A new preparation scheme for a difficult-to-float coking coal by column flotation following grinding

by Yinfei Liaoa*, Yijun Caoa*, Zhongbo Hub†, and Xiuxiang Taoc‡

Synopsis

A new preparation scheme for a difficult-to-float coking coal from the Kailuan Mine, Tangshan, China was investigated. The results showed that grinding followed by column flotation was beneficial for obtaining products with low ash content. The positive effect of grinding on the coal floatability is attributed to the liberation of intergrowths and coal surface improvement. Tests indicated that 10 minutes was the optimum grinding time, and overgrinding resulted in a deterioration in flotation performance. With a grinding time of 10 minutes, conventional flotation had potential to yield a product with around 12.42% ash content and 69.15% combustible recovery. Column flotation can reduce the product ash content to 11.15% and increase combustible recovery to 74.47%. Consistently better flotation results reveal that column flotation is more efficient than conventional flotation for such fines.

Keywords
coal flotation, column flotation, grinding, difficult-to-float, liberation.

Introduction

Coking coal is scarce in China, accounting for less than 10% of total coal reserves (Tao *et al*., 2009). Most of these valuable resources are difficult to wash, with high ash content in the product and low recoveries. Reduced availability of good quality coking coal has resulted in Chinese steel plants using low-ash imported coal as a sweetener in coal blends (Liu *et al*., 2009). Ash has a highly adverse effect on blast furnace productivity and coke consumption (Dey and Bhattacharyya, 2007). Thus it is necessary to develop an efficient preparation scheme to produce coking coal with a low ash content.

Froth flotation, which is the most common separation technique used for cleaning fine coals, has been widely applied since the 1920s (Hacifazlioglu and Toroglu, 2007; Hacifazlioglu and Sutcu, 2007). Column flotation has been developed into an efficient technology in the past few decades. In many studies, it has been claimed that column flotation can give a higher recovery with lower ash content (Jena *et al*., 2008; Jena, Biswal, and Rudramuniyappa, 2008; Tao Luttrell, and Yoon, 2000). Furthermore, recent work has proved that coal floatability can be improved considerably by grinding (Sokolovic, Stanojlovic, and Markovic, 2012; Xia *et al*., 2012; Feng and Aldrich, 2000). The floatability of low-rank coal is enhanced through grinding in the presence of bituminous coal pitch (Atesok and Celik, 2000). Grinding has also been adopted to improve the floatability of oxidized coal (Xia, Yang, and Zhu, 2012). Clean coals have been successfully produced from Mecsek bituminous coal by flotation following ultrafine liberation (Bokanyi and Csoke, 2003). Thus by combining the two processes of grinding pretreatment and column flotation, the separation efficiency may be greatly improved.

In this investigation, we compar the ash content and combustible matter recovery by conventional flotation with that by column flotation for different grinding times. A cyclonic-static microbubble flotation column (Cao *et al*., 2012; Li *et al*, 2010), a novel column developed by China University of Mining and Technology, was used for the flotation tests. Size analysis, density analysis, and contact angle measurements were used to investigate the effects of grinding pretreatment. The efficiency of combined grinding pretreatment and column flotation is also discussed.

Experimental procedure

Materials

A coking coal sample of -0.5 mm size fraction was collected from Kailuan Mine, Tangshan, China. An SPB200 vibrating Taylor screen was used for size analysis. Size fractions of +0.5, -

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0.5+0.25, -0.25+0.125, -0.125+0.074, -0.074+0.045, and -0.045 mm were generated. Each size fraction was analysed for ash. Ash content (%) = Wt. of baked ash/Wt. of unbaked coal. A GL-21 M high-speed centrifuge was used for density analysis, with a centrifuge speed of 3000 r/min. The organic solutions with densities of 1.3, 1.4, 1.5, 1.6, and 1.8 g/cm^3 were prepared. Each density fraction was washed, filtered, dried, weighed, and analysed.

Grinding pretreatment

A cylindrical laboratory ball mill with a diameter of 160 mm and a length of 200 mm was used for wet grinding in all the experiments. The ball mill was run at a constant speed of 120 r/min and a media filling of 20%. A 500 g coal sample was added to the ball mill at a pulp concentration of 40%. The grinding times were 10, 20, 30, and 40 minutes. Size analysis, density analysis, and contact angle measurements were carried out on the grinding products. Contact angles were determined by the sessile drop method using a digital goniometer (Drop Shape Analysis System, DSA100, Krüss GmbH, Hamburg, Germany). The measurements were repeated three times for every sample.

Flotation tests

The flotation tests were carried out in both a conventional flotation cell and a laboratory flotation column. Diesel oil was used as collector and 2-octanol as frother. Sodium hexametaphosphate was used as silica depressant as well as dispersant.

About 100 g of coal was taken for conventional flotation experiments using a 1.5 L XFD flotation cell with an impeller speed of 1590 r/min and an air flow rate of 2 L/min. The slurry was prepared with 6.25% solid concentration and conditioned with sodium hexametaphosphate (1.5 kg/t) for 2 minutes. It was then treated with the required amount of diesel oil (310 g/t) for an additional 2 minutes. 2-octanol (120 g/t) was then added and the slurry further conditioned for 1 minute. The flotation was carried out by introducing air and the froth was collected for 3 minutes. The flotation products were filtered, dried, weighed, and analysed for ash. The organic solution was comprised by carbon tetrachloride, benzene and tribromethane.

The column flotation study was carried out employing a 100 mm diameter by 2000 mm tall laboratory flotation column. A schematic diagram of the experimental set-up is shown in Figure 1. The slurry was treated with sodium hexametaphosphate (1.5 kg/t) at 6.25% solid concentration in the conditioner for 2 minutes. It was then agitated with diesel oil (310 g/t) for an additional 2 minutes, after which 2-octanol (120 g/t) was then added and the slurry further conditioned for 1 minute. The slurry was fed with a peristaltic slurry pump at a specified rate to the column. The required air rate was monitored by flow meter. The slurry flow rates of feed and tailings were checked, and when both remained more or less constant, the concentrate and tailings were collected simultaneously at a certain time interval. The operating parameters for column flotation are presented in Table I. The samples were analysed using a similar procedure to that followed for the conventional flotation products. Each experiment was replicated to ensure the reproducible of data within the acceptable experimental error.

Results and discussion

Characterization of coal samples

The particle size characterization data is given in Table II. It can be seen that the ash content increases with decreasing coal particle size. The lowest ash content was found in the +0.5 mm fraction. The majority of the material falls in the size range -0.074 +0.045 mm, with 34.27% yield. The fines and ash contents of the fine-grained samples are both high. The -0.074 mm fraction accounted for 39.78% of yield at an ash content of 26.27%, which is significantly higher than the other fractions. The fine particles with high ash content float into clean coals easily through mechanical entrainment, thus generating a high-ash concentrate. The selective recovery of fine fractions has thus become the key to preparation of these types of coal.

The density analysis results for this fine coal are shown in Table III. It can be observed that the major yield of the coal is in the density range of -1.5 g/cm^3. At a theoretical separation density of 1.4 g/cm^3 and 1.5 g/cm^3, the content of

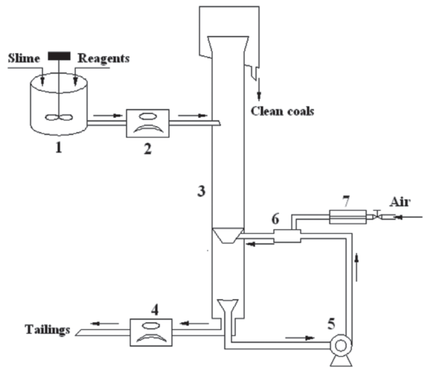

Figure 1—Schematic diagram of the flotation column. 1. Conditioner 2. Feed peristaltic pump 3. Flotation column 4. Tailing peristaltic pump 5. Circulation pump 6. Bubble generato; 7. Flow meter

Table I

Operating parameters of column flotation

Operating parameters	Index
Collector	Diesel oil (310 g/t)
Frother	2-octanol (120 g/t)
Dispersant	Sodium hexametaphosphate (1.5 kg/t)
Solid concentration	6.25%
Feed rate	3 l/min
Air rate	5-6 l/min
Froth depth	400 mm
Circulating pressure	0.20 Mpa

Table II

Size analysis results

Size fraction (mm)	Weight (%)	Ash content (%)	Combustible recovery (%)
+0.5	3.21	15.31	2.34
-0.5+0.25	15.73	16.66	12.48
-0.25+0.125	24.75	17.05	20.09
-0.125+0.074	16.53	19.47	15.33
-0.074+0.045	34.27	26.06	42.53
-0.045	5.51	27.58	7.24
Total	100.00	21.00	100.00

Table III

Density analysis results

Density (g/cm³)	Yield (%)	Ash (%)	Float		Sink		Content of δ±0.1	
			Yield (%)	Ash (%)	Yield (%)	Ash (%)	Density (g/cm³)	Yield (%)
-1.3	8.56	4.32	8.56	4.32	100	20.87	1.3	40.37
1.3-1.4	31.81	11.36	40.37	9.87	91.44	22.42	1.4	68.36
1.4-1.5	36.55	19.62	76.92	14.5	59.63	28.32	1.5	46.49
1.5-1.6	9.94	24.35	86.86	15.63	23.08	42.1	1.6	13.55
1.6-1.8	7.21	41.94	94.07	17.64	13.14	55.53	1.7	7.21
+1.8	5.93	72.05	100	20.87	5.93	72.05	1.8	9.54
Total	100	20.87	—	—	—	—		

δ±0.1 is 68.36% and 46.49%, respectively. This demonstrates that the washability of such fine coal is poor. The ash content in the density range of 1.6 g/cm³ to 1.8 g/cm³ is relatively low. It can be indicated that there are numerous non-liberated intergrowth particles formed by gangue minerals and coals. It was supposed by the above density analysis result that a microscopic investigation will be done for further verification. It is therefore difficult to obtain low-ash clean coals and high-ash tailings by direct conventional flotation.

Grinding properties

Figure 2 shows that the yield of the +0.074 mm size fraction decreases with the grinding time. However, the yield of the -0.074 mm size fraction increases with grinding time. Moreover, it is interesting to note that the yield changes quickly during the first 10 minutes, and changes slowly when the grinding time is more than 20 minutes. This indicates that the grinding efficiency is high during the first 10 minutes and then declines.

Figure 3 presents the relationship between cumulative yield and ash content, according to the density analysis of grinding products, for different grinding times. It can be seen that the yield increases with ash content. Moreover, the yield increases with the grinding time for a given ash content. This indicates that intergrowth particles are liberated in the grinding process and some coals are enriched to a certain extent.

Contact angle has been extensively used to characterize the hydrophobicity and floatability of coal samples. Figure 4 shows that the contact angle initially increases with grinding time, then decreases slowly, decreasing more rapidly when the grinding time is more than 30 minutes. This phenomenon indicates that the hydrophobicity of this fine coal can be improved by appropriate grinding, but will deteriorate with overgrinding. Some coals are liberated and enriched through grinding. The coal floatability is improved in the attrition process. However, if the grinding time is too long, the coal will be overground and the coal surface will be covered by high-ash slime.

Flotation results

Figure 5 shows that combustible matter recovery by both column flotation and conventional flotation increases initially with grinding time and then decreases when the grinding time is more than 10 minutes. However, the combustible matter recovery by column flotation is consistently higher than that by conventional flotation at all grinding times. In both cases, the concentrate ash content at first decreases with grinding time and then increases when the grinding time is more than 10 minutes. The ash content of the column flotation product is lower than that of conventional flotation at all grinding times. These results indicate that the scheme of column flotation following grinding is beneficial for obtaining products with low ash content.

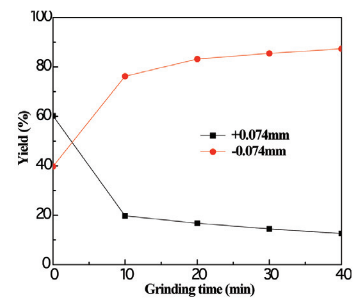

Figure 2—Effect of grinding time on size composition of coal samples

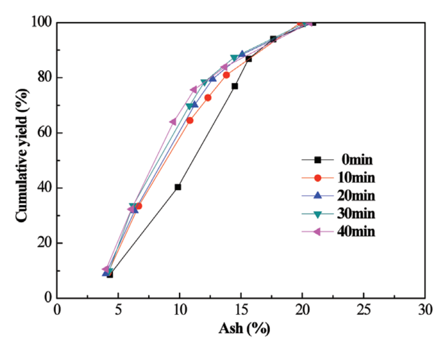

Figure 3—Effect of grinding time on the relationship between cumulative yield and ash content

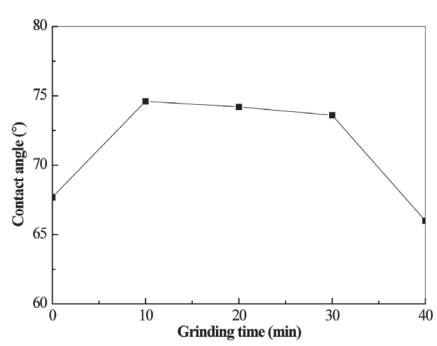

Figure 4—Effect of grinding time on contact angle of coal samples

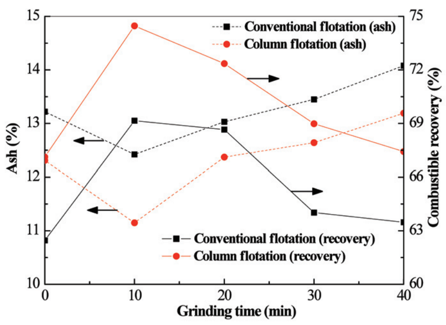

Figure 5—Effect of grinding time on flotation performance

It is worth mentioning that both column flotation and conventional flotation achieve the best performance at a grinding time of 10 minutes. The contact angle at first increases with the grinding time, and then decreases as shown in Figure. 4. Appropriate grinding can be used to improve flotation performance, but overgrinding will exacerbate mechanical entrainment, leading to a deterioration in flotation performance. Therefore, these results demonstrate that 10 minutes is the optimum grinding time.

Conventional flotation reduces the ash content from 21.36% to 12.42%, with 69.15% combustible recovery. Using column flotation, the ash content of the clean coals is reduced to 11.15%, with 74.47% combustible recovery. It is obvious that column flotation is superior to conventional flotation, producing cleaner coals in terms of lower ash content and higher combustible matter recovery. Column flotation has advantages in recovering valuable fines at a better grade due to the minimization or prevention of hydraulic entrainment of undesirable fines (Li *et al*., 2012; Demir *et al*., 2008; Finch, 1995). The fine fractions increase after grinding pretreatment. Column flotation can selectively separate these fines to obtain clean coals of lower ash content. Moreover, the cyclonic-static microbubble flotation column features multiple mineralization steps, including countercurrent mineralization, cyclone mineralization, and pipe flow mineralization, which provide sufficient retention time to ensure fines recovery (Zhang *et al*., 2013).

Conclusions

Investigations carried out on coking coal collected from Kailuan Mine indicate that it is difficult to obtain low-ash clean coals and high-ash tailings through direct conventional flotation. Improved hydrophobicity and floatability can be achieved by appropriate grinding. The effect of grinding on coal floatability is attributed to the liberation of intergrowths and improvement of the coal surface properties.

It is concluded that 10 minutes is the optimum grinding time, and overgrinding results in a deterioration in flotation performance. With a grinding time of 10 minutes, conventional flotation has the potential to yield a product with approximately 12.42% ash content and 69.15% combustible recovery, while the product ash content can be further reduced to 11.15% with 74.47% combustible recovery in case of column flotation. Flotation tests results show that column flotation is more efficient than conventional flotation for such coking coal fines. The scheme of column flotation following grinding is beneficial for obtaining products with low ash content.

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