Effect of Nanobubbles on the Slime Coating of Kaolinite in Coal Flotation

Panwu Li, Ming Zhang,* Wang Lei, Wei Yao,* and Rong Fan

ABSTRACT: Understanding the coating behavior of fine gangue slimes in the presence of nanobubbles (NBs) is important for the application of NB technology in flotation. In this study, slime coating of kaolinite in the flotation of a low-ash coal using deionized (DI) water and NB water was investigated. Kaolinite was found to depress coal flotation by the formation of coating on coal surfaces, but its deleterious effect was less pronounced in the NB water with mitigated slime coating. Setting tests, Brunauer–Emmett–Teller surface area measurements, and dynamic light scattering were conducted to understand the underpinning mechanism. In comparison with DI water, the degree of kaolinite aggregation was enhanced in the NB water. The intensified self-aggregation of kaolinite platelets which appears to be induced by the presence of NBs reduces the solid–liquid interfacial area as well as the number of free kaolinite particles in the suspension, mitigating the coating of kaolinite on coal surfaces in NB water flotation.

1. INTRODUCTION

Clay minerals present many challenges in the flotation of a large range of sulfides, phosphates, and coal in that they can dilute the concentrate through mechanical entrainment because of their small particle sizes, cause high pulp viscosity with poor flotation hydrodynamics, and coat the valuable mineral surface resulting in low-flotation recovery. Coating of clay slimes on mineral surfaces in froth flotation has been extensively studied over the past several decades, and the following two main mechanisms are proposed: (1) electrostatic attraction between clay and mineral surfaces when they are oppositely charged and (2) van der Waals attraction, which is irrespective of the sign of the net surface charge of the minerals. Plate-like clay particles expose two different surfaces: the basal planes (silica tetrahedral face and alumina octahedral face) and the edges. The basal planes carry a permanently negative charge arising from isomorphic substitutions, whereas the sign and density of the charges at the edge surface depend on the pH of the system. Peng and Zhao noted that the anisotropic charging behavior on edges and basal faces allows clay slime coating on a range of positively and negatively charged mineral surfaces. Nevertheless, Oats et al. showed that clay slimes could coat the coal surface even when they carried the same sign of charges. At a close distance (<5 nm), the van der Waals attraction was strong enough to overcome the repulsive double layer forces, resulting in a net attraction between coal and clay particles. Li et al. adopted density functional theory calculations to interpret the mechanisms of clay coating in coal flotation and found that the van der Waals force governs the interaction between coal model molecules and kaolinite particles.

Nanobubbles (NBs), in recent years, have gained great interest in forth flotation because of to their unique properties, such as the large surface area and high aqueous stability. When introduced to the flotation system, NBs can rapidly attach to the hydrophobic surfaces of valuable minerals and enhance the probability of bubble–particle adhesion by promoting the aggregation of fines and ultrafines through the capillary effect and also by acting as adherable nuclei to conventional flotation-sized bubbles, resulting in an increase in the flotation efficiency and recovery. While the role of NBs in the flotation of valuable minerals has been well studied, how NBs interact with fine gangue materials such as clay minerals and further alter the coating behavior of fine gangue slimes in flotation remains unclear. Zhou et al. showed that NBs generated by hydrodynamic cavitation enhanced the aggregation of hydrophobilized ultrafine scheelite particles (<10 μm) in suspension. Another study by Zbik and Horn found that similar to hydrophobic particles, the presence of NBs on the edges of clay crystals may cause hydrophilic clay particles...
particles in study investigated the entrainment behavior of kaolinite found that enhanced entrainment of kaolinite particles in NB been demonstrated that in a aggregates of clay particles can be formed.22 Our previous particles in coal fl fi figure 1. PSD of pure coal and pulverized kaolinite. Ne-grained clay particles and hence their coating behavior. Kaolinite is a typical gangue mineral and is commonly found in coal, oil sands, and a wide range of industrial ores. It is a nonswelling 1:1 structured clay with low chemical reactivity, but capable of initiating slime-coating problems in flotation.21,24 Given the widespread occurrence of kaolinite in various ore deposits, improving the understanding of the coating behavior of kaolinite in the presence of NBs is important for applying the NB technology in flotation practice. In this study, we investigate the effect of NBs on the association and subsequent slime coating of fine kaolinite particles in coal flotation. To our best knowledge, the experimental results are the first of this kind and may provide a valuable reference for applying the NB technology in field flotation systems dealing with other fine gangue slimes.

2. MATERIALS
The coal sample was collected from a coking coal preparation plant in Inner Mongolia, China, and crushed to <0.5 mm. The crushed sample was then dry-screened to collect <0.25 mm particles for flotation tests. The overall ash content of the <0.25 mm fraction was 8.39 wt %, and this fraction was hereafter referred to as "pure coal" in this study.

A high-purity kaolinite sample was obtained from Jiangsu Province, China, and pulverized once received. The phase purity of the kaolinite sample was confirmed by quantitative X-ray diffraction analysis, which shows that the sample contains 98.8 wt % kaolinite and 1.2 wt % quartz. Particle size distributions (PSDs) of the pure coal and pulverized kaolinite were measured by laser diffraction (Malvern Mastersizer 2000). The PSD results, as shown in Figure 1, illustrate the fine nature of the pulverized kaolinite with D50 (cumulative particle size at 50% distribution) and D90 being 2.42 and 10.16 μm, respectively. In comparison, the coal particles are considerably coarser with D90 being 212 μm.

Kerosene and 4-methyl-2-pentanol (MIBC, 99%, Energy Chemical) were used in flotation tests as a collector and a frother, respectively. Deionized (DI) water and NB water were used in parallel in the study. Pure water was produced using a UPY water purification system (UPY-III-103, Chengdu Ultrapure Technology Co., Ltd., Sichuan, China) with a conductivity of 18.2 MΩ cm.

3. EXPERIMENTAL SECTION
3.1. Generation and Characterization of NBs. NBs were generated by pumping water through a micro–nano-bubble nozzle (model BT-50, Riverforest Corporation, California, USA) based on the hydrodynamic cavitation method.18,25,26 The NB generation system is schematically shown in Figure 2. To generate NBs, a 20 L container was filled with 18 L of DI water, which was circulated through the nozzle at a high speed by a water pump (model 3YU60A, Dayton, USA), resulting in a swirling flow. The swirling water creates a vacuum inside the nozzle where air is sucked in and subsequently broke into microbubbles and NBs by the dynamic forces within the vortex. The system was operated at a water pressure of 0.2 MPa, a water flow rate of 18 L min⁻¹, and an air flow rate of 0.18 L min⁻¹ for 5 min. The resultant "milky" gas–liquid mixture was kept still for another 5 min allowing microbubbles to disappear.17 Samples were then collected from approximately 2 cm beneath the surface of the liquid using a thoroughly washed 1 mL syringe at a low speed to limit the introduction of air bubbles. Once air bubbles were removed from the syringe, it was immediately submitted to nanoparticle tracking analysis (NTA) for the characterization of NBs. A similar setup has been reported to generate abundant bulk NBs.13

NTA is a relatively new technique that can measure the size and number concentration of particles ranging from 30 to 1000 nm in suspension based on their light-scattering capabilities and Brownian motion. Herein, NanoSight 300 (NS300, Malvern Instruments), an NTA-based instrument, equipped with a blue laser 488 nm and a high-speed sCMOS camera was used and the movie was captured at 25 frame s⁻¹ for 60 s. The camera level and threshold were set at 12 and 4, respectively, and the solution viscosity was 0.9 cP. It should be noted that when measuring the size and number concentration of NBs, pure water was used in the container before cavitation (Figure 2) instead of DI water because NTA is not capable of distinguishing between NBs and the impurities in DI water. All

Figure 1. PSD of pure coal and pulverized kaolinite.

Figure 2. Schematic of the experimental setup for the NB generation system.
aggregates. After being naturally dried in air, the remaining μsuspension could be separated from the coal process, the suspended unattached kaolinite particles in the used in this study was 10.16 and rinsed mildly with each water type. As D90 of the kaolinite predominantly distributed in the size range of 100–200 μm with a maximum size of 32.0 μm with a maximum size of 32.0 μm. From the screening analysis, the kaolinite was dispersed in a beaker with 350 mL of DI water or NB water by stirring with a magnetic stirrer for 5 min, and the resultant kaolinite suspension was washed into a 500 mL pulp. The pulp was premixed for 5 min and then conditioned successively with kerosene (180 g t−1) and MIBC (150 g t−1) for 2 min each. After conditioning, flotation was operated at an air flow rate of 0.17 m3 h−1, and the speed of the impeller was kept constant at 1800 rpm. In flotation, five concentrates were collected after cumulative times of 10, 30, 60, 120, and 180 s. Flotation concentrates and tailings were filtered, dried at 80 °C, and weighed. The ash contents of dried samples were analyzed for combustible recovery calculation.

3.2. Flotation Test. Flotation of the pure coal with and without kaolinite using DI water and NB water was conducted on a 0.5 L XFD lab-scale single-flotation cell. For each test, 40 g of pure coal alone or 30 g of pure coal with the addition of 10 g of kaolinite was transferred to the flotation cell which was then filled with DI water or NB water to make up the flotation pulp. The pulp was premixed for 5 min and then conditioned successively with kerosene (180 g t−1) and MIBC (150 g t−1) for 2 min each. After conditioning, flotation was operated at an air flow rate of 0.17 m3 h−1, and the speed of the impeller was kept constant at 1800 rpm. In flotation, five concentrates were collected after cumulative times of 10, 30, 60, 120, and 180 s. Flotation concentrates and tailings were filtered, dried at 80 °C, and weighed. The ash contents of dried samples were analyzed for combustible recovery calculation.

3.3. SEM Analysis. Scanning electron microscopy (SEM) analysis was conducted using a Hitachi SU8010 field emission scanning electron microscope. The pure coal was wet-screened to obtain the particles in the size fraction of 74–125 μm. 3 g of the screened coal was then mixed with 1 g of kaolinite in 50 mL of DI water or 50 mL of NB water separately for 10 min using a magnetic stirrer, allowing the slime coating to occur. The resultant suspensions were subjected to a 74 μm screen and rinsed mildly with each water type. As D90 of the kaolinite used in this study was 10.16 μm with a maximum size of 32.0 μm (Section 2), it was expected that during the screening process, the suspended unattached kaolinite particles in the suspension could be separated from the coal–kaolinite aggregates. After being naturally dried in air, the remaining materials on the screen were ready for SEM analysis. This SEM sample preparation technique was adopted from Yu et al. Prior to examination, the SEM sample was first mounted onto a metal stub with a double-sided adhesive carbon tape, followed by the sputtering of gold.

3.4. Settling Test. The association of kaolinite particles under different solution conditions can be reflected by their settling behaviors. Batch settling tests on 6 wt % kaolinite suspensions were conducted at natural pH. For each test, 30 g of kaolinite was dispersed in a beaker with 350 mL of DI water or NB water by stirring with a magnetic stirrer for 5 min, and the resultant kaolinite suspension was washed into a 500 mL self-made graduated cylinder with 150 mL of corresponding water. The cylinder was turned upside down four times to obtain a uniform suspension. Once the cylinder was placed on a solid, flat surface, the settling test commenced with no further disturbances permitted. After settling for a given time length, the top 250 mL suspension was taken out via a valve in the wall of the cylinder and dried at 80 °C to measure the mass of remaining solids. The results of the settling tests were recorded as settling efficiency (E), which is defined as

\[
E = \frac{m - m_0}{m} \times 100\%
\]

where \(m\) and \(m_0\) represent the mass of solids in the top 250 mL suspension before and after settling, respectively.

3.5. Characterization of Kaolinite Particles. The specific surface area of the kaolinite treated with different solutions (DI water vs NB water) was determined by the multipoint nitrogen adsorption–desorption measurements [Brunauer–Emmett–Teller (BET) method] at the temperature of liquid nitrogen (−196 °C), with an ASAP 2460 surface area and porosimetry analyzer (Micromeritics Instrument Corporation, USA). Treatment of kaolinite with DI water and NB water was done by adding 6 g of kaolinite to 100 mL

Figure 3. Signal graph of NBs generated by hydrodynamic cavitation (left) and the concentration distribution of NBs as a function of NB size (right).

Figure 4. Cumulative combustible recovery as a function of flotation time in the flotation of pure coal with and without the addition of kaolinite in DI water (A) and NB water (B).
of each water separately. After mixed with a magnetic stirrer at room temperature for 5 min, the suspensions were filtered and the residues were then dried in an oven at 50 °C for 24 h. The final products were used for BET surface area measurements. It should be mentioned that although nitrogen condensates in pores formed by the aggregation of clay particles, the nitrogen-BET surface area measurement can provide useful information on the external specific surface of nonswelling clay minerals such as kaolinite and illite.\textsuperscript{32}

The hydrodynamic size distributions of kaolinite particles in DI water and NB water were measured with a dynamic light scattering (DLS) instrument (Malvern Zetasizer Nano ZS90). Its size range limit is 0.3 nm–5 μm in diameter. Fine kaolinite particles (<5 μm) were obtained by the sedimentation method developed by Mekhamer et al.\textsuperscript{33}

4. RESULTS AND DISCUSSION

4.1. Flotation Performance. Figure 4 shows the flotation results of pure coal with and without the addition of kaolinite using DI water and NB water. In the DI water system (Figure 4A), compared to the baseline flotation in the absence of kaolinite, the addition of kaolinite reduced the overall combustible recovery from 73.68 to 60.07% with a significant drop of 13.61%. The depression of coal flotation by kaolinite may be due to coating of hydrophilic kaolinite slimes on the coal surfaces, which reduced the natural hydrophobicity of coal and inhibited the coal particles from direct contact with collectors and/or air bubbles.\textsuperscript{34,24}

While using DI water, the presence of 25% kaolinite in the feed was detrimental to coal flotation, the negative effect of kaolinite on coal flotation was less pronounced in the NB water. As shown in Figure 4B, when flotation tests were conducted with NB water, addition of a same amount of kaolinite reduced the overall combustible recovery from 79.51% (in the absence of kaolinite) to 70.91% and the gap was 8.60%. While using the DI water, the depressed coal flotation in the presence of kaolinite was attributed to the slime coating of kaolinite on coal surfaces, and the mitigated impact of kaolinite on coal flotation performance using NB water may result from the altered coating behavior of kaolinite in the NB water, which is studied in the next section.

4.2. Slime Coating. SEM analysis has been widely used to observe the slime coating phenomenon in flotation, and the preparation of SEM samples which are normally flotation concentrates or tailings involves filtration and drying.\textsuperscript{3,28} It has been pointed out that during these two processes, the natural sedimentation of abundant fine clay particles suspended in water in the flotation products could lead to the "false attachment" of clay particles onto coal surfaces, rendering the observed slime coating questionable.\textsuperscript{5,28} The sample preparation procedure described in Section 3.3, was first proposed by Yu et al.,\textsuperscript{28} and it was noted in their study that this well-designed technique successfully avoided the defect arising from the traditional filtration and drying.

Figure 5 shows the SEM images of coal–kaolinite aggregates formed in the DI water and NB water. It is evident that slime coating of fine kaolinite on the coal surface occurred in DI water (Figure 5A). Similar observations have also been reported by Yu et al.\textsuperscript{28} and by Wang and Peng.\textsuperscript{35} Thus, it is safe to conclude that slime coating of kaolinite contributed to the depressed coal flotation in DI water (Figure 4A). The occurrence of kaolinite coating on coal particles could be driven by the van der Waals attraction between coal and clay particles.\textsuperscript{9} The interaction energy between these two types of particles has previously been calculated by Yu et al.,\textsuperscript{28} and it was concluded that the external kinetic energy input to the coal–kaolinite suspension provided by agitation can enable particles to overcome the energy barrier, aggravating kaolinite coating. On the other hand, the extent of kaolinite coating on the coal surface appears to be less severe in the NB water than in the DI water (Figure 5B). As seen in Figure 5, while a considerable volume of individual kaolinite platelets as well as their aggregates accumulated on the coal surface in the DI water, they were found to be scarcely scattered on the coal surface in the NB water. This is consistent with the flotation results and confirms that the coating behavior of kaolinite was indeed modified in the NB water.

4.3. Association of Kaolinite Platelets in DI Water and NB Water. In our previous study, it was found that NBs exacerbated the entrainment of kaolinite in flotation because of the variation in particle association.\textsuperscript{3} The difference in the coating behavior of kaolinite in the DI water and NB water observed in this study may also be related to various clay particle associations. To prove this, setting tests, BET surface area measurements, and DLS were employed to examine the effect of NBs on the association of kaolinite particles.

Figure 6 shows the settling efficiency of kaolinite particles as a function of settling time in DI water and NB water. The settling efficiency increased with increasing settling time for both water types. What is more, compared to settling in the DI water, the efficiency in the NB water was observed to be smaller, and their difference became progressively larger over settling time. For instance, after 20 min of settling, the efficiency of the same kaolinite sample was 71.1% in the DI water, compared to 65.3% in the NB water. The lower settling efficiency for the NB water implies the formation of porous kaolinite aggregate structures in the suspension. These microporous aggregates trapped more intraaggregate water inside and therefore tend to settle more slowly.\textsuperscript{36} The formation of porous kaolinite clusters in NB water may be
induced by the presence of NBs. The role of NBs in clay aggregate formation has been previously proposed in Zbl and Horn where a slower settling of kaolinite particles was observed in CO$_2$-saturated water compared with degassed water.

To further characterize the kaolinite particles as well as their association (aggregates) under different solution conditions, BET surface area measurements were conducted, and the results are summarized in Table 1. In general, the results of BET surface area measurements were in good agreement with the results of settling tests. For DI water, the specific surface area, pore volume, and mean diameter of the kaolinite sample were 25.0 m$^2$ g$^{-1}$, 11.6 $\times$ 10$^{-2}$ cm$^3$ g$^{-1}$, and 18.5 nm, respectively. Pulverized kaolinite used in this study had a higher specific surface area than the refined natural kaolinites, which are generally in the range of 10–20 m$^2$ g$^{-1}$, as noted by Rouquerol et al. Compared to DI water, treatment with NB water at room temperature resulted in a reduced specific surface area of the kaolinite sample, 20.3 m$^2$ g$^{-1}$. This may be attributed to increasing self-aggregation of kaolinite particles in the presence of DI water. It has been reported that during calcination, the kaolinite specific surface area decreased as a result of the aggregation of kaolinite particles. Moreover, when treated with NB water, the pore volume and the mean diameter of the same kaolinite sample were increased, which were 14.1 $\times$ 10$^{-2}$ cm$^3$ g$^{-1}$ and 27.3 nm, respectively, further indicating that aggregates of high porosity were formed in the NB water.

The changing association of kaolinite particles under different solution conditions (DI water vs NB water) was also illustrated by the hydrodynamic size distribution results (Figure 7). It is evident that kaolinite appears to be greater in size in the NB water than in the DI water. For instance, the size distribution of the same kaolinite in the NB water was centered at 955 nm, approximately 1.34 times greater than that in the DI water (712 nm), revealing a promoted aggregation of kaolinite at 955 nm, approximately 1.34 times greater than that in the DI water. For instance, the size distribution curve of the same kaolinite sample to greater diameters when the DI water was replaced by the NB water was not caused by the size contributions of NBs in the NB water, otherwise the size distribution of NBs plus kaolinite should be centered between 190 and 712 nm (Figure 7). The increased degree of aggregation of kaolinite platelets when the DI water was replaced by the NB water could be related to an increase in the hydrophobicity of kaolinite particles in the presence of NBs. Previous studies have indicated that while the alumina octahedral face and edge surfaces of kaolinite are hydrophilic, the silica tetrahedral face surface has a moderate level of hydrophobicity. Moreover, Saada et al. measured the adsorption of asphaltene on three kaolinites of different origins and revealed that only 25–28% of the kaolinite surface is hydrophilic. It is believed that in the NB water where the number density of NBs was sharply increased after 5 min of cavitation (Figure 3), a considerably large number of NBs would be attached to the (moderately) hydrophobic surfaces of kaolinite, rendering these surfaces more hydrophobic. Driven by the long-range hydrophobic attractive force between the hydrophobic surfaces originating from the coalescence of NBs, kaolinite platelets may attach to their neighboring particles, forming three-dimensional clusters. Previous research using cryo-SEM revealed a coagulated spanning network of E–E linked kaolinite particles possibly resulting from the coalescence of NBs on clay crystals. In our previous study, it was reported that the apparent viscosity of kaolinite suspension was increased in the presence of bulk NBs, which also suggested the formation of network structures.

A model was proposed comparing the coating behaviors of two coal/kaolinite systems in the DI water and in the NB water (Figure 8). For coal flotation with the addition of fine kaolinite using the DI water, as shown in Figure 8A, slime coating of kaolinite on the coal surface is formed, possibly due to the van der Waals attraction between coal and clay particles. As a result, the hydrophobicity of the coal surface is decreased. In contrast, Figure 8B shows an advantageous variation in the association of kaolinite particles induced by the presence of NBs, where the degree of kaolinite aggregation is enhanced in the NB water in comparison to the DI water. This means that more of the kaolinite particles are trapped in clay aggregate structures with reducing number of free clay grains in the system, resulting in less free clay particles as well as a lower total clay surface area for slime coating on the coal surface to occur. In a previous study, Tao et al. found that several commercial clay binders were able to agglomerate clay particles
with enlarged apparent size, reduce their surface area, and remove coal surface slime coating to restore its hydrophobicity. From the model (Figure 8), it can be seen that NBs act as a binder for fine kaolinite in coal flotation, which is responsible for the less affected coal flotation in the NB water.

It is worth noting that in addition to slime coating, the consumption of flotation reagents by fine clay particles could contribute to the affected coal flotation performance.\(^1\) When this is the case, because of the promoted kaolinite aggregation in NB water, as revealed in this section, clay clusters with larger apparent sizes and lower specific surface area are expected to consume less reagents. Hence, it is reasonable to suggest that by initiating clay aggregation, NBs can reduce not only slime coating but also potential reagent consumption by kaolinite. Continued work is needed to better understand the role of reagent consumption by clay minerals in coal flotation using NB water.

5. CONCLUSIONS

In this current study, NBs were produced by hydrodynamic cavitation and their effect on the coating behavior of kaolinite particles in coal flotation was investigated. It was found that, compared to DI water, the coating of kaolinite on the coal surface and its detrimental effect on coal flotation were less significant in NB water. Owing to the enhanced self-aggregation of kaolinite particles, which may be induced by the presence of NBs, the solid–liquid interfacial area as well as the number of free kaolinite particles in the suspension reduced, and the extent of coating of kaolinite on the coal surface was mitigated accordingly. Although further testing on other types of fine gangue materials and at an industrial scale is still needed, the results of this study may help advance the application of NB technology in froth flotation.

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Notes

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