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Grinding flotation of bituminous coal of different oxidation degrees

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ABSTRACT

H_2O_2 solutions of different concentrations were added to a bituminous coal to obtain samples of different oxidation degrees. Flotation and concentrate screening experiments were conducted. It was found that the yield and ash content of the concentrate both decrease after oxidation and particles finer than 0.074 mm are more likely to be oxidized. Grinding flotation experiments were conducted at grinding time of 10, 20 and 30 min. The size distribution and Zeta potential of samples after grinding were detected. The results showed that grinding can effectively improve flotation performance. The optimum grinding time increases with the oxidation degree. And the relative extent of external and internal oxidation changes with the oxidation degree.

1. Introduction

Froth flotation is a method for fine coal separation that depends on the difference of surface hydrophobicity of coal and gangue minerals. Fresh coal particles with hydrophobic surface are easy to float with low flotation reagent dosage but they would become difficult to float when they are oxidized (Pis et al., 1996; Jia et al., 2000; Polat et al., 2003; Wang et al., 2003; Dey et al., 2013; Xia et al., 2013a). The oxidation of coals starts with the physical adsorption of oxygen on the surface to form an oxycomplex, followed by chemical adsorption of oxygen to form polar phenolic-OH, carbonyls, phenols and peroxide type oxygenated moieties (Jia et al., 2000; He et al., 2003; Ge and Li, 2003; Lu et al., 2008; Polat et al., 2003; Dey et al., 2013). These newly formed hydrophilic functional groups lead to poor flotation performance. Coals can be oxidized to different extents according to coal ranks. Low rank coals are more readily to be oxidized (Gutierrez-Rodriguez and Aplan, 1984; Polat et al., 2003; Wang et al., 2003; Zhou et al., 2005; Yu et al., 2010; Xia and Yang, 2013b).

Approaches of overcoming the flotation problem of oxidized coal have been investigated extensively. New collectors, regulators and surfactants are concluded as follows. Cationic collectors and collectors with oxygenated functional groups can enhance the flotation performance of oxidized coals. Sarikaya and Ozbayoglu (1995) used cationic collectors to float oxidized coal. The Zeta potential of oxidized coal was changed from negative to positive and the contact angle increased. Jia et al. (2000, 2002) added oxygenated polar groups to the collector molecule to form hydrogen bonds with the oxygenated surface sites on coal. Regulators could also improve the flotation effect of oxidized coal. Electrolytes, such as barium chloride, chromic chloride and ferric chloride, can decrease the surface potential of coal particles and make the surface less negative at low concentration. Electric double layer is compressed and surface free energy is decreased by adding electrolytes (Sarikaya and Ozbayoglu, 1990; Bolat et al., 1998; Sun et al., 2002). Jena et al. (2008) used aliphatic alcohols, i.e., ethanol and butanol, to deoxidize the coal surface and then added black oil as promoter to enhance the floatability of the oxidized coal. Aliphatic alcohols were also attempted as activator to improve flotation performance of oxidized coal by Chen (2012). Researchers also used surfactants to improve floatability of oxidized coal (Lin et al., 2001; Sun et al., 2002; Dey, 2012). Surfactants can reduce the surface tension of oil and make them disperse into small droplets. The energy required to spread the oil onto the coal surface is decreased. Recently, biodiesel and oxidized diesel oil were also used to improve flotation of oxidized coals (Xia et al., 2013c; Xia and Yang, 2013a).

Pretreatments were also raised to enhance the oxidized coal flotation. As Xia et al. (2013a) has summarized, the methods of pretreatments are grinding, premixing/preconditioning, ultrasound, thermal, microwave and direct contact mixing of the reagents with dry coal before wetting. Among these pretreatment methods, grinding, premixing/preconditioning and ultrasound treatment can remove the oxide layer on the oxidized coal surface (Buttermore and Slomka, 1991; Piskin and Akgun, 1997; Feng and Aldrich, 2005; Sokolovic et al., 2012a, 2012b; Xia et al., 2012a). Thermal and microwave treatments are attributed to the removal of pore water, hydration water and some hydroxyl functional groups (Xia et al., 2013b). The direct contact procedure makes the oxidized coal avoid the prevention of the collectors absorbing on the coal surface and this procedure was successfully proved (Ahmed and Drzymala, 2004, 2012). Xia et al. (2012b) investigated the flotation of oxidized coal dry-ground with collector and found the optimum grinding time to improve the flotation performance.

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Grinding pretreatment to improve the flotation performance of oxidized coal was investigated recently (Xia et al., 2012a, 2012b, 2012c, 2013a). The materials in the study of Xia are oxidized anthracite. Bituminous coal is softer than anthracite and much more ultra-fine particles will emerge after grinding. The ultra-fine coal particles finer than 10 μm are usually difficult to float (Polat et al., 2003; Forbes, 2011). So grinding of oxidized bituminous coal might deteriorate flotation performance due to the increase of ultra-fine particles. In addition, the grinding process is usually at a high temperature (Hadad and Sadeghi, 2012). In this condition, coal samples might be re-oxidized especially for lower rank coals (Wang et al., 2003; Zhang, 2004). This may play a negative part in the grinding flotation of oxidized bituminous coal. In this work, flotation of bituminous coal samples of different oxidation degrees before and after grinding is conducted. It is found that finer particles are more likely to be oxidized. In addition, the relative extent of external and internal oxidation is discussed based on the flotation tests (Sarikaya, 1995).

2. Experimental

2.1. Materials

The flotation feed from Xingtai coal preparation plant was sampled for this study. The proximate and size analyses are shown in Tables 1 and 2.

2.2. Preparation of samples oxidized by H_2O_2 solution

Artificial oxidation methods were applied to obtain oxidized coal samples. H_2O_2 solution and oxidizing acids are extensively used agents for coal oxidation (Liu et al., 2000, 2003; Wang et al., 2004). Oxidation can also be performed in air, in oxygen or even in solutions dissolved of oxygen with or without heating (Phillips et al., 1986; Sarikaya, 1995; Pis et al., 1996; Liu et al., 2000; Lin et al., 2001; He et al., 2003; Wang et al., 2003; Zhou et al., 2003; Yan and Zhang, 2005; Wang et al., 2012; Xia et al., 2014). H_2O_2 solution method is quick and easy to control, so it was used to prepare the oxidized coal samples in this paper.

H_2O_2 solutions of different concentrations were mixed and every 500 mL of them was added to 500 g of bituminous coal sample. The five mixtures were kept being agitated for 24 h at 20 °C. To obtain a very deeply oxidized coal, Sample 6 added with H_2O_2 solution of 30% was agitated for 24 h at 80 °C. The detailed treatment of samples is listed in Table 3. The properties of the coal samples after treatment have changed. The proximate analysis of coal samples before and after oxidation is shown in Table 4. The moisture and volatile increase from Samples 0 to 6, while the ash content decreases. This can be explained by the newly formed oxygenated groups of the coal particles.

Table 1
Proximate analysis of coal sample.

M_{ad} (%)	V_{ad} (%)	FC_{ad} (%)	A_{ad} (%)
1.46	20.92	52.79	24.83

Table 2
Size analysis of coal sample.

Size fraction (mm)	Rate (%)	Ash content (%)	Accumulative rate (%)	Accumulative ash content (%)
0.5–0.25	9.75	5.20	9.75	5.20
0.250–0.125	13.04	6.59	22.79	5.99
0.125–0.074	14.16	10.86	36.95	7.86
0.074–0.045	8.74	17.06	45.69	9.62
−0.045	54.31	38.23	100.00	25.16
Total	100.00	25.16	–	–

Table 3
Samples obtained by different treatment.

Name of samples	Concentration of H_2O_2 solution (%)	Temperature of agitation (°C)
Sample 0	No addition	No agitation
Sample 1	0 (tap water)	20
Sample 2	3	20
Sample 3	9	20
Sample 4	15	20
Sample 5	30	20
Sample 6	30	80

2.3. Grinding flotation

Dry grinding of the oxidized samples was conducted in a laboratory rod mill at grinding times of 10, 20 and 30 min. The size distribution of Sample 0 and Zeta potential of all coal samples after different grinding times were measured by a laser particle size analyzer and Zeta potentiometer. Flotation experiments before and after grinding were all conducted in a XFD flotation cell of 1.5 L. N-dodecane and 2-octanol were used as the collector and frother. The dosages were 1200 and 200 g/ton of coal, respectively. The impeller speed of flotation cell was 1900 r/min. The aeration rate was 0.25 m^3/h. Each sample of 90 g was first added into tap water in flotation cell and agitated for 2 min. Then the collector was added and the pulp was conditioned for another 2 min. After that the frother was added and an additional 30 s of conditioning was kept. Then 3 min of flotation was kept. The concentrates and tailings were filtered and then dried in an oven for 5 h for the preparation of analysis. The concentrates of flotation before grinding were screened to investigate the oxidation of particles of different sizes.

3. Results and discussion

3.1. Flotation result before grinding

Fig. 1 shows the flotation results of seven coal samples of different oxidation degrees. The yield and ash content of concentrate both reduce

Table 4
Proximate analysis of coal samples before and after oxidation.

Name of samples	M_{ad} (%)	V_{ad} (%)	FC_{ad} (%)	A_{ad} (%)
Sample 0	1.46	20.92	52.79	24.83
Sample 1	1.48	21.01	52.68	24.83
Sample 2	1.49	21.36	52.50	24.65
Sample 3	1.54	21.77	52.27	24.42
Sample 4	1.65	21.81	52.23	24.31
Sample 5	1.77	21.88	52.06	24.29
Sample 6	2.12	22.74	51.35	23.79

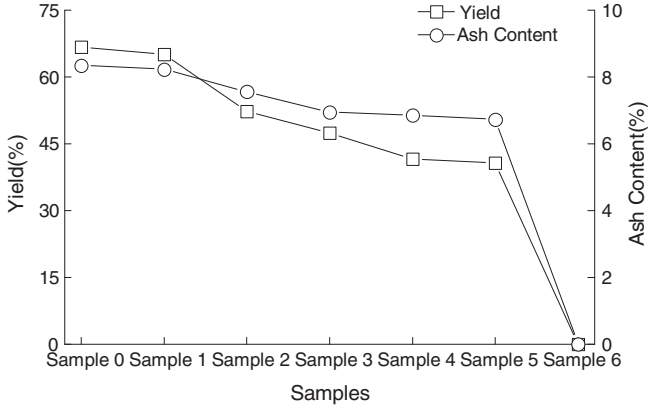

Fig. 1. Flotation result before grinding.