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DEASHING AND DESULPHURIZATION OF FINE OXIDIZED COAL BY FALCON CONCENTRATOR AND FLOTATION

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Abstract: Flotation and enhanced gravity separation based on different separation principles were carried out to investigate the desulphurization and deashing efficiency of fine oxidized coal. Surface properties of fresh and oxidized coals were tested by XPS and results showed that the contents of hydrophobic functional groups decreased while the content of hydrophilic functional groups increased after oxidation. Floatability and density analysis results showed that the floatability of coal samples decreased sharply because of oxidation, however, density composition of coal sample only had slight changes. Separation results showed that yields of gravity concentrates outclassed that of flotation concentrates, meanwhile, ash contents and sulfur contents of gravity concentrates were far lower than that of flotation concentrates. Yields and ash contents increased with the collector dosage and achieved to be 17.83 and 26.94% respectively when the collector dosage was 1600 g·Mg⁻¹. Yields and ash contents of gravity concentrates decreased with the centrifugal force and increased with the recoil water flow with similar sulfur content. Clean coal with yield of 53.86%, ash content of 9.81%, sulfur content of 1.47% and with a corresponding desulphurization efficiency of 44.53% was achieved at centrifugal force of 107 g and recoil water flow of 13.3 dm³·min⁻¹. For fine oxidized coal, enhanced gravity separation has a significant advantage of the separation efficiency compared with flotation.

Keyword: *fine oxidized coal, desulphurization, deashing, flotation, enhanced gravity separation*

Introduction

Coal oxidation, especially for the low rank coal, can be caused in the process of storage and transportation. The physicochemical properties of coal surface will change significantly due to oxidation, which lead to sharp increase of hydrophilic groups and decrease of hydrophobic function groups (Boylu and Laskowski, 2007; Xia et al., 2013a). That is, the hydrophobicity of coal decrease while the hydrophilicity increase

(Xia et al., 2013b). Fine oxidized coal is difficult to clean by conventional flotation processes (Harris et al., 1995; Jia et al., 2000; Jena et al., 2008). Therefore, pretreatment methods have been discussed to improve the floatability of oxidized coal in previous studies (Pi Kin and Akgun, 1997; Xia et al., 2012a; Xia et al., 2012b). Oxidation surface can be eliminated by premixing and ultrasound treatment. For mild oxidized coal, oxidation process only occurs on the surface of coal particles and the internal structure has no significant change. The floatability of oxidized coal has remarkable improvement by appropriate grinding time. Hydrophobic surface can be exposed. Moreover, the flotation of oxidized coal can be well improved by dry-grinding with collector.

Resulting from dehydration and reorientation of functional groups on the coal surface, pretreatment method, such as attrition and microwave pretreatments, were available to improve the flotation of oxidized coal (Cinar, 2009; Royaei et al., 2012; Sokolovic et al., 2012a; Sokolovic et al., 2012b; Xia and Yang, 2013). Thus, the above methods were employed in the previous studies to enhance flotation process by improving the hydrophobicity of coal surface. From another point of view, density difference between the coal particles has no significant change in the oxidation process, which can be used to clean the oxidized coal based on gravity separation.

Enhanced gravity separation is considered as cost effective and environmentally friendly separation process and extensively utilized in the fine-particle mineral processing (Galvin et al., 2010). Falcon concentrator which utilizes the high gravitational forces to reduce the settling time and achieve a rapid separation has been proved to be efficient for the separation of fine materials with density difference (Kroll-Rabotin et al., 2012, 2013, 2014). The fine coal with high sulfur content can be efficiently cleaned in the enhanced centrifugal field. The desulphurization efficiency can be achieved over 71%, and even up to 87% (Tao et al., 2006; Ibrahim et al., 2014). Enhanced gravity separators make it possible for the efficient separation of fine coal, ash and sulfur rejection can be 85 and 70% respectively with 85% combustible material recovery (Honker, 1998; Oruc et al., 2010).

However, comparative tests between enhanced gravity separation and flotation of oxidized coal were seldom discussed in previous studies. Therefore, the desulfurization and de-ash performance of fine oxidized coal with high-sulfur content was investigated by a SB40 Falcon concentrator in this paper. For comparison, flotation tests were also carried out to study the effect of separation methods on the separation efficiency of oxidized coal. Surface properties and density compositions of fresh and oxidized coals were also discussed. Moreover, the effects of the operation variables were evaluated.

Experimental methods and procedure

Materials

The coal samples were supplied by the Hua Heng coal preparation plant from China. The coal samples were stored in natural weathering processes for six months and oxidized at high temperature. The coal was gas coal. Proximate analysis of coal in air dry condition is as follows shown as: moisture content 3.68%, volatile content 16.73%, fixed carbon content 45.08%, ash content 34.51%, total sulfur content 2.95%. As shown in Table 1, yield of +0.074 mm size fraction accounted for 74.9%, meanwhile, yield of the ultrafine fraction (-0.045mm) was only 9.45%. In addition, ash and sulfur contents of different size fractions had no significant difference. Sulfur composition of the coal sample was tested and the results showed that organic sulfur content accounted for 47.82% of the total sulfur content, the rest was inorganic sulfur, including pyritic sulfur (49.32%) and sulfate sulfur.

Table 1. Size analysis of coal samples

Size (mm)	Yield (%)	Ash (%)	Std (%)	Σ Yield (%)	Σ Ash (%)	Σ Sulfur (%)
0.5–0.25	30.16	31.25	3.01	30.16	31.25	3.01
0.25–0.125	22.56	32.42	2.98	52.72	31.75	3.00
0.125–0.074	22.18	32.55	2.88	74.90	31.99	2.96
0.074–0.045	15.65	33.71	2.77	90.55	32.29	2.93
–0.045	9.45	55.83	3.11	100.00	34.51	2.95
Total	100	34.51	2.95			

X-ray photoelectron spectroscopy (XPS) measurements

In order to study the changes of elements composition and chemical states of C in the oxidization process, XPS detection of fresh coal and oxidized coal were carried out in an ultra-high vacuum (UHV) system with the surface analysis system (ESCALAB 250Xi, U.S.) at room temperature. The base pressure of the analysis chamber during the measurements was lower than $1.0 \cdot 10^{-9}$ mbar. Al K α radiation ($h\nu = 1486.6$ eV) 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 $900\mu\text{m}$. 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 Peakfit software. The binding energies were corrected by setting the C1s hydrocarbon (-CH₂-CH₂-bonds) peak at 284.6 eV.

Flotation measurements

Regressive release flotation test of fresh coal and oxidized coal were carried out to indicate the flotation changes in the oxidization process. To evaluate the effect of

grinding on the flotation improvement of oxidized coal, flotation of oxidized coal that was ground for 20 minutes was tested. XFD-63 flotation machine with a 1.5 dm³ XFG flotation cell was employed for the regressive release flotation tests. Positive twelve alkane and methyl isobutyl carbinol were used as collector and frother respectively. The positive twelve alkane collector dose was 1000 g·Mg⁻¹ and methyl isobutyl carbinol frother dose was 100 g·Mg⁻¹. The impeller speed of the flotation machine was 1800 rpm and the air flow rate was 0.15 m³·h⁻¹. Samples mass of 150 g with a pulp density of 9.68% solids was employed in each test. According to requirements of the regressive release flotation test, coal samples were sorted for five times, that is, flotation concentrate that obtained from the first round sorting was sorted for five times without adding flotation reagent. Six separation products were finally obtained. Generally speaking, the better the floatability of coal is, the higher yield of the final concentrate with same ash content will be. According to national standards, repeat tests were conducted to ensure the accuracy of the experiments. If the relative difference between a set of test values was over 10% or the absolute difference was more than 1%, the test needs to be re-conducted.

Density composition measurement

Float/sink analysis of fresh and oxidized coal were conducted to evaluate the density composition change in the oxidization process. Density solutions of 1.30, 1.40, 1.50, 1.60, 1.80 g·cm⁻³ with different proportions of benzene, carbon tetrachloride and tribromomethane were used to analyze the density composition of samples. Speed of centrifuge was 1200 rpm and each separation was conveyed for 12 min. Testing started at the lowest density and proceeded towards the highest density. Yields and ash contents of coals with different density could be determined subsequently. Cumulative yield vs. density curve was obtained from the dates of density composition measurement test.

Flotation tests

Flotation tests were conducted for the separation of fine oxidized coal. Flotation machine models and operating parameters were mentioned in section 2.3. In those tests, different frother dosages and collector dosages were tested to optimize the separation efficiency of flotation. Clean coals and tailings were obtained after each experiment.

Enhanced gravity separation tests

Falcon centrifugal concentrator, model SB40, was used to clean oxidized coal. The slurry was fed through the center feed pipe to the revolving impeller. Rapid deposits and stratification of particles occurred on the wall of separation rotor and particles moved up under the centrifugal force. The light products were expelled by overflowing, while the heavy products were concentrated in grooves. In this

investigation, the influence of operation parameters, namely centrifugal force and recoil water flow, on the separation efficiency were investigated.

Separation concentrates were analyzed using the indexes: ash content, concentrate yield, sulfur content and desulphurization efficiency. Equation (1) was used to calculate the desulphurization efficiency of the flotation experiments and enhanced gravity separation:

$$\text{Desulphurization efficiency (\%)} = \frac{\gamma_j \times (S_y - S_j) \times 100}{S_y \times (100 - A_y - S_y)} \quad (1)$$

where γ_j is yield of clean coal (%), S_j is the sulfur content of clean coal (%), A_y is the ash content of feed (%), S_y is the sulfur content of feed.

Results and discussion

XPS analysis

X-ray photoelectron spectroscopy spectra of different elements of fresh coal and oxidized coal are shown in Fig.1.

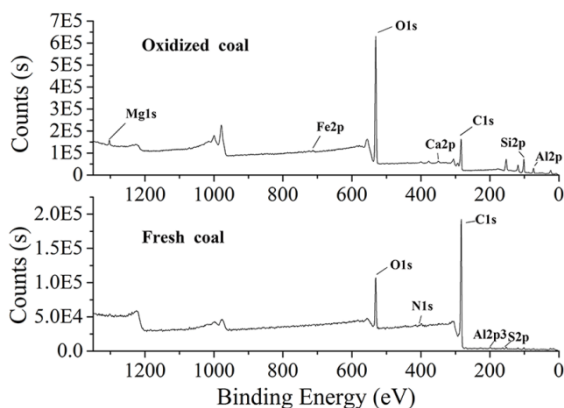


Fig. 1. XPS wide energy spectrum of oxidized coal and fresh coal

As shown in Fig.1, intensity of C1s peak decreased in the oxidization process compared with fresh coal, however, the intensity of O1s increased. Intensity changes of other elements were not evident due to the low content. It indicated that the carbon atoms on coal surface was replaced by oxygen atoms due to oxidation. The C/O atom ratio decreased after oxidation processes.

The C1s spectra shows four types of peaks, that is, peaks with binding energies of 284.6, 285.6, 286.6, 289.1eV were corresponding to the C-C/C-H, C-O, C=O and

O=C-O respectively (Xia et al., 2014; Zhang et al., 2014). Quantitative analysis of these four groups could be conducted by the XPS peak fit analysis technology and the results are shown in Table 2.

Table 2. Fraction of C groups of fresh coal and oxidized coal

Coal types	C-C, C-H (%)	C-O (%)	C=O (%)	O=C-O (%)
Fresh coal	78.44	9.76	2.17	9.63
Oxidized coal	53.02	25.36	10.64	10.97

Significant changes of the four group contents were observed. In the oxidization process, the content of C-C/C-H groups on coal surface decreased from 78.44 to 53.02%. However, the content of C-O groups increased from 9.76 to 25.36%. Meanwhile, the content of C=O increased from 2.17 to 10.64% and the content of O=C-O increased from 9.63 to 10.97%. Therefore, the essence of oxidation was the recombination process of functional groups due to the change of atom content. The C-O, C=O and O=C-O groups were the primary hydrophilic groups in coal while the C-C/C-H groups were hydrophobic groups (Xia and Yang, 2014). Hydrophobicity of fresh coal was weakened by oxidization, which adversely affected flotation.

Flotation analysis

The results of regressive release flotation tests are shown in Fig. 2.

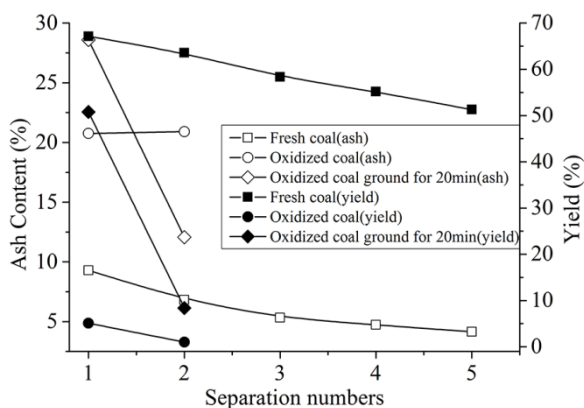


Fig. 2. Flotation of fresh coal, oxidized coal and ground products of oxidized coal for 20 min

As shown in Fig. 2, fresh coal had greater concentrate yields with lower ash contents. For fresh coal, final clean coal with yield of 51.28% and ash content of 4.15% could be achieved after five times of separations, which indicated that bubble could be firmly adhered on the surface of coal particle, that is, fresh coal had ideal

flotation. However, concentrate yields of oxidized coal were less than 2% only after two separation times, which indicated that hydrophobicity decreased sharply because of oxidization and oxidized coal was difficult to float. Furthermore, clean coal with 8.38% yield and 12.06% ash content of oxidized coal that ground for 20 min was obtained. It could be concluded that grinding could only slightly improved the effect of flotation for the seriously oxidized coal. Grinding generated fresh surface, so an improvement was achieved.

Density change analysis

Density composition of fresh coal and oxidized coal are shown in Fig. 3.

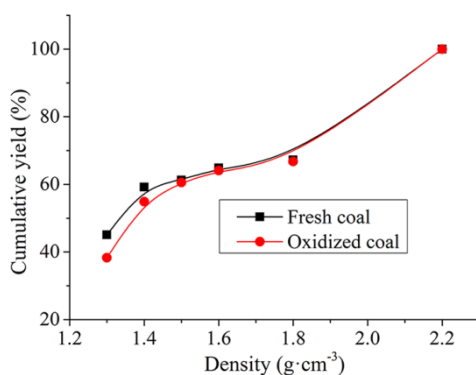


Fig. 3. Density composition of fresh coal and oxidized coal

As shown in Fig. 3, there was a slight change in density composition of coal after oxidization. For coal with density lower than $1.4 \text{ g}\cdot\text{cm}^{-3}$, the yields of fresh coal and oxidized coal were 56.51 and 52.19% respectively. It indicated that the low density fractions ($-1.4 \text{ g}\cdot\text{cm}^{-3}$) decreased to some extent after oxidization, however, yields of low density fractions ($-1.4 \text{ g}\cdot\text{cm}^{-3}$) and high density fractions ($+1.8 \text{ g}\cdot\text{cm}^{-3}$) were still much greater than that of the intermediate density fractions ($1.4\text{-}1.8 \text{ g}\cdot\text{cm}^{-3}$). The density differences remained significant, which could be used in the subsequent gravity separation process.

Separation results

Different frother (60, 80, 100, 120, 140 $\text{g}\cdot\text{Mg}^{-1}$) and collector dosages (1000, 1200, 1400, 1600, 1800 $\text{g}\cdot\text{Mg}^{-1}$) were tested to optimize the separation efficiency of flotation. The results showed that frother dosages had little effect on the yields and ash contents of flotation concentrates. Thus, this section only presented the effect of collectors on flotation. The effects of collectors on the de-ashing and desulphurization of fine oxidized coal are shown in Fig. 4 and Fig. 5 respectively.

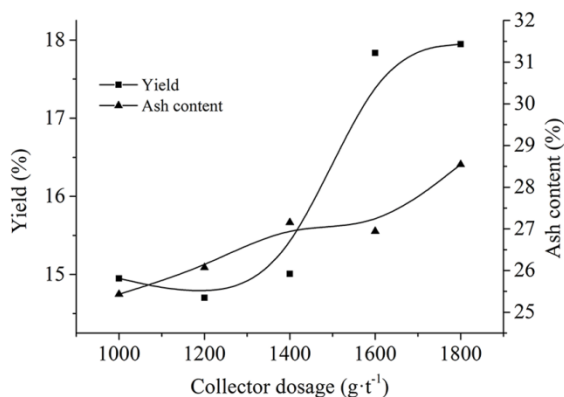


Fig. 4. Yield and ash content of flotation concentrates with different collector dosages

As shown in Fig. 4, yields and ash contents of flotation concentrates increased with the collector dosages, yields were between 14.95 and 17.96% and ash contents were between 25.43 and 28.55%. Comparing with flotation results of fresh coal (collector dosage: 600–1200 g·Mg⁻¹, yields of clean coal: 54–75%, ash contents of clean coal: 6.5–12.6%), it suggested that the effect of collector on the flotation was significantly decreased. The reasonable explanation of this phenomenon was that the sensitivity of particle surface to the collector decreased significantly due to oxidation, which was mainly caused by the increase of hydrophilic groups. Obviously, such low yields and high ash contents of clean coal were difficult to meet the industry requirements.

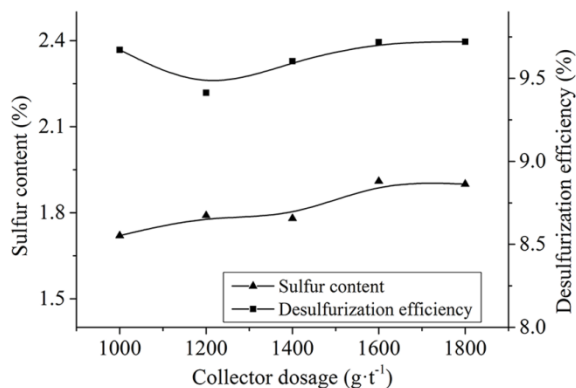


Fig. 5. Desulfurization efficiency and sulfur content of flotation clean coals with different collector dosages

As shown in Fig. 5, sulfur contents slightly increased with the collector dosage, values of desulfurization efficiency almost maintains stable and fluctuating between

9.41-9.72%, which was approximate regarded as equal. Sulfur contents were between 1.72 and 1.95%. It could be concluded that the desulfurization effect of flotation was relatively poor for the separation of fine oxidized coal. This was mainly because pyrite with good hydrophobicity had favorable flotation and it was easy to attach to the air bubble with the help of collector. Thus, it is difficult to separate pyrite and coal by flotation (Honker et al., 1996; Sivrikaya, 2014).

Deashing and desulfurization results of gravity separation tests of fine oxidized coal at different frequencies with fluidizing recoil water flow of $9.6 \text{ dm}^3 \cdot \text{min}^{-1}$ are shown in Fig. 6 and Fig. 7. The calculated radius was 0.044 m.

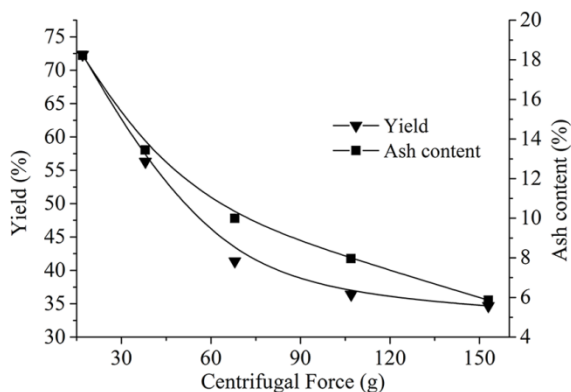


Fig. 6. Yield and ash content of clean coal of gravity concentrates at different centrifugal forces

As shown in Fig. 6, yields and ash contents of clean coal of gravity separations decreased with the centrifugal force and yields were between 35 and 75%, ash contents were between 6 and 13%. Compared with Fig. 4, Fig. 6 showed that clean coals obtained by gravity separation had higher yields with lower ash content than that of flotation concentrates. In centrifugal force field, particles were subjected to greater centrifugal force at higher bowl rotation, leading to more particles with relatively low density retained in circles and causing the decrease of separation density. Thus, yield of clean coal decreased with the centrifugal force associated with lower ash content. It indicated that enhanced gravity separation had better the deashing efficiency than flotation for oxidized coal.

As shown in Fig. 7, sulfur contents of clean coals of gravity separation were relatively stable and fluctuating at around 1.4%, meanwhile, desulfurization efficiency decreased with the rotational frequency because of the lower yields. Thus, gravity concentrates had greater desulfurization efficiency with lower sulfur content than that of flotation clean coals. The density difference between pyrite and coal was greater

than $3 \text{ g}\cdot\text{cm}^{-3}$, which caused the greater desulfurization efficiency of enhanced gravity separator in centrifugal force field than that of flotation.

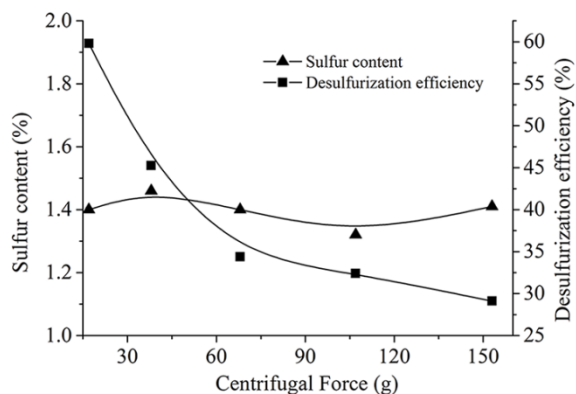


Fig. 7. Desulfurization efficiency and sulfur content of gravity concentrates at different centrifugal forces

In the case of a centrifugal force of 107 g, test indexes were further optimized by adjusting the recoil water flow. Deashing and desulfurization results of different recoil water flows with centrifugal force of 107 g are shown in Fig. 8 and Fig. 9.

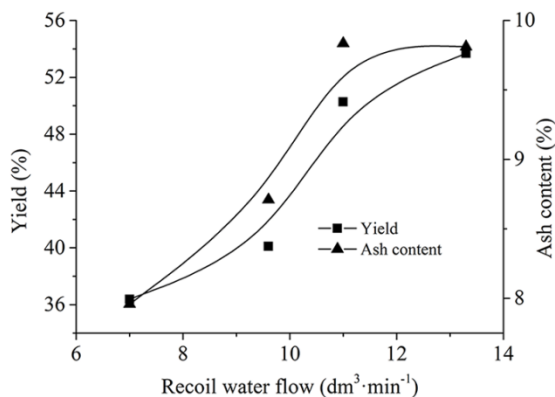


Fig. 8. Yield and ash content of gravity concentrates at different recoil water flows

As shown in Fig. 8, yields and ash content of gravity concentrates increased with the recoil water flow, yield achieved to be 53.68 with 9.81% ash content at recoil water flow of $13.3 \text{ dm}^3\cdot\text{min}^{-1}$. In the separating process, particle group could be easily decentralized with greater recoil water flow, leading to more light particles into the overflow, that is, the yield of clean coal could be improved.

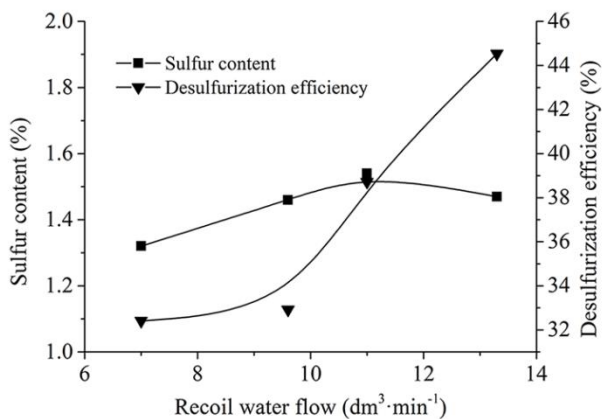


Fig. 9. Desulfurization efficiency and sulfur content of gravity concentrates at different recoil water flows

As shown in Fig. 9, at first sulfur content increased with the recoil water flows and then tended to be stable. Desulfurization efficiency increased with the recoil water flows because of the greater clean coal yields. Desulfurization efficiency reached 44.53% with 1.47% sulfur content at centrifugal force of 107 g and recoil water flow of $13.3 \text{ dm}^3 \cdot \text{min}^{-1}$.

Conclusions

Enhanced gravity separation based on density difference and flotation that utilizes surface properties differences were carried out to investigate the deashing and desulfurization efficiency of fine oxidized coal. In addition, surface properties, density composition and floatability of fresh coal and oxidized coal were also discussed.

In the process of oxidization, the content of hydrophobic functional groups (C-C/C-H) group was decreased while the content of hydrophilic functional groups (C-O, C=O and O=C-O) was increased significantly.

Flotation of fresh coal decreased sharply after oxidation, however, density compositions only had slight changes in the oxidization process.

For flotation, yields, ash content and sulfur content of clean coals were increased with the collector dosage, meanwhile, desulphurization efficiency were relatively steady. Yields of clean coal were less than 19%, ash contents were greater than 25% and sulfur contents were greater than 1.72%, that is, qualified clean coal could not be obtained by flotation.

For enhanced gravity separation, yields, ash contents and desulfurization efficiency of clean coals were decreased with the centrifugal forces because the greater centrifugal force particles were subjected to, the more likely particles remained in action circles and became tailings. Yields, ash contents and desulfurization efficiency

of gravity clean coals increased with the recoil water flow with similar sulfur content. Particle group in separation area could be easily decentralized with the greater recoil water flow, leading to more light particles into the overflow.

A final gravity clean coal with 53.68% yield, 9.81% ash content, 1.47% sulfur content and 44.53% desulphurization efficiency was achieved at centrifugal force of 107 g and recoil water flow of $13.3 \text{ dm}^3 \cdot \text{min}^{-1}$.

The results showed that enhanced gravity separation had a significant advantage of separation efficiency compared with flotation for fine oxidized coal.

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