



Full Length Article

Flotation behavior of raw and oxidized fine coal when mixed with coarse particles



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ABSTRACT

It is well-known that ultra-fine particles, especially those with oxidized surface, are difficult to float using conventional methods. Flotation of oxidized ultra-fine particles has been a serious problem which should be well solved as the demand for energy is increasing. In this investigation, the flotation behaviors of fresh and oxidized fine coal ($< 45 \mu\text{m}$) and the effect of mixing with coarse particles (250–500 μm) in equal proportions were discussed. Zeta potential and contact angle results indicated the surface hydrophobicity of oxidized fine coal was much lower than that of fresh fine coal. When floating alone, yields of fresh, moderately oxidized, deeply oxidized and very deeply oxidized fine coal were 77.88%, 62.36%, 34.66% and 16.67%, respectively. However, the yields for the above four fine coals were increased to 82.83%, 87.04%, 82.72% and 50.07%, respectively when mixed with coarse particles. The yield improvement indicator values for the four fine coals were 4.95%, 24.68%, 48.06% and 33.40%, respectively. Obviously, the yield improvement indicator values for oxidized fine coals were much higher than fresh fine coal. A similar trend was also observed in the experiments without collector. Throughout this research, mixing with hydrophobic coarse coal is proved to be an effective method to improve the flotation behavior of oxidized fine coal.

1. Introduction

Coal, especially ultra-fine particle, is easily oxidized in air [1–4]. The oxidation of coal starts with the physical adsorption of oxygen to form an oxycomplex on the surface of the coal, and then the chemical adsorption of oxygen to form polar phenolic-OH, carbonyls, phenols and peroxide type oxygenated moieties [5–7]. These newly formed functional groups are usually hydrophilic, leading to poor flotation performance of oxidized coal. In addition, oxidation process increases the roughness of coal surface, and produces some scraps and fragments on oxidized coal surface [8]. As a result, oxidized coal is difficult to float with commonly oily collectors [5,9–12].

Grinding pretreatment has been proved to be an effective method to improve the flotation performance of oxidized coal [2,9,13–15]. After grinding, the oxidized surface containing scraps and fragments is removed from coal particle, producing both ultra-fine particles with oxidized surface and coarse particles with newly appearing fresh hydrophobic surface. Coarse particles can be recovered easily. However, ultra-fine particles with oxidized surface are still difficult to be recovered. The recovery of oxidized ultra-fine particles cannot be ignored due to their high proportion. Tan et al. [2] reported that ultra-fine particles are more likely to be oxidized. Xia et al. [16] revealed that

coal particle size decreases after oxidation, resulting in enhanced proportion of oxidized ultra-fine coal. In addition, the crush and attrition effects during dense medium process for separating oxidized lump coal could produce oxidized ultra-fine particles since oxidized coal tends to crumble [17]. Therefore, flotation of oxidized ultra-fine coal has been a serious problem which should be well solved as the demand for energy is increasing.

It is known that attractive forces occur when two hydrophobic surfaces approach each other in water. The strong adhesion of hydrophobic particles can be explained by the formation of cavities or nanobubbles in the interfacial region between hydrophobic surfaces [18]. As reported by Hou et al. [19] and Nalaskowski et al. [20], the hydrophobic force of particle surfaces in water is affected significantly by surface roughness. They thought that surface roughness increases the range of these forces by promoting the formation of vapor or gas cavities between interacting surfaces. Therefore, considering the high surface roughness of oxidized coal, it can be supposed that adhesion between oxidized ultra-fine coal and hydrophobic coarse coal may occur when they are mixed together, which may improve the recovery of oxidized ultra-fine coal by coating hydrophobic coarse particles. Hence, mixing with hydrophobic coarse coal may be an effective method to improve the flotation behavior of oxidized ultra-fine coal.

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Interactions between ultra-fine particles and coarse particles have been reported by many researchers. Liang et al. [21] studied the froth stability and flotation performance of various mixtures generated from coal particles < 74 μm at different density fractions and particles between 250 and 500 μm . They found that the highest overall yield was obtained from the mixture of < 74 μm coal with density between 1.5 and 1.8 g/cm^3 and 250–500 μm coal, which might be because fine moderate hydrophobic particles greatly increased the froth stability. However, Hou et al. [19] showed that ultra-fine particles depressed coarse coal flotation regardless of their density, while the ultra-fine particles with intermediate density had the greatest influence on coarse coal flotation since they could cover coarse particle surface via hydrophobic forces provoking their hydrophobic group interact with coarse particles and their hydrophilic group interact with water. Peng et al. [22] and Tao et al. [23] presented that low-ash ultra-fine particles could promote the detachment of coarse particles from bubbles because of competitive adsorption on frother, but high-ash ultra-fine particles did not show similar impact. Cheng et al. [24] reported that wide particle size range exhibited a better flotation effect than narrow particle size. The interactions between ultra-fine particles with different density fractions and coarse particles during flotation have been extensively investigated. However, the interaction between oxidized ultra-fine particles and coarse coal during flotation is rarely reported. In addition, the supposition about the enhancement of oxidized ultra-fine coal flotation by mixing with coarse particles is not reported yet, which needs to be validated.

In this research, flotation experiments for fresh and oxidized fine coal and hydrophobic coarse coal before and after mixing with each other were conducted. Interaction effects of fine particles with different oxidation degrees and hydrophobic coarse coal were discussed, and a new approach to improve the flotation behavior of oxidized fine coal was put forward.

2. Experimental

2.1. Materials and preparation of the samples

The anthracite coal sample used in this study was clean coal prepared with heavy-medium process, obtained from No. 2 Coal Preparation Plant in Yangquan, Shanxi Province of China. The ash content of this clean coal sample was 10.09%. The sample was first crushed using a jaw crusher to reduce the particle size to < 1 mm, and then sieved with a 500 μm screen. The > 500 μm particles were ground using a rod mill and sieved again employing the 500 μm screen and this process was repeated until all particles passed through the 500 μm screen. All the < 500 μm particles were mixed together and wet-sieved using a 45 μm screen and the sample with particle size < 45 μm was separated. The > 45 μm particles were sieved using a 250 μm screen to obtain a granulometry of 250–500 μm . The ash content of the two samples, < 45 μm and 250–500 μm are 11.53% and 9.42%, respectively.

Artificial oxidation method is widely applied for producing oxidized coal samples, and H_2O_2 solution is an extensively used agent for coal oxidation in laboratory, however, coal is usually oxidized in air, e.g. in an underground coal mine, or when stored in coal bunkers, or even during long distance transport [1,3]. Therefore, heating in the air is considered to be a more realistic coal oxidation method [8,25]. In this paper, oxidized coal samples with different oxidation degrees were obtained by heating the fine particles (< 45 μm) in an electric thermostatic drying oven at 200 $^\circ\text{C}$ for 20 h, 50 h and 100 h, and labeled moderately oxidized coal, deeply oxidized coal and very deeply oxidized coal, respectively. Coal sample without oxidation was named fresh coal.

Table 1

Proximate analyses of < 45 μm fresh and oxidized coal particles.

Samples	Mad (%)	Vdaf (%)	FCdaf (%)	Ad (%)
Fresh	1.01	7.60	92.40	11.70
Moderately oxidized	1.34	11.12	88.88	11.42
Deeply oxidized	1.48	13.56	86.44	11.31
Very deeply oxidized	1.91	16.78	83.22	11.08

Note: ad = air dry basis; daf = dry ash-free basis; d = dry basis; M = moisture content; V = volatile matter content; FC = fixed carbon content; A = ash content.

2.2. Proximate analysis

Proximate analysis was conducted according to Chinese standard (Proximate analysis of coal, GB/T 212-2008). Procedures are as follows:

Moisture content analysis was conducted in an oven. The oven temperature was first risen to 110 $^\circ\text{C}$, then two samples of 1.0000 ± 0.0010 g were put into the oven and heated for 1.5 h. The samples were taken out, weighed, and then reheated in the oven for further 30 min period until any change in mass did not exceed 0.0010 g. The average value of the two moisture contents was used for experimental analysis.

Ash content analysis was conducted in a muffle. Two samples of 1.0000 ± 0.0010 g were put into the muffle. The temperature was risen to 500 $^\circ\text{C}$ within 30 min, and kept for 30 min, then risen to 815 $^\circ\text{C}$ and kept for 1 h. The samples were taken out and weighed. The average value of the two ash contents was used for experimental analysis.

Volatile matter content analysis was conducted in a muffle, too. The muffle temperature was first risen to 920 $^\circ\text{C}$, then two samples of 1.0000 ± 0.0010 g were put into the muffle in a short time and then heated for 7 min. The samples were taken out and weighed. The average value of the two volatile matter contents was used for experimental analysis.

2.3. Sink and float separation tests

Sink and float separation tests were conducted in a centrifugal using heavy liquids with various densities prepared by mixing benzene, carbon tetrachloride and bromoform at appropriate ratios. Firstly, divided the representative particles (60.00 g) equally into two portions and put them into two centrifuge tubes. Poured 150 mL of heavy liquid of lowest relative density into each tube. Secondly, mixed the contents of the tubes by stirring and transferred the tubes to the centrifuge holders and rotated for 10 min at a rotational frequency of 2500 r/min. Thirdly, removed mixture of floating solids and heavy liquid from the tubes, carefully avoiding disturbing the sinks material. Filtered the floats, remained the sinks material in the centrifuge tubes and repeated the procedure using the heavy liquid of next higher relative density. When all the relative density levels had been completed, removed the final sinks material and filtered. All the obtained particle fractions were dried in an oven for 5 h at 60 $^\circ\text{C}$ and analyzed for yield and ash content.

2.4. Zeta potential and contact angle measurements

The zeta potential measurements for coal samples were carried out using a micro-electrophoresis apparatus (JS94H, Powereach). First, about 5 g of fine coal sample (< 45 μm) were conditioned in deionized water at natural PH using a magnetic stirrer at 500 rpm for 15 min, and the obtained suspension standing for 30 min made the coarse particles settle down. Then, 0.5 mL of the supernatant was taken out and transferred to the measurement cell by a pipette. Ten measurements for each fine coal sample were performed, and the average value of the ten measurements was obtained. The experiments were carried out at room temperature (25 $^\circ\text{C}$).

Representative fine particles (fresh or oxidized) were sampled for

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