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Flotation characteristics and particle size distribution of micro-fine low rank coal

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Abstract

In this work, attempts to float the micro-fine low rank coal and its particle size distribution in the flotation were made. Then, standard screening, FT-IR, XRD and SEM were adopted to characterize the size distribution and flotation of micro-fine Shendong low rank coal. The results indicated that the size fraction of -0.045 mm was the dominant size fraction in raw coal with a yield of 91.65% and ash content of 46.25%. Flotation of Shendong low rank coal required a larger dosage of collector, 50 kg/t of diesel oil, to achieve a higher combustible matter recovery (63.25%) and flotation efficiency index (40.70%) accompanied with a significant decrease in ash content (22.44 percentage points) due to the hydrophilicity of coal surface. Under this condition, concentrate contained 83.38% of -0.045 mm size fraction (38.04% of total particles in feed) with ash content of 24.98%. In comparison, tailing was almost consisted of -0.045 mm fraction (93.63%) with a higher ash content of 60.82%. It seems that the higher ash particles in feed were largely migrated in tailing at a proper collector dosage. The analysis of FT-IR, XRD and SEM would contribute to the understanding of the flotation and size distribution.

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Keywords: Micro-fine low rank coal; flotation characteristics; particle size distribution

1. Introduction

Currently, flotation of low rank coal is a new hot issue of coal preparation. A low rank coal is difficult to float

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in moderate additions of reagents due to its high hydrophilic character [1,2]. The main reason for its high hydrophilicity, poor flotability, is the formation of the surface oxygen-containing functional groups, such as hydroxyl, carbonyl and carboxyl [3,4].

Particle size is one of the important factors in coal flotation. The effects of size fractions on coal flotation had been investigated. Li et al. [5] found that the best flotation selectivity was obtained from the middle size fraction, -0.250+0.075 mm, while the selectivity of -0.500+0.250 and -0.075 mm particles was decreased. The coal slime size fraction of -0.125+0.074 mm possessed the highest flotation rate [6]. Xia et al. [7] showed that a better particle size for the flotation of heavily oxidized coal ranged from 11 to 74 μm. For the coal maceral group's separation using flotation, the vitrinite was mainly concentrated in the fine size fractions (-40+25 and -25 μm) [8]. In addition, the effects of particle size distribution on the strength parameters of German brown coal [9] and the rheology of coal-water slurries (CWS) [10] were also investigated. But the particle size distribution and its characterization of low rank coal, expecially for the micro-fine low rank coal, was less studied.

This investigation aims to seek the proper dosage for the flotation of micro-fine low rank coal and the particle size distribution before and after flotation. Then the micro-fine size fraction, -0.045 mm, of them were characterized by FT-IR, XRD and SEM.

Nomenclature

M_{ad}	air dried moisture content (%)
A_{ad}	air dried ash content (%)
V_{ad}	air dried volatile matter content (%)
FC_{ad}	air dried fixed carbon content (%)
M_C	weight of concentrate (kg)
M_F	weight of feed (kg)
A_C	ash content of concentrate (%)
A_F	ash content of feed (%)

2. Experimental Materials and Methods

2.1. Materials

The coal sample (coal slime, -0.5 mm), the subbituminous coal, was obtained from Shigetai Coal Preparation Plant in Shendong mine area of China. It was dried and prepared, then sealed for experiments. The proximate analysis of dried sample was exhibited as follow: M_{ad}=2.70%, A_{ad}=43.84%, V_{ad}=20.49%, FC_{ad}=32.97%.

2.2. Flotation test

In this study, diesel oil was used as the collector and the MIBC was used as the frother. The pulp density of the flotation test was fixed at 60 g/L. All the experiments were performed in a 1.5 L XFG-63 type flotation cell with the impeller speed of 1800 r/min and the airflow rate of 3 L/min. The dosage of frother was kept at 200 g/t. The variable dosages of collector were 5, 10, 15, 20, 25, 50, and 100 kg/t of coal, respectively.

For each test, the pulp was agitated with coal sample in the flotation cell for 2 min. Next, the required collector was added and the pulp was conditioned for another 2 min. Then, fixed dosage of frother (400 g/t) was added and the pulp was stirred for additional 1 min. After air was introduced into cell, the flotation concentrate and tailing were collected. The result was analyzed by combustible matter recovery and flotation efficiency index. Eq. (1) and (2) were adopted to calculate them [11]:

$$\text{Combustible matter recovery}(\%) = \frac{M_C(100 - A_C)}{M_F(100 - A_F)} \times 100\% , \tag{1}$$

$$\text{Flotation efficiency Index}(\%) = \frac{M_C(A_F - A_C)}{M_F A_F(100 - A_F)} \times 100\% \cdot \tag{2}$$

2.3. Sieve tests

According to the fine coal size analysis of the Method for Size Analysis of Coal of China (GB/T 477-2008), sieve tests of the raw coal was carried out by the standard sieves with mesh size of 0.250, 0.125, 0.074 and 0.045 mm. Due to the high content of fine coal particles, the standard sieves with mesh size of 0.250 and 0.125 mm were omitted for concentrate and tailing, and their fractions were combined into the fraction of -0.500+0.740 mm.

2.4. Characterization

The surface functional groups of Shendong low rank coal samples were tested by a Nicolet 380 type Fourier Transform Infrared Spectrometer (Thermo) with a range of 4000-400 cm^{-1}. Scans were at a resolution of 4 cm^{-1} with a frequency of 32.

Mineral matters in coal samples were indentified using a D8 Advance type X-ray Diffractometer (Bruker). The voltage of X-ray turb was 40 kV, and the current was 30 mA. The anode target material was Cu with Kα radiation. The measuring range (2-theta) was 3-70°.

The surface morphology of coal samples was determined with a Quanta 250 type Scanning Electron Microscope (FEI). Before the measurement, each sample was sputter-coated with a layer of gold. The magnification times were fixed at 6000 and 12000. The measuring mode was high vacuum (HV, 30.00 kV), and the spot was 2.5.

3. Results and Discussion

3.1. Flotation results

As shown in Fig. 1, with the increase of addition of diesel oil, the combustible matter recovery and flotation effciency index increased rapidly, but the ash content of concentrate was somewhat unchanged. When the dosages of diesel oil exceeding 50 kg/t of coal, the increase in combustible matter recovery was slowed down, and the flotation efficiency index began to decrease. This may be attributed to the decrease in growth rate of yield and the increase in ash content of concentrate caused by the collection of high ash content particles. Compared with the raw coal, the decrease in ash content of concentrate was 22.44 percentage points, and the combustible matter recovery and the flotation efficiency index were 63.25% and 40.70% separately with the diesel addition of 50 kg/t of coal.

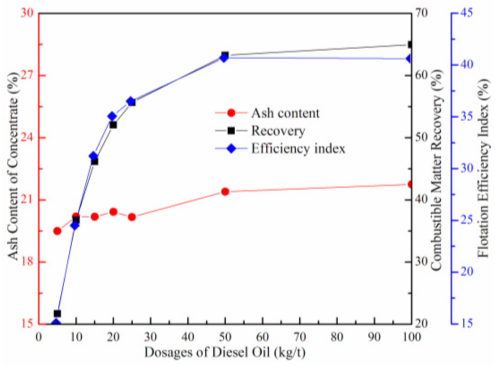


Fig. 1. The relationship among addition of diesel oil and flotation responses

3.2. Particle size distribution

Under former optimal condition (50 kg/t of diesel oil), the concentrate and tailing were screened by standard sieves. Table 1 showed the results of size distribution of raw coal. The dominant size fraction of the raw coal was -0.045 mm size fraction with a yield of 91.65% and ash content of 46.25%. This high content of micro-fine particle would increase the difficulty of flotation by attachment of high ash particle on coal surface [12].

Table 1. Size distribution of sieving for the raw coal.

Size fractions, mm	Yield (%)	Ash content (%)
-0.500+0.250	0.23	6.95
-0.250+0.125	1.01	6.42
-0.125+0.074	1.25	8.93
-0.074+0.045	5.86	6.78
-0.045	91.65	46.25
total	100	42.98

As compared in Fig. 2, a similar tendency was exhibited in concentrate and tailing. The concentrate contained 83.38% of -0.045 mm fraction (38.04% of total particles in feed) with ash content of 24.98%. In comparison, tailing was almost composed of -0.045 mm size fraction (93.63%) with a higher ash content of 60.82%. It seems that the higher ash particles in feed were greatly migrated in tailing at a proper collector dosage.

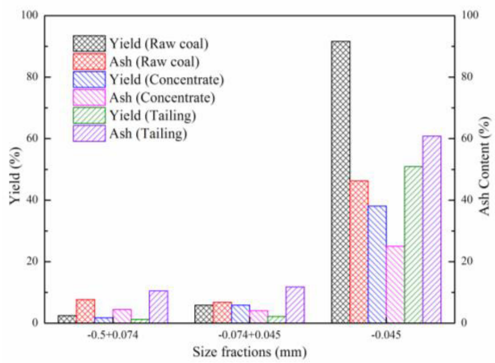


Fig. 2. Particle size distribution of raw coal, concentrate and tailing.

Therefore, the micro-fine fractions, -0.045 mm, of raw coal, concentrate and tailing were characterized by FT-IR, XRD and SEM.

3.3. FT-IR analysis

Fig. 3 showed the spectra of raw coal, concentrate and tailing. According to the literatures [13,14,15], the widest strong absorbance peak of coal sample was the weavenumber of 3428 cm^{-1} which was the stretching vibration of the hydroxyl group. The wavenumbers of 2951, 2921 and 2853 cm^{-1} were the absorbance peaks of methyl and methylene. The absorbance peaks of carbonyl associated hydrogen bond were presented in the wavenumber of 1622 cm^{-1}. The wavenumber of 1404 cm^{-1} was the absorbance of aromatic C=C. The ash-forming mineral matters exhibited in the wavenumber of 1034 and 1008 cm^{-1}. The existence of hydroxyl and carbonyl groups increased the hydrophilicity of the coal surface and decreased the flotability of coal particles. Because of the increase and decrease of organic matter in concentrate and tailing separately, the hydroxyl and carbonyl groups were enriched in concentrate and decreased in tailing at a greater addition of collector. The result coincided with the flotation and size sieve.

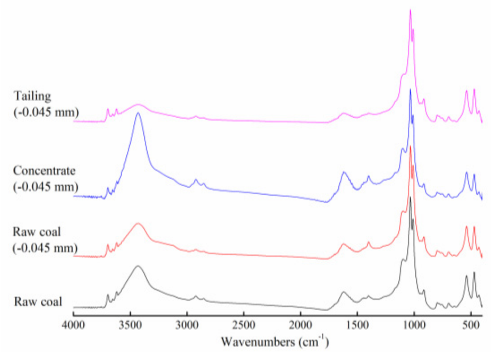


Fig.3. FT-IR spectra of raw coal, concentrate and tailing with fractions of -0.045 mm.

3.4. XRD analysis

The XRD spectra of coal samples were indicated in Fig. 4. Quartz and kaolinite were the major composition of mineral matters in all coal samples, and muscovite, clinochlore, montmorillonite and pyrite were the minor mineral matters. Compared with the raw coal of -0.045 mm, the diffraction intensity of concentrate with size fraction of -0.045 mm was deduced and the diffraction intensity of tailing was strengthened. This was due to the enrichment of ash-forming mineral matters in tailing. Owing to the entrainment of hydrophilic mineral matters in foam product [16], there were also some mineral matters, such as quartz and kaolinite, presented in concentrate.

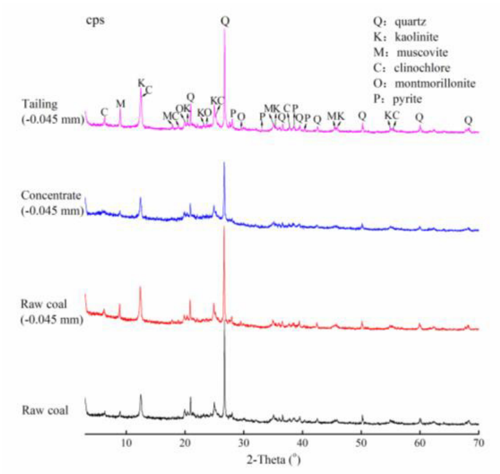
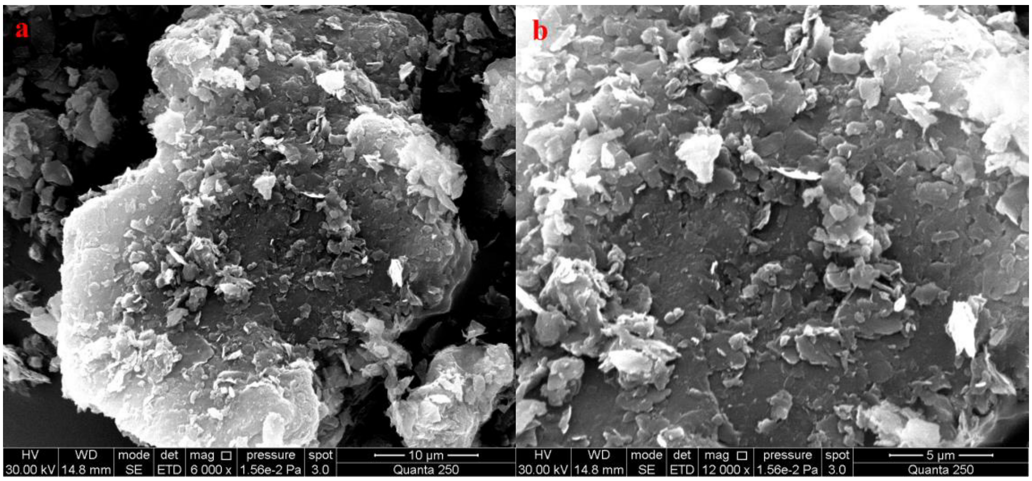


Fig. 4. X-ray diffraction spectra of raw coal, concentrate and tailing with fractions of -0.045 mm.

3.5. SEM analysis

As displayed in Fig. 5, there were abundant micro-fine particles attached to the surface of coal and/or gangue for all samples. The surfaces of both full and -0.045 mm size fraction raw coal (Fig. 5a-d) were crude and presented some fissures and hollows. The attachment of micro-fine particles to concentrate with size fraction of -0.045 mm (Fig. 5e-f) was less than raw coal (Fig. 5c-d) and tailing (Fig. 5g-h). Combined with energy dispersive X-ray spectroscopy (EDS), numerous fine schistous mineral matters were attached to the surface of fine tailing. The results were consisted with the analysis of XRD.



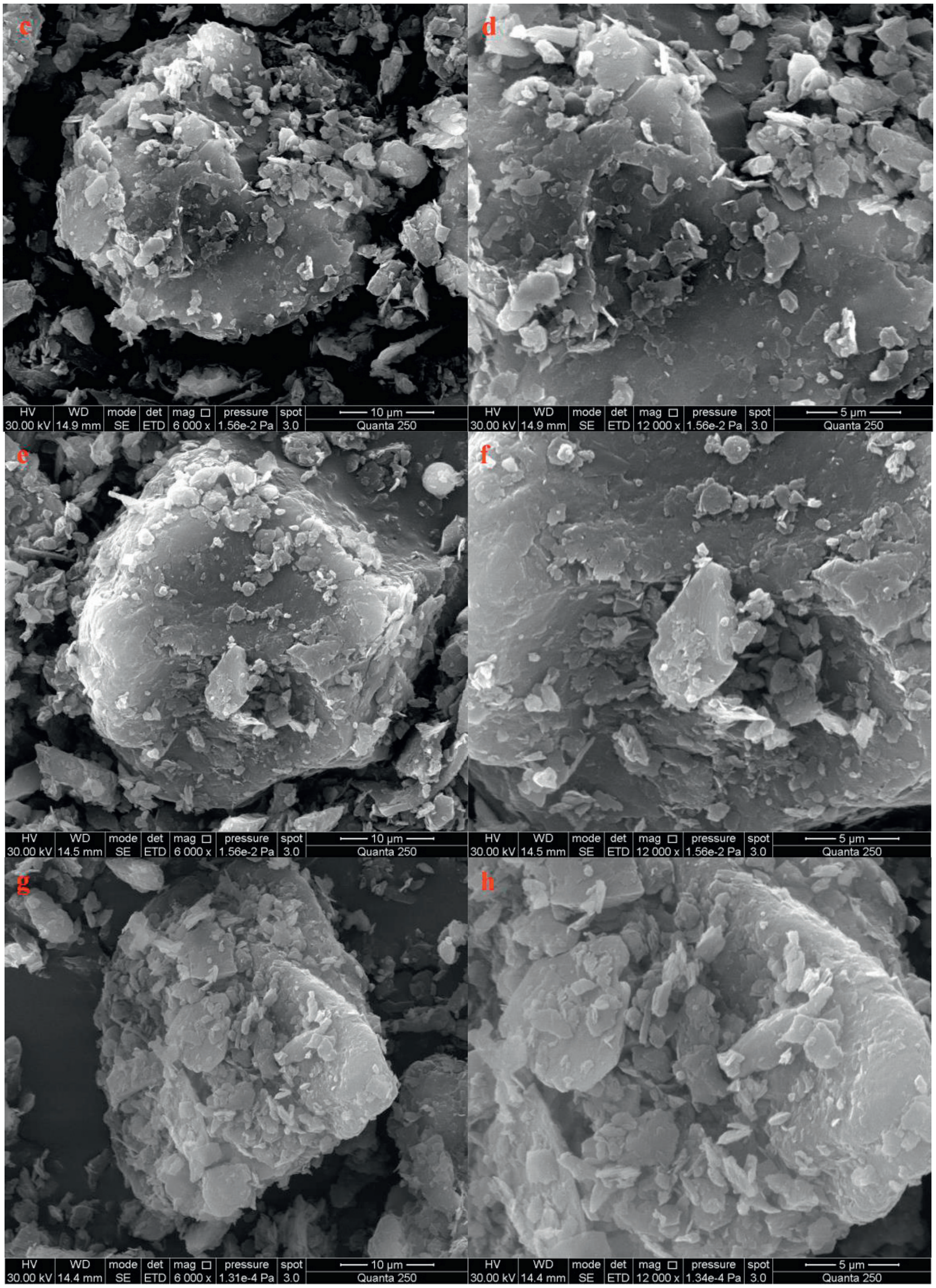


Fig. 5. SEM photography of (a) & (b) raw coal; (c) & (d) raw coal with size fraction of -0.045 mm; (e) & (f) concentrate with size fraction of -0.045 mm; (g) & (h) tailing with size fraction of -0.045 mm.

4. Conclusions

The dominant size fraction of the Shendong low rank raw coal was -0.045 mm size fraction with a yield of 91.65% and ash content of 46.25%. Flotation of this micro-fine coal required a greater dosage of collector (diesel oil, 50 kg/t) to achieve a higher combustible matter recovery (63.25%) and flotation efficiency index (40.70%) accompanied with a significant decrease in ash content (22.44 percentage points) due to the hydrophilicity of coal surface. Under this condition, the concentrate contained 83.38% of -0.045 mm size fraction (38.04% of total particles in feed) with ash content of 24.98%. Comparatively, tailing was almost consisted of -0.045 mm size fraction (93.63%) with a higher ash content of 60.82%. It seems that the higher ash particles in feed were largely migrated in tailing at a proper collector dosage. The analysis of FT-IR, XRD and SEM showed good agreement with the flotation and size distribution.

Acknowledgements

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References

[1] G. Ateşok, M.S. Çelik, A new flotation scheme for a difficult-to-float coal using pitch additive indry grinding, Fuel 79 (2000) 1509-1513.
[2] K. Ceylan, M.Z. Küçük, Effectiveness of the dense medium and the froth flotation methods in cleaning some Turkish lignites, Energy Convers. Manage. 45 (2004) 1407-1418.
[3] Y. Cebeci, The investigation of the floatability improvement of Yozgat Ayridam lignite using various collectors, Fuel 81 (2002) 281-289.
[4] A. Gürses, K. Doymuş, Ç. Doğar, M. Yalçn, Investigation of agglomeration rates of two Turkish lignites, Energy Convers. Manage. 44 (2003) 1247-1257.
[5] Y.F. Li, W.D. Zhao, X.H. Gui, X.B. Zhang, Flotation kinetics and separation selectivity of coal size fractions, Physicochem. Probl. Miner. Process. 49(2) (2013) 387-395.
[6] X.H. Gui, J.T. Liu, X.X. Tao, Y.T. Wang, Y.J. Cao, Studies on flotation rate of a hard to float fine coal, J. China Coal Soc. 36(11) (2011) 1895-1900. (in Chinese)
[7] W.C. Xia, J.G. Yang, B. Zhu, The improvement of grindability and floatability of oxidized coal by microwave pre-treatment, Energy Source Part A 36(1) (2014) 23-30.
[8] E.Jorjani, S. Esmaeili, M. Tayebi Khorami, The effect of particle size on coal maceral group's separation using flotation, Fuel 114 (2013) 10-15.
[9] S. Zhong, F. Baitalow, P. Nikrityuk, H. Gutte, B. Meyer, The effect of particle size on the strength parameters of German brown coal and its chars, Fuel 125 (2014) 200-205.
[10] F. Boylu, H. Dincer, G. Ateşok, Effect of coal particle size distribution, volume fraction and rank on the rheology of coal–water slurries, Fuel Process. Technol. 85(4) (2004) 241-250.
[11] W.C. Xia, J.G. Yang, C. Liang, Improving oxidized coal flotation using biodiesel as a collector, Int. J. of Coal Prep. Util. 33(4) (2013) 181-187.
[12] X.H. Gui, G. Cheng, J.T. Liu, S.L. Li, Y.T. Wang, Y.J. Cao, Process characteristics of heterogeneous fine mud in the coal flotation, J. China Coal Soc. 37(2) (2012) 301-309. (in Chinese)
[13] S.F. Wen, Spectral Analysis of Fourier Transform Infrared Spectroscopy, second ed., Chemical Industry Press, Beijing, 2010. (in Chinese)
[14] R.M. Silverstein, F.X. Webster, D.J. Kiemle, Spectrometric Identification of Organic Compounds, seventh ed., Wiley, Hoboken, 2005.
[15] H. Zhu, H.L. Li, Z.S. Ou, D.Z. Wang, X.L. Lv, Study on surface modification of different rank coals by using FTIR, J. China Univ. Min. Technol. 30(4) (2001) 366-370. (in Chinese)
[16] B.J. Arnold, F.F. Aplan, The effect of clay slimes on coal flotation, part I: The nature of the clay, Int. J. Miner. Process. 17 (1986) 225–242.