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Effect of pre-wetting time on oxidized coal flotation

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ABSTRACT

In this investigation, we discussed the flotation behavior of oxidized coal treated by different pre-wetting times. The pre-wetting time is 1 min, 2 min, 3 min, 4 min and 5 min, respectively. X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and contact angle measurements were used to indicate the surface properties of oxidized coal. XPS results show that the hydrophobicity of oxidized coal is very low and the oxidized coal should be pre-wetted quickly as there are many hydrophilic functional groups and few hydrophobic functional groups on the oxidized coal surface. In addition, there are also many cracks and holes on the oxidized coal surface from SEM pictures. The cracks and holes should be covered or filled up with water during pre-wetting process. Contact angle measurement results show that the contact angle between the water drop and oxidized coal surface decreases rapidly with the increase of measurement time. Both of the combustible matter recovery and concentrate ash content decrease with the increase of pre-wetting time.

1. Introduction

Oxidized coal is usually difficult to float using the oily collectors [1–8]. In China, Taixi oxidized coal is also difficult to float through our previous studies [9–15]. However, Taixi oxidized coal can achieve a good flotation recovery after grinding pretreatment [10–12]. At the same time, the surface attrition has also been used to improve the floatability of waste (oxidized) coal [7,8]. The flotation regents usually play a very important role in flotation of oxidized and low rank coals [1,2,4,5,14–17]. In addition, the floatability of oxidized or low rank coals can be enhanced by pretreatments, such as microwave treatment, heat treatment and premixing treatment [6,13,18,19].

However, the effect of pre-wetting time on coal flotation has not been well researched. The pre-wetting time may have a significant effect on the flotation of Taixi oxidized coal. Piskin and Akgun considered that a short premixing time, just 1 minute, could remove the oxidized layer on the coal surface and also improve the floatability of oxidized Amasra coal [6]. A long premixing time had a negative effect on the flotation behavior of oxidized coal. It might be that a long premixing time reduced the floatability of Amasra coal because a long premixing time could pre-wet the oxidized coal completely. In flotation pulp, the hydration shell on the oxidized coal surface may be much thicker at a longer premixing or pre-wetting time than that at a shorter premixing or pre-wetting time.

In this investigation, we discussed the flotation behavior of oxidized coal treated by different pre-wetting times. The pre-wetting time was 1 min, 2 min, 3 min, 4 min and 5 min, respectively. X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and contact angle measurements were used to indicate the surface properties of oxidized coal. XPS, SEM and contact angle measurements results will indicate why the oxidized coal is not suitable for long pre-wetting times. The difficult-to-float reason for Taixi oxidized coal will be explained. And also, the effect of pre-wetting time on the flotation of oxidized coal will be discussed.

2. Experimental method and procedure

2.1. Materials

The oxidized coal samples were provided by Taixi Coal Preparation Plant in China. The coal mine was oxidized heavily due to the spontaneous combustion. The unoxidized coal cannot be obtained due to this coal mine which has been oxidized completely. The coal samples were screened to pass 0.5 mm firstly and then dry ground in a laboratory rod mill to get 90% coals pass 74 micron. The proximate analysis of coal samples can be shown as: $Mad = 5.75\%$, $Vad = 7.64\%$, $FCad = 66.50\%$, $Aad = 20.11\%$, $St = 0.60\%$. Where *Mad* is the moisture content, *Vad* the volatile content, *FCad* the fixed carbon content, *Aad* the ash content, and *St* is the total sulfur content.

2.2. X-ray photoelectron spectroscopy

For the indication of surface properties of Taixi oxidized coal, the oxidized coal samples were pressed into the plate. The XPS experiments were carried out at room temperature in an ultra high vacuum (UHV) system with the surface analysis system (ESCALAB 250Xi, America). The base pressure of the analysis chamber during the measurements was lower than 1.0×10^{-9} mbar. Al Ka radiation ($h\nu = 1486.6$ eV)

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from a monochromatized X-ray source was used for XPS. For all analyses, the take-off angle of the photoelectrons was 90° and the spot size was 900 μm. The spectra of survey scan were recorded with the pass energy of 100 eV; the energy step size was 1.00 eV. High resolution spectra were recorded with the pass energy of 20 eV, and the energy step size was 0.05 eV. The data processing (peak fitting) was performed with XPS Peak fit software. The binding energies were corrected by setting the C1s hydrocarbon (—CH_2—CH_2— bonds) peak at 284.6 eV.

2.3. SEM analysis

The FEI Quanta 250 SEM was used to analyze the surface morphology of oxidized coal. The magnification time was fixed at 2000, 4000, 10,000 and 20,000, respectively. The oxidized coal samples were prepared by surface cleaning using absolute ethyl alcohol. After surface cleaning, the coal samples were dried in air. Before SEM, the coal samples were sputter-coated with a layer of gold. The details operating parameters of SEM were as follows: HV was 30.00 kV; WD was 20.2 mm; Pressure was $1.21e^{-4}$ pa; Spot was 2.5.

2.4. Contact angle measurements

The oxidized coal was firstly pressed into the plates. The plates of oxidized coal were measured using water contact angle analyzer (JC2000D), such as a water droplet on the surface of coal plate in air. The contact angles were obtained at different measurement times as a water droplet contacted on the coal plates. The measurement time ranged from 0 s to 20 s. The measurement time of 0 s meant that the water droplet contacted with the coal plate at the exact moment.

2.5. Pre-wetting and flotation processes

In this investigation, the oxidized coal was pre-wetted at a flotation cell and the impeller speed of flotation machine was 1910 rpm. The pre-wetting time was 1 min, 2 min, 3 min, 4 min and 5 min, respectively. After the pre-wetting process, the collector with the dosage of 5 kg/t coal was added into the flotation pulp and the pulp was conditioned for 6 minutes. At last, 2-octanol frother (1 kg/ton) was added and the pulp was conditioned for another 1 minute. The flotation tests were conducted in a 1.5 L XFG flotation cell using 100 g of coal. The airflow rate was 2.5 L/min. The flotation concentrate was analyzed using the indexes: combustible matter recovery and concentrate ash content. Eq. (1) was used to calculate the combustible matter recovery:

$$\text{Combustible matter recovery}\ (\%) = [M_C(100-A_C)/M_F(100-A_F)] \times 100 \quad (1)$$

Where M_C is weight of the concentrate (%), M_F weight of the feed (%), A_C the ash content of the concentrate (%), and A_F is the ash content of the feed (%).

3. Results and discussion

3.1. XPS results

The binding energies corresponding to appropriate peaks in coals are as follows:

C1s: Peaks at binding energies of 285.3 eV, 284.6 eV, 286.1 eV, 287.6 eV and 289.1 eV are corresponding to the following groups: C—C or C—H, C C, C—O (alcohol, phenol or ether), C O (carbonyl or chinone) or O—C—O (in low rank coals) and COO^- (carboxyl) [20–22].

The C1s peaks are fitted as shown in Fig. 1. The relative contents of carbon forms on the surface of oxidized coal are shown in Table 1. The

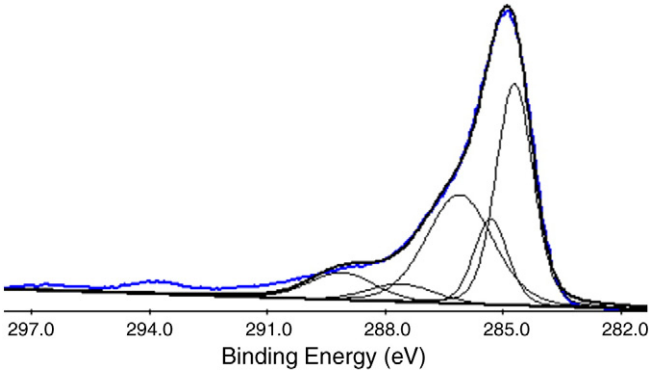

Fig. 1. C1s peaks for oxidized coal surface.

content of C—C and C—H is 13.87% which is very low compared with the content of C C, 40.03%. The C C group is usually more hydrophilic than the C—C and C—H groups. The content of oxygen-containing functional groups, such as C—O, C O and COOH, is 46.10%. The C—O, C O and COOH groups are the most hydrophilic functional groups in the coal. It can be concluded that the oxidized coal has many hydrophilic functional groups, such as C O, C—O and COOH. However, there are few hydrophobic functional groups, such as C—H and C—C. The hydrophilic functional groups will be bonded with the water by hydrogen bond and the oxidized coal surface will be covered by a thick hydration shell if the oxidized coal is pre-wetted in the flotation pulp. Therefore, the oxidized coal is difficult to float. The pre-wetting time should have a significant effect on oxidized coal flotation.

3.2. SEM results

As shown in Fig. 2, the surface morphology of oxidized coal is very rough with lots of cracks or holes. The cracks are evenly distributed. It indicates that these cracks are not man-made. The crack size is primarily between 1 and 3 micrometers. Taixi oxidized coal samples were oxidized due to the spontaneous combustion at a higher temperature. The oxidation processes of this oxidized coal should include the processes of the oxidation and pyrolysis. During the pyrolysis, the volatile component releases out from coal particles. Furthermore, coal particles may expand at high temperatures and contract when they are cooled. Therefore, the cracks are produced in this process.

3.3. Contact angle results

Fig. 3 shows the contact angle of oxidized coal decreases quickly with the increase of measurement time. The contact angle of oxidized coal is about 0 while the measurement time is as short as 5 s. There were lots of cracks and holes on the oxidized coal surface as shown in Fig. 2, so the water may be attributed to the infiltration of water into the cracks. While the oxidized coal is pre-wetted in flotation pulp, the coal surface will be wrapped by the hydration shell which makes the oxidized coal difficult to contact with the oily collectors and bubbles. It

Table 1
Relative content of carbon forms on oxidized coal surface (relative % of C1s).

Carbon forms	C—C, C—H	C C	C—O	C O	COOH
Content (%)	13.87	40.03	29.95	6.17	9.98