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FLOTATION BEHAVIOR OF HARD-TO-SEPARATE AND HIGH-ASH FINE COAL

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Abstract: The flotation behavior of hard-to-separate and high-ash fine coal was investigated using conventional flotation with constant power input. A new flotation process, based on energy input and distribution, was designed to lower the ash content of concentrate. The results obtained using Fourier transform infrared (FTIR) analysis show that the coal samples have good floatability because of many hydrophobic and few hydrophilic functional groups. Under a constant power input, a large number of ash-forming materials floated into a froth product at the start of flotation. Based on the Fuerstenau upgrading curves, it was determined that the 0.25-0.074 mm size fraction range showed the worst selectivity when compared with 0.50-0.25 mm and -0.074 mm size fractions. The desired concentrate with an ash content of 13.98%, 27.59% of ash recovery, and 80.01% combustible matter recovery could be obtained by transferring the excess energy of the flotation-conditioning stage to the pre-conditioning stage and increasing the power input step-by-step in the flotation-conditioning stage at equal total energy consumption.

Keywords: *flotation behavior, fine coal, Fuerstenau upgrading curve, energy input, selectivity*

Introduction

Froth flotation is an effective separation method for fine coal cleaning based on differences in the surface hydrophobicity between the organic and mineral matters (Vanangamudi et al., 1988; Tao et al., 2002; Barraza et al., 2013). However, when the flotation difference between particles is either minimal or fine slimes content in coal samples is high, conventional froth flotation methods mainly suffer from lack of selectivity for fast-floating coals because of flotation of middlings and entrainment of fines in the froth (Polat et al., 2003). To maximize coal production and utilization, coal mining operations and preparations have been highly mechanized. It resulted in high

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proportions of ash matters in the raw coal, such as clays and middlings, which posed a severe challenge to fine coal flotation (Jia et al., 2000; Xu, 2003; Tao et al., 2009). High-efficiency flotation of hard-to-separate and high-ash slime has been a research interest in China. It is necessary to search for a new method for enhancing the flotation performance of hard-to-separate and high-ash fine coal.

Many studies have been conducted to investigate the selectivity and kinetics of hard-to-separate and high-ash fine coal flotation. Fines recovery by entrainment has a detrimental effect on the flotation selectivity (Akdemir and Sonmez, 2003; Xu et al., 2005; Wang and Peng, 2013; Liu and Peng, 2014). It is well known that the particle size of fine coal is the most important parameter that effects the recovery and entrainment behavior of coal and ash materials. It was shown recently by Gui et al. (2014a, 2014b) that higher flotation efficiency index could be obtained through an early low-energy input for the recovery of easy-to-float materials and late high-energy input for the recovery of difficult-to-float materials. Entrainment could be significantly depressed using a new flotation process based on the energy input and distribution (Gui et al., 2014a; 2014b). Kinetics is an important aspect of fine coal flotation (Wills and Napier-Munn, 2006; Li et al., 2012). The flotation rate determines the time needed for removal of ash from raw coal. Coal particles with different sizes and densities have different flotation rates. Most flotation rate tests show that the fine coal particles can be described by the first-order kinetic model (Chelgani et al., 2010; Abkhoshk et al., 2010). Moreover, Li et al. (2012) found that the combustible matter recovery could be approximated with the first-order kinetic equation and flotation of ash forming minerals by the second order equation. The flotation behavior of oxidized coals has also been extensively studied (Dey, 2012; Xia et al., 2014). However, the complex behavior of hard-to-separate and high-ash slime flotation is not well understood.

The present investigation was undertaken to search for an effective separation method for hard-to-separate and high-ash slime. In this study, the flotation behavior of different size fractions was analyzed. A new flotation process was designed based on the energy input and distribution, and a comparison of flotation performance at constant and variable power input was carried out.

Experimental

Materials

Dry fine coal samples were obtained from Wuhai City, Inner Mongolia, China. The composition of the sample is shown in Table 1. The particle size is the main factor that affects flotation of slime. A wet-screening test of the coal samples was conducted with a set of standard sieves (0.500, 0.250, 0.125, 0.074, and 0.045 mm). The particle size and ash content distributions are presented in Table 2. It indicates that the ash content gradually increases as the particle size decreases. The yield and ash contents of the fine-grained fractions are high. Fractions less than -0.074 mm account for 35.28% of the yield with an ash content of 40.41%, which is 8.41% higher than the total ash content of samples. It should be noted that these fine fractions with high ash can easily degrade concentrates through mechanical entrainment during the flotation process.

Table 1. Proximate analysis of coal samples (air dried)

Moisture ,%	Volatile matter ,%	Fixed carbon,%	Ash ,%
2.37	15.79	48.73	33.11

Table 2. Particle size and ash content distributions of coal sample

Size fractions, mm	Yield,%	Ash,%	Oversize		Undersize	
			Yield,%	Ash,%	Yield,%	Ash,%
0.50-0.25	16.38	29.16	16.38	29.16	100.00	31.99
0.25-0.125	32.85	26.25	49.24	27.22	83.62	32.55
0.125-0.074	15.49	28.01	64.72	27.41	50.76	36.63
0.074-0.045	10.08	34.52	74.80	28.37	35.28	40.41
-0.045	25.20	42.76	100.00	31.99	25.20	42.76
Total	100.00	31.99	-	-	-	-

The density composition of the coal samples is presented in Table 3. It illustrates that the yield of +1.8 g/cm^3 is 27.10%, which is higher than those of the other fractions. Coal at an intermediate density of fraction 1.5 g/cm^3 to 1.8 g/cm^3 is 22.35%, with an ash content reaching 30.26%. Theoretically, obtaining qualified product with an ash content of 14% is not difficult because the density±0.1 content is lower than 20%.

Table 3. Density analysis data of samples

Density fractions, g/cm^3	Yield, %	Ash content, %	Cumulative of floating objects		Cumulative of sediments		Content of density±0.1	
			Yield, %	Ash, %	Yield,%	Ash, %	Density, g/cm^3	Yield, %
-1.3	10.08	3.16	10.08	3.16	100.00	31.34	1.30	29.37
-1.3+1.4	19.29	7.73	29.37	6.16	89.92	34.50	1.40	40.47
-1.4+1.5	21.18	15.00	50.55	9.87	70.63	41.81	1.50	33.13
-1.5+1.6	11.95	24.44	62.50	12.65	49.45	53.29	1.60	17.15
-1.6+1.8	10.40	36.96	72.90	16.12	37.50	62.48	1.70	10.40
+1.8	27.10	72.27	100.00	31.34	27.10	72.27	1.80	32.30
Total	100.00	31.34	-	-	-	-	-	-

Fourier transform infrared (FTIR) spectra of coal samples with low-density (< 1.5 g/cm^3), moderate-density (1.5 g/cm^3 to 1.8 g/cm^3), and high-density (> 1.8 g/cm^3) fractions are shown in Fig. 1. The peak at 1030 cm^{-1} is attributed to ashbearing minerals, and the peaks at 3694 and 3618 cm^{-1} are attributed to kaolinite, which is detrimental to flotation (Jia et al., 2000; Jena et al., 2008). The CH_3 and CH_2 groups were characterized by peaks approximately at 2850 cm^{-1}–2930 cm^{-1} and 1435 cm^{-1}. The peaks at 3420 and 1650 cm^{-1} are attributed to -OH and C=O groups, respectively (Pietrzak and Wachowska, 2003; 2004; Grzybek et al., 2006; Xia and Yang, 2013; Xia et al., 2013). The peak at 1596 cm^{-1} is attributed either to benzene rings or to C=C. The peak at 915 cm^{-1} may be a phenolic hydroxyl group. The peaks at 800 –650 and 3030 cm^{-1} are attributed to C-H in aromatic and benzene rings. The weak peaks of C=O and –OH and the considerably strong and sharp peaks of CH, CH_2, and CH_3 suggest good floatability of the coal samples, which have relatively few hydrophilic functional groups and a number of hydrophobic functional groups. Nevertheless, coal particles with good hydrophobicity may lead to particle aggregations because the particles are difficult to disperse in the flotation pulp, and could result in the low selectivity as the ash materials are entrapped in the agglomerated structure (Polat et al., 2003).

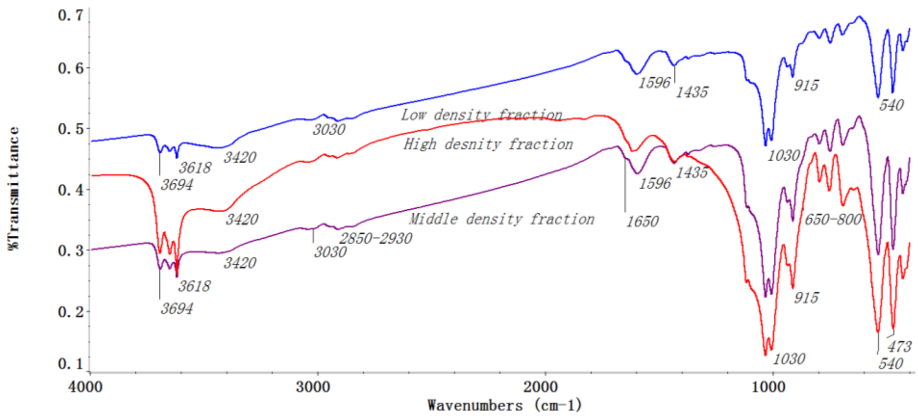


Fig. 1. FTIR spectra of coal samples with different density fractions

Method

Flotation rate tests were conducted in an XFDIII 0.75 dm^3 laboratory flotation machine. Kerosene and octanol were used as the collector and frother, respectively. They are commonly used in industry. The impeller rotation speed, collector dosage, frother dosage, solid content, and air flow rate were kept constant at 2100 rpm, 300 g/Mg, 100 g/ Mg, 100 g/ dm^3, and 2.5 dm^3/min, respectively. Firstly, the pulp was pre-wetted for 2 min. After that, first collector and then frother were added to the pulp. The collector and frother conditioning periods were 1 and 0.5 min, respectively. The flotation process was divided into four stages and continued for 3 min. Four flotation concentrate products, namely, concentrate 1 (C1), concentrate 2 (C2), concentrate 3 (C3), and concentrate 4 (C4) were consecutively collected after 20, 40, 100, and 180 s, respectively. Flotation concentrates and tailings were filtered, dried at 80°C and weighed. For the ash analysis, approximately 10 g of the dried sample from each product was first ground using a mortar and pestle. Then, approximately 2 g of a ground sample from each product was burned in an oven at 815 °C for 2 h. The remaining ash was weighed to calculate the ash content (William et al., 2010).

Performance evaluation

The required ash content of clean coal in this experiment was 14%. The combustible matter and ash recoveries, which are used to evaluate the flotation performance, were calculated using the following general equations (Wills et al., 2006; Gupta et al., 2009):

$$\text{Combustible recovery (\%)} = \frac{(F-T)(100-C)}{(C-T)(100-F)} \cdot 100 \tag{1}$$

$$\text{Ash recovery (\%)} = \frac{(F-T)C}{(C-T)F} \cdot 100 \tag{2}$$

where F, C, and T are the ash content of the feed, concentrates, and tailings, respectively.

In this paper, the Fuerstenau upgrading curves are used for characterization, comparison and analysis of the separation process. The curves are very useful in the analysis of de-ashing and desulfurization of coal flotation results (Fuerstenau et al., 1992). The kinetic equations relating the recoveries of two components in separation products and time, when combined together to eliminate the time parameter, provide Fuerstenau upgrading curves, which relate, for instance, the recoveries of two components in concentrate. Additionally, there are simple empirical equations which can be used to approximate the separation results (Drzymala and Ahmed, 2005; Drzymala et al., 2010).

Results and discussion

Flotation behavior analysis using constant power input

The results of flotation kinetic experiments at the constant impeller rotation speed of 2100 rpm are shown in Table 4. The cumulated ash content, combustible matter and ash recoveries increased as the flotation time increased. Clean coal with an ash content of 19.03%, 43.19% of ash recovery, and 86.57% of combustible matter recovery can be obtained with the flotation time up to 180 s, at which point the flotation process was nearly completed. Interestingly, the ash content of concentrates rapidly increased to 17.41%, which exceeded the required ash content of 14% within 20 s of flotation. The ash recovery of the first concentrate C1 was 17.38%, which was much higher than that of the others. This indicates that a large number of ash-forming materials floated into the froth products at the onset of flotation, and completely deteriorated the selectivity. The total ash of clean coals did not significantly increase as the flotation time increased from 20 s to 180 s. However, the floated ash content of the products after 20 s remained at a high level. The ash contents of concentrates C2, C3, and C4 were 18.95, 20.10, and 23.15%, respectively. The ash recovery of C2, C3, and C4 decreased because the yield decreased. The results similar are to flotation of oxidized coals. It is difficult to obtain a product with the ash content of 14%.

The flotation behavior of different particle sizes of the clean coal was also investigated in this paper. As shown in Table 5, the recovery of all size fractions decreased as the flotation proceeded. With a three-fold increase in flotation time, C3 recovery of -0.074 mm was higher than that of C2. Meanwhile, most materials in the concentrate rapidly floated within 40 s of flotation, thus illustrating the high flotation rate of coal samples. However, the ash content of all size fractions, totally exceeded the initial product requirement for ash content of 14%, especially for the -0.125 mm size fraction. Thus, the high ash content of cleans was the result of the flotation behavior of all size fractions. The unliberated coarse particles floated and this was the reason why the ash content of fraction of +0.125 mm was high. The high ash content of the -0.125mm size fraction is likely attributed to nonselective flotation of a large number of fine mud or ash particles. These high ash fractions floated into froth at the beginning of flotation.

Table 4. Results of coal flotation

Products	Ash content,%	Combustible matter recovery,%	Ash recovery,%	Cumulated ash content,%	Cumulated combustible matter recovery,%	Cumulated ash recovery,%
C1	17.41	38.85	17.38	17.41	38.85	17.38
C2	18.95	19.40	9.63	17.93	58.25	27.01
C3	20.10	18.35	9.80	18.46	76.60	36.81
C4	23.15	9.98	6.38	19.03	86.57	43.19
Tailings	66.60	13.43	56.81	32.03	100.00	100.00
Total	32.03	100.00	100.00	-	-	-

Table 5. Results of different size fractions flotation

Products	Combustible matter recovery,%					Ash content,%				
	0.50-0.25	0.25-0.125	0.125-0.074	0.074-0.045	-0.045	0.50-0.25	0.25-0.125	0.125-0.074	0.074-0.045	-0.045
C1	36.82	43.32	49.51	29.72	28.76	15.92	16.91	16.99	19.15	19.95
C2	18.61	20.40	19.17	21.53	17.39	16.55	18.82	18.74	19.69	21.49
C3	18.31	16.64	16.61	25.08	19.61	15.35	18.22	20.64	20.26	24.82
C4	5.99	9.30	8.35	13.36	14.00	14.71	18.58	24.93	24.18	28.91
Tailings	20.27	10.34	6.36	10.31	20.24	56.32	61.06	73.10	74.28	71.43
Total	100.00	100.00	100.00	100.00	100.00	29.16	26.25	28.01	34.52	42.76

To evaluate the separation process, a selective parameter from the Fuerstenau recovery-recovery upgrading curve was used. A mathematical formula for the approximation of the Fuerstenau curves could be derived from kinetic considerations. Commonly, the flotation process can be explained as a rate process, because the rate of recovery of either coal or ash particles is proportional to the concentration of either coal or ash particles remaining in the pulp (Gupta et al., 2009). The classical first-order equation was used to express the flotation kinetics of both the combustible matter and ash recoveries:

$$\varepsilon = \varepsilon_{\infty}(1 - e^{-k_1 t}) \tag{3}$$

$$\varepsilon_a = \varepsilon_{a\infty}(1 - e^{-k_2 t}) \tag{4}$$

where ε and ε_a are the combustible matter and ash recoveries in the concentrate, respectively; ε_{∞} and $\varepsilon_{a\infty}$ are the maximum combustible matter and ash recoveries in the concentrate, respectively; k_1 and k_2 are the first order flotation rate constant of combustible matter and ash forming material, respectively; and t is the flotation time. Matlab7.0 was used to calculate the values of k_1 and k_2. The kinetics fitting results of different size fractions are shown in Table 6.

Table 6. The kinetics fitting results of different size fractions

Materials	Size fraction，mm	ε_{∞}	k_1	Correlation coefficient square (R^2)
Combustible	0.50-0.25	78.82	1.84	0.9939
	0.25-0.125	86.96	1.99	0.9725
	0.125-0.074	90.61	2.23	0.9638
	0.074-0.045	91.02	1.19	0.9944
	-0.045	79.16	1.27	0.9758
	Total	84.38	1.76	0.9767
	Size fraction，mm	$\varepsilon_{a\infty}$	k_2	Correlation coefficient square (R^2)
Ash	0.50-0.25	36.12	1.88	0.9959
	0.25-0.125	53.00	1.86	0.9775
	0.125-0.074	53.65	1.84	0.9556
	0.074-0.045	44.67	1.05	0.9901
	-0.045	33.62	0.91	0.9771
	Total	42.27	1.50	0.9735

The combustible matter rate of the 0.25-0.074 mm size fraction was higher than those of -0.074 mm fine fraction and +0.25 mm coarse fraction. This result can probably be attributed to the low collision probability of fine particles and the high detachment probability of coarse fraction. However, the ash materials rate decreased with the particle size decreasing. It should be noted that the ash materials rate was only slightly lower than that of the combustible matter, which likely illustrates the poor flotation selectivity. Table 6 also shows that a good fit for the flotation kinetics results of both combustible and ash materials was obtained using the first order dynamic model as the correlation coefficient square R^2 values exceeded 0.9500. These results agree well with other researchers (Vapur et al., 2010). Therefore, the combinations of two first-order kinetic Eqs (3) and (4) gives :

$$\varepsilon = \varepsilon_{\infty}\left[1-\left(\frac{\varepsilon_{a\infty}-\varepsilon_a}{\varepsilon_{a\infty}}\right)^{\frac{k_1}{k_2}}\right]. \tag{5}$$

Assuming that ε_{∞}=$\varepsilon_{a\infty}$=100% and $=\frac{k_1}{k_2}$, which can evaluate separation selectivity, then Eq (3-3) can be simplified to the following equation (Bakalarz and Drzymala, 2013):

$$\varepsilon = 100\left[1-\left(\frac{100-\varepsilon_a}{100}\right)^{k}\right]. \tag{6}$$

The fitting results of the Fuerstenau upgrading plots of different size fractions, fixed starting and ending points are shown in Fig. 2 and Table 7. High R^2 values were obtained using Eq. (6), which indicates that the size range of 0.25-0.074 mm show the worst selectivity compared with those of 0.50-0.25 and -0.074 mm size fractions. This is probably due to high collision and low detachment probabilities of the middle-sized fraction 0.25-0.074 mm with good hydrophobicity.

The phenomena observed above may be attributed to two factors: (i) an excessively high-energy input during flotation, especially at the early stage, increased the number of collisions among coal, ash-forming particles, and air bubbles, which led to the mechanical entrapment and entrainment of ash with coal; and (ii) good hydrophobicity of the coal samples based on the FTIR resulted in the formation of particle aggregates. A large amount of ash particles were entrapped in the agglomerate structure, reducing the flotation selectivity. A new flotation process based on energy input and distribution was proposed to attempt to reduce the ash content of clean coal.

Table 7. Fitting results of different size fractions

Size fraction, mm	k	Correlation coefficient square (R^2)
0.50-0.25	2.97	0.9866
0.25-0.125	2.29	0.9892
0.125-0.074	2.60	0.9900
0.074-0.045	3.04	0.9850
-0.045	3.73	0.9970
Total Size	2.97	0.9913

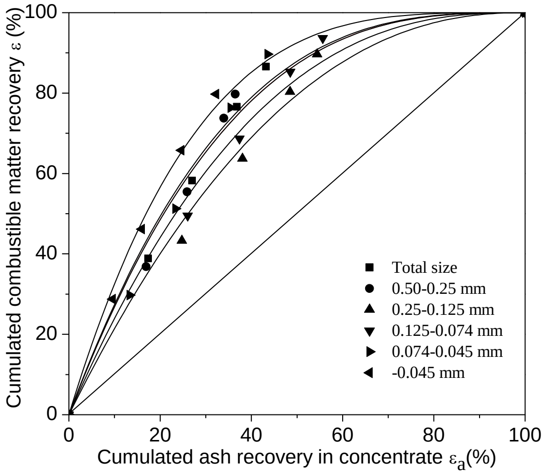


Fig. 2. The Fuerstenau upgrading curve of different size fractions

Flotation process based on energy input and distribution

Experimental design

The total energy input in flotation is composed of pre-conditioning and flotation-conditioning energy. Pre-conditioning was conducted for 210 s, and it involved pre-wetting (120 s), collector (60 s) and frother (30 s) conditioning. Flotation was conducted for 180 s. The flotation energy consumption test and energy input calculation were performed according to the method proposed by Gui et al. (2014a). Power data with different flotation shaft speeds are shown in Table 8. The new flotation process design, based on energy input and distribution is as follows. Under conditions of equal total energy consumption, the excess energy from the flotation-conditioning stage was transferred to the pre-conditioning stage. The other conditions were held constant. The impeller rotation speed was increased to 2500 rpm in the pre-conditioning stage. The power input increased in the flotation-conditioning stage (900, 1200, 1500, and 1800 rpm at different flotation time). The total energy input under the condition of energy input and distribution was 2898 J, which was similar to 2890 J under constant power condition. The experimental design scheme is presented in Fig. 3. High pre-conditioning power input and high-shear conditioning can promote dispersion of pulp and prevent excessive aggregation. However, the new energy input pattern during the flotation-conditioning stage, where low energy was applied early and high energy was applied late based on the floatability variation characteristics of the materials, can improve the flotation selectivity.

Table 8. Power data with different flotation shaft speeds

n, rpm	300	600	900	1200	1500	1800	2100	2400	2500
P, W	0.17	0.29	0.53	0.75	1.29	3.49	7.41	10.20	11.98

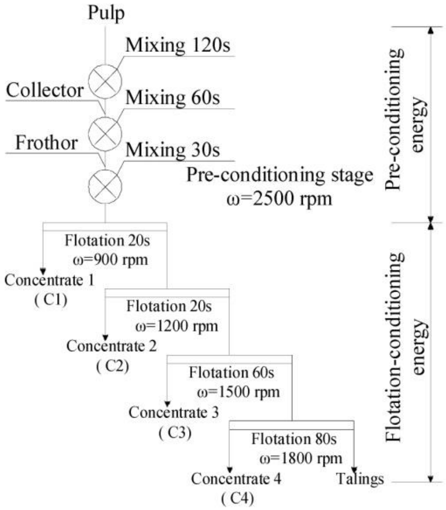


Fig. 3. Experimental design

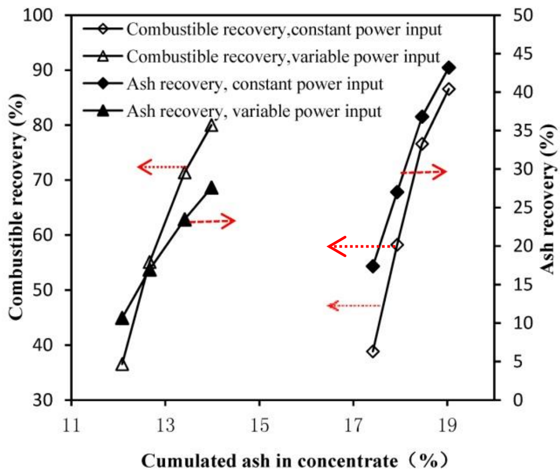


Fig. 4. Flotation results using variable and constant power inputs

Flotation behavior analysis using variable power input

The kinetics flotation results using variable and constant power inputs are shown in Fig. 4. Qualified clean coal with an ash content of 13.98%, ash recovery of 27.59%, and 80.01% combustible matter recovery can be obtained. This indicates that a variable-power input flotation pattern shows better selectivity and could lower the concentrate ash content by 5.05%. The ash and combustible matter recoveries decreased by 15.60 and 6.56%, respectively. However, the combustible matter recovery remained above 80%, which is still acceptable in practical production. Additionally, the Fuerstenau upgrading curves of variable power input and constant power input are shown in Fig. 5. Obviously, the selectivity improved, as the k values of variable power input and constant power input were 4.51 and 2.97, respectively.

A laser particle size analysis meter was used to measure the coal superficial size distribution in the pulp at two different pre-conditioning power input conditions. The circulation flow rate of laser particle size analysis meter was decreased to a minimum to prevent damage to the agglomerate structure. Each sample was repeated 3 times. The effect of pre-conditioning power input on the coal superficial size distribution in the pulp is presented in Fig. 6. The coarse particles reduced while the fine fractions increased. The superficial average size d_{50} decreased by 16.95 μm as the pre-conditioning power input increased to 11.98 W. This illustrates that aggregations were broken up by the high-shear pre-conditioning pattern. The ash particles entrapped in the agglomerate structure were released. As a result, entrapment was controlled to a lower degree. The flotation selectivity was improved.

Scanning electron microscopy (SEM) photographs of first concentrate C1 at two different energy input patterns are shown in Fig. 7. Aggregations and ash materials significantly decreased on the surface of concentrate. This finding indicates that lower ash content could be obtained by decreasing the total flotation-conditioning energy and increasing the power, given that this energy pattern was suitable for floatability variation characteristic of materials in the flotation-conditioning stage. The low-energy input in the early stage of flotation decreased the number of collisions among the coal, ash-forming particles, and air bubbles. Only easy-to-float materials with low ash content could float using the low energy input in the early stage. Mechanical entrapment and entrainment of ash with coal were suppressed. A qualified concentrate could be obtained using a new flotation process by transferring the excess energy of the flotation-conditioning stage to the pre-conditioning stage as well as increasing the power input step by step in the flotation-conditioning stage under the condition of equal total energy consumption.

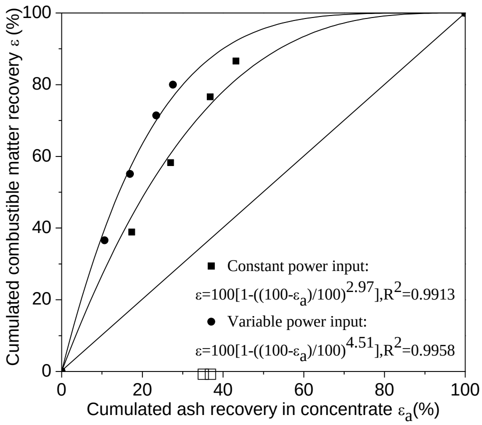


Fig. 5. The Fuerstenau upgrading curve of variable power input and constant power input

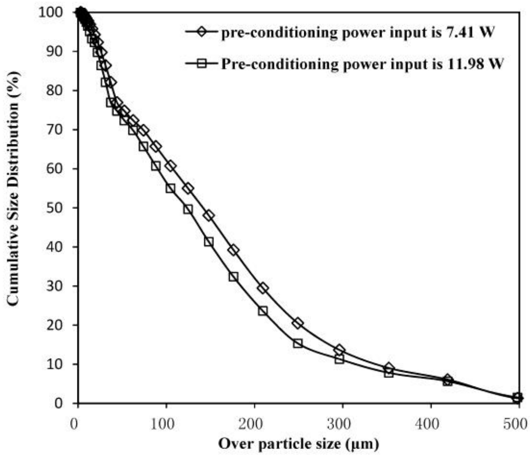
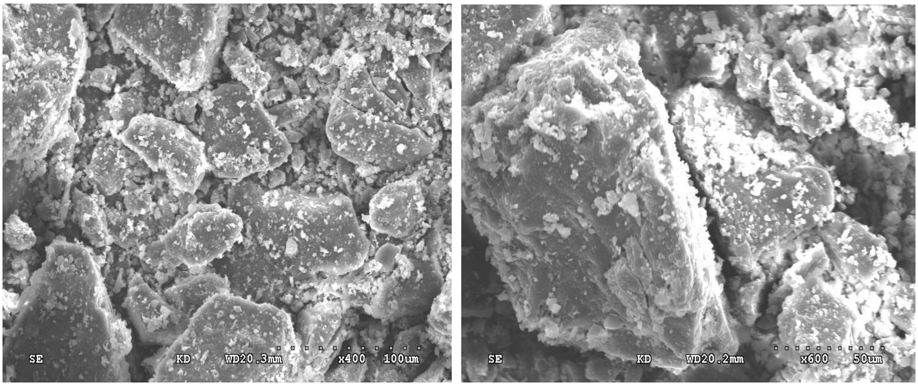
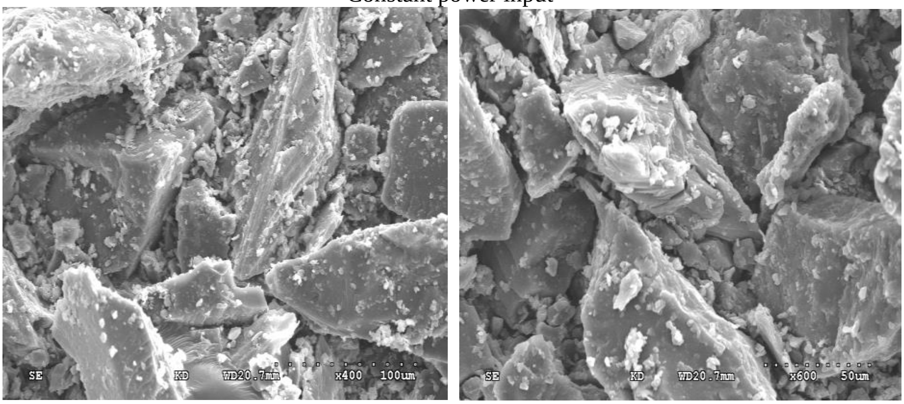


Fig. 6. Effect of pre-conditioning power input on coal superficial size distribution in pulp



Constant power input



Variable power input

Fig. 7. SEM pictures of concentrate C1 at two different energy input patterns

Conclusions

Based on FTIR analysis it can be said that coal samples exhibited good flotation because of the presence of a large number of hydrophobic and few hydrophilic functional groups,. A large number of ash-forming materials floated into froth products at the beginning of flotation under constant power input. Ash content of all size fractions exceeded the initial product requirement for ash of 14%. However, the 0.25-0.074 mm particles had the worst selectivity compared with that of 0.50-0.25 mm and -0.074 mm, as determined from the Fuerstenau upgrading curves. These observations could be attributed to the excessively high-energy input during the flotation-conditioning stage, which increased the number of collisions among the coal, ash-forming particles, and air bubbles that led to the mechanical entrapment and entrainment of ash materials with coal. Moreover, the relatively good hydrophobicity of the coal samples resulted in formation of particle aggregates, which reduced the flotation selectivity of all size fractions.

The desired concentrate could be obtained using a new flotation process by transferring the excess energy from the flotation-conditioning stage to the pre-conditioning stage and increasing the power input stepwise in the flotation-conditioning stage under equal total energy consumption. A qualified clean coal with an ash content of 13.98%, 27.59% of ash recovery, and 80.01% of combustible matter recovery was obtained. The aggregates were broken up with a high-shear pre-conditioning pattern based on laser particle size analysis. The new energy pattern in the flotation-conditioning stage, low energy was applied early and high energy was applied late. It was suitable for floatability variation characteristic of the materials. Low-energy input in the early stage of flotation decreased the number of collisions among coal, ash-forming particles, and air bubbles. Only easy-to-float materials with the low ash content could float using the low energy input at the early stage. Mechanical entrapment and entrainment of ash with coal particles was suppressed.

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