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EFFECT OF INTERGROWN PARTICLE LIBERATION ON DIFFICULT-TO-SEPARATE COKING COAL FLOTATION

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Abstract: Flotation tests with intergrown particle liberation were conducted to explore a separation method of difficult-to-separate coking coal from the Tangshan Kailuan mine in China. The particle size distribution, density and coal petrography were investigated. The difficult-to-separate coking coal sample resulted in intergrown particles, such as non-liberated coal and rocks. Thus, intergrown coal particle liberation and re-separation tests were conducted. The results showed that grinding time had a great effect on the flotation performance. Grinding prompted coal to dissociate and improve the surface hydrophobic properties of minerals. However, heterogeneous fine silt covered the surface of coal particles when coal was ground too long. The inorganic mineral particles were over-ground and reduced the contact angle of coal. The results of coal rock dissociation and laboratory re-separation tests showed that clean coal combustible recovery increased through intergrown particle liberation and re-separation.

Keywords: *liberation, particle size, coking coal, hydrophobicity*

Introduction

This study focuses on fine coal from coking and fat coal samples. As an indispensable raw material used in a coke-making industry in China, coking and fat coal resources are quite depleted. Prime coking coal from coking and fat coal samples have reserves of approximately 100 petagrams (billion tons). Coking coal accounts for 5.39% of the total coal resources and 22.07% of coal resources for coking. Fat coal resources are scarce, accounting for only 3.38% of the total coal resources and 12.58% of coal resources for coking. China is the largest steelmaking coking coal producer and consumer in the world. China consumes nearly one petagrams of coke-making coal annually, and uses only about 10% of coking coal resources to support about half of

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the world steel production. Coking and fat coals are mined faster than other types of coal. Coking and fat coals yield more than double in proportion compared with total coal resources, which rapidly reduced the country coking and fat coal resources. Coking and fat coals are becoming rare in China (Xu, 2003; Du, 2006; Ma, 2006; Wu, 2007).

The most rare coal resource in China is high-ash and difficult-to-separate coal because of the intergrowth of coal and gangue, very finely disseminated size of high-ash coal and very large supply of middle-density coal (Cheng et al., 2013; Gui et al., 2013). A large amount of middlings, which account for about 10 to 30% of raw coal, is produced by separation. The ash content of middlings is generally from 25 to 35%. Thus, middlings are only used as a fuel. An estimated two petagrams per year of coking coal is used as power coal, which causes a great waste of scarce coal resources.

Some feeds to flotation of fine coal are middlings, intergrown coal and gangue of which the surface hydrophobicity generally ranges between that of coal and inorganic minerals. The middlings of intergrown particles are recovered as either flotation concentrate or tailing, depending on their floatability. However, the content of ash in a product is generally close to the content of ash in a feed, and may enter to tailings in actual production. The flotation middlings are then obtained by classification recovery operations and used as power coal, resulting in a coking coal resource waste. The coal middlings are primarily inter-band coal, which is an accretion of minerals and coal, and has a density between that of clean coal and rock. The conventional coal preparation methods do not effectively separate interlocked minerals. Petrographic components of coal, such as macerals or group macerals, should be dissociated to obtain low-ash clean coal from fine inter-band coal. However, the existing coal slime flotation technology only performs flotation progress without dissociation (Harris, et al., 1992; Giroux, et al., 2006; Jena, et al., 2008; Jorjani, et al., 2009; Vapur et al., 2010; Gui, et al., 2012). Fu and Wang (1996) studied recovery of high-quality clean coal from flotation coal middlings. They adopted the hydrophobic flocculation flotation process to recover low-ash clean coal from coking coal middlings by pulverizing coal middlings using a JM series stirred mill. The tests showed that a concentrate with ash content of 11.32% could be obtained from coking coal middlings with 57.78% ash content and yield of 38.31%. In addition, grinding was used to increase floatability of oxidized coal and some difficult-to-separate coal (Shu et al., 2002; Man et al., 1998; Tao et al., 2009; Hu et al., 2011; Li et al., 2013; Xia et al., 2012,a ,b, 2013; Zhang and Tang, 2014).

From the perspective of coal petrology, difficultness in separating of organic and inorganic matters in coal is determined by coal slime washability, which mainly depends on the composition, particle size, density and distribution state of inorganic matter in coal. Dissociation could be easily performed when the inorganic matter is gathered in layers, large nodulars, lenticulars or monomers. However, dissociation is very difficult and leads to poor washability when mineral dissemination size is very fine and is distributed in the organic substrate as either disseminated or in thin strips or even filled in plant cells. This study focuses on rare fat coal and explores the methods for improving coal rock liberation of middlings through the coal petrology analysis and grinding floating experiments.

Experimental

The representative –0.5 mm coal sample used for testing was obtained from the Tangshan Kailuan mine in China. The collected sample was subsampled to obtain a representative sample. The samples were selected for an X-ray diffraction test. The instrument used was a Rigaku D/Max-RA rotating anode X-ray diffraction. The working conditions were as follows: Cu target, operating voltage: 40 kV, current at 50 mA, graphite monochromator, scattering slit at 1°, slit acceptance at 0.3 mm, step scan, preset time at 0.1 s, and pace: 0.02 °/step.

A SPB200 vibrating Taylor screen was used for size analysis to determine the ash content at various particle sizes. A GL-21M high-speed centrifuge was used for density analysis at a centrifuge speed of 3000 rpm. Specification of centrifuge tube was 4×250 cm^3. Four centrifuge tubes with volumes of 250 cm^3 were arranged symmetrically in the centrifuge. The centrifugal liquid was prepared by mixing an organic solution composed of carbon tetrachloride, benzene and tribromethane.

A ZEISS imager M1m type microscope spectrophotometer was used for coal maceral analysis and testing. The working conditions were as follows: immersion oil refractive index (Ne): 1.518, standard material for sapphire and yttrium aluminum garnet, determination target: 90% of telocollinite and 10% of desmocollinites. The test was performed at ambient temperature (23 °C).

A contact angle of coal sample was determined by fast photo-angle measurement. The coal sample was formed into a tablet under a pressure of 30 MPa for 10 seconds. The diameter and thickness of tablet were 10 and 2 mm, respectively. The contact angle of right and left sides were measured three times and the average value was considered as the final contact angle.

A crushed sample was ground in a laboratory XMB-68 type 160×200 ball mill. The mill cylinder diameter and length were 160 and 200 mm, respectively. The cylinder volume was 4.02 dm^3, cylinder speed was 120 rpm and feeding quantity was from 300 to 800 g. The test was completed through wet grinding to simulate grinding-flotation industrial processes at the production site. An XFD 0.75 dm^3 laboratory-scale flotation machine was used for flotation tests.

The flotation concentrates were analyzed using combustible matter recovery and ash contents. The combustible matter recovery was calculated by using formula:

$$\text{Combustible matter recovery}(\%) = [M_c(100 - A_c) / M_F(100 - A_F)] \cdot 100 \tag{1}$$

where M_C and M_F are weights of concentrate and feed (%), respectively, A_C and A_F are ash contents in concentrate and feed (%), respectively

A coal rock liberation test had an open circuit flow and consisted of one rougher, one scavenger, middling liberation and re-separation. To ensure the maximum recovery of clean coal combustible matter, the scavenger process (wherein numerous hard-to-recycle intergrown particles float) was applied to the rougher tailings, followed by liberation and re-separation. Figure 1 shows the flow-sheet. Using a single factor experiment and considering comprehensive ash concentrate and clean coal combustible matter recovery, the test conditions for preparation of grinding middlings were as follows: diesel as the collector (rougher, 190 g/Mg; scavenger, 310 g/Mg), 2-octanol used as the frother (rougher, 90 g/Mg; scavenger, 125 g/Mg), stirring speed during flotation 1590 rpm, and air flow 0.37 m^3/min.

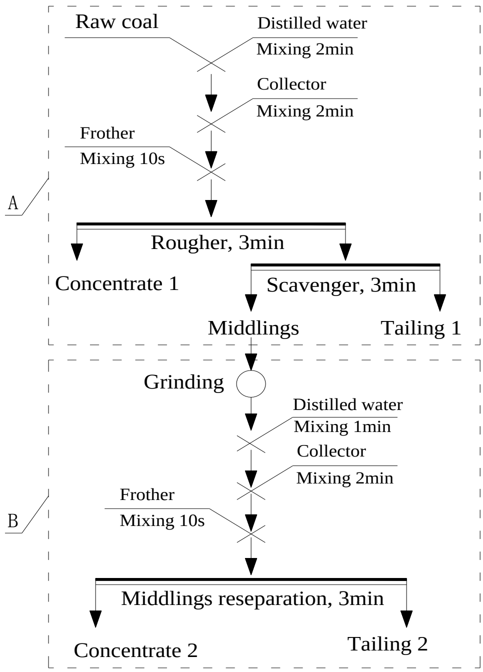


Fig. 1. Test flow-sheet

Results

Mineralogical and chemical analysis of the sample

The X-ray diffraction patterns of the −0.5 mm coal sample (Fig. 2) show that the main gangue mineral in the coal slime was kaolinite, with small amount of quartz, illite and pyrite. Kaolinite $Al_4[Si_4O_{10}](OH)_8$, a very fine mineral is the major component of clay and argillaceous rocks. The exchange capacity of anions in kaolinite is relatively high. Organic reagents are adsorbed on the particle surface. It allows kaolinite to enter easily to the concentrates through bubbles, which results in the high ash content in the concentrates.

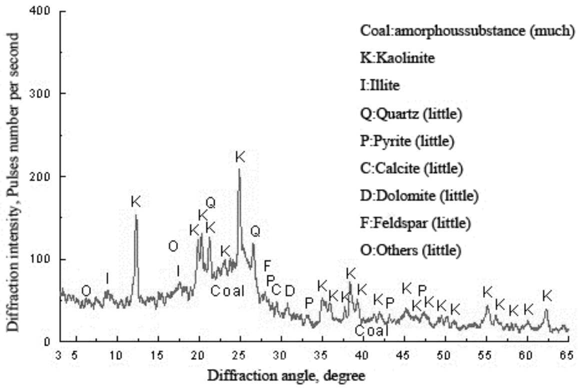


Fig. 2. X-ray diffraction patterns of -0.5mm coal sample

Density and size analyses

Figure 3 shows results of density and size analyses. It can be seen that the low density fraction of investigated coal was mainly located in the coarse particle size fraction. For instance, the density fraction less than 1.3 g/cm^3 was mainly located in particles coarser than 0.045 mm. However, the yield of density fractions from 1.3 to 1.5 g/cm^3 was uniform. The coal slime of density fractions of 1.5-1.8 g/cm^3 was the main cause of intergrowth of coal and gangue, which was verified in later petrological composition analysis.

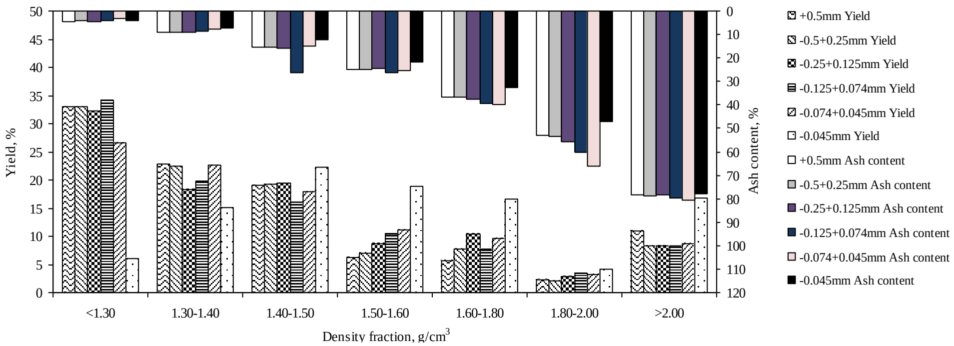


Fig. 3. Density analysis of narrow size fraction

Intergrown coarse particles easily entered the concentrates during flotation because of high desorption probability. Thus, mineral liberation prior to separation increased the recovery of combustible matter. Table 1 shows the yield and ash content of the middle density range (1.5–1.8 g/cm^3) of +0.074 mm size fraction. It can be seen that the middle density range was 10.75% of raw coal with 31.50% ash content. This fraction was composed mostly of intergrown particles of coal and mineral matter. Thus, full liberation was a key to efficient separation.

Table 1. Yield and ash of middle density fraction

Density, g/cm^3	Size fraction, mm	Yield of size fraction, %	Yield of raw coal, %	Ash, %
1.5-1.8	+0.5	11.93	0.60	30.65
	-0.5+0.25	14.72	1.34	30.97
	-0.25+0.125	19.07	4.86	31.44
	-0.125+0.074	18.16	3.94	31.88
Total			10.75	31.50

A mean-value curve can be used to show washability of coal (Fig. 4). The increasing trend of cumulative yield was weakened when the ash content was higher than 10%. The cumulative yield of five size fractions coarser than 0.045 mm, that is 0.5, 0.25–0.5, 0.125–0.25, 0.045–0.074 mm, was close for the same ash content. For a yield of about 80% the ash content was under 12%. The cumulative yield of fine size fraction (−0.045 mm) was the lowest.

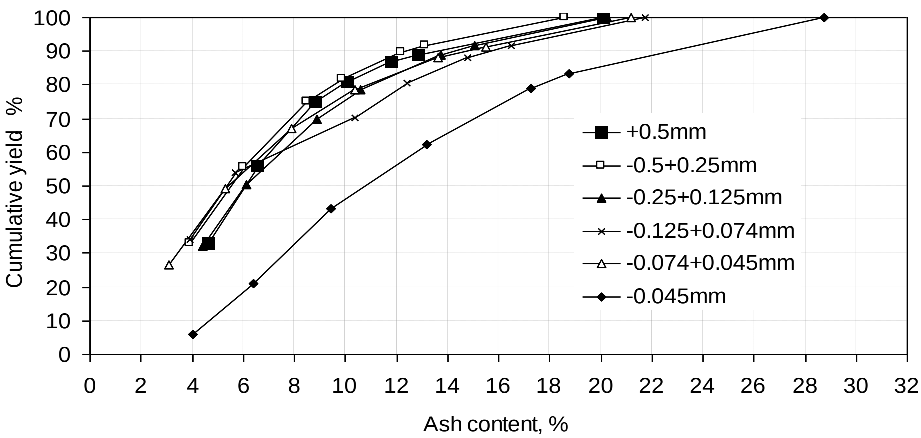


Fig. 4. Cumulative yield – ash content curve of narrow size fractions

Coal petrography

Table 2 shows that the coal sample mainly consists of vitrinite (56.34%) and inertinite (32%). The proportion of sample composition determined floatability due to differences in the physical properties of each maceral. Inertinite exhibits a very low floatability.

Table 2. Coal petrography analysis of particle sample

Sample	Organic maceral			Inorganic maceral			
	Vitrinite	Inertinite	Exinite	Clay	Pyrite	Calcite	Quartz
Content, %	56.34	32.44	1.76	6.38	2.15	—	0.93

Figure 5 shows the petrological composition of different density fractions. Inertinite, clay and pyrite contents increased with increase in density, whereas content of vitrinite gradually decreased. The majority of fine inorganic minerals in the middle density range (1.5–2.0 g/cm^3) were disseminated throughout the coal particles. Clay minerals always appeared to fill the plant cell in an aggregate form.

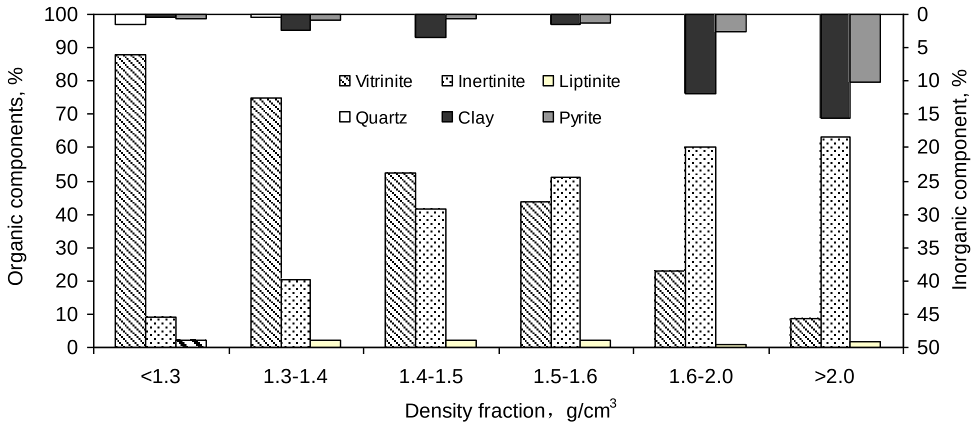


Fig. 5. Petrological composition of different density fractions

Figure 6 shows the petrological composition of different size fractions. The organic maceral content of each size fraction was very uniform. The vitrinite content ranged from 55 to 60%, and the content of inertinite was approximately 30%, with trace amounts of exinite. The content distribution of inorganic maceral was high in the coarse and fine fractions and low in the middle size fraction. A large amount of clay and quartz were disseminated in the coarse coal particles. The coal particles in +0.125 mm size fraction filled the cell cavities and holes in the form of particle aggregates. The dissemination size of clay and pyrite was generally less than 10 μm. Minerals almost appeared in the form of monomer in the fine particle size fraction (−0.045 mm). Dispersed clay particles of micro-fine size were occasionally observed in the coal particles (Fig. 7). The size analysis showed that the ash content of coarse size fraction was low. However, coarse coal particles disseminated with a large amount of inorganic minerals such as clay. Thus, liberation of coarse particles was clearly significant to the intensification process of fine coal.

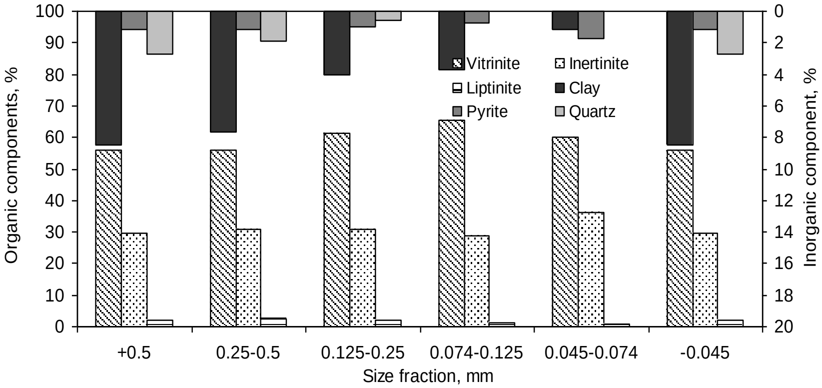


Fig. 6. Petrological composition of narrow size fractions

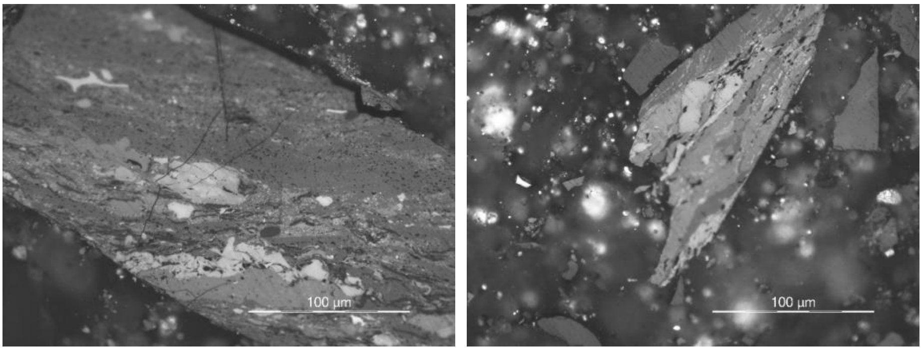


Fig. 7. Maceral composition photos of raw coal

Grinding test for the middlings

Middlings were first prepared as indicated in the flow-sheet in A-block diagram for the single-factor experiment (Fig. 1). The test conditions were as follows: 20% filling rate, 120 rpm wheeling speed, 40% grinding density, 300 g grinding dried slime, 5, 10, 15, and 20 min grinding time. Figure 8 shows changes in the yield and ash content of size fraction of −0.074 mm. The results show that the fine-sized middlings in the first 10 min were quickly reduced. The yield was 83.23% after 10 min of grinding. The slope of yield-time curve became significantly small and size reduction of materials became difficult in the next 10 min. The ash content decreased in the first 5 min. The organic matter in the coal was easier to grind than gangue. A considerable amount of low-ash clean coal among the inorganic matter was liberated and entered the next size fraction, which decreased the ash content. The decreasing trend of ash content of fine-sized fraction weakened with grinding time.

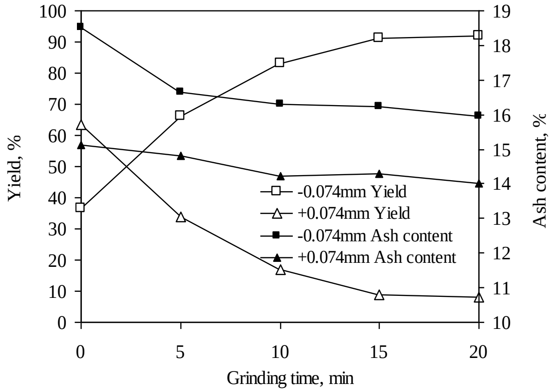


Fig. 8. Relationship between grinding time, yield and ash content

Table 3 shows that the yield of middle density fraction and ash content of low density fraction gradually decreased over grinding time. The density slightly changed when grinding time exceeded 15 min. These phenomena show that grinding was clearly effective by liberation of the middlings.

Table 3. Density analysis of different grinding times

Density fraction	0 min		5 min		10 min		15 min		20 min	
g/cm^3	Yield, %	Ash, %	Yield, %	Ash, %	Yield. %	Ash, %	Yield, %	Ash, %	Yield, %	Ash, %
<1.3	21.58	3.68	24.39	3.85	21.18	3.47	23.29	3.41	23.71	3.43
1.3-1.4	39.41	11.42	26.41	8.79	22.53	7.00	21.96	7.10	21.75	6.88
1.4-1.5	25.43	20.59	29.08	17.65	18.94	11.74	24.67	13.53	20.70	12.69
1.5-1.6	7.35	32.15	10.55	28.34	24.05	22.67	9.52	20.99	17.75	21.83
1.6-2.0	2.85	46.55	4.09	43.33	6.22	37.66	6.11	30.96	4.53	34.12
>2.0	3.38	54.04	5.48	50.60	7.08	49.85	14.45	43.73	11.56	46.66

In this work a four-factor and three-level orthogonal test was designed. The effect of different grinding conditions, such as filling rate, grinding time, wheeling speed and grinding density, on liberation of inorganic matter was comprehensively explored. Table 4 provides the results of the orthogonal test.

Table 5 shows the results of the variance analysis of yield and content of ash with density less than 1.4 g/cm^3. The filling rate had the most obvious effect on the yield of clean coal. It can also be seen that the wheeling speed did not affect the yield. The grinding conditions with the greatest effect on the yield of clean coal after grinding were as follows: 20% filling rate, 130 rpm wheeling speed, 40% grinding density and 5 min grinding time. Grinding time had the most obvious effect on the ash content of clean coal. The grinding conditions with the greatest effect on the ash content of clean coal were as follows: 40% filling rate, 120 rpm wheeling speed, 40% grinding density and 5 min grinding time.

Table 4. Data of the grinding orthogonal test

Group	Filling rate	Wheeling speed, rpm	Grinding density	Grinding time, min	-1.4 g/cm^3		+1.4 g/cm^3	
					Yield, %	Ash, %	Yield, %	Ash, %
1	20	110	30	5	24.72	8.61	75.28	19.76
2	20	120	40	10	26.32	7.08	73.68	20.78
3	20	130	50	15	23.52	7.51	76.48	19.54
4	30	110	40	15	13.61	6.52	86.39	17.81
5	30	120	50	5	15.14	7.88	84.86	18.45
6	30	130	30	10	12.44	7.04	87.56	18.08
7	40	110	50	10	19.64	7.64	80.36	18.80
8	40	120	30	15	13.90	8.20	86.10	17.71
9	40	130	40	5	27.00	14.55	73.00	16.07

Table 5. Variance analysis of clean yield and content of ash with density less than 1.4 g/cm^3.

	Filling rate		Wheeling speed		Grinding density		Grinding time		Error	
	Yield, %	Ash, %	Yield, %	Ash, %	Yield, %	Ash, %	Yield, %	Ash, %	Yield, %	Ash, %
Square of deviance	187.17	14.99	9.94	8.39	42.08	5.04	41.83	18.22	281.03	46.64
Degree of freedom	2	2	2	2	2	2	2	2	8	8
F	2.664	1.286	2.664	0.72	0.599	0.432	0.595	1.562		

F denotes the ratio of average sum of squared deviations and error average deviation squared error caused by changes in factor/level

The filling rate had different effects on the yield of low-density products. The yield of clean coal with density less than 1.4 g/cm^3 decreased with increase in filling rate, whereas the ash content increased. The effect of grinding density and time on the products yield with low density were in a good agreement with each other. Thus, the optimum grinding conditions for liberation of middlings were determined to include the low filling rate and high wheeling speed.

Re-separation test of middlings

The re-separation test of liberated middlings was conducted as indicated in the flow-sheet in B block diagram of Fig. 1. The test conditions included collector (310 g/Mg), frother (125 g/Mg) and stirring speed of 1590 rpm, with flow of 0.37 m^3/min. Table 6 shows the results of the test.

Table 6. Re-separation results of dissociated middling coal

Group	Filling rate, %	Wheeling speed, rpm	Grinding density, %	Grinding time, min	Clean coal		Physical Properties of feed middlings				
					Ash, %	Combustible recovery ,%	Ash, %	$-1.4\ g/cm^3$		-0.074mm	
								Yield,%	Ash,%	Yield,%	Ash,%
8	40	120	30	15	12.28	31.49	16.37	13.90	8.20	69.99	17.02
4	30	110	40	15	12.33	37.25	16.60	13.61	6.52	93.42	16.64
3	20	130	50	15	12.25	46.57	16.49	23.52	7.51	96.73	16.55
6	30	130	30	10	12.27	46.91	16.42	12.44	7.04	85.53	16.88
2	20	120	40	10	12.04	48.45	16.50	26.32	7.08	86.39	17.05
7	40	110	50	10	11.99	61.62	16.71	19.64	7.64	70.59	17.11
1	20	110	30	5	12.18	67.87	16.43	24.72	8.61	64.79	17.84
9	40	130	40	5	12.49	76.66	16.15	27.00	14.55	45.37	17.47
5	30	120	50	5	12.51	76.72	16.17	15.14	7.88	52.46	17.33
Direct flotation	0	0	0	0	12.50	67.70	/	/	/	/	/

The ash content of liberated middlings was kept as 12.5% to fulfill the ash requirement of desired clean coal based on the re-separation test results of liberated middling coal (Table 6) in the Qianjiaying Coal Preparation Plant of Tangshan Kailuan Mine. The tests of Groups 1, 9, and 5 obtained the clean coal combustible recovery of approximately 70% and ash content of about 12.5%. The results of Groups 4 and Groups 8 were worsened, with the clean coal combustible recovery of just about 35% and ash content of 12.3%. The best three groups were found in the initial stage with grinding time of 10 min, whereas the worst two groups were observed at the latter stage at grinding time of 15 min. Therefore, longer grinding time did not result in higher recovery rate. The increase in grinding time led to overgrinding. A large amount of overground clay minerals covered the surface of coal particles and caused competitive adsorption of flotation reagents. It decreased the probability of collecting by bubbles the low ash content coals, resulting in low clean coal combustible matter recovery.

The grinding density had the most obvious effect on flotation. The sliming probability of clay minerals in middlings decreased as the grinding density increased. The yield of low density fraction of middlings floated after liberation increased. It suggests that high grinding density was beneficial for selective liberation of minerals. Based on the results of size analysis of liberated middlings the yield of fine particles (−0.074 mm) were: 50% in Group 1, Group 9 and Group 5, 93.42% in Group 4, 69.99% in Group 8 and 70.59% in Group 7. A significant difference existed between flotation of Groups 7 and 8, what indicated that different grinding conditions resulted in different size compositions and mineral liberations. The results of the liberation and re-separation tests for middlings showed that better flotation results were obtained when the yield of size fraction of -0.074 mm after liberation was approximately 50%.

Figure 9 shows the quantitative and qualitative flow-sheet of middling coal re-separation based on coal and rock liberation. Based on calculation of the whole flow-sheet, the ash content of total concentrates was 12.41%, and the total concentrate combustible recovery was 75.82%. This value increased by 8.12 percentage when compared to raw coal direct flotation without liberation and re-separation of middlings.

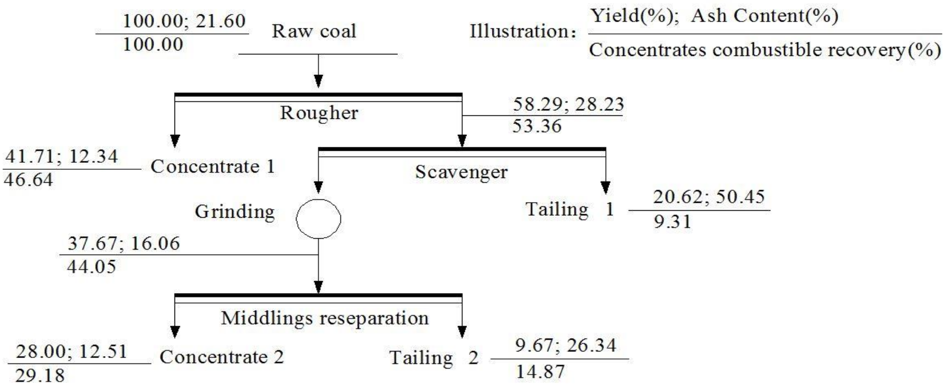


Fig. 9. Quantitative and qualitative flow-sheet of middling coal re-separation based on dissociated coal and rock

Physical characteristics of middlings

Middlings of Group 2 and Group 9, which are typical middling coal products in the grinding orthogonal test, were selected to determine mineral morphology of middling surface after dissociation. Figures 10–12 show that the content of coarse particle in middlings of Group 9 was larger than Group 8. A large number of micrometers grade clay minerals were observed on the middling coal surface and in the fissure of fractured ground particles after dissociation. Overly crushed coal particles dissociated and only little fine clay covered their smooth surfaces.

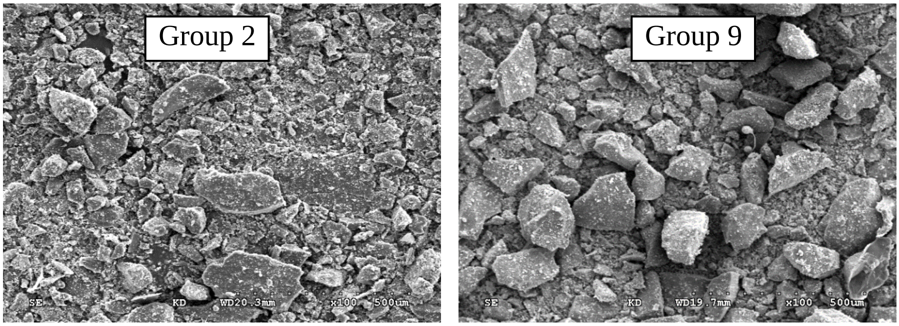


Fig.10. SEM photos of middlings of Group 2 and Group 9 coal after dissociation

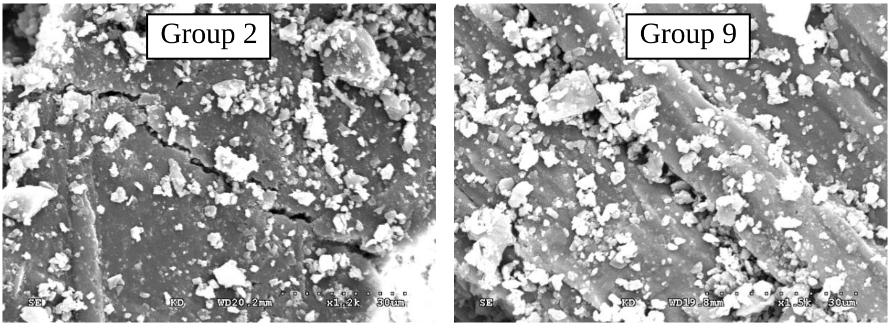


Fig. 11. Fracture SEM photos of middlings of Group 2 and Group 9 coal after dissociation

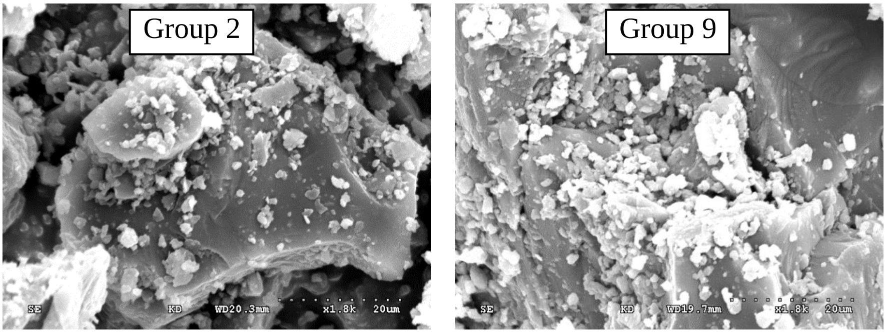
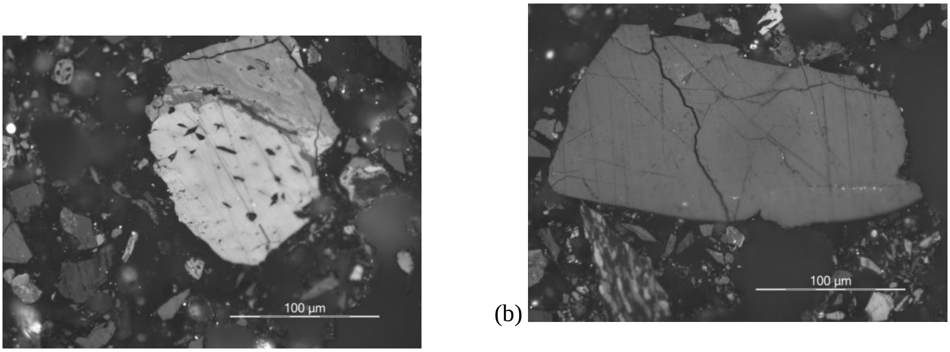


Fig. 12.Cleaves layer SEM photos of middlings of Group 2 and Group 9 coal after dissociation

Middlings of Group 9 in the grinding orthogonal test was selected for the density analysis. The petrography analysis was conducted for sample products with density less than 1.4 g/cm^3 (Table 7 and Fig. 13). The liberated middlings sample had a large particle size that ranged from micrometers to millimeters. Telocollinite and desmocollinite account for 96% and 4%, respectively. Invitrinites are the primary macerals, followed by telinite and a small amount of corpovitrinite. Semivitrinite is mainly composed of half-desmocollinite and includes some half-telinite. Inertinite mainly consists of macrosome and includes small amounts of fusinite and semifusinite. Exinite is mainly composed of liptodetrinite, resinite and sporophyte. Clay minerals are finely dispersed. Sulfide minerals are mainly dispersed. Figure 13a shows that middling coal is ground into micrometers. Figure13 b shows a large number of cracks and scratches on the particle surface, indicating that mineral grinding phenomenon occurred.

Table 7. Maceral composition of middlings of Group 9 after dissociation

Organic maceral, %				Inorganic maceral, %		
Vitrinite	Semivitrinite	Inertinite	Exinite	Clay	Sulphide	Carbonate
83.45	3.50	10.45	1.15	1.20	0.25	—



(a) (b)

Fig. 13. Macerals map of middlings of Group 9 with density less than 1.4 g/cm^3 after dissociation

Table 9 shows that the contact angle of raw coal, measured by the sessile drop technique, was 68°, and increased to 85° with grinding time. Then, the contact angle decreased as the time increased after 15 min of grinding. This result suggests that grinding prompted coal to dissociate from the mineral and improved hydrophobic properties on its surface. However, heterogeneous fine silt covered the surfaces of coal particles when coal was ground too long. Also overground inorganic mineral particles reduced contact angle.

Table 9. Hydrophobicityof coal middlings over grinding time

Grinding time, min	0	5	10	15	20
Contact angle, °	68	86.4	85.6	84.6	75.1

Conclusion

The results presented in this paper showed that the gangue mineral in the hard-to-separate coal slime of the Tangshan Kailuan mine was mostly kaolinite. The dominant size fraction of coal slime was –0.074 mm. The yield of middle density fraction was high and equal to 35%. The conventional separation process increased the difficulty-to-obtain high ash tailings and low ash concentrates.

The data on middling coal rock liberation in Group 9 showed that the maceral content in the low density fraction (less than 1.4 g/cm^3) was high and vitrinite enrichment was at its highest degree. Only a few inorganic minerals were found in low-density materials. Such finding was favorable to the flotation process. However, fine mud of clay minerals with high ash content was obtained during the liberation process.

The middlings coal rock liberation and re-separation tests were conducted in a laboratory scale. Grinding time had the greatest effect on the flotation performance. A high grinding density enhanced selective mineral liberation. The best flotation performance was obtained when the yield of −0.074 mm size fraction after liberation was 50%. The clean coal combustible recovery after re-separation based on intergrown particles liberation increased by 8.12 percentage points when compared to raw coal direct flotation for the equal clean coal ash content.

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