

The Influence of Kaolinite and Quartz on Stability of Coal Froths – A Rheology and Structure Study

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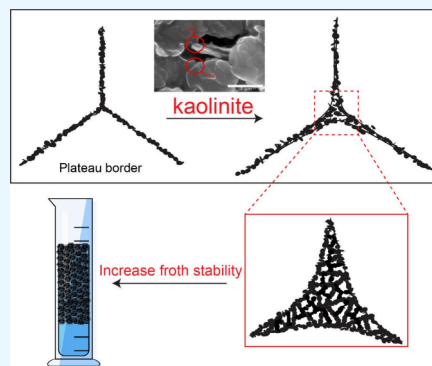
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ABSTRACT: Kaolinite and quartz are the common gangue minerals found in raw coal; however, their effects on stability of coal froths and subsequent settling of coal flotation products have not been investigated. In this study, in the coal froths batch settling tests, the amount of froth floating on top of water was 275, 325, 355, and 405 mL for coal concentrates generated with 0, 20, 40, and 60 wt % kaolin Q38, respectively, while that was almost the same (300–306 mL) for coal froth concentrates generated with 0, 20, 40, and 60 wt % quartz added in flotation, respectively, which turned out that the kaolinite could increase the stability of coal froth, while quartz could not. To investigate the mechanism, oscillatory rheology and scanning electron microscopy (SEM) were applied. The results of the oscillatory rheology suggested that the structural strength in coal froth was strengthened with the addition of kaolinite. In addition, images of Plateau borders by SEM illustrated that the addition of kaolinite in flotation increased the size of Plateau borders and generated network structures in the Plateau borders. However, as a comparison, the addition of quartz did not cause an obvious change for the oscillatory rheology and SEM results of coal froth. Based on the results, it can be concluded that network structures were generated in the Plateau border of coal froth with the addition of kaolinite, which increased its structural strength and retarded the drainage in froth. As a result, the stability of the coal froth increased.



1. INTRODUCTION

In coal processing plants, dewatering is an important step in determining the quality of final coal concentrates. The water percentage in coal concentrates affects coal's thermal value and a high moisture content in coal concentrates increases the freight charges and causes handling problems.^{1–3} Thickening and filtration are two steps in dewatering. In thickening, particles are separated from water by sedimentation under the force of gravity. The settled particles in thickeners are transported to filters, and thus, the quality of thickened products affects the efficiency of filtration. The froth stability affected the dewatering of froths.⁴ There is a general problem in coal preparation plants that ultrastable froth products float on top of thickeners, which causes the loss of coal concentrates and the decrease of dewatering efficiency.

The effect of coal froths on settling of coal flotation products has been investigated.⁵ Compared with the coal slurry without going through the flotation process, the particle interactions in coal froths are much stronger in the presence of air bubbles, driven by the capillary force. As a result, most coal particles will be attached on bubbles and stay in froths rather than settling down. It has also been found that saline water deteriorates the problem by promoting more froths to float on top of water in settling.⁶ This is because saline water can promote the formation of coagula among coal particles, and the size of coagula increases with an increase in water salinity. Addition-

ally, saline water increases the capillary attraction force between particles on bubbles, so the structural strength of coal froths is increased. As a result, capillary and buoyancy forces counteract the gravity forces and hold coal particles in froths.

The previous studies used purified coal particles and did not consider gangue minerals, which may be recovered to the froth concentrate through mechanical entrainment. The entrained gangue minerals may affect particle interactions in coal froths and their settling behavior. In fact, kaolinite and quartz are the major gangue minerals in raw coals.⁷ The composition of kaolinite is $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. Kaolinite is a nonswelling clay mineral with low chemical reactivity. Its structure is composed of 1:1 Al–OH octahedral to Si–O tetrahedral layers.⁸ The pseudo-hexagonal clay platelets have two surfaces, the flat basal surface (F) and the edge surface (E). The basal surface carries permanent negative charges due to isomorphous substitution of Si^{4+} by Al^{3+} groups or positioning of the electro-dependent

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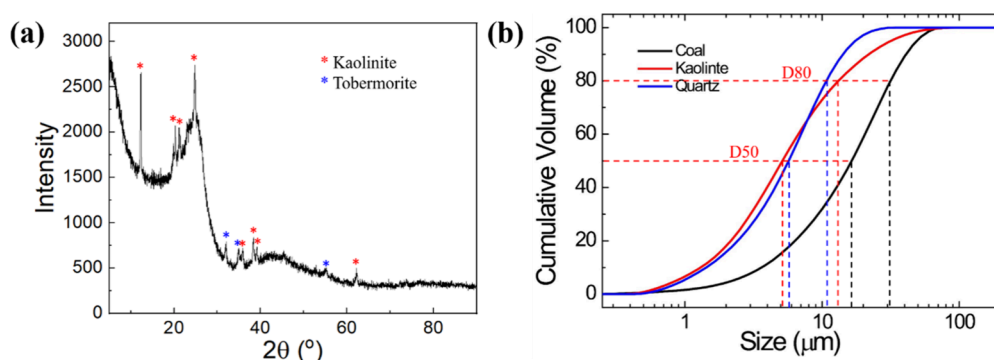


Figure 1. (a) XRD result of the purified coal. (b) Size distribution of coal, kaolinite Q38, and quartz.

charge above the surface,⁹ while the charge on the edge surface depends on the bulk pH due to the protonation or deprotonation of edge surface Al–OH and Si–OH groups.¹⁰ The isoelectric points (iep) of the Al–OH and Si–OH edge sites are from 5.0 to 7.0, so the edge surface is negative beyond pH 7.0.⁹ The platelets can connect with each other through face to face (F–F), edge to face (E–F), and edge to edge (E–E) associations depending on the balance between van der Waals attractive forces and electrostatic repulsive forces on different surfaces.^{11,12} Quartz is a kind of silica mineral (SiO₂) that is made of SiO₄ tetrahedral units. The crystalline quartz is hard and strong. Quartz is also negatively charged in the neutral pH range, which is the same as coal particles.^{13,14}

Oscillatory rheology is a nondestructible method for fragile coal froths.¹⁵ The rheology of froths depends on the surface tension, which is related to particle interactions in froths.¹⁶ Rheological responses of froths have been efficiently employed to investigate the interactions of particles in froths in the absence of gangue minerals in the previous studies.^{5,6} In this study, the settling of coal flotation products generated with different concentrations of kaolinite or quartz was conducted. Oscillatory rheology was still applied to investigate how kaolinite and quartz affect the properties of coal froths together with scanning electron microscopy (SEM). The target of this study was to figure out the effects of quartz and kaolinite on stability of coal froths and subsequent settling of coal flotation products, which would have guiding significance on the elimination of stable coal froths in dewatering.

2. MATERIALS AND METHODS

2.1. Materials. A raw coal sample was supplied from Bulli Mine, New South Wales, Australia, crushed in a cone crusher and then the coal particles with a size of several millimeters was ground in a stainless steel rod mill. After grinding, particles were filtered through a sieve with a size of 38 μm. To purify the raw coal, the ground coal particles (<38 μm) were floated in a 3.0 L flotation cell without an addition of any chemicals. The purified coal particles were filtered and then put into the oven at 70 °C overnight. The ash content for the purified coal was 5.62%, and the main gangue minerals were kaolinite and tobermorite, as shown in the XRD result (Figure 1a). Its size distribution was measured using Mastersizer 2000 (Malvern Instrument Ltd., UK). As shown in Figure 1b, 50% of purified coal particles are under 16.5 μm and 80% of them are under 32.3 μm.

The kaolinite tested in this study was Q38 purchased from Sibelco Australia Ltd. It consists of 85% kaolinite, 4% quartz, and 11% muscovite.^{9,17} Q38 is poorly crystallized and its

surface structure is complex with a high specific surface area.⁹ As shown in Figure 1, 50% of the Q38 particles are under 5.1 μm, and 80% of them are under 13.6 μm.

Quartz (milky, > 99.3% SiO₂) was purchased from Ward's Science, US.¹⁸ It was introduced to compare with kaolinite. The quartz sample was crushed in a cone crusher, and then a lab ball mill was used to grind the quartz to get a particle size distribution similar to that of Q38. As shown in Figure 1, 50% of ground quartz particles are under 5.7 μm and 80% are under 11.0 μm.

Fresh water was used in this study. Its pH was 7.4, and conductivity was 475 μS. Methyl isobutyl carbinol (MIBC) was used as the frother. Industry-grade diesel was used as collector.

2.2. Flotation. 0 g (0 wt %), 7.5 g (20 wt %), 20.0 g (40 wt %), and 45 g (60 wt %) of Q38 or quartz were mixed with 30.0 g of purified coal and 1.5 L of fresh water in a 1.5 L flotation cell and stirred at 1000 rpm for 4 min. 0.01 mL of diesel was added into the flotation cell and then stirred for 1 min. Then, 2 mL of MIBC (1% v/v) was added in the flotation cell and conditioned for 30 s. Then, air was introduced at a rate of 3.0 L/min. Froths were generated and collected every 10 s for 2 min. The recovery of coal particles was over 95%. The coal concentrates collected were ready for settling tests.

2.3. Settling Tests. The coal concentrates collected from flotation were transferred to 1000 mL measuring columns. All of the samples' weights were kept the same. They were placed on a flat bench overnight at room temperature. Then, the volumes for the settled particles and floating froths were recorded. After settling tests, all samples were filtered and then dried at 70 °C in an oven. 1.0–2.0 g dried sample was then put into a muffle furnace and burned at 815 °C for 5 h to determine the ash content or the content of Q38 or quartz.

2.4. Oscillatory Rheology. The TA Discovery HR-1 Rheometer (TA Instruments Ltd., USA) was applied to conduct the oscillatory rheology measurements. A vane in the cup geometry was adopted. The diameter and length of the vane were 28.0 and 42.0 mm, respectively. The inner diameter and height of the cup were 60.3 and 100.0 mm, respectively. The temperature was set at 25 °C during measurements.

To avoid the damage of coal froths, coal froths were generated in the cup for oscillatory rheology measurements. 100 mL of slurry was taken from the flotation cell after conditioning of the collector and frother, transferred in the cup, and stirred for 4 min. The cup was then put on the rheometer, and the mixture was stirred by the vane (with a gap of 6 mm) for 1 min at the speed of 100 rad/s. Then, air flow was introduced at a rate of 1.0 L/min, and a froth was

generated. The air flow and the vane were stopped when the froth height was 4.5 cm. Then, the gap between the vane and the bottom of the cup was altered to 400 mm followed by the rheology measurement.

Amplitude sweeps were performed from 0.01% to 1% at a frequency of 1 Hz. Frequency sweeps were conducted from 0.1 to 10 rad/s at a shear strain of 0.02%. Storage modulus G' and loss modulus G'' were recorded.

2.5. Scanning Electron Microscopy (SEM). The images of Q38, quartz, and froth structures were captured on SU3500 (Hitachi Ltd. Japan). A carbon tab was attached to the sample holder ($d = 12.6$ mm). Q38 and quartz samples were collected from 0.01 wt % suspensions. One drop of sample was applied on the carbon tab and put into the vacuum oven at 70 °C for 2 h. The frozen samples were collected from flotation. A teaspoon of coal froth generated between 20 and 30 s in flotation was put on the sample holder, and then, they were kept in the fume hood overnight. When the sample was dried naturally, the edge of the sample holder was knocked to remove any froth on its top, and a new fracture surface of the coal froth was obtained. The prepared samples were coated in the Iridium Coater Q150TS (Quorum Technologies Ltd., UK) and put into SEM. The images of them were captured at 20 kV accelerating voltage, 20 spot size, and 20 mm working distance.

3. RESULTS AND DISCUSSION

3.1. Settling of Coal Concentrates. Q38 or quartz was added to the flotation cell together with the purified coal. In the flotation, some Q38 or quartz was entrained in the froth concentrate as ash. As shown in Table 1, the ash content

Table 1. Content of Q38 or Quartz in Coal Froth Concentrates Generated with the Additions of 20, 40, and 60 wt % Q38 or Quartz in Flotation

percent of Q38 or quartz in flotation	20%	40%	60%
percent of Q38 in concentrate	1.3 ± 0.03%	3.5 ± 0.41%	7.7 ± 0.16%
percent of quartz in concentrate	1.4 ± 0.22%	3.6 ± 0.42%	8.3 ± 0.28%

increased with the increase of the concentration of Q38 or quartz added with the coal. Compared with the amount of Q38 or quartz added in flotation, the amount of Q38 or quartz entrained into the froth concentrate was much less. The amounts of Q38 and quartz entrained were also similar when the same amount of Q38 and quartz was added in the flotation cell.

The froth concentrates were then allowed to settle in cylinders. The ideal settling is that all froths break into coal slurry and the coal particles settle down to the bottom of the cylinder so that the particles and water are separated. However, in this study, only part of the coal particles settled down, and the rest of the coal particles stayed in froths and floated on top of water, which simulates the phenomenon occurring in the coal preparation plants where froths float on top of thickner. Due to the small amount of particles that settled and the turbidity of the water phase caused by ultra fine particles, the settling rate of particles was hard to measure. Therefore, the amount of froths floating on top of water in the cylinder was measured as an indicator of the settling efficiency and froth stability in this study.

The settling of coal froth concentrates with the addition of 0, 20, 40, and 60 wt % Q38 in flotation is shown in Figure 2a. With an increase in the Q38 concentration, the volume of froth floating on top of water increased in settling. The average volume of froth floating on top of water was 275, 325, 355, and 405 mL for coal concentrates generated with 0, 20, 40, and 60 wt % Q38, respectively. The volume of froth floating on top of water was plotted as a function of Q38 concentration in flotation and a linear relationship was obtained with a slope of 2.03 as inserted in Figure 2a.

The addition of quartz in coal flotation had little influence on the settling of coal froth concentrates, as shown in Figure 2b. The volume of froth floating on top of water was almost the same for coal froth concentrates generated with 0, 20, 40, and 60 wt % quartz added in flotation, the average of which was between 300 and 306 mL. A linear relationship was also obtained between the volume of froth floating on top of water and quartz concentration but with a small slope of 0.05.

According to the settling results, the entrained Q38 in flotation had an obvious influence on the settling of coal froth concentrates, but the entrained quartz had little influence, although their contents in coal froth concentrates were almost the same when the same amount was added in flotation. Additionally, Q38 and quartz had a similar size distribution in this study and at pH 7.4, the pH used in this study, both were negatively charged.^{12,19} However, their microstructures were totally different. For Q38, large flakes were formed by clay platelets, as shown in Figure 3a. The basal and edge surfaces were both negatively charged in fresh water at pH 7.4.¹⁶ The type of platelet aggregation depends on the balance between van der Waals attractive forces and electrostatic repulsive forces on different surfaces.²⁰ Because of the low electrolyte concentration in fresh water, clay platelets tend to deflocculate, but E–E structures still form due to hydrogen bonding between Si–OH and Al–OH edge site. Hydrogen bonded E–F and F–F structures also possibly generate on the basal surface of poorly crystallized kaolinites, such as Q38.^{9,12} As shown in Figure 3c, E–E and E–F aggregations dominated the flocculation, forming honeycomb structures. Some small clay platelets attached on big ones through F–F structures. However, for quartz, the minimum unit that could be observed was a single particle as shown in Figure 3b,d. The microstructure of quartz was a surface contacted with some small particles, as shown in Figure 3d. Therefore, it was inferred that the microstructure of kaolinite aggregates in the flotation products caused the rise of froth volume floating on top of water in the settling of coal concentrates.

3.2. Froth Characterization by Oscillatory Rheology.

Oscillatory rheology has been used to determine the structural strength of froth in the previous study.^{5,6} In this study, amplitude sweeps were first performed to determine the linear viscoelastic (LVE) range where the deformation of sample is reversible. In the LVE range, the change of storage modulus G' is subtle, while G' decreases steeply outside the LVE range where the deformation of sample is irreversible. Frequency sweeps were then performed at a strain within the LVE range to make sure that the deformation was reversible. The structural strength of the sample would be inferred through the change of storage modulus (G') and loss modulus (G'') with frequency.

Figure 4a,d presents the amplitude sweeps for coal froth concentrates generated with different concentrations of Q38 or quartz added to the purified coal. The changes of G' and G''

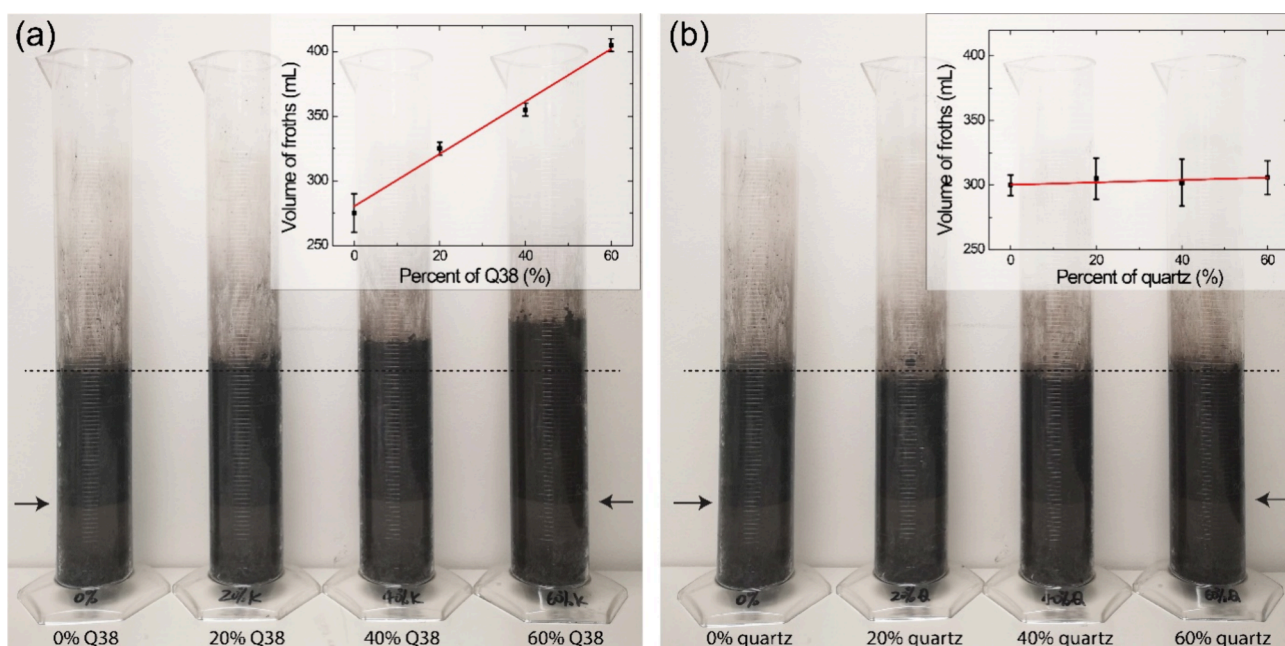


Figure 2. Settling of coal concentrates generated with 0, 20, 40, and 60 wt % of Q38 (a) or quartz (b) added in flotation. The interface between coal froth and water was labeled by black arrows. The volumes of coal froths floating on top of water were plotted as a function of Q38 or quartz concentration, which was inserted in the figure.

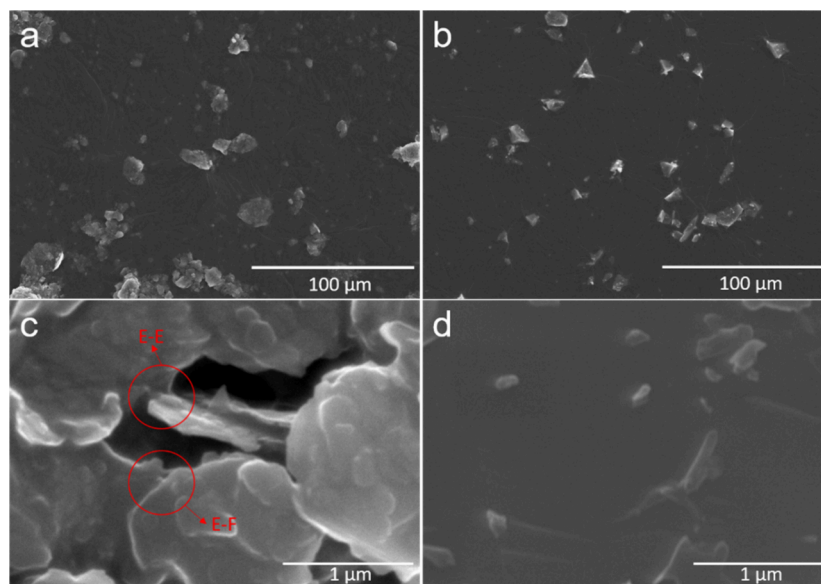


Figure 3. Scanning electron micrograph of Q38 (a, c) and quartz (b, d) at different magnifications.

with strain were all the same for all samples. The LVE range was defined through the dashed lines in Figure 4a. The LVE range for all samples was within the strain of 0.1%, which was called yield strain. As shown in Figure 4a,d, the change of G' was gentle in the LVE range, while outside the LVE range, the downward slope of G' increased. G'' had the tendency to increase along with oscillation strain, but the change was very subtle. G' was much larger than G'' . The value of $\tan \delta$ ($\tan \delta = G''/G'$) was less than 0.1 in the LVE range, which illustrates that most of the energy during deformation was stored other than dissipated. Based on this, it could be inferred that coal froth concentrates were solid-like and had rigidity.^{6,21,22} For coal froth concentrates generated with Q38 additions, the G' increased obviously from 237 to 442 Pa with Q38. Meanwhile,

for coal froth concentrates generated with quartz additions, G' only increased subtly from 270 to 341 Pa with quartz.

The frequency sweeps of froths generated with Q38 and quartz additions are shown in Figure 4b,e, respectively. The frequency sweeps were performed in the LVE range, where shear strain was 0.02%. The changes of G' and G'' were all the same for all samples. G' increased gently with an increase in frequency, and G'' had the tendency to decrease, but on the whole, the frequency only had a small impact on the changes of G' and G'' . G' was still much larger than G'' , and $\tan \delta$ was less than 0.2. The properties of network structures in froth would be inferred based on the change of G' and G'' in frequency sweeps.⁵ The value of G' at high or low frequency had a subtle difference, which shows that the network structures in coal

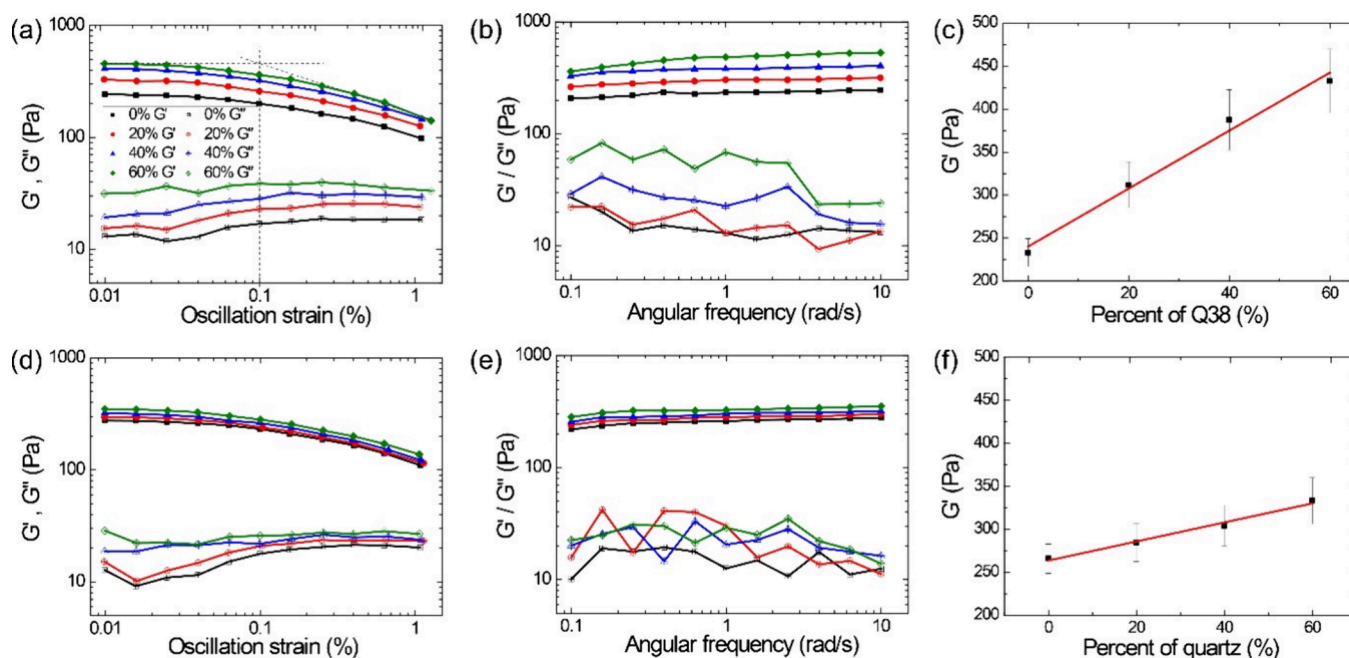


Figure 4. Oscillatory rheology results of coal froths generated with Q38 (a–c) or quartz (d–f) additions. The storage (G') and loss (G'') moduli were recorded in amplitude sweeps (a, d) and frequency sweeps (b, e). The three black dash lines in panel (a) defined the linear viscoelastic (LVE) range. G' and G'' were presented with solid and hollow syllables, respectively. The average of G' in LVE range was plotted as a function of Q38 or quartz concentration (c, f).

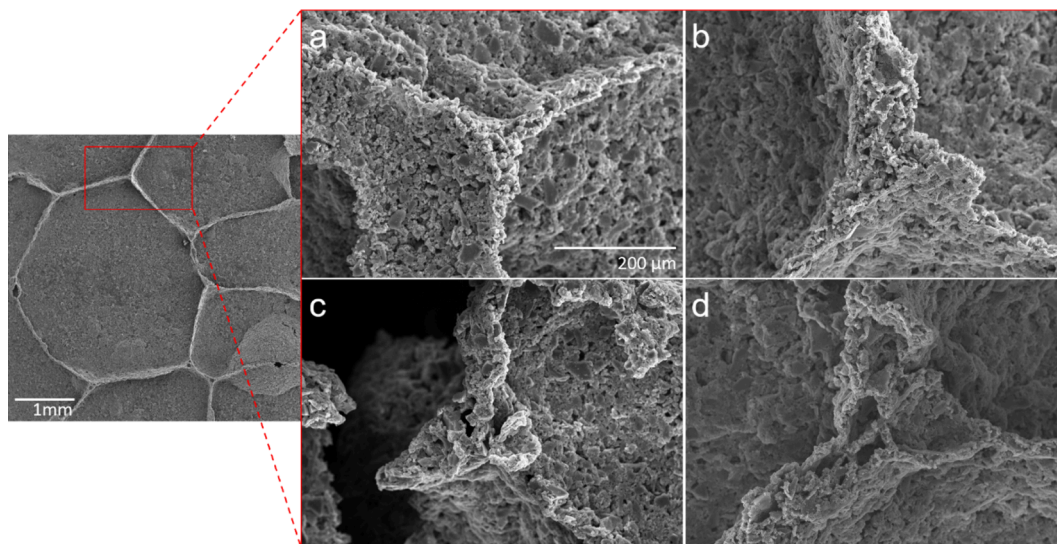


Figure 5. SEM images of Plateau borders for coal froths generated with the addition of 0 wt % (a), 20 wt % (b), 40 wt % (c), and 60 wt % (d) Q38. The figure in the right side showed the panorama of the Plateau border in a large scale.

froths were strong enough to resist the changes caused by external forces, so coal froths were physically stable. The value of G' increased with the increase of Q38 or quartz concentration, the same trend observed in amplitude sweeps. G' increased obviously with the addition of Q38 but gently with the addition of quartz.

The average values of G' in the LVE range in amplitude sweeps for froths generated with Q38 and quartz additions are presented in Figure 4c,f, respectively. The slope of the fitting line was 3.4 in Figure 4c for Q38 and 1.1 in Figure 4f for quartz. The influence of Q38 on G' of coal froths was three times more than that of quartz. The structure strength would be proportionally related to the value of G' , so the increased

value of G' represented the increase of structural strength in froths.^{6,12} It would be concluded that the entrained Q38 in coal froths strengthened the froths to a large extent, while the entrained quartz imposed less influence on the structural strength of coal froths. This would explain the settling results as shown in Figure 2. The strengthened structure of coal froths by entrained Q38 led to their increased stability, which resulted in an increased volume of froths floating on top of water in settling. While the entrained quartz also increased the structural strength of froths, the effect was too weak to show up in settling results.

3.3. Characterization of Froth Structures by SEM. As mentioned that kaolinite and quartz is hydrophilic and

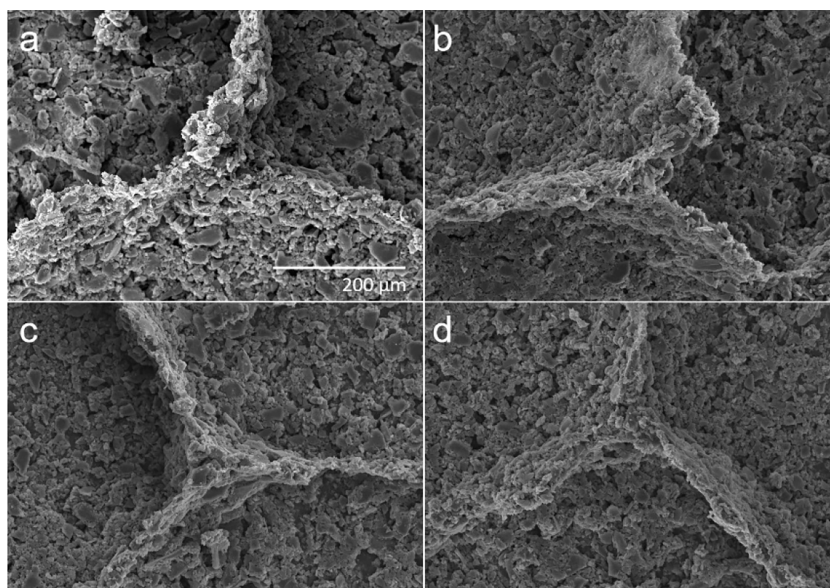


Figure 6. SEM images of Plateau borders for coal froths generated with 0 (a), 20 (b), 40 (c), and 60 wt % (d) quartz.

negatively charged in fresh water at pH 7.4,^{12,23} so the most likely place they may stay in froths would be Plateau borders.²⁴ In this way, they might affect the properties of froths by influencing the structures of the Plateau borders. Therefore, the structures of Plateau borders for froths generated with Q38 or quartz additions were observed through SEM in this study.

Figure 5 presents the Plateau borders of froths generated with the addition of 0, 20, 40, and 60 wt % Q38 in flotation. Plateau borders were the intersections of three bubbles, and the angle for each section was around 120°. When Q38 was not added in flotation, the size of Plateau border was about 50 μm (Figure 5a). With the addition of Q38, more and more particles accumulated in the area of Plateau borders, and the width of Plateau borders increased to 140, 150, and 220 μm with 20, 40, and 60 wt % Q38 as shown in Figure 5b–d, respectively. With the increase of the width, the structure of Plateau borders became complex. Based on Figure 5b–d, it can be seen that Plateau borders were not all stuffed by particles. There were some cavities in Plateau borders, and the cavities were more and more obvious with the increase of the Q38 amount in froth. In this way, with the increase of the amount of Q38, network structures were generated in Plateau borders.

Because of the low percent of Q38 entrained compared with that added in flotation (Table 1), the particles in the Plateau borders were almost all coal particles. Although Q38 particles with a size of several micrometers were hardly found in Plateau borders, it could be inferred that the clay platelets could form E–E and E–F aggregations between coal particles in Plateau borders based on the structure of Q38 in Figure 3c, which generated the network structure in Plateau borders.

The structures of the Plateau borders for coal froths produced with quartz additions are shown in Figure 6. There was little difference for the Plateau borders of coal froths generated with different quartz concentrations. The angle for each section in Plateau borders was around 120°. The sizes of Plateau border were all skinny, around 50 μm. No particles accumulating in Plateau borders, and there was no network structures in Plateau borders either. It can be concluded that quartz had no influence on the structure of Plateau borders.

The problem in settling caused by ultrastable froths is attributed to the structure of coal froths as shown in Figure 7.

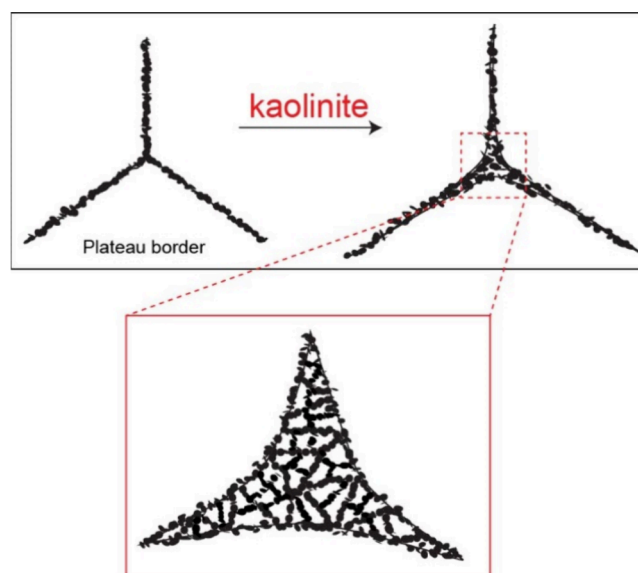


Figure 7. Effect of kaolinite on the structure of the Plateau border in coal froth.

The structure of coal froths affects the drainage of water and thus has an influence on the stability of coal froths.^{25,26} Plateau borders are the interconnected network in froths for liquid flow, and most of the water in froth is in the Plateau border.^{27,28} The drainage of water in froth affects its stability, and high drainage rate is detrimental to the froth stability.²⁹ Based on the SEM images, it was obvious that Q38 changed the structure of the Plateau borders. The size of Plateau borders increased, and network structure was generated with the entrainment of Q38. Therefore, Plateau borders were the root for the effect of Q38 on the settling of coal concentrates.

The presence of Q38 and the formation of a network structure in Plateau borders increased the viscosity of this area. The drainage depended on the geometry and surface chemistry

of Plateau border.^{30,31} For the drainage in foam, the Boussinesq number (Bo) is a criterion to assess the liquid velocity in the Plateau border. The liquid velocity was inversely proportional to Bo .³¹

$$Bo = \frac{\mu_s}{\mu R} \quad (1)$$

where μ_s is the shear interfacial viscosity, R is the radius of the circular arcs in Plateau border, and μ is the shear viscosity of bulky fluid. Based on eq 1, the drainage velocity was inversely proportional to μ_s .

The introduction of Q38 in coal froth increased the shear interfacial viscosity μ_s , and so the drainage velocity was reduced. The effect of particles was not considered in foam, but for froths, the formation of network structure by particles in the Plateau border blocked the water channel and retarded the drainage. As a result, the froth stability increased.

Overall, the reasons for the increased volume of froths that floated on top of water in settling of coal flotation products with the presence of kaolinite could be concluded as that the formation of network structures in the Plateau borders strengthened the structure of froths and retarded the drainage in froths.

4. CONCLUSIONS

The effects of kaolinite and quartz on the stability of coal froths and settling of coal flotation products was investigated. The entrainment of Q38 in coal froths increased the volume of froths that floated on top of water in settling from 275 to 405 mL, while the effect of quartz on it was negligible, which was 300–307 mL. The results of rheology that G' increased from 237 to 442 Pa with the increase amount of Q38 added illustrated that Q38 strengthened the structure of froths. SEM images directly illustrated the effect of Q38 on the structure of the Plateau borders. Based on the results, the mechanism of how kaolinite affects the settling of coal concentrates was obtained. Network structures were formed in the Plateau border under the effect of kaolinite, and the structure of the Plateau border was more complicated with more kaolinite entrained. The network structure in the Plateau border increased the structural strength of froths; besides, the viscosity in the Plateau border increased, which decreased the drainage velocity. In addition, the network structure in the Plateau border blocked the drainage channels and retarded drainage. As a result, the addition of kaolinite increased the stability of the froths, which made the volume of froths that floated on top of water increase in settling.

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Notes

The authors declare no competing financial interest.

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