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**EFFECT OF SURFACE OXIDATION IN AIR AND WATER ON HYDROPHOBICITY
AND FLOATABILITY OF A BITUMINOUS COAL****WPŁYW UTLENIANIA POWIERZCHNI WĘGLA W POWIETRZU I WODZIE NA WŁAŚCIWOŚCI
HYDROFOBOWE I ZACHOWANIA WĘGLA BITUMICZNEGO W TRAKCIE FLOTACJI**

Fresh bituminous coal was stored in air-/water-oxidation conditions after 20 and 80 days, respectively. FTIR results show that the Hydrophilicity Index (HI) value of air-oxidized coal is higher than that of water-oxidized coal. SEM results show that the surface roughness of water-oxidized coal is higher than that of air-oxidized coal and water-oxidized coal surface has more holes and chips than air-oxidized coal surface. Flotation results show that the floatability of water-oxidized coal is worse than that of air-oxidized coal. The flotation performance of both air-/water-oxidized coals can be improved at larger collector dosages. The air-oxidation processes changed the HI value of coal greater than the water-oxidation processes while the water-oxidation processes changed the surface morphology of coal greater than the air-oxidation processes. Both the changes in HI value and surface roughness of coal determine the flotation behavior of oxidized coal. The changes in the surface morphology of coal particles after oxidation processes may be the primary factor determining the floatability of coal particles while the changes in the HI value of coal particles may be the inferior factor.

Keywords: Fresh coal; Oxidized coal; Flotation; Floatability; FTIR; Hydrophilicity Index

Świeży węgiel bitumiczny przechowywano w warunkach sprzyjających procesom utleniania, w obecności wody i powietrza, odpowiednio przez 20 i 80 dni. Badania metodą spektroskopii w podczerwieni z transformatą Fouriera (FTIR) wykazały że wskaźnik hydrofilności (HI) węgla podlegającego utlenianiu w obecności powietrza jest wyższy niż w przypadku węgla poddanego oddziaływaniu wody. Wyniki badań z użyciem mikroskopu elektronowego wykazują, że powierzchnia węgla który uległ utlenieniu pod wpływem powietrza i wody wykazuje większą ilość nierówności i szczelin niż powierzchnia węgla poddanego oddziaływaniu powietrza. Badania procesu flotacji wskazały, że flotacyjność węgla podlegającego utlenianiu pod wpływem wody jest wyższa niż w przypadku węgla poddanego oddziaływaniu powietrza. W przypadku obydwu węgli poddawanych oddziaływaniu powietrza i wody, ich flotacyjność

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można poprawić poprzez zwiększenie dozowania w dużych kolektorach. Procesy utleniania w obecności powietrza prowadzą do zmiany wskaźnika HI węgla w większym stopniu niż ma to miejsce w przypadku węgla poddanego działaniu wody; z kolei utlenianie w obecności wody w znacznie większym stopniu wpływa na strukturę powierzchni węgla. Zarówno zmiany wartości wskaźnika HI jak i nierówności powierzchni w dużej mierze określają zachowanie węgla w trakcie procesu flotacji. Zmiany w strukturze powierzchni cząstek węgla po procesie utleniania mogą okazać się być głównym czynnikiem decydującym o zachowaniu węgla w trakcie procesu flotacji; z kolei wydaje się że zmiany wartości wskaźnika HI dla cząsteczek węgla mają znaczenie drugorzędne.

Słowa kluczowe: świeży węgiel, węgiel po utlenianiu, flotacja, flotacyjność, spektroskopia w podczerwieni z transformatą Fouriera (FTIR), wskaźnik hydrofilności

1. Introduction

Bituminous coal has natural hydrophobicity and floatability and is easy to float with common oily collectors in coal preparation plants (Xia et al., 2018). However, bituminous coal is easy to be oxidized in natural conditions, such as in raw coal bunkers, in open coal yards as well as in coal tailings refuse ponds. After oxidation, the hydrophobicity and floatability of bituminous coal will reduce, resulting in the difficulty in recovering fine oxidized coals during flotation processes with common oily collectors (Dey, 2012; Dey et al., 2013; Wang et al., 2013; Xia et al., 2017). For instance, the weathering and oxidation processes usually increase the number of oxygen containing functional groups, whereas decrease the number of hydrophobic functional groups on coal surface, as a result the wettability of coal surface increases due to the weathering and oxidation processes (Fuerstenau et al., 1983; Pilawa et al., 2002; Pietrzak et al., 2005; Grzybek et al., 2006; Sokolovic et al., 2006). In addition, the surface morphology of coal can be also changed after the oxidation processes. The surface roughness of bituminous coal will increase after the weathering processes, and many fragments are produced on the oxidized coal surface and these fragments primarily consist of inorganic materials, resulting in lower floatability of oxidized coal (Xia & Yang, 2014; Xia et al., 2014).

Researchers have paid much attention to achieve an effective flotation of oxidized coals. In the literature, ultrasonic treatments (Kang et al., 2008), grinding (Song & Tras, 1997; Tan et al., 2015), high intensity conditioning (Piskin & Akgun, 1997; Feng & Aldrich, 2005) and surface attrition pretreatments (Sokolovic et al., 2012a; Sokolovic et al., 2012b) are used to remove the oxidized layers from oxidized coal surface in order to improve coal surface hydrophobicity. In addition, some effective flotation collectors/reagents are used to enhance the floatability of oxidized coals (Attia et al., 1993; Jia et al., 2000; Eisele & Kawatra, 2002; Jena et al., 2008; Xia & Yang, 2013). For example, Jia et al. (2000) used the improved Tetrahydrofurfuryl (THF) butyrate series of collectors to increase the floatability of oxidized and low rank coals successfully because these improved collectors could be bonded with the surface oxygenated sites on oxidized coal through hydrogen bonding.

However, how to store fresh coal to avoid the oxidation damage on coal surface hydrophobicity and floatability has not been investigated sufficiently. In coal preparation plants, fresh coal is usually stored in the raw coal bunkers (in a relative dry-air environment) and open coal yards (suffering the rain and sunshine). Sometimes, coal fines are stored in the coal tailings refuse ponds in the case of production accident. It is necessary to investigate the effects of storage and oxidation conditions on the surface hydrophobicity and floatability of coal.

A review of literature has shown that oxidation and the use of various oxidizing agents and changes occurring during these processes (Pietrzak & Wachowska, 2003; Grzybek et al., 2004; Pietrzak & Wachowska, 2004). The main aim of this study was to determine the effects of oxidation of bituminous coal on its floatability and surface properties. The experiments of air-/water oxidation process were conducted. FTIR, SEM and flotation tests have been employed in our research works. The results are compared for fresh and air- and water- oxidized coal. Throughout this paper, a recommendation is proposed for the better store of coal in order to avoid the decrease in the hydrophobicity and floatability of bituminous coal.

2. Experimental method and procedure

2.1. Materials

Fresh bituminous coal bulks (ash content of 7.37%) were obtained from Xuzhou Jiahe Coal Preparation Plant in Jiangsu Province of China. The elemental analysis of raw coal is shown in Table 1. Fresh coal samples were crushed and sieved to pass 0.5 mm sieve. Then, coal samples were classified into three parts. The first part was stored in a pocket by nitrogen protection for the fresh coal samples. The second part was oxidized in the room (inside) for obtaining the air-oxidized coal samples. The third part was oxidized in the tubs (filled with water and coal fines, in a condition of pulp) for obtaining the water-oxidized coal samples. The oxidation temperature was room temperature about 20~25°C. The oxidation times were 20 and 80 days.

TABLE 1

The elemental analysis of raw coal

<i>C_{daf}</i> (%)	<i>H_{daf}</i> (%)	<i>O_{daf}</i> (%)	<i>N_{daf}</i> (%)	<i>S(t, daf)</i> (%)
85.6	5.9	7.2	1.1	0.2
<i>C_{daf}</i> , <i>H_{daf}</i> , <i>O_{daf}</i> , <i>N_{daf}</i> and <i>S(t, daf)</i> are the element (<i>C</i> , <i>H</i> , <i>O</i> , <i>N</i> and <i>S</i>) contents on a dry-ash-free basis.				

2.2. FTIR Measurement

The FTIR spectras of fresh, air-oxidized and water-oxidized coals were obtained with KBr pellets prepared with various coal samples. For the FTIR analyses, a Perkin Elmer Spectrum 2000 model spectrometer was used while the spectra were obtained at 2 cm⁻¹ resolution which is between 4000 and 400 cm⁻¹.

2.3. SEM Measurements

The FEI Quanta 250 SEM was used to analyze the surface morphology of fresh, air-oxidized and water-oxidized coals. The magnification was fixed at 10000. The three coal samples were prepared by the surface cleaning using absolute ethyl alcohol. After the surface cleaning processes, the coal samples were dried in air. Before the SEM measurements, the three coal samples were sputter-coated with gold layer.

2.4. Flotation tests

The flotation experiments were conducted in a 1.5 L XFG flotation cell using 100 g/1.5 L of coal each time. The airflow rate was 2.0 L/min. First, fresh, air-oxidized and water-oxidized coals were pre-wetted in the flotation cell for 1 min at the impeller speed of 1800 rpm. After the pre-wetting processes, the collector was added into the flotation pulp and the pulp was conditioned for 3 min. Then, 2-octanol frother was added and the pulp was conditioned for 1 min. At last, the air inlet was opened and foam was collected. The collector dosages were 0, 150, 300, 500 and 600 g/t while, the frother dosage was 300 g/t in a constant value.

The flotation concentrate was analyzed using the concentrate ash content and the combustible matter recovery. 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 mass yield of the concentrate (%), M_F mass yield 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. FTIR Analysis

The FTIR spectra of studied coal show a certain number of common absorption bands, different shapes and intensities, with characteristic peaks. As shown in Figure 1, the primary hydrophilic groups, such as -OH and -COOM are at the peaks of 3400 cm^{-1} and 1600 cm^{-1} . The primary hydrophobic functional groups such as Ar-H and R-H are at the peaks of 3030 cm^{-1} and 1435 cm^{-1} (Yuh & Wolt, 1983; Cinar, 2009). The changes and the presence of different functional groups in the structure and surfaces coal was confirmed by the FTIR study on the fresh and oxidized coal.

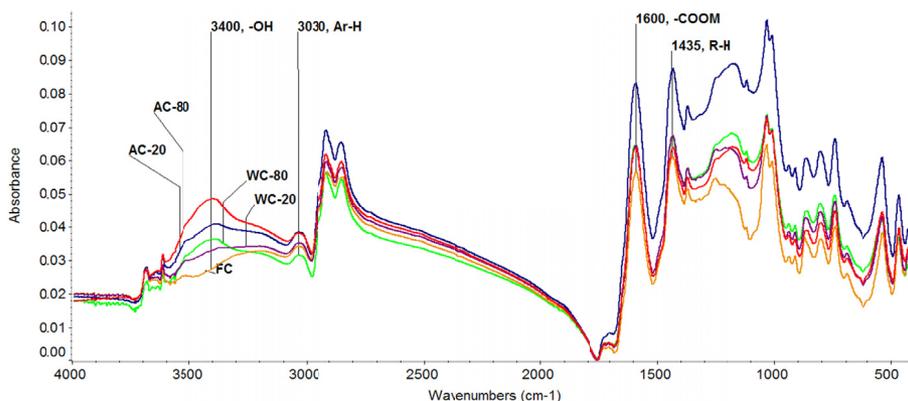


Fig. 1. FTIR spectra of fresh, air-oxidized and water-oxidized coals (FC is Fresh coal; AC-20 is air-oxidized coal by 20 days; AC-80 is air-oxidized coal by 80 days; WC-20 is water-oxidized coal by 20 days; WC-80 is water-oxidized coal by 80 days)

Comparing the FTIR spectra of fresh and oxidized coal provide the following conclusions: on the surfaces of oxidized coal, as a result of air- and water-oxidation, there is a formation of different functional groups. After oxidation, the increase in the intensities of the primary hydrophilic groups, such as –OH and –COOM was observed on the coal surface. The extent of this process was highly dependent on the oxidizing medium. The most changes were observed in the intensity of the band at 3400 cm^{-1} for water-oxidized coal by 80 days. Significant differences in the intensity of –COOM and R-H bands were noted in the range $1400\text{--}1600\text{ cm}^{-1}$ after air-oxidized coal by 20 days. Higher intensity of these vibrations in the spectrum of water- and air-oxidized coal confirms changes during oxidation process.

Hydrophilicity Index (HI) values were calculated in accordance with Equation (2), and are shown in Table 2.

$$HI = [\lambda(-COOH) + 2\lambda(-OH)] / [\lambda(R - H) + \lambda(Ar - H)] \quad (2)$$

Where (–COOM), (–OH), (R-H) and (Ar-H) are the adsorption intensity values as expressed by the Kubelka-Munk function (Yuh & Wolt, 1983; Cinar, 2009).

TABLE 2

Hydrophilicity Index values of fresh, air-oxidized and water-oxidized coals

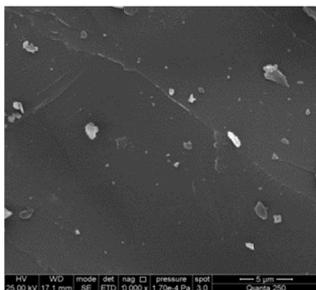
Coal samples	Hydrophilicity Index value
Fresh coal	1.17
Air-oxidized coal 20 days	1.30
Water-oxidized coal 20 days	1.27
Air-oxidized coal 80 days	1.57
Water-oxidized coal 80 days	1.40

Table 2 indicates that the HI value of coal increased after oxidation processes. The HI values of water-oxidized coals are lower than those of air-oxidized coals after the same time oxidation. The water-oxidized coals were obtained in the tubs (filled with water and coal fines) and the oxygen in water was limited, until water prevents the effective reaction between oxygen and coal surface. The natural air and natural water were used for air oxidation and water oxidation in this paper. It means the the oxygen content for air oxidation of coal is 21% while the oxygen content for water oxidation of coal was determined by the air dissolved in water at the temperature of room temperature. The surface oxidation degree of water-oxidized coals may be milder than that of air-oxidized coals. Compared the HI values of coal particles after air- and water- oxidation process, it can be concluded that air-oxidation process increased HI value more than water-oxidation. In most cases, the HI value determines the floatability of coals. Based on the above-mentioned results, the floatability of air-oxidized coals should be lower than that of water-oxidized coals. However, this deduction may be not right because the flotation performance of air-oxidized and water-oxidized coals is contrary, which do not agree with the results of HI value analysis. The detailed explanation will be given in Section 3.3 combined with Section 3.2.

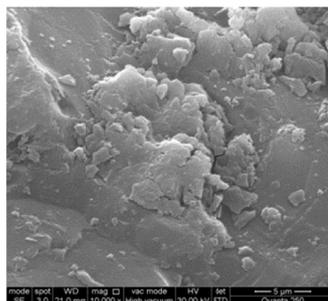
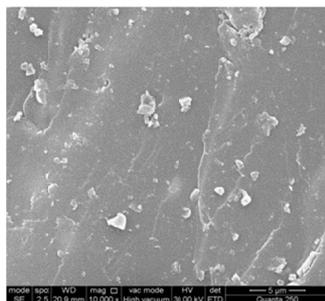
3.2. SEM Analysis

As shown in Figure 2, the fresh coal surface is very smooth while the oxidized coal surface has many holes and chips. The surface roughness of coal surface are increased after the oxidation

processes. In addition, the water-oxidized coal surface shows more holes and scaly slices than the air-oxidized coal surface with the same oxidation periods. These slices may be crispy and easy to be scraped off during the conditioning and flotation of coal fines in the flotation pulp.



Fresh coal (0 day)



Air-oxidized coal (80 days) Water-oxidized coal (80 days)

Fig. 2. SEM pictures of fresh, air-oxidized and water-oxidized coals

During the oxidation processes, the organic materials are oxidized by oxygen while the inorganic materials are usually not oxidized. The organic materials can be oxidized and a part of organic materials can be oxidized to gas components (CO and CO₂) and water (Xia & Yang, 2014; Xia et al., 2014). As a result, the content of organic materials on coal surface is reduced after the oxidation processes. The inorganic materials remain on the coal surface like the scales and chips.

However, the water-oxidized process may produce more scales and chips on the coal surface than the air-oxidized processes because the coal was immersed in the water (tap water) in the tubs. There are many calcium and magnesium ions in the tap water and these calcium and magnesium may be deposited and adsorbed on the coal surface. The deposited calcium and magnesium makes the coal surface more hydrophilic. Furthermore, the coal may have an ion exchange with the water because the organic and inorganic materials may be soaked and some organic and inorganic materials may be released from the coal surface into the water. More holes are produced after the water-oxidation process compared with the air-oxidation process. These holes will be filled up with the water in the flotation pulp and these water-filled holes make the coal surface more hydrophilic and difficult to be attached by air bubbles (Laskowski, 1994; Myint et al., 2013).

The surface morphology of coal was greatly changed by the water-oxidized processes while the air-oxidized process changes the surface morphology, only lightly. The changes in the surface morphology of coal during the water-oxidized processes should have a greater effect on the coal floatability than the changes in the functional group composition of the surface expressed by HI value of coal during the water-oxidized processes. The detailed analysis will be discussed in Section 3.3.

3.3. Flotation results

Figure 3 illustrates combustible matter recoveries of fresh, air-oxidized and water-oxidized coals at the various collector dosages. The combustible matter recovery increases with the increasing of collector dosage. However, Figure 3 gives us an insight that the combustible matter recovery of water-oxidized coal is much lower than that of air-oxidized coal with the same oxidation periods, which has not been sufficiently investigated in the coal flotation science and does not agree with the changes in HI values in Section 3.1. The combustible matter recovery of fresh coal is the highest while that of water-oxidized coal (80 days) is the lowest. The combustible matter recovery of air-oxidized coal is much higher than that of water-oxidized coal.

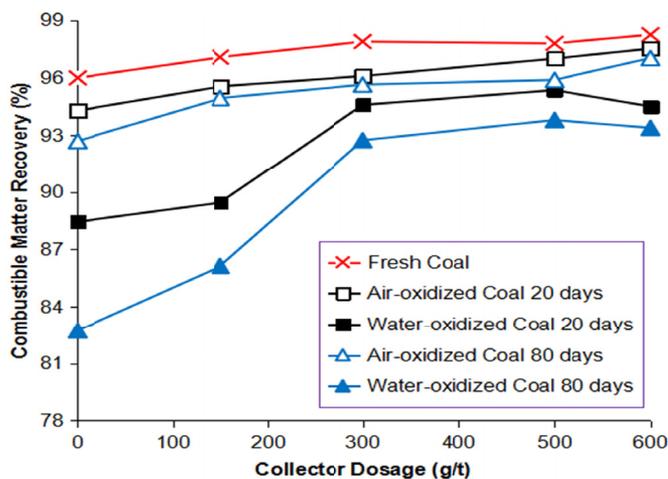


Fig. 3. Combustible matter recoveries of fresh, air-oxidized and water-oxidized coals at various collector dosages

It seems that the air-oxidized process have a milder effect on the coal floatability, while water-oxidized processes have a greater effect on the coal floatability. The combustible matter recovery of air-oxidized coal (20 days) is similar to that of air-oxidized coal (80 days). However, the combustible matter recovery of water-oxidized coal (20 days) is much higher than that of water-oxidized coal (80 days). The flotation performance of water-oxidized coal (80 days) is deteriorated much more than that of water-oxidized coal (20 days). It indicates that the coal floatability will be greatly reduced in the water-oxidation environment if the oxidation period is so long.

Figure 3 also indicates an interesting phenomenon that the difference in the combustible matter recovery between fresh, air-oxidized and water-oxidized coals becomes much smaller at the high collector dosages. It means that a higher collector dosage can recover the oxidized coal effectively (Tao et al., 2002). In the absence of collectors, the flotation is achieved only by the natural floatability. However, the natural floatability of coal should be greatly reduced after air-/water-oxidized processes. Therefore, the collectors can promote the floatability of oxidized coal and the collector dosage is the most important parameter which determines the effective flotation of oxidized coal as it is well known.

As shown in Figure 1 and Table 1, both air-oxidation and water-oxidation processes can produce many oxygen functional groups (considered as primary hydrophilic groups) on coal surface which makes coal surface more hydrophilic. According to the calculation of Hydrophilicity Index values, the air-oxidation processes should have a greater effect on the decrease of hydrophobicity than the water-oxidation processes because the Hydrophilicity Index values of air-oxidized coals are much higher than those of water-oxidized coals. However, the floatability of air-oxidized coal is higher than that of water-oxidized coal, according to the flotation results as shown in Figure 3. It is contradiction if based on Table 1 and Figure 3. However, it becomes reasonable if we built a relationship between Figure 2 and Figure 3. SEM pictures show that the water-oxidation processes change the surface morphology of coal greater than the air-oxidation processes. More holes are produced after the water-oxidation processes compared with the air-oxidation processes. The holes will be filled up with the water in the flotation pulp and the water-filled holes will make the coal surface more hydrophilic and difficult to be attached by air bubbles. In addition, the deposited calcium and magnesium from the water makes the coal surface more hydrophilic. Therefore, the increased number of holes and chips on the water-oxidized coal surface reduce the coal floatability greatly. The floatability of coal should be determined by both the HI value (chemical composition) and the surface morphology of coal. In this investigation, it was found that the changes in the surface morphology of coal particles reduced the floatability of coal particles greater than the changes in the HI values of coal particles.

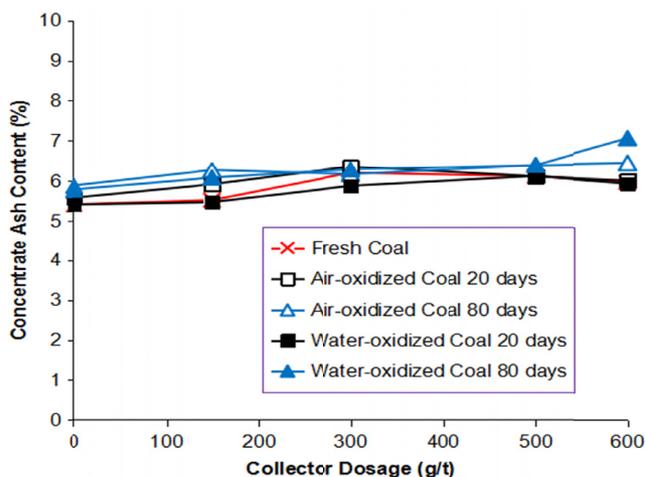


Fig. 4. Concentrate ash contents of fresh, air-oxidized and water-oxidized coals at various collector dosages

Figure 4 illustrates the concentrate ash contents of fresh, air-oxidized and water-oxidized coals at various collector dosages. The concentrate ash content does not show obvious differences as opposite to combustible matter recovery between fresh, air-oxidized and water-oxidized coals. With the increase of collector dosage, the concentrate ash content has a little increase. It has been well known that the hydrophilicity/floatability of some middling coal particles can be also enhanced at a higher collector dosage. These middle ash coal particles will be floated at the higher collector dosages. Therefore, the concentrate ash content increases with the increase of collector dosage. It is also difficult to obtain the regular a between different types of coal samples.

4. Conclusions

The following conclusions can be drawn from this paper.

- (1) The FTIR spectra of studied coal show formation of different functional groups on the surface of fresh and oxidized bituminous coal, as a result of oxidation by water and air. The primary hydrophilic groups, such as -OH and -COOM are at the peaks of 3400 cm^{-1} and 1600 cm^{-1} while the primary hydrophobic functional groups such as Ar-H and R-H were noted at the peaks of 3030 cm^{-1} and 1435 cm^{-1} . After oxidation, the increase in the intensities of the primary hydrophilic groups, such as -OH and -COOM was observed on the coal surface.
- (2) The Hydrophilicity Index (HI) value of coal is increased after both air-oxidized and water-oxidized processes. The HI values of water-oxidized coals are lower than those of air-oxidized coals. It indicates that the estimated surface hydrophobicity of air-oxidized coal is lower than that of water-oxidized coal.
- (3) The surface roughness of coal surface is increased after the oxidation processes. The water-oxidized coals have more holes and chips on the surface than the air-oxidized coals because many calcium and magnesium from the tap water might deposit and adsorb on the coal surface. Furthermore, differences in water chemistry due to Ca^{2+} and Mg^{2+} , in consort with slimes, may affect the coal flotation (Zhijun et al., 2013). In addition, the coal surface may have a mass exchange with the tap water as the organic and inorganic materials may be soaked. Some organic and inorganic materials may release from the coal surface into the water. More holes are produced after the water-oxidation processes compared with the air-oxidation processes. The holes may be filled up with water in the flotation pulp and the water-filled holes make the water-oxidized coal surface more hydrophilic and difficult to be attached by air bubbles.
- (4) The combustible matter recovery of oxidized coal is much lower than that of fresh coal. The combustible matter recovery of water-oxidized coal is much lower than that of air-oxidized coal with the same oxidation periods. A higher collector dosage can improve the floatability of oxidized coal. The floatability of coal is determined by both the surface hydrophobicity and the surface morphology of coal. In our research it was found that the changes in the surface morphology of coal particles reduce the floatability of bituminous coal particles greater than the changes in the surface hydrophobicity of coal particles during the oxidation.

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