China
[4] Guizhou Key Lab of Comprehensive Utilization of Nonmetallic Mineral Resources, Guizhou, Guiyang 550025, China

Corresponding authors: 18212106728@163.com (A. Chen), zq6736@163.com (Q. Zhang)

Abstract: This research aims to explore how the flotation process conditions influence the rheological properties of phosphate rock slurry. The apparent viscosity of phosphate rock slurry was measured by Brookfield DVNext rheometer. Different mineral types, grinding time, slurry mass concentration and reagent systems were adopted to study the viscosity behavior of phosphate rock slurry. The results showed that under the same conditions, the apparent viscosity of apatite and dolomite slurry was basically the same, and the apparent viscosity of the slurry after mixing the two minerals was basically the same as that of single mineral. For the same slurry concentration, the mineral particle size had a significant effect on the rheological behavior of the slurry, and its apparent viscosity increased exponentially with the decrease of particle size. In addition, phosphate rock slurry showed shear thickening fluids characteristics when the slurry concentration is 20%-40%, but changed to pseudoplastic fluid at high concentration (60%). Sulfuric acid as pH regulator and inhibitor had little effect on the rheology of slurry; when GJBW and NaOL were added as collectors, the rheology of slurry changed, and the effect of GJBW was more obvious.

Keywords: phosphate rock, grinding, concentration, rheological properties, apparent viscosity

1. Introduction

Phosphate rock is a non-renewable resource. As an important chemical raw material extracted from phosphate rock, phosphorus is mainly used to produce phosphate fertilizer, phosphate and phosphoric acid (Zhou et al., 2020). Apatite is the main recovered useful mineral in phosphate rock, while silicate minerals and carbonate minerals (dolomite and calcite) are the main gangue minerals. Apatite and gangue minerals usually coexist (Zhang et al., 2021). In order to make effective use of phosphate rock resources, the selective separation of apatite and other gangue minerals is very important (Yang et al., 2020). China is rich in phosphate rock resources, but it is characterized by its less rich ore. Most of them are medium and low-grade collophanite (Yang et al., 2021). Collophanite and gangue mineral dolomite are highly symbiotic and are difficult to separate due to their similar physical, chemical properties and mineral composition on the mineral surface (Liu et al., 2017b). At present, the beneficiation processes for treating phosphate rock include flotation, roasting, leaching, and gravity separation, among which flotation is the most commonly used method for phosphate rock beneficiation (Ruan et al., 2019).

Flotation is the use of physical and chemical properties of minerals to separate useful minerals from gangue minerals, and is the most effective method for removing dolomite from phosphate rock (Liu et al., 2021). Flotation is divided into positive flotation and reverse flotation. Usually, direct flotation with anionic collectors and reverse flotation with cationic collectors are used to remove gangue minerals in phosphate rock (Zheng et al., 2022). Both positive flotation and reverse flotation are suitable for silicate-containing gangue minerals, while carbonate-containing gangue minerals are more suitable for reverse flotation. The most effective separation of dolomite and apatite in collophanite is the reverse flotation process (Liu et al., 2017a). The use of reagents in the flotation process is crucial to the flotation of phosphate rock. Long-chain fatty acids and their salts are the most widely used collectors in the flotation process of phosphate rock, such as oleic acid and Sodium oleate(NaOL). However, fatty acid collectors have poor selectivity and cannot selectively separate apatite and dolomite (Pan et al., 2020; Zeng et al., 2022). Inhibitors can prevent the adsorption of collectors on mineral surfaces and enhance mineral hydrophobicity, and sulfuric acid and phosphoric acid are the most commonly used inhibitors in phosphate rock flotation (El-Mofty and El-Midany, 2018; Liu et al., 2017c).

In recent years, due to the improvement of the basic theory of rheological properties and the innovation of research methods, rheological properties has been widely used and developed in the chemical industry, metallurgy, materials and mineral processing industries (Cruz et al., 2019; Freitas et al., 2018; Lefebvre et al., 2020). In the field of mineral processing, more and more researchers apply rheological properties to flotation (Wang et al., 2015; Zhang and Peng, 2015). The flotation separation of minerals may be affected by specific interaction. Slurry is a heterogeneous solid-liquid composed of mineral particles and water, and the rheological behavior of slurry indicates the interaction and aggregation degree between particles. The interaction between particles, between bubbles and particles, and between agents and minerals will affect the rheological properties of slurry. Therefore, rheological properties can be used as a useful control parameter in the process of mineral flotation (Cruz et al., 2015a; Farrokhpay, 2012).

And some studies have found that the ore type, particle shape and size, and mineral content will affect the rheological properties of the slurry during the flotation process (Cruz et al., 2015b; Mueller et al., 2009). Cruz et al. (Cruz et al., 2013) studied how pH regulators, collectors and frothers commonly used in copper gold flotation affect rheological properties. The result showed that pH regulators, collectors and frothers changed the rheological behavior of kaolinite and bentonite slurry, and the effect of pH regulators was more obvious. Osorio et al. (2015)studied the viscosity of quartz slurry under different grinding time and different solid concentration. They found that the solid concentration had a significant impact on its rheological properties, and the reduction of particle size would not significantly change the rheological behavior of slurry with low concentration (less than 40%). Chen et al. (2020) studied the effects of montmorillonite, kaolinite and illite on pyrite flotation. Through rheological measurement, it was found that with the increase of kaolinite content, the slurry viscosity increased slightly and the recovery of pyrite decreased slightly. The addition of illite had no obvious effect on the apparent viscosity, and thus the effect of illite on pyrite flotation can be ignored to a great extent. Montmorillonite significantly increased the slurry viscosity, which greatly reduced the recovery and grade of pyrite.

In this paper, Guizhou phosphate rock was selected as the research object, and representative apatite and dolomite were manually selected to stady the influence of mineral types on the rheological properties of slurry. Phosphate rock with different fineness was obtained by different grinding time, and the rheological properties of phosphate rock slurry with different fineness was tested. The study included the following steps. Configure different concentrations of slurry, test its rheological properties, and explain the influence of slurry concentration on flotation through rheological properties. Moreover, in the flotation process, pH regulator is usually added to adjust the flotation pH, while inhibitor, collector, and frother are added to adjust the slurry. The adsorption of these agents on the mineral surface will change the interaction between mineral particles and change the rheological properties of the slurry. The influence of agents on the rheological properties of the slurry is explored by using the process conditions of phosphate rock reverse flotation.

2. Experiment

2.1. Materials and reagents

The phosphate rock used was taken from a phosphate rock mining area in a certain area of Guizhou, China. The useful minerals in the ore are mainly collophanite, the gangue minerals are mainly dolomite, with a small amount of calcite, siliceous minerals and limonite. It belongs to typical calcium magnesium collophanite. The analysis results of the main chemical components of the test are shown in Table 1. Crush the massive phosphate rock to less than 3 mm, grind it with a ball mill, and the ground sample was used for rheological test.

Table 1. Chemical composition of raw materials

Elements	P_2O_5	CaO	MgO	SiO_2	Fe_2O_3	Al_2O_3
Content / %	22.36	41.21	7.73	4.57	0.46	1.22

This experiment explored the rheological properties of slurry according to the experiment conditions of reverse flotation of collophanite. Sulfuric acid is often used as flotation inhibitor and pH regulator in reverse flotation. Therefore, sulfuric acid was selected as inhibitor and pH regulator, and the concentration of sulfuric acid was 20%. GJBW and NaOL were used as collectors, with the concentration of 1%. GJBW is the code name of the collector. It is a phosphate rock flotation collector independently developed by the school. The water used in the test was laboratory tap water.

2.2. Slurry preparation

The main minerals in collophanite were apatite and dolomite. Representative apatite and dolomite were selected manually. After grinding, the sample was configured into a slurry with a concentration of 25%. The particle size distribution of apatite and dolomite after grinding was measured by laser particle size analyzer. The sample was mixed with water to form a very thin slurry, which was then dispersed by ultrasound for 30 s and poured into the diluter of the laser particle sizer for particle size measurement. The cumulative particle size distribution of apatite and dolomite is shown in Fig.1. The particle size distribution of apatite and dolomite is similar, but there are more micro fine minerals in dolomite.

The particle size of the sample was controlled by setting different grinding time. The grinding time was set as 1 min, 2 min, 3 min, 4 min, 5 min and 6 min. 150 g phosphate rock was weighed each time, with the grinding concentration of 60%. The particle size distribution of samples with different grinding time was measured by a laser particle size analyzer. The cumulative particle size distribution of different grinding time is shown in Fig. 2, and the statistical data of particle size distribution is shown in Table 2. The longer the grinding time was, the smaller the particle size distribution of phosphate rock appeared. The median particle size D_{50} of 1 min grinding was 68.40 μm. The median particle size D_{50} of grinding for 6min was 21.27 μm. There appeared a big difference between the two. The rheological properties of slurry with different grinding fineness were investigated.

The crushed samples below 3 mm were ground with a ball mill for 2 min. The fixed capacity of the ground samples was 500 mL, and the ore slurry with concentrations of 20%, 25%, 30%, 35%, 40%, and 60% were respectively prepared to test the rheological properties of slurry with different concentrations.

The samples ground for 2 min were configured into slurry with a concentration of 25%, which was similar to the slurry concentration in the flotation of mechanical stirring flotation machine in the laboratory. The dosage of the inhibitor was 8 kg/t, 10 kg/t, 12 kg/t and 14 kg/t. The rheological properties of slurry were tested after being mixed with sulfuric acid and stirring for 1min. The dosage of sulfuric acid is 12 kg/t, NaOL and GJBW were used as collectors, and the addition amounts of collectors were 100 g/t, 200 g/t, 300 g/t and 400 g/t. Firstly, sulfuric acid was added to adjust the slurry for 1 min, and then GJBW and NaOL with different dosage being added to adjust the slurry for 2 min to test the rheological properties of the slurry. The influence of inhibitors and collectors on the rheological properties of the slurry was studied.

2.3. Rheological properties measurement

The rheological property was measured by Brookfield DVNext rheometer. After the slurry was prepared, the slurry was stirred for a certain time before the viscosity of the slurry was measured. Each group of slurry was tested for 20 s, and the average value was obtained by testing three groups of data. The measurement error is less than 1%. The rheological properties of the slurry were tested at room temperature at about 14 ℃. Select a suitable rotor and completely immerse the rotor in the slurry for slurry rheological properties test. The torque range of the rotor for testing slurry rheological properties was kept between 10-100%, which proved to have the best effect. For unknown fluid, the process of selecting rotor and speed should be tested repeatedly. LV61 rotor was finally selected according to the test. When the slurry concentration was 60%, LV61 rotor exceeded the torque range of 100%, so LV62 rotor was taken to measure its apparent viscosity. It was found that when the speed of LV61 rotor for slurry was lower than 125 RMP, the torque of the rotor was less than 10%. Thus, the speed for the test was selected to be 125-250 RMP.

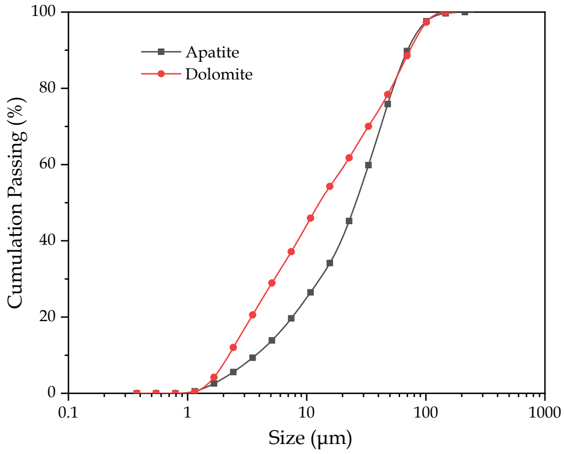

Fig. 1. Cumulative particle size distribution of apatite and dolomite

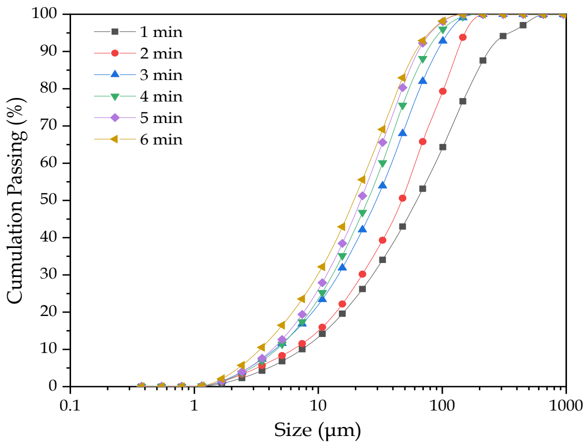

Fig. 2. Cumulative particle size distribution of samples after grinding

Table 2 Particle size distribution of samples with different grinding times

Sample	Range（μm）	d_{10}（μm）	d_{50}（μm）	d_{90}（μm）
1min	0.87-863.87	8.12	68.40	258.61
2min	0.87-256.94	6.89	51.76	145.39
3min	0.87-234.05	4.91	32.25	99.54
4min	0.87-234.05	5.07	27.51	87.27
5min	0.87-176.91	4.67	24.14	69.98
6min	0.87-176.91	3.73	21.27	66.99

3. Results and discussion

3.1. Effect of mineral types on rheological properties of slurry

Fig. 3 shows the effect of mineral types on the rheological properties of slurry. It can be seen that different mineral compositions have little effect on rheological properties of slurry. When there are only apatite and dolomite in the slurry, the apparent viscosity of the slurry is similar. Besides, when dolomite and apatite were mixed in different proportions, the apparent viscosity of slurry had no obvious change. It shows that the mineral types in phosphate rock slurry are not the main factors affecting the apparent viscosity of phosphate rock slurry.

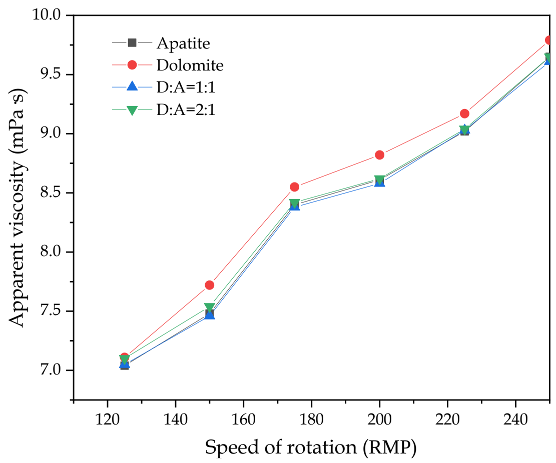

Fig. 3.Effect of mineral types on rheological properties of slurry

3.2. Effect of grinding fineness on rheological properties of slurry

Fig. 4 shows the apparent viscosity of slurry with different grinding fineness at different rotating speeds. Fig. 5 shows the relationship between different grinding fineness and rheological properties of slurry at rotating speed of 200 RMP. It can be seen from Figure 4 and Figure 5 that the apparent viscosity of the slurry is different at different speeds, and the slurry is a non-Newtonian fluid; The apparent viscosity increases with the increase of rotating speed, indicating that the slurry is a shear thickening fluids. When the mass fraction of -0.075 mm particles in the slurry exceeds 90%, the apparent viscosity of the slurry decreases with the increase of rotating speed. With the increase of -0.075 mm mass fraction in the slurry, the fine particles in the slurry increase. The properties of the slurry will change with the increase of fine particles.

When the rotating speed is 200 RMP, the apparent viscosity of the slurry after grinding for 6 min is 7.56 mPa · s, the apparent viscosity of the slurry after grinding for 1 min is 5.37 mPa · s, and the apparent viscosity of the slurry increases by 2.19 mPa · s. With the increase of grinding fineness, the overall particle size of particles in slurry becomes finer, and the apparent viscosity of slurry shows an upward trend. This indicates that the particle size has a great influence on the viscosity of the slurry. When the particle size in the slurry is too large, the interaction and friction between the particles are small, the particles precipitate too fast in the slurry, the surface area of the particles is small, and the combination with water molecules is less, which reduces the effective concentration in the slurry and reduces the viscosity of the slurry. It is observed that the smaller the particles are in the slurry, the greater the interaction and friction appear between the particles, and the slower the settling speed of the particles and the increase of the surface area. Combined with more water molecules, the effective concentration of the slurry increases, so the apparent viscosity of the slurry increases (Kawatra and Eisele, 1988). The apparent viscosity of slurry under different grinding fineness is low, which indicates that the interaction between particles in phosphate rock slurry is small and the particles are in a good dispersion state in flotation.

3.3. Effect of slurry concentration on rheological properties

Fig. 6 shows the change of apparent viscosity with rotating speed under different concentration. Fig. 7 shows the effect of concentration on the rheological properties of slurry. It can be seen from Fig. 6 that the apparent viscosity of slurry increases with the increase of rotating speed, and the apparent viscosity of slurry changes significantly with the increase of concentration. The concentration is controlled in the range of 20-40%, and the properties of the slurry have not changed. It is a shear thickening fluids. When the concentration of slurry is 60%, the apparent viscosity of slurry decreases with the increase of rotating speed. It can be deduced that the properties of slurry will change from shear thickening fluids to pseudoplastic fluid at high concentration (Laboratories, 2006). It can be seen from Fig. 7 that the apparent viscosity of slurry increases with the increase of concentration, which is

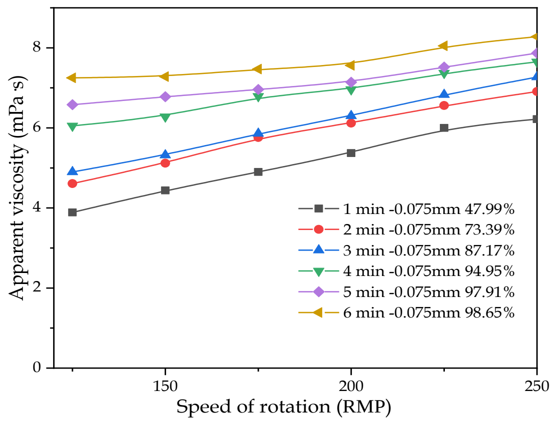

Fig. 4. Apparent ore viscosity at different grinding speeds

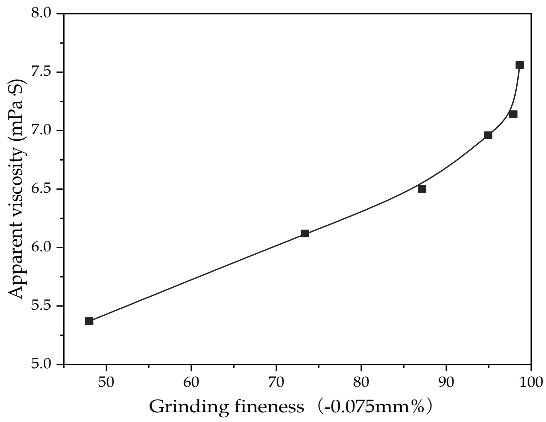

Fig. 5. Relationship between different grinding fineness and slurry rheological properties at rotating speed of 200 RMP

mainly due to the increase of interaction and friction between particles in the fluid. The slurry concentration is in the range of 20%-30%, and the apparent viscosity of the slurry only increases slightly with the increase of the concentration. When the slurry concentration＞30%, the slurry viscosity rises sharply. Through exponential fitting, it is found that the apparent viscosity of slurry increases exponentially with the increase of concentration, which is similar to the phenomenon observed by Rutgers (Rutgers, 1962). According to the conclusion in 3.2, it can be found that the grinding fineness and slurry concentration are the main factors affecting the rheology of phosphate rock slurry, which is similar to the conclusion obtained by Moçayd when studying the physicochemical parameters affecting the rheology of the phosphate slurry pipeline transportation (El Moçayd and Seaid, 2021). Whether in the field of pipeline transportation or flotation, slurry concentration and particle fineness will have a greater impact on the viscosity of phosphate rock slurry. The apparent viscosity of the slurry increases with increasing concentration, mainly due to the increase in particle-particle interactions. At low concentration, the influence of hydrodynamic interactions is dominant, and the apparent viscosity of slurry increases slowly; with the increase of slurry concentration, the frictional interactions between particles per unit volume increases, which makes the apparent viscosity of slurry increase sharply (Cheng 1980; Mangesana et al., 2008).

3.4. Effect of pH on rheological properties of slurry

Fig. 8 shows the effect of different addition amounts of sulfuric acid on the rheological properties of phosphate rock slurry. Fig. 9 shows the effect of different addition amounts of sulfuric acid on the apparent viscosity of phosphate rock slurry when the rotating speed is 200 RMP. It can be seen from Fig. 8 and Fig. 9 that the apparent viscosity of the slurry is different at different speeds, indicating that the change of pH value in the slurry will not change the properties of the slurry, and the slurry is a shear

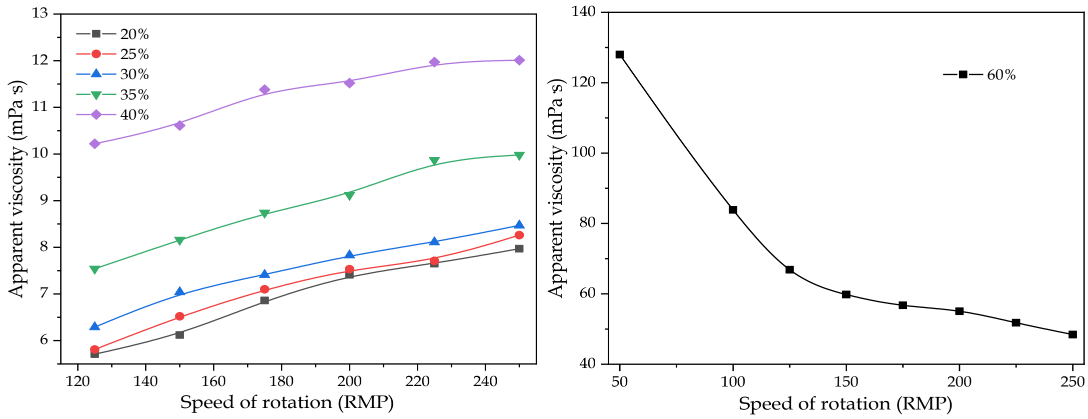

Fig. 6. Effect of slurry concentration on rheological properties of slurry

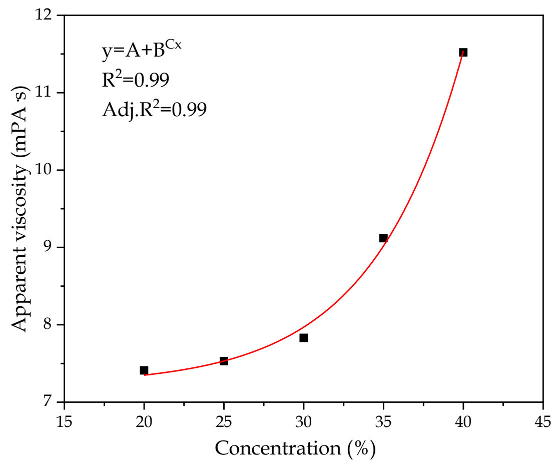

Fig. 7. Effect of slurry concentration on rheological properties at rotating speed of 200RMP(Model parameters: A=7.218, B=0.00395, C=0.175)

thickening fluids. With the increase of sulfuric acid dosage, the pH value of slurry decreases continuously, and the apparent viscosity of slurry increases only slightly with the decrease of pH. It shows that the apparent viscosity of phosphate rock slurry will not change significantly by changing the pH in the slurry. The reason for this phenomenon may be that sulfuric acid mainly combines with Ca atoms on the surface of apatite in phosphate rock slurry, which hinders the adsorption of collector on the surface of apatite and keeps apatite hydrophilic (Zhang et al., 2020; Zou et al., 2019). However, there are other minerals in the phosphate flotation slurry, which will not have a great impact on the interaction between particles in the slurry.

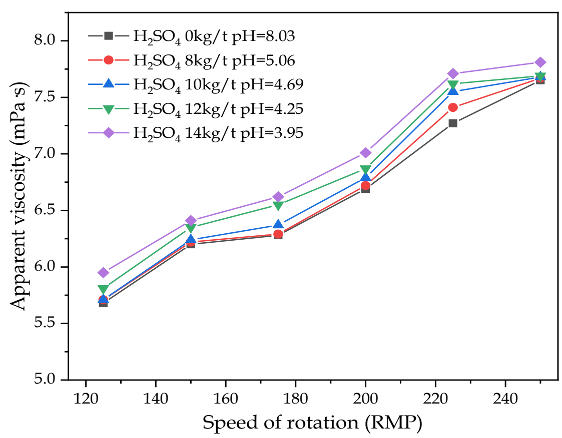

Fig. 8. Effects of sulfuric acid on the rheological properties of slurry at different rotational speeds

3.5. Effect of collector on rheological properties of slurry

Fig. 10 shows the effect of different addition amounts of GJBW and NaOL on the rheological properties of phosphate rock slurry, and Fig. 11 shows the effect of different addition amounts of GJBW and NaOL on the apparent viscosity of phosphate rock slurry at 200 RMP. As can be seen from Fig. 10, with the increase of rotating speed, the apparent viscosity of slurry increases, indicating that the properties of slurry have not changed and still belong to shear thickening fluids. After adding NaOL and GJBW, the apparent viscosity of slurry increases slightly. When the dosage of reagent is 100 g/t, the viscosity of

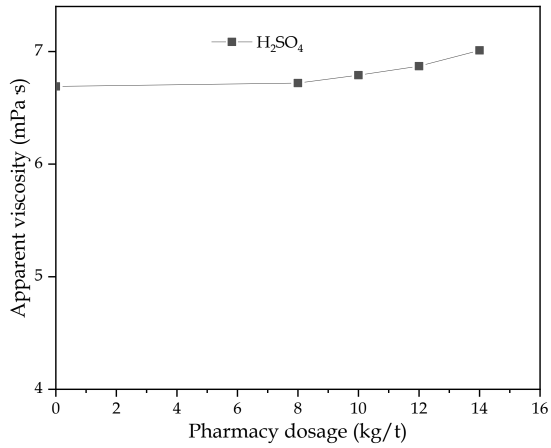

Fig. 9. Effect of sulfuric acid and on the apparent viscosity of slurry at 200RMP

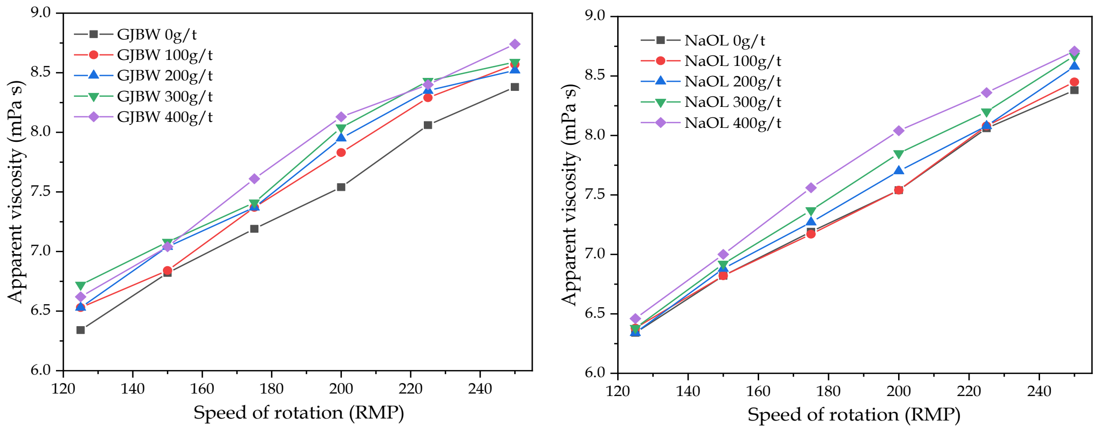

Fig. 10. Effects of GJBW and NaOL on the rheological properties of slurry at different rotational speeds

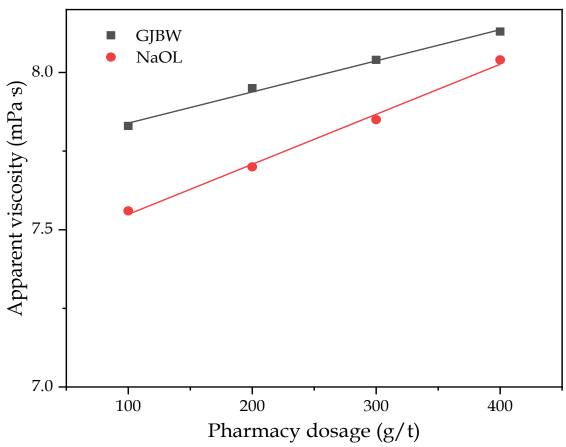

Fig. 11. Effects of GJBW and NaOL on rheological properties at 200 RMP

slurry added with GJBW increases obviously, and the apparent viscosity of slurry added with NaOL does not change significantly. The apparent viscosity of slurry increases linearly with the increase of collector dosage. It can be seen from Fig. 11 that when the dosage of GJBW and NaOL is 100-400 g/t, the apparent viscosity of slurry increases, but the increase is not more than 1 mPa·s. The apparent viscosity of slurry increases linearly with the increase of collector dosage. Among them, GJBW has a greater impact on the apparent viscosity of slurry than NaOL. Since both GJBW and NaOL have foaming properties, it was observed in the test that a large amount of froths would be produced when the slurry was stirred after adding GJBW, and the slurry was slightly foamed after adding NaOL. With the increase of the dosage of the agent, the amount of froths generated increases, which may be the reason that the apparent viscosity of the slurry increases slightly after adding GJBW and NaOL, and it is also the reason that the apparent viscosity of the slurry after adding GJBW is greater than that of NaOL.

4. Conclusions

This research has the following findings. The apparent viscosity of apatite and dolomite slurry is found to be basically the same, and the apparent viscosity of slurry does not change significantly after mixing. In the collophanite flotation system, the mineral types have little effect on the rheology of slurry. Besides, the longer the grinding time, the smaller the average particle size of the ore and the more fine minerals in the slurry. With the increase of fine minerals, the surface area increases, so are the effective concentration of slurry and the apparent viscosity of slurry. The apparent viscosity of slurry is too large, which is not conducive to flotation, so the grinding time should not be set too long.

The concentration of slurry has a great influence on the apparent viscosity. At low concentration, the hydrodynamic interactions effect dominates, and the apparent viscosity of slurry increases slowly. When the concentration exceeds 30%, the particles in the unit volume of slurry increase. At this time, the frictional interactions between particles increases, and the apparent viscosity of slurry increases sharply. This well explains the reason why the slurry concentration should not be too high in the flotation process. When the slurry concentration is 20%-40%, the slurry behaves as shear thickening fluids, and when the slurry concentration is too high (60%), the slurry will change from shear thickening fluids to pseudoplastic fluid.

The addition of reagents has little effect on the apparent viscosity of the slurry. Adding sulfuric acid as inhibitor and pH regulator has little effect on the apparent viscosity of slurry; after adding GJBW and NaOL, the apparent viscosity of the slurry increased slightly, and the increase was less than 1mPa·s. In the case of the same concentration and the same particle size, the apparent viscosity of the slurry added with the agent increased slightly, indicating that the slurry as a whole was in a good dispersion state and would not adversely affect the flotation.

Acknowledgments

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