Effective Separation of High-Ash Fine Coal Using Water Containing Positively Charged Nanobubbles and Polyaluminum Chloride

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ABSTRACT: The coal industry is facing the challenge of treating high-ash fine coal. In this study, we proposed an effective method to handle high-ash fine coal using water containing positively charged nanobubbles (PCNBs) and polyaluminum chloride (PAC). For comparison, normal nanobubble (NB) water was tested in parallel. Flotation results of a modeled high-ash fine coal showed that compared to the use of NBs alone, an enhanced combustible recovery with a simultaneous reduction in ash recovery was obtained when using water containing PCNBs and PAC. Particle size distribution together with particle video microscopy (PVM) and the degree of entrainment analysis were conducted to understand the underpinning mechanism. It was found that the presence of PCNBs intensified the aggregation of fine coal particles, which accounted for the boosted combustible recovery. It was interesting that PAC could disrupt coal flocs induced by NBs, leading to the release of trapped kaolinite particles with alleviated clay recovery by entrapment.

1. INTRODUCTION

With depleting ore resources and increasing demand for high-quality coal worldwide, the coal industry has started to process increasing amounts of low-grade coal ores using froth flotation, which is based on the difference in the surface hydrophobicity of coal and gangue minerals. To fully liberate coal from the mineral impurities in low-grade coal, fine grinding is required. As a result, a large amount of fine and ultrafine particles is generated in the low-grade feed for flotation. Because of the fine size and high ash content of such a feed, the collision probability between the fine coal particles and bubbles is decreased, whereas the nonselective entrainment recovery of fine mineral impurities, such as clay, is exacerbated.1,2 In the literature, many studies have attempted to improve the efficiency and selectivity in the flotation separation of high-ash fine coal. Wang and Peng3 reported that clayey fine coal flotation using saline water remarkably increased the combustible recovery from 58% in freshwater to 89% in saline water while also increasing the recovery of unwanted mineral matter by 34%. Fu et al.4 tried reverse flotation of kaolinite from high-ash fine coal using N,N-dimethyl dodecyl amine (DRN12) as a kaolinite collector and found that the beneficiation of kaolinite from the raw coal with DRN12 was effective only in a highly acidic pulp (pH = 3). Polyaluminum chloride (PAC), a cationic coagulant widely used in the water purification process was first introduced by Liang et al.5 to reduce clay entrainment in high-ash coal flotation. Their results showed that PAC successfully reduced kaolinite entrainment, but with the trade-off of lower combustible recovery.5

In recent years, nanobubbles (NBs) have gained much interest in froth flotation. Bulk NBs generated in pure water are negatively charged.6 They can rapidly attach to the hydrophobic mineral surfaces and thereby enhance the flotation efficiency and recovery by promoting the aggregation of fine and ultrafine mineral particles through capillary effect and by acting as nuclei for conventional flotation-sized bubbles to adhere.7−9 Li et al.10 revealed that the use of the water containing NBs increased the combustible recovery because of the increased apparent size of fine coal particles in the presence of NBs, while simultaneously increasing the entrainment recovery of clay particles to the concentrate. In the same study, PAC was then introduced to NB water flotation, and it was found that while the direct addition of PAC to NB water did alleviate the high kaolinite entrainment, this method also reduced the combustible recovery because PAC had a detrimental effect on the surface hydrophobicity of coal particles.10 Clearly, these reported strategies were either not

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practical or had the common shortcoming of not being able to achieve high recovery of fine coal and low gangue entrainment at the same time. There is an urgent need for improved approaches to separate high-ash fine coal.

While the role of NBs in mineral flotation has been increasingly well established, positively charged NBs (PCNBs), which can be obtained by adding salts and ionic surfactants to the NB generator, have shown great potential in wastewater treatment. Bui and Han\(^{11}\) attributed the improved decolorization rate of dark green Rit dye using positively charged NBs (PCNBs) to the electrostatic attraction between the negative dye components and positive NBs, the surface charge of which was reversed by adding coagulant Al\(_2\)(SO\(_4\))\(_3\) to the NB generation system. Moreover, Xia et al.\(^{12}\) reported that positively charged microbubbles enhanced the flotation recovery of fine low-rank coal. In this study, we propose to separate high-ash fine coal using water containing PCNBs and PAC. Given the ability of PAC to reduce clay entrainment and the negative surface charge of coal at a neutral pH,\(^{5,10}\) it was expected that NBs would become more capable of enhancing the flotation recovery after reversing the charge and a decent concentrate grade could be maintained in the presence of PAC.

2. EXPERIMENTAL SECTION

2.1. Materials and Reagents. A coking coal sample was collected from a coal washing plant in Inner Mongolia, China. It was crushed and ground to obtain a fine coal powder sample. The ash content of the ground coal sample was 8.36 wt %, which is hereafter referred to as “clean coal” in this study. High-purity kaolinite lumps (>95%) were obtained from Jiangsu province, China, and pulverized once received. The particle size distribution of the clean coal and pulverized kaolinite was determined using laser diffraction (Malvern Mastersizer 2000), and their D50 was found to be 57.90 and 5.01 μm, respectively.

PAC (Al\(_2\)O\(_3\) ≥ 28%) in powder form was supplied by Aladdin Industrial (China). PAC solution (3 wt %) was prepared with deionized water at room temperature before being introduced to an NB generator to produce PCNBs or flotation as a clay depressant. During flotation, kerosene and octanol were used as collector and frother, respectively.

2.2. Preparation of NBs and PCNBs. To compare the flotation performances of NBs and PCNBs, NBs were first generated using a commercialized micro-nanobubbles generator (LF-1500, Xingheng Technology, China) based on the decompression method.\(^{13,14}\) To do this, 1 L of tap water was circulated through the NB generator at the water pressure of 0.4 MPa and airflow rate of 0.02 L/min for 10 min (Figure 1). The resultant “milky” solution was left still for another 3 min, allowing microbubbles to escape. The nanoparticle tracking analysis results (Malvern Nanosight NS300) showed that NBs generated in pure water following this procedure have a mean diameter of 222.7 nm and a number concentration of 2.7 × 10\(^8\) mL\(^{-1}\).

PCNBs were produced by preadding a known amount of PAC solution to the NB generator before starting the experiment. The following generation procedure was the same as that for NBs. The ζ-potential of the resulting NBs was measured by a ζ-potential analyzer (Malvern Zetasizer Nano ZS90) and compared to that of NBs generated without PAC (Figure 2). While pure NBs were negatively charged at pH > 4, the presence of PAC (10 mg/L) shifted the ζ-potential curve positively and the absolute value of their charges decreased with increasing pH. A similar observation of NBs in the presence of Al\(^{3+}\) or Fe\(^{3+}\) (1 mM) was previously reported by Bui et al.\(^{14}\) To explain this, the authors\(^{14}\) illustrated the species that could be formed in the Al\(^{3+}\) or Fe\(^{3+}\) solution in relation to pH and found that positively charged [Al\(^{3+}\)] and [Fe\(^{3+}\)] were predominant at low pHs. They concluded that it was their adsorption that caused higher positive ζ-potential values of PCNBs at lower pH values.\(^{14}\) PAC is a cationic coagulant that is composed of three groups of intertransformable Al species, namely, monomer Al\(_\nu\) rapid reaction polymer Al\(_g\) (mainly including Al\(_{13}\) (Al\(_{13}\)O\(_{42}\)(OH)\(_{24}\))\(^{1+}\)), and slow reaction colloid Al\(_c\).\(^{15,16}\) Wang et al.\(^{16}\) investigated the effect of solution pH on the speciation transformation of PAC by Ferron assay and observed a rapid decrease in the positively charged Al\(_\nu\) and Al\(_c\).
species in the alkaline pH region. Hence, the decrease in ζ potential with the solution pH in the case of PAC could be explained by the decreasing dominance of Al₅ and Al₆ species at higher pHs, and the results reveal the adsorption of positively charged Al₅ and Al₆ at the NB interface and thus the formation of PCNBs in the solution.11

2.3. Flotation Test. Batch flotation tests were conducted with a 0.5 L XFD laboratory-scale flotation cell at nature pH with no pH modifier added. Tap water, NB water, and PCNB water containing both PCNBs and PAC were used in parallel in this study. To model high-ash, fine real coal, a total mass of 40 g of clean coal and kaolinite mixture (at a mass ratio of 3:1) was added to the flotation cell, which was then filled with each water to make up the flotation pulp.12 The mixture was premixed for 4 min and conditioned successively with kerosene (180 g/t) and octanol (150 g/t) for 2 min each. After conditioning, flotation was operated at the airflow rate of 4 L/min and constant impeller speed of 1500 rpm. In flotation, four concentrates were collected after cumulative times of 30, 60, 120, and 180 s. To compare the effects of PAC addition before and after NB generation on flotation, a fourth group, referred to as “NB water + PAC,” where PAC solution was added to the pulp (10 mg/L) in the first minute of the premixing stage in the flotation using NB water, was investigated, and the remaining was kept the same. The flowchart of the flotation tests carried out under different solution conditions is illustrated in Figure 3.

![Figure 3. Batch flotation flow sheet for coal-kaolinite mixture.](image)

2.4. Particle Video Microscope (PVM). The interaction between fine coal particles in different water environments was examined with a PVM system (Mettler-Toledo ParticleView V19). Dilute coal dispersions were prepared by adding 0.5 g of coal samples (<54 μm) to 0.5 L of tap water, NB water, and PCNB water, respectively, and then mixed by a magnetic stirrer for 5 min. For each test, the beaker with the corresponding coal slurry was placed on a lifting platform with agitation (600 rpm), and the PVM probe was immersed in approximately two-thirds of the height of the dispersion.17 During testing, enlarged images of the observed field were transmitted to the computer in real time for 13 min, and the relative backscatter index (RBI) of each image was calculated by the iC PVM software.18 RBI is defined as

\[ \text{RBI} = \frac{\text{image brightness}}{\text{intensity of light input}} \] (1)

RBI decreases as the particles get finer and as the concentration of particles increases, i.e., image brightness decreases.

3. RESULTS AND DISCUSSION

3.1. Flotation Performance. Figure 4 shows the cumulative combustible recovery as a function of flotation time from the flotation of clean coal—kaolinite mixture using different waters with and without the addition of PAC. Compared to the flotation using tap water, the use of NBs alone increased the overall combustible recovery from a baseline of 54.2−67.1% in NB water. The beneficial effect of NBs on coal flotation has been widely acknowledged in the literature.20−22 However, after adding PAC to NB water, the ability of NBs to enhance coal flotation was compromised, as suggested by the relatively lower cumulative combustible recoveries in the group of NB water + PAC. A similar decline in combustible recovery using NB water after PAC addition has previously been reported by Li et al.,10 who attributed it to the reduced surface hydrophobicity of coal in the presence of PAC. Interestingly, the same dosage of PAC (10 mg/L), when added to tap water prior to the generation of NBs (PCNB water) rather than being directly added to NB water, not only abrogated the detrimental effect of PAC on coal flotation but further boosted the overall combustible recovery by 11.0−78.1% as compared with the use of NB water alone.

Ash recovery, in addition to the combustible recovery, was accessed to justify the flotation performance using different waters (Figure 5). It can be seen that after the completion of 180 s of flotation, the ash recovery increased from 16.9% in tap water to 23.4% in NB water. Given the fine and hydrophilic nature of kaolinite particles and the low ash content of the clean coal, the majority of the mineral matter (ash) recovered should be via entrainment.23 Exacerbated entrainment recovery of kaolinite particles using NB water was also identified in our previous study.24 The negative effect of NBs on ash recovery was mitigated by the addition of PAC to NB water as the overall ash recovery dropped back to 16.6%. The effect of PAC on reducing the concentrate ash content in the flotation separation of coal from kaolinite has previously been reported in a couple of published studies.25 In contrast, in PCNB water, where PAC was added to tap water prior to NB generation, the efficiency of the same amount of PAC in reducing concentrate ash content was reduced as the overall
ash recovery rose to 20.6%. This was likely due to the consumption of PAC during the formation of PCNBs. Nevertheless, compared to the use of NBs alone, the combination of PCNBs and PAC (PCNB water) had a less deleterious effect on the concentrate grade while still being more beneficial for the combustible recovery. In fact, the concentrate ash content in PCNB water was comparable to the lowest ash content obtained in the case of NB water + PAC, being 9.13 and 9.15 wt %, respectively. This can be explained by the most significant enhancement of combustible recovery by PCNBs, which dominated their detrimental effect on ash recovery.

3.2. Effect of PCNBs and PAC on the Interaction between Coal Particles. While in the previous research, the enhanced combustible recovery in coal flotation using water containing NBs was attributed to the intensified aggregation of fine coal particles induced by NBs,\textsuperscript{10,21} the boosted combustible recovery using PCNB water could also be related to a modified interaction between coal particles. To justify this, the particle size distribution of clean coal in different waters with and without the addition of PAC was measured, and the results are shown in Figure 6. As can be seen that when tap water was replaced by NB water, the size distribution of the same coal sample shifted to the right and the D90 increased to 265.66 μm. This may be due to the accommodation of NBs onto coal surfaces, and the hydrophobic attraction originating from the bridging of NBs causes more coal particles to aggregate.\textsuperscript{21} More importantly, the D90 of the coal sample was further increased to 312.69 μm when immersed in the PCNB water, the highest in the four different water solutions, suggesting the most intensified aggregation of coal particles in the presence of PCNBs. While it was revealed that PCNBs are positively charged at neutral pH by the \(\zeta\)-potential measurement, the coal surface was noted to be negatively charged at the same pH.\textsuperscript{5,10} The electrostatic attraction might therefore facilitate the adsorption of PCNBs onto the coal particle surfaces; this, in turn, further promoted coal aggregation through the capillary effect.\textsuperscript{12,25} It is interesting that after the addition of PAC to the NB water in which coal particles were added and mixed beforehand, the presence of PAC shifted the size distribution curve in NB water to the left, indicating that a portion of the initially formed coal aggregates was disrupted by PAC. The disruption of coal aggregates by adding PAC to NB water was further justified by the RBI trend of −54 μm clean coal before and after PAC addition shown in Figure 7. Note that PAC was added at the time point of 4 min. RBI can qualitatively monitor the changes of particle size and concentration in a suspension in real time.\textsuperscript{26} It is clear from Figure 7 that for coal particles (aggregates) in NB water without the addition of PAC, their value of RBI remained stable during the entire 13 min of testing (mixing) period, suggesting that the coal aggregates induced by NBs were stable enough to sustain agitation (600 rpm). By contrast, in the NB water + PAC group, a sharp decline in the value of RBI from approximately 75−60 was observed in the first 2 min after PAC addition, after which the value of RBI decreased gradually and linearly to approximately 50 at the end of the experiment. A higher RBI is associated with larger particle size, whereas an increase in the concentration of particles, due to the reduction in particle size, decreases the RBI values.\textsuperscript{19} Here, the sharp
decrease in the RBI values after the addition of PAC is believed to display the disruption of coal aggregates by PAC, which dissociates coal particles from their aggregates and objectively increases the concentration of fine particles.

On the other hand, the effect of PCNBs on the aggregation of fine coal particles was also illustrated by comparing the PVM morphologies of −54 μm clean coal in different waters (Figure 8). It was observed that while the fine coal particles were widely dispersed in tap water, they appeared to be in more aggregated form in NB water. This coincided with the particle size distribution results in Figure 6. More importantly, the fine particles in PCNB water were largely agglomerated with greater aggregate size than those in NB water, confirming that PCNBs are more capable of initiating the aggregation of coal particles than NBs. Similar visual observation for a coarser coal sample (−125 + 250 μm) immersed in water containing positively charged microbubbles was previously reported by Xia et al.12

3.3. Effect of PCNBs and PAC on the Degree of Entrainment of Mineral Matter. The degree of entrainment is known as ENT, which uses water as a reference to define the classification effect of the drainage of entrained particles in the froth phase.23 To better understand the effect of PCNBs and PAC on the entrainment behavior of mineral matter, ENT of the four concentrates of the batch flotation using different water was calculated on an unsized base using the following equation27

\[
\text{ENT}_{\text{con:tail}} = \frac{M_{\text{gangue}}}{M_{\text{water}}} \frac{M_{\text{water}}}{M_{\text{gangue}}} \text{con} \text{tail}
\]

(2)

where \(M_{\text{gangue}}\) and \(M_{\text{water}}\) represent the mass of free gangue and water, respectively (g).

Since the added fine kaolinite was the predominant mineral matter in the feed and liberated in flotation, \(M_{\text{gangue}}\) was estimated by the mass of total ash in the concentrates and tailing.23

ENT of the four concentrates of the flotation of clean coal–kaolinite mixture under different solution conditions is shown in Figure 9. As can be seen, the ENT values in the four different water solutions decreased with increasing flotation time. In a previous study, Wang et al.28 investigated the variation of ENT of quartz or hematite gangue with time in the flotation of chalcopyrite and found that the ENT values for both gangue minerals decreased with flotation time with a trend similar to that observed in this study. More importantly, it is evident from Figure 9 that the presence of NBs increased the ENT over the flotation process as compared to the baseline flotation in tap water. In particular, the ENT of the first two concentrates increased from around or below 1 in tap water to above 1 in NB water. An ENT greater than 1 means that in addition to entrainment, there could be the entrapment mechanism that contributed to the reporting of mineral matter to the concentrate.5,29 It has been demonstrated by both size distribution and PVM results that NBs accelerated the aggregation of coal particles in NB water. Hence, in flotation using NB water, the intensive aggregation of coal particles may lead to the undesirable entrainment of fine kaolinite particles, the predominate mineral matter in the feed, within the flocs or aggregates of hydrophobic coal particles with higher ash recovery (see Figure 5).5

While the use of NBs alone exacerbated the entrainment of mineral matter, accompanied by entrainment, PAC reduced the elevated entrainment using NBs. As shown in Figure 9, the ENT in NB water decreased sharply after the addition of PAC. It has been reported that the presence of PAC may facilitate the selective aggregation of kaolinite particles, and thus reducing their entrainment in coal flotation.5,10 However, it should be noted that the reduced ash recovery in the NB water + PAC group shown in Figure 5 could be partially attributed to the disputation of coal aggregates/flocs by PAC addition as suggested by the size distribution and PVM results, which releases the trapped fine kaolinite particles in coal flocs and thus reduces their chance of being recovered via entrainment.
This is supported by the fact that the value of ENT for the first two concentrates in NB water flotation declined from well above 1 without PAC to close to or below 1 after PAC addition. In the meantime, the use of PCNBs led to similar aggravated entrainment of the mineral matter when compared to the baseline flotation. Nevertheless, the ENTs in PCNB water were always lower than those in NB water without PAC over the entire flotation process, suggesting that excluding those consumed by the formation of PCNBs, the remaining PAC species in PCNB water were still effective in reducing the entrainment of mineral matter.

CONCLUSIONS
The current study found that water containing PCNBs and PAC can be easily prepared by preadding PAC to an NB generator. Flotation of a modeled high-ash fine coal using this water further enhanced the beneficial effect of NBs on the combustible recovery with a simultaneous reduction in ash recovery as compared to using pure NB water. While the former was attributed to an intensified aggregation of coal particles induced by PCNBs, the latter was evidenced by the reduced degree of entrainment in the presence of PAC. It was also found that PAC could disrupt coal aggregates induced by NBs, resulting in the release of trapped kaolinite particles with lower entrainment. Overall, the PCNBs plus PAC flotation technique is a promising option for the effective separation of high-ash fine coal.

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Notes
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