Understanding the difficult selective separation characteristics of high-ash fine coal

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Abstract: As the supply of high-quality coals decreases and mechanical coal mining becomes more widespread, the high selective recovery of high-ash fine coal has become a prominent problem in the flotation process. Herein, we discuss the main reasons why the selective separation of high-ash fine coal is difficult. The analysis of high-ash fine coal properties shows that coarse particles (0.25-0.5 mm) account for 22.53% of the total size fraction and that 57.90% of the coal is moderate- or high-density (+1.4 g/cm³) intergrowth. Grinding experiments show that the traditional rod mill has little impact on the liberation of the intergrowth. Instead, its main function is to adjust the particle size composition to ensure that the particle sizes of high-ash fine coal are within the particle size range suitable for flotation. The flotation results show that a clean coal yield of 30.42%, with a 12.46% ash content, is obtained with the optimal flotation parameters through the roughing and cleaning flotation process. However, the flotation results also show that in the separation of high-ash fine coal, it is difficult to obtain clean coal with a high yield and low ash content at the same time. This is mainly due to the similar floatability of moderate-density and low-density coal particles, which allows a large number of moderate-density coal particles to be recovered, and a significant slime coating of clay on the coal's surface that is generated during the flotation process. The results of this work provide valuable guidance for high-ash fine coal industrial flotation applications.

Keywords: high-ash fine coal, flotation, rod grinding, floatability, slime coating

1. Introduction

Coal is a major fossil fuel and a primary raw material in many countries. Nearly 60% of the world's electricity is generated using coal (Hacifazlioglu, 2016; Yang et al., 2019), with an annual consumption of over 5.0 billion metric tons (Lee et al., 2019). In general, coal is not a clean source of energy; harmful impurities, gangue, ash, sulfur, and water need to be removed or reduced to improve its quality (Demirbaş, 2002; Xing et al., 2017a; Xu et al., 2019). In addition, as the supply of high-quality coals gradually diminishes, low-quality coals, such as high-ash coal and high-sulfur coal, constitute an increasing proportion of coal production. In response, some enterprises have investigated the recovery of qualified clean coal from middlings and tailings (Yang et al., 2019; Yang et al., 2018). Furthermore, the increase in mechanical coal mining and the rapid development of heavy-medium coal preparation has enabled micronization, which can reduce the particle size of the flotation feed (Xing et al., 2017a). These factors have led to new scientific and production challenges not only in the amount of the separation of high-ash fine coal but also in its low selectivity (Gui et al., 2013; Xu et al., 2019). Therefore, it is very important to investigate methods for the efficient selective separation of high-ash fine coal.

To this end, many studies have been conducted to improve the selective performance of high-ash

fine coal, focusing on energy input (Gui et al., 2013; Xing et al., 2016), ultrasonic pretreatment (Ghadyani et al., 2018; Peng et al., 2018), flocculation flotation (Song and Valdivieso, 1998; Zou et al., 2019), oil agglomeration (Sahinoglu, 2018; van Netten and Galvin, 2018), and new flotation flowsheets (Çiçek et al., 2008; Xing et al., 2016; Yang et al., 2019). In recent decades, many scholars have found that one of the main reasons for the poor selectivity of high-ash fine coal is that clay minerals, such as kaolinite and montmorillonite, coat the surface of the coal or entrainment phenomenon. For example, Zhang et al. (2013) found a coat of kaolinite on coal surfaces via a scanning electron microscope (SEM). However, Xu et al. (2003) found that montmorillonite can inhibit coal flotation but not kaolinite clay. These inconsistent results may be due to the different chemical solutions used in the experiments. Xing et al. (2017b) reported that in the presence of 0.1 mol/L Ca^{2+}, the flotation recovery of fine coal with coats of either kaolinite or montmorillonite increased significantly, while the selectivity deteriorated.

The results mentioned above have provided profound insights into how to improve the process of selective separation of high-ash fine coal. However, considering its complex composition, multiple perspectives (not simply one single perspective, such as slime coating) are required to explain its poor selectivity. Therefore, in this work, the difficult selective separation characteristics of high-ash fine coal are studied using a wide range of components. The results provide valuable guidance for high-ash fine coal industrial flotation applications.

2. Materials and methods

2.1. Materials

A high-ash fine coal sample with an ash content of approximately 36% was collected from flotation tailing of a coal preparation plant in Hebei Province, China. The proximate analysis of the coal sample revealed that it had an air-dried moisture content of 1.35%, an ash content of 36.24%, a dry ash-free volatile matter content of 19.35%, and an air-dried fixed carbon content of 43.03%. In this study, the particle size distribution and particle density distribution of all coal samples were measured according to the previously reported details in the literature (Xie et al., 2013; Yang et al., 2019).

2.2. Rod milling test

Owing to the high-ash fine coal sample was collected from flotation tailing, which generally contains a large number of 0.25-0.5 mm coarse particles and 1.4-1.6 g/cm^3 intergrowth (Yang et al., 2018). Compared with ball milling, rod milling tends to achieve selective liberation of the intergrowth more easily (Li et al., 2018). Therefore, an XMB-67—200 × 240 mm roll mill (Wuhan Exploring Machinery Factory, Wuhan city, China) was used to liberate the intergrowth and reduce the particle size of the high-ash fine coal sample. The rod grinding test flowsheet was prepared based on our previously reported methods (Yang et al., 2018). The conditions for the grinding experiment were as follows: 30% slurry density, 100 kg/h capacity, 53 r/min mill speed, and media-filling rates of 11, 13, 15, 17, and 19%.

2.3. Progressive release flotation tests of high-ash fine coal

To evaluate the liberation of the high-ash fine coal after the rod grinding, progressive release flotation tests were conducted using an XFDII 1.0-L laboratory flotation machine (Jianfeng mill, Nanchang city, China). The progressive release flotation flowsheet is shown in Fig. 1(a). During the progressive release flotation tests, diesel oil (Zhuzhou Flotation Reagent & Chemicals Co., Ltd., Hunan province, China) and 2-octanol (Xilong Science Co. Ltd., Guangdong province, China) were used as the collector and frother, respectively. The pulp density, collector dosage, frother dosage, impeller speed, and air flow rate were maintained at 80 g/L, 518 g/t, 240 g/t, 1200 r/min, and 0.12 m^3/h, respectively. After flotation, the froth concentrates and tailings were filtered, dried at 80 °C for 12 h, and weighed. The ash contents of all the products were measured using a muffle furnace.

2.4. Batch flotation tests of high-ash fine coal

The high-ash fine coal sample was separated using two semi-industrial scale cyclonic-static microbubble flotation columns (FCSMCs): an FCSMC-300×3000 (diameter: 300 mm, height: 3000 mm) for the

roughing process and an FCSMC-200×3000 (diameter: 200 mm, height: 3000 mm) for the cleaning flotation process. The separation mechanism of FCSMCs is described in detail by Liu (1999), and the flotation flowsheet for these tests is shown in Fig. 1(b). Single factor tests were applied to determine the best separation conditions for the recovery of clean coal from the high-ash fine coal. To obtain the best separation results, the collector dosage, frother dosage, circulation pressure, and foam height were evaluated in the roughing stage. In addition, the foam height was evaluated in the cleaning stage. The concentrates of the roughing and cleaning processes were named C1 and C2, respectively.
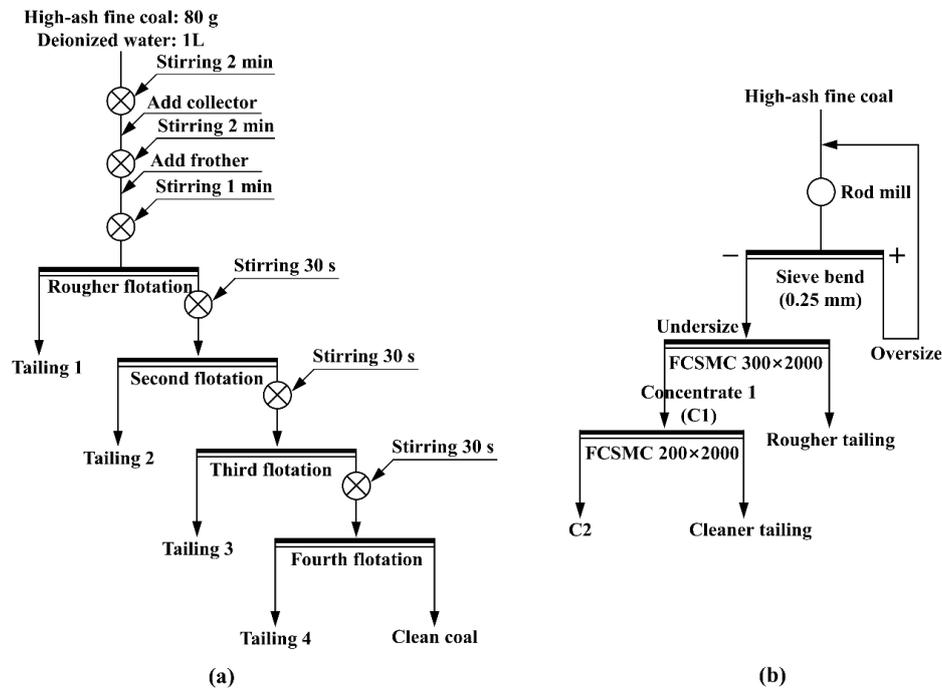
Fig. 1. Flowsheet of (a) progressive release flotation tests and (b) batch flotation tests of high-ash fine coal

2.5. SEM-EDS analysis

A Quanta TM250 scanning electron microscope with energy-dispersive X-ray spectroscopy (SEM-EDS, FEI Company, USA) was used to analyze the surface morphology and element composition of the C2 particles. First, a portion of C2 was collected and transferred to a test table. Then the surface of the coal sample was sprayed with a thin (~2 nm) layer of gold coating to improve the image quality. The SEM-EDS working parameters were as follows: accelerating voltage = 25 kV, pressure < $6×10^{-4}$ Pa, working distance ~14 mm.

3.   Results and discussion

3.1. High-ash coal characterization

Table 1 lists the particle size distribution of the high-ash coal sample. It shows that the size distribution of the coal particles was relatively uniform, while the ash content gradually increased with decreasing particle size. In particular, the -0.074 mm coal particles accounted for 42.03% of the yield, with a 47.60% ash content. The average ash content of the -0.074 mm fraction was 11.25% higher than the total ash content, which means that the high-ash fraction of -0.074 mm particles may have caused significant contamination during the flotation process.

Table 2 presents the particle density distribution of the high-ash fine coal sample. The low-density particles of -1.4 g/cm³ accounted for approximately 42% of the total and had a total ash content of 12.50%, which is acceptable for most industrial applications. The moderate-density particles of 1.4-1.6 g/cm³ composed 26.25% of the total and had an ash content of 34.01%, indicating that the coal sample contained many intergrowths. Lastly, 31.65% of the sample was made up of the high-density (+1.6

g/cm³) particles, which had an ash content greater than 70%. This implies that the coal sample contained a large number of gangue particles.

Table 1. Particle size distribution of high-ash fine coal sample

Particle size, mm	Yield, %	Ash, %	Oversize		Undersize	
			Yield, %	Ash, %	Yield, %	Ash, %
0.5-0.25	22.53	25.23	22.53	25.23	100.00	36.35
0.25-0.125	19.24	27.11	41.77	26.10	77.47	39.58
0.125-0.074	16.20	33.58	57.97	28.19	58.23	43.70
0.074-0.045	15.58	39.56	73.55	30.60	42.03	47.60
-0.045	26.45	52.34	100.00	36.35	26.45	52.34
Total	100.00	36.35				

Table 2. Particle density distribution of high-ash fine coal sample

Density, g/cm³	Yield, %	Ash, %	Cumulative floats		Cumulative sinks	
			Yield, %	Ash, %	Yield, %	Ash, %
-1.3	28.53	10.56	28.53	10.56	100.00	36.77
1.3-1.4	13.57	16.58	42.10	12.50	71.47	47.23
1.4-1.5	15.69	29.65	57.79	17.16	57.90	54.41
1.5-1.6	10.56	40.51	68.35	20.76	42.21	63.61
1.6-1.8	8.30	53.68	76.65	24.33	31.65	71.32
+1.8	23.35	77.59	100.00	36.77	23.35	77.59
Total	100.00	36.77				

3.2. Liberation characteristics of high-ash fine coal

Figs. 2(a) and 2(b) show the particle size distribution and progressive release flotation results, respectively, of the coal sample for different media-filling rates. As shown in Fig. 2(a), the particle size distribution curves shift gradually to the top left as the media-filling rate of the rod mill increases. This demonstrates that as the media-filling rate increased, the coarse particle fraction decreased while the fine particle fraction increased. Further analysis indicated that there was an inverse linear relation between the d_{50} and d_{80} passing size values and the media-filling rate. This can be explained by the fact that a higher media-filling rate increased the probability of collision between the coal and rod. In general, the particle size range suitable for fine coal flotation is 0.074-0.25 mm. For a media-filling rate of 15%, d_{50} and d_{80} were 0.11 and 0.26 mm, respectively, and for a media-filling rate of 17%, d_{50} and d_{80} were 0.09 and 0.24 mm, respectively. Therefore, the particle sizes of the coal sample matched the particle size range suitable for fine coal flotation (Fuerstenau et al., 2007).

Fig. 2(b) shows the results of progressive release flotation for the grinded coal products. At the required maximum coal ash content of 12.5%, the clean coal yields of the grinded coal products were 28.23, 24.69, 36.46, 33.56, and 32.18% for media-filling rates of 11, 13, 15, 17, and 19%, respectively. However, the qualified clean coal yield did not significantly increase with a change in the particle size distribution, which suggests that the rod milling did not significantly affect the intergrowth liberation of the high-ash fine coal. This phenomenon is possibly due to minerals such as quartz, pyrite, kaolinite, and maceral, which were closely combined in the high-ash fine coal sample (Yang et al., 2019). Therefore, it can be concluded that traditional rod grinding process has little impact on the liberation of minerals and maceral components in the high-ash fine coal. Instead, its main function is to adjust the

particle size distribution to ensure that the particle size of the grinded products meets the flotation requirements.
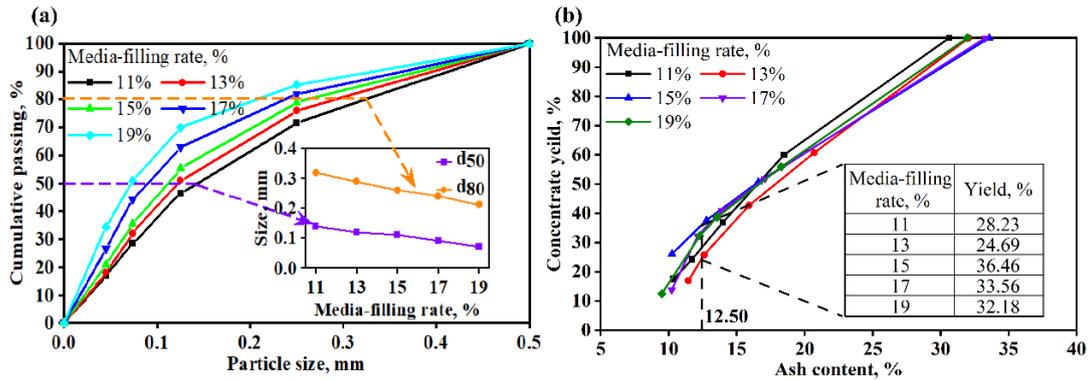
Fig. 2. The liberation characteristics of high-ash fine coal for different media-filling rates: (a) particle size distribution and (b) curves of progressive release flotation for grinded products

3.3. Condition experiments of roughing flotation

The capacity determines the residence time of the coal particles in the pulp in the flotation column. Fig. 3(a) displays the effect of capacity on the flotation performance of the high-ash fine coal sample. As the capacity of the pulp increased, the recovery of combustible matter decreased. However, the ash content decreased slightly and then increased sharply. This was because many fine slime minerals with high ash content required unselective recovery at a higher capacity, resulting in an increase in the ash content of C1. When the capacity of the pulp was 1 m^3/h, a superior result of approximately 72% combustible matter recovery with a 16% ash content was obtained. Therefore, further experiments were conducted at a capacity of 1 m^3/h.

Fig. 3(b) presents the combustible matter recovery and ash content of C1 for different collector dosages ranging from 1200 to 2400 g/t and a constant capacity of 1 m^3/h. Both the combustible matter recovery and ash content of C1 increased as the collector dosage increased. In particular, when the collector dosage was increased from 1200 to 2100 g/t, the recovery rate of the combustible matter increased more rapidly than that of the ash content. However, for collector dosages ranging from 2100 to 2400 g/t, the ash content of C1 increased more sharply than that of the combustible matter. This can be ascribed to the fact that, for a higher collector dosage, the collector also enhanced the hydrophobicity of coal particles with high ash content. Therefore, the optimal separation of 63.77% combustible matter recovery and 16.11% ash content corresponded to a diesel oil dosage of 2100 g/t.
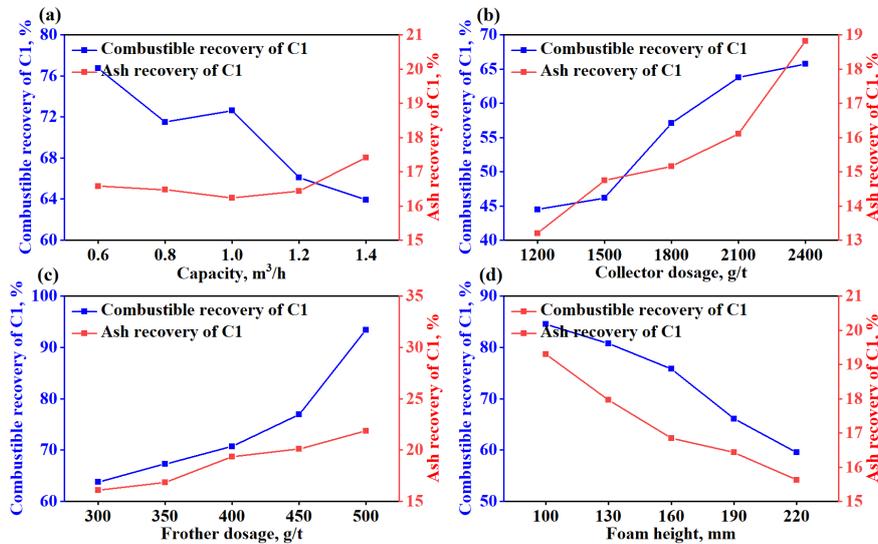
Fig. 3. Combustible matter recovery and ash content of C1 with various flotation parameters: (a) capacity, (b) collector dosage, (c) frother dosage, and (d) foam height

Fig. 3(c) demonstrates the effect of the frother dosage on the flotation performance of the high-ash fine coal sample. The capacity and collector dosage of the diesel oil were held constant at 1 m³/h and 2100 g/t, respectively. As the frother dosage was increased from 300 to 350 g/t, the recovery of combustible matter ranged from 60 to 70%, with a corresponding ash content of approximately 16%. When the frother dosage was increased from 350 to 500 g/t, the combustible matter recovery increased from 70 to 93% while the ash content remained above 20%. The increase in combustible matter recovery and ash content in C1 was mainly due to the higher frother dosage, which produced a more stable foam layer. This helped to form more bubble-particle aggregate and also promoted the entrainment of clay slime (Han et al, 2014). To ensure that the ash content was as close as possible to the upper limit of 12.5% required in the coke industry while maximizing the combustible matter recovery, a frother dosage of 350 g/t was chosen.

Fig. 3(d) displays the effect of the foam height in the FCSMC 300×3000 on the flotation performance of the high-ash fine coal sample. The capacity, collector dosage, and frother dosage were held at 1 m³/h, 2100 g/t, and 350 g/t, respectively. The results clearly show that as the foam height increased, the combustible matter recovery and ash content of C1 decreased. This occurred primarily because coarse coal particles or weak hydrophobic particles fell off the bubbles as the foam height increased. In addition, the secondary enrichment of the coal was facilitated by a higher foam layer, which helped to reduce the entrainment of high ash materials and decrease the ash content of C1. The optimal separation of 78.85% combustible matter recovery and 16.84% ash content corresponded to a foam height of 160 mm.

3.4. Condition experiments of cleaning flotation

The condition tests of roughing flotation show that the optimal separation of the high-ash fine coal (approximately 70% combustible matter with a 15-17% ash content) was achieved at a capacity of 1 m³/h, a collector dosage of 2100 g/t, a frother dosage of 350 g/t, and a foam height of 160 mm. To meet the 12.5% ash content requirement of qualified clean coal, it was necessary to further investigate the cleaning flotation process. The ash content of C1 was able to be decreased significantly by controlling the foam height parameter in the flotation column. Therefore, the effect of foam height on the flotation index was investigated in the cleaning flotation flowsheet based on the roughing flotation results described above.

Table 3. Effect of cleaning separation foam height on flotation index

Foam height, mm	C2			Cleaner tailing		Rougher tailing	
	Yield, %	Ash, %	Recovery of combustible, %	Yield, %	Ash, %	Yield, %	Ash, %
200	64.51	15.89	65.25	1.39	60.78	34.09	48.93
250	64.75	15.03	65.80	2.78	47.82	32.47	50.72
300	66.67	14.91	67.46	3.72	33.64	29.61	60.38
350	48.21	13.78	49.57	13.12	24.81	38.66	48.03
400	38.04	12.85	39.65	25.77	21.59	36.19	47.00
450	30.42	12.46	31.61	38.58	27.41	30.99	40.36

The results are presented in Table 3. As the height of the foam layer increased, the yield of C2 and the combustible matter recovery decreased significantly, while the cleaner tailing showed the opposite trend. Meanwhile, the ash content of C2 and the cleaner tailing decreased continuously with increasing foam height. Specifically, when the foam height was 300 mm or less, a yield of approximately 65% combustible matter with a 15% ash content was obtained as a result of the cleaning flotation process. This result was largely attributable to the recovery of a large amount of intergrowth with high ash content, which was the result of the cleaner tailing (column second from the right in Table 3). However, the yield of the cleaner tailing was only 1-4%. As the foam height increased to more than 300 mm, the yield and the combustible matter recovery for C2 decreased rapidly from 66.67 to 30.42% and from 67.46

to 31.61%, respectively. Meanwhile, the ash content of C2 decreased from 13.78 to 12.46%. The ash content of the C2 clean coal met the requirements only when the foam height was 450 mm. In addition, the yield of the cleaner tailing increased steeply from 3.72 to 38.58%, with an average ash content of ~24.50%. This value was a result of the fact that a significant secondary enrichment of a high foam layer prevented the intergrowth with a high ash content from rising to the top of the foam layer (Gui et al., 2010; Ni et al., 2015). That is, a high foam layer was needed to ensure the ash content of the clean coal was less than 12.5%, but this resulted in a low yield of clean coal. On the contrary, a yield of more than 60% clean coal could be achieved at a low foam layer height, but the ash content of the clean coal exceeded 12.5%. Thus, the cleaning flotation tests indicated that it was difficult to achieve a high yield and a low ash content at the same time.

3.5. Separation characteristics of clean coal

To further investigate the difficult selective separation characteristics of high-ash fine coal, the clean coal samples generated with foam heights of 250 and 450 mm were analyzed in terms of their particle size distributions, particle density distributions, and SEM images.

Table 4 shows the particle size distributions of the clean coal samples generated using foam heights of 250 mm and 450 mm. The two samples exhibited similar particle size distributions, as both were dominated by fine particles with sizes of -0.074 mm, which were responsible for more than 60% of the yield. However, the ash contents of the 250 mm sample were approximately 1-4 percentage points higher than those of the 450 mm sample. In general, the finer the particle size, the larger the absolute difference in ash content. This indicates that higher foam layers reduced the ash content of the clean coal, especially for fine particle sizes.

Table 4. Particle size distributions of clean coal produced using foam heights of 250 and 450 mm

Size, mm	250 mm		450 mm		Ash difference, %
	Yield, %	Ash, %	Yield, %	Ash, %	
0.25-0.5	7.83	9.83	8.53	8.37	1.46
0.25-0.125	10.88	10.42	11.26	9.75	0.67
0.125-0.074	16.85	13.65	14.73	11.63	2.02
0.074-0.045	27.68	15.38	30.84	13.52	1.86
-0.045	36.76	18.67	34.64	14.23	4.44
Total	100.00	15.32	100.00	12.62	

Table 5 shows the density distribution of the clean coal samples generated at foam heights of 250 mm and 450 mm. For the 250 mm sample, the yields of the low-density fraction (-1.4 g/cm^3), moderate-density fraction (1.4-1.6 g/cm^3), and high-density fraction (+1.6 cm^3) were 52.52, 34.60, and 12.88%, respectively. For the 450 mm sample, the yields of the corresponding density fractions were 69.06, 19.02, and 11.92%, respectively. In general, the higher the density of the coal particles, the more the coal contained minerals (such as kaolinite, quartz, and montmorillonite) and the weaker the coal's surface hydrophobicity. This resulted in a smaller adhesion force between the coal particles and bubbles during the flotation process. After increasing the height of the foam layer, the upper layer of bubbles coalesced more vigorously, leading to the desorption of the moderate- and high-density coal particles from the bubbles. In turn, this led to a decrease in the proportion of the moderate- and high-density coal particles in the clean coal. It can be concluded that the ash content of the clean coal produced using a foam layer height of 250 mm exceeded the requirement (12.5%) primarily because the moderate-density materials with high ash contents were recovered in the flotation process. Furthermore, for the foam layer height of 250 mm, the yields of the clean coal for the three density fractions of 1.4-1.5, 1.5-1.6, and 1.6-1.7 g/cm^3 were 23.76, 18.75, and 15.85%, respectively. The corresponding ash contents were 10.54, 14.38, and 22.46%, respectively. The small difference between yields demonstrates that the components with different densities in high-ash fine coal have similar floatability characteristics. This can cause difficulty

in accurately separating the high-ash fine coal from the ash content so that the clean coal exceeds the standard (Gui et al., 2017; Wang et al., 2018).

Table 5. Particle density distributions of clean coal produced at foam heights of 250 and 450 mm

Density	250 mm		450 mm	
g/cm³	Yield, %	Ash, %	Yield, %	Ash, %
-1.3	28.76	5.42	35.42	4.89
1.3~1.4	23.76	10.54	33.64	9.42
1.4~1.5	18.75	14.38	11.45	12.68
1.5~1.6	15.85	22.46	7.57	20.43
1.6~1.8	7.46	31.95	8.20	31.00
+1.8	5.42	54.67	3.72	52.80
Total	100.00	15.67	100.00	12.41
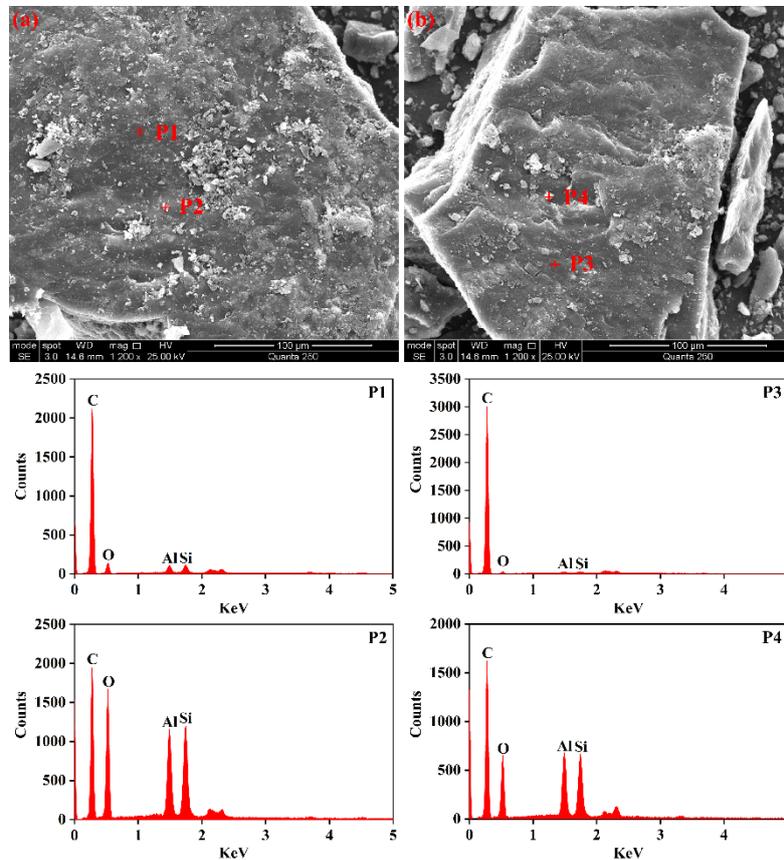
Fig. 4. Surface morphology and elemental spectrograms of clean coal produced at a foam layer height of (a) 250 mm and (b) 450 mm

Figs. 4(a) and 4(b) present the surface morphology and elemental spectrograms of the clean coal samples produced at foam layer heights of 250 mm and 450 mm, respectively. The two clean coal surfaces were coated with a large quantity of fine particles. According to the SEM images, points P1 and P3 are clean areas. The elemental spectrum analysis shows that the main elemental composition of those areas was carbon (C), which can be characterized as coal. Points P2 and P4 represent the fine particles, and the spectrogram indicates that those fine particles were clay because the main elemental compositions were oxygen (O), aluminum (Al), and silicon (Si). The carbon was detected in P2 and P4 mainly because the diameter of the electron beam spot (~10 μm) produced by the SEM was larger than

the particle size of clay, which is often a few microns or even smaller. Therefore, it is inevitable that some clean areas would be detected. In summary, the SEM images indicated that heterogeneous condensation of fine clay onto the surface of the coal occurred during the flotation process. The slime coating on the surface of the coal was also a cause of the high ash content of the clean coal and the poor selectivity of the high-ash fine coal (Ali et al., 2018; Peng et al., 2018; Xu et al., 2003). On the other hand, the density of the fine clay coat on the surface of the clean coal produced using a foam layer height of 450 mm was less than that of the clean coal produced using a foam layer height of 250 mm. This indicates that a high foam layer reduced the floating of coal particles covered by slime minerals.

4. Conclusions

The selective separation characteristics of high-ash fine coal were analyzed comprehensively in this study. The main conclusions of this study are the following:

(1) The particle size distribution of the high-ash fine coal sample was relatively uniform. The ash content gradually increased with decreasing particle size, while the ash content of -0.074 mm coal particles was 11.25% higher than that of the whole sample. This caused undesirable effects during the flotation process. The total yield of moderate- and high-density particles in the high-ash fine coal was more than 55%, with an average ash content of 54.10%. This indicates that there were large quantities of intergrowth and clay minerals.

(2) After rod grinding was complete, the particle size range of the high-ash fine coal was suitable for flotation requirements at media-filling rates of 15 or 17%, but the qualified clean coal yield did not significantly increase. This shows that it is difficult to liberate the intergrowth in high-ash fine coal by traditional grinding processes, and that the main function of grinding is to adjust the particle size distribution of high-ash fine coal.

(3) The qualified clean coal (less than 12.5% ash content) was obtained from high-ash fine coal using two FCSMCs with roughing and cleaning flotation flowsheets. A good flotation performance (30.42% yield, 12.46% ash content, and 31.61% combustible matter recovery) was achieved with a 1 m^3/h capacity, a 2100 g/t collector dosage, a 350 g/t frother dosage, a 160 mm foam height for roughing, and a 450 mm foam height for cleaning.

(4) It was difficult to obtain a high yield and a low ash content for the clean coal at the same time in the high-ash fine coal flotation process when diesel oil and 2-octanol were used as the collector and frother, respectively. The main reasons for this were that the similar floatability of moderate- and low-density coal particles allowed a majority of the particles to be recovered and that the heterogeneous condensation of slime minerals onto the surface of the coal during the flotation process caused the high ash content of the clean coal and the poor selectivity of the high-ash fine coal.

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