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Utilization of coal slime: Coal and kaolinite separation by classification, forward and reverse flotation method

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Abstract: Coal slime is not only a solid waste, but also a source of energy. With the improvement of environmental protection requirements, the comprehensive utilization of slime has become an urgent problem for coal preparation plants. In this paper, we put forward a promising way of coal slime resource utilization. X-ray diffraction (XRD), X-ray fluorescence spectrometer (XRF) and laser particle sizer was used to analyze the properties of coal slime. Obtained results showed that the slime was mainly composed of the coal, kaolinite and quartz with a particle size of -100 μm. Most kaolinite minerals can be enriched in overflow when the feed pressure is 0.2 MPa by using hydrocyclone. 21.3% clean coal with ash content of 12.3% and 33.46% kaolinite with particle size of -5 μm can be recovered by forward flotation and reverse flotation respectively. Coal water slurry with 61% concentration can be prepared from reject of forward flotation and concentrate of reverse flotation at shear rate of 100 s^{-1}. This study has an important practical application value in clean and efficient utilization of coal.

Keywords: coal slime, classification, forward flotation, reverse flotation

1. Introduction

Coal is a source of energy, and plays a leading role in the energy consumption system in China. In 2020, China's coal output was about 4000 Tg and it is estimated that China’s coal consumption will remain at 2000 Tg until 2050 (Guo et al., 2021). With the improvement of coal mining mechanization, the output of slime in raw coal is higher and higher. At the same time, with the continuous large-scale equipment of coal preparation plant, the amount of slime produced in the process of raw coal separation is also increasing rapidly (Haibin and Zhenling, 2010; Xie et al., 2010, 2009). It is roughly estimated that China's annual output of slime is more than 200 Tg since 2013 (Li et al., 2018). However, the comprehensive utilization rate of slime is less than 10%.

Coal slime is a by-product in the coal preparation process (Haibin and Zhenling, 2010; MA et al., 2008; Zhou et al., 2017). According to different varieties and formation mechanism, its properties are very different. If the coal slime is mainly obtained by crushing clean coal, the ash content of the coal slime is low and the particle size is large. On the contrary, if the coal slime is mainly obtained by mineral sliming, the ash content of the coal slime is high and the particle size is fine. The slime in coal preparation plant mainly comes from the flotation tail coal, and its ash content is usually more than 55%. Due to the high moisture and ash content of coal slime, its price is usually low, and sometimes even the coal preparation plant needs to pay extra transportation and treatment fees for coal slime (MA et al., 2008; Zhou et al., 2017).

Research shows that the environmental pollution caused by coal slime is higher than other coal mine waste. The particle size of coal slime is very fine, so dust will be produced during stacking or transportation, especially under the influence of wind (Haibin and Zhenling, 2010). In addition, due to the high water content of coal slime, in order to improve energy efficiency, it must be dried before use. However, a large amount of dust is inevitably produced in the drying process, leading to secondary

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pollution. It is worth noting that the inhalation of harmful trace elements in ultrafine particles will cause respiratory diseases, and even increase the risk of cancer, thus endangering human health (Pope et al., 2002; Song et al., 2015; Xu et al., 2021; R. Zhang et al., 2021). Some studies have shown that the stacking of coal slime will cause serious soil and water pollution (Fu et al., 2012).

With the improvement of environmental protection requirements, the comprehensive utilization of slime has become an urgent problem for coal preparation plants. So far, the comprehensive utilization of slime has included power generation, briquette production, building materials and chemical products (Szpyrka et al., 2012). Lutynski et al evaluated the energy potential and possible utilization of coal slurry sediments in detail (Lutynski et al., 2014). Guo et al. discussed the feasibility of anaerobic fermentation technology of coal slime. The results showed that organic matter in various coal slimes could be converted into biomethane (H. Guo et al., 2021). Meng et al. proposed a new way to dispose of coal slime and studied the effects of coal slime on the slurry ability of a semi-coke water slurry (Meng et al., 2020). Coal slime can replace clay as raw material or auxiliary material for cement production, providing necessary silicon and aluminum components (Qiu et al., 2010; Yang et al., 2016). Coal slime contains valuable mineral resources such as kaolinite and quartz. Therefore, it is an important way to extract silicon and aluminum from coal slime to produce chemical products (Guo et al., 2016; Xiao et al., 2015). However, some comprehensive utilization technologies have certain requirements for the properties of slime, and not all slimes can meet these requirements. For example, in order to extract aluminum and silicon from coal slime, the content of alumina and silica should be more than 35% and 30% (Guo et al., 2014; Mahi et al., 1991; Xiao et al., 2015; Y. Zhang et al., 2021). At present, most coal slimes are still burning as fuel (Huang et al., 2020; Niu et al., 2017; Song et al., 2021; Wang et al., 2017; Wzorek et al., 2010). Through previous studies, it can be seen that coal slime is not only a pollutant, but also a source of energy. However, in the process of comprehensive utilization of slime, not only its economic benefits should be considered, but also the secondary pollution should be avoided.

According to the literature research, most scholars mainly study how to recover clean coal from coal slime, and the method used is usually forward flotation (Shen et al., 2019; Yang et al., 2017). The combination of classification, forward flotation, and reverse flotation is less researched. In this paper, XRD, XRF, as well as laser particle sizer (LPSA) were used to analyze the characteristics of coal slime particles. A process of comprehensive utilization of slime was proposed, in which hydrocyclone was used for the classification of coal slime particles, and forward flotation and reverse flotation were used to recover clean coal and kaolinite respectively.

2. Materials and methods

2.1. Materials and reagents

The slime sample was obtained from a coal preparation plant in Huainan City, Anhui Province, China, which produces 3000 Mg of slime per day. Diesel was used as collector and methyl-isobutyl-carbinol (MIBC) as frother in the forward flotation. Dodecylamine Hydrochloride (DAH) was used as collector and dextrin as depressant in reverse flotation. DDA neutralized with HCl regarded as DAH, the solution concentration was 0.135 mol/dm^3. All reagents were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. and used as received.

2.2. Methods

2.2.1. Utilization process

The flow chart of the slime comprehensive utilization process was shown in Fig. 1. Firstly, the coal slime and water were added into the agitating vessel to form a slurry with a concentration of 100 g/dm^3. Then the slurry is pumped into the hydrocyclone by slurry pump for classification. The underflow product of hydrocyclone containing coarse particles was used as the feed for forward flotation, and the overflow product of hydrocyclone containing fine particles is used as the feed for reverse flotation. The float products in forward flotation was clean coal and the sink products in reverse flotation was kaolinite. While, the sink products in forward flotation and the sink products in reverse flotation were combined as middings.

2.2.2. Classification process

During the experiments, the slurry was firstly pumped into the hydrocyclone. After 5 mins, when the hydrocyclone worked stably, the overflow and underflow of the hydrocyclone were collected at the same time. The collection time was 10 secs. The feed pressure is 0.1 MPa and 0.2 MPa respectively. In order to obtain larger capacity and higher classification efficiency, five hydrocyclones with different specifications were selected. Schematic diagram of the classification test system was shown in Fig. 2.

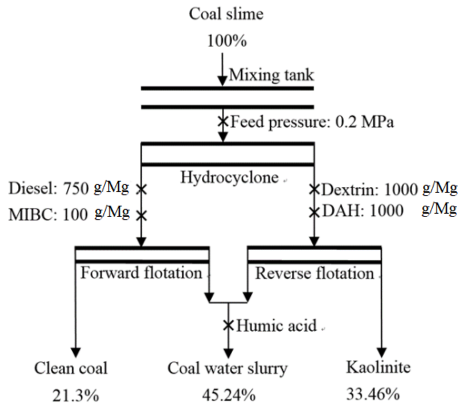

Fig. 1. Flow chart of the slime comprehensive utilization process

2.2.3. Flotation experiment

During the forward flotation process, the coal slime was firstly added into the flotation cell and conditioned for 2 mins. Then diesel was added and conditioned for 2 mins (W. Li et al., 2021; Liao et al., 2020; Yang et al., 2021). Finally add MIBC and open the intake valve after 30 secs, and start collecting foam products. The collection period last 5 mins. While for reverse flotation process, depressant was added first, and then collector was added. The other operations were the same as those of forward flotation process. The pulp concentration were 50g/dm^3, 75g/dm^3 and 100g/dm^3, respectively. The dosage of diesel was 750 g/Mg, 1000 g/Mg and 1250 g/Mg. The dosage of DAH was 1000 g/Mg, 1500 g/Mg and 2000 g/Mg. The dosage of MIBC and dextrin was kept at 100 g/Mg and 1000 g/Mg, respectively. All flotation tests were conducted in an XFD-1.5 L flotation machine.

2.2.4 Preparation of coal water slurry (CWS)

The experimental CWS was prepared by a dry slurry technology. The reject in the forward flotation and the concentrate in the reverse flotation were mixed firstly. Then, 0.5 wt% humic acid and deionized water were added and stir for 15 mins at a speed of 1000 r/min. Apparent viscosity of CWS was measured by an NXS-4C viscometer (Chengdu Instrument Factory, CN).

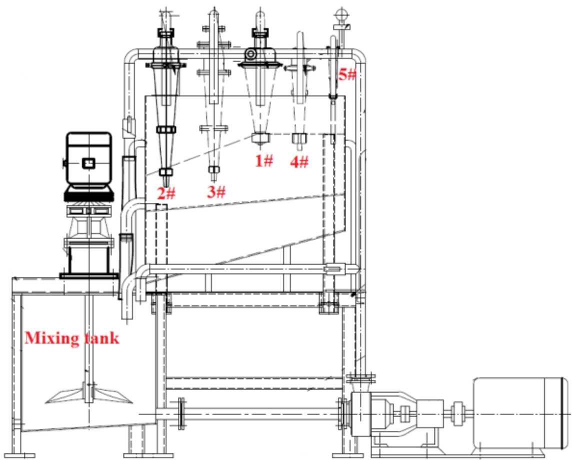

Fig. 2. Schematic diagram of the classification test system (The diameter of 1#, 2#, 3#, 4# and 5# hydrocyclone is 125, 100, 75, 50 and 25 mm respectively)

2.2.4. Utilization process

The flow chart of the slime comprehensive utilization process was shown in Fig. 1. Firstly, the coal slime and water were added into the agitating vessel to form a slurry with a concentration of 100 g/dm^3. Then the slurry is pumped into the hydrocyclone by slurry pump for classification. The underflow product of hydrocyclone containing coarse particles was used as the feed for forward flotation, and the overflow product of hydrocyclone containing fine particles is used as the feed for reverse flotation. The float products in forward flotation was clean coal and the sink products in reverse flotation was kaolinite. While, the sink products in forward flotation and the sink products in reverse flotation were combined as middings.

3. Results and discussion

3.1. Properties of the coal slime

The proximate analysis of the slime was shown in Table1. As shown in Table 1, the ash content of coal slime reached 53.64%, which indicated that there are many non-combustible substances in coal slime. If the slime is burned directly, a large number of dust particles will be produced.

The XRD and XRF analysis results of the slime were presented in Table 2 and Fig. 3. As shown in Fig. 3, the main gangue minerals in slime were kaolinite and quartz. Table 2 showed that the main chemical components in slime were SiO_2 (28.46 wt%) and Al_2O_3 (19.28 wt%). However, the theoretical molar ratio of Al_2O_3 to SiO_2 in pure kaolinite is 1:2, which indicated that there was quartz in the slime. In addition, a small amount of Fe_2O_3 in coal slime will affect the whiteness of kaolinite, so it also needs to be removed.

The particles size distribution of the slime was shown in Fig. 4. It can be seen that the slime was mainly composed of fine particles, and most of the particles were less than 10 μm. Kaolinite is easy to mud into very fine particles in solution due to its own structural characteristics. Therefore, it can be effectively enriched by classification method.

Table 1 Proximate analysis of coal sample (Air dried)

M_{ad} (%)	A_{ad} (%)	V_{ad} (%)	FC_{ad} (%)
6.72	53.64	19.26	20.38

Table 2 XRF analysis of the slime (wt%) (Low content components were not listed)

Compound	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	LOI
Content (%)	28.46	19.28	1.56	1.22	0.99	47.12

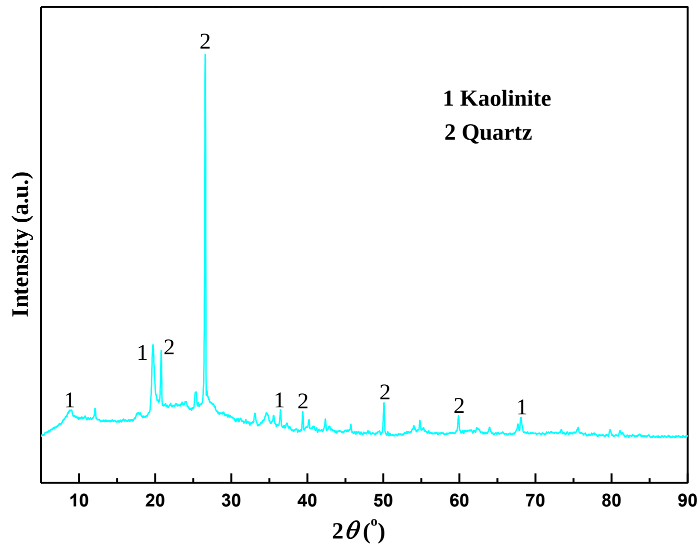

Fig. 3. XRD patterns of the slime

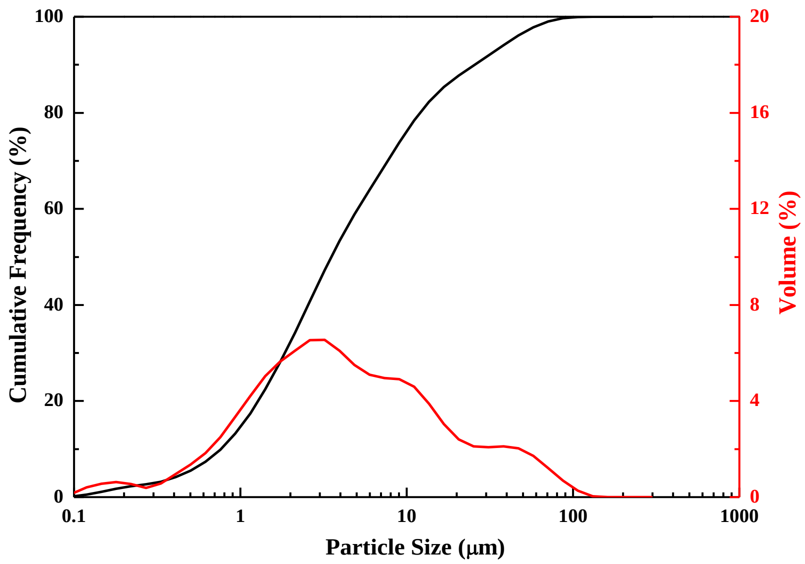

Fig. 4. Particles size distribution of the slime

3.2. Classification results

The classification results of the slime with five different hydrocyclone were shown in Table 3. As shown in Table 3 that as the diameter of hydrocyclone decreased, the overflow yield decreased and the ash content increased. Because with the decrease of the diameter of hydrocyclone, the classification particle size gradually decreases, and the material contained in the overflow was mainly uncombustible fine kaolinite and quartz. In addition, it can be seen that with the decrease of hydrocyclone diameter, the molar ratio of SiO_2 and Al_2O_3 in the overflow gradually decreased, indicating that a part of quartz in the slime entered the underflow. It can be seen from table 3 that when the feed pressure is 0.2 MPa, the processing capacity of 3# hydrocyclone is 2.1 times and 6.8 times more than that of 4# and 5# respectively. For the coal preparation plant, the amount of slurry to be treated exceeds 100 dm^3/h. If 5# hydrocyclone is selected, the production and maintenance cost of the coal preparation plant will increase sharply. In addition, with the decrease of hydrocyclone diameter, the content of fine particles in underflow is gradually increasing, which has an adverse impact on the forward flotation process. In order to obtain high clean coal recovery, and considering the process capacity of hydrocyclone, 3# hydrocyclone was selected as slime classification equipment in the experiment. Particles size distributions of overflow and underflow of all five hydrocyclones were given in Fig. 5. As shown in Fig. 5, with the decrease of hydrocyclone diameter, the overflow particle size decreased gradually, which is consistent with the analysis results in Table 3. With the increase of feed pressure, the particle size of underflow increased, which indicated that more fine particles entered into the overflow, which is conducive to the recovery of clean coal from underflow by flotation later. Therefore, the feed pressure of hydrocyclone was set as 0.2 MPa.

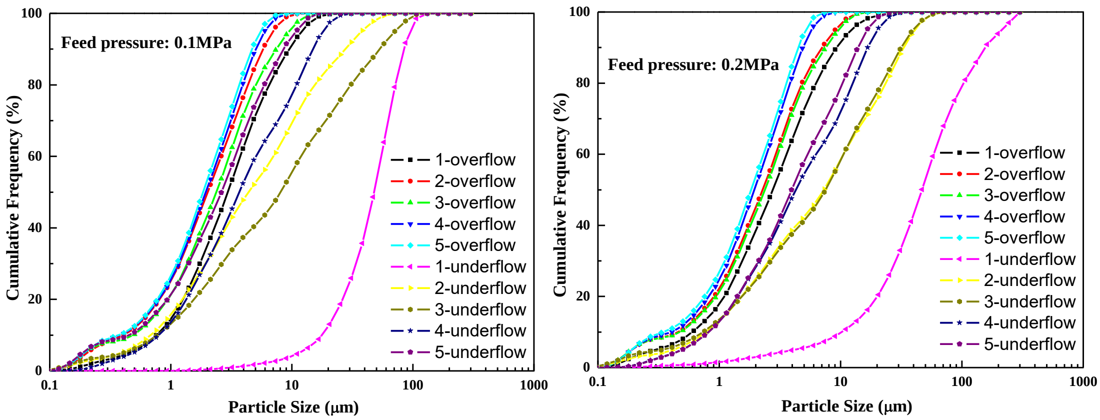

Fig. 5. Particles size distribution of the slime

Table 3. XRF analysis of the slime (wt%)

Hydrocyclone	Capacity (dm^3/h)	Feed pressure (MPa)	Overflow				Underflow	
			Yield (%)	Ash content (%)	SiO_2 (%)	Al_2O_3 (%)	Yield (%)	Ash content (%)
1#	9.2	0.1	84.34	56.50	28.68	19.63	15.66	38.26
	11.8	0.2	92.96	54.93	28.74	19.72	7.04	36.57
2#	6.1	0.1	65.14	57.72	29.83	20.21	34.86	46.01
	8.7	0.2	65.95	58.54	30.12	20.64	34.05	44.14
3#	2.1	0.1	54.84	59.31	32.78	22.01	45.16	46.75
	3.4	0.2	57.29	60.12	32.92	22.24	42.71	44.95
4#	0.9	0.1	33.21	60.45	34.28	23.69	66.79	48.97
	1.6	0.2	42.48	63.02	34.42	23.77	57.52	48.61
5#	0.2	0.1	15.40	63.85	34.78	24.03	84.60	51.78
	0.5	0.2	25.86	65.42	35.41	24.98	74.14	49.53

3.3. Forward flotation results

Effect of diesel dosages on the forward flotation under different feed concentration were presented in Fig. 6. As shown in Fig. 6 that the yield and ash content of the concentrate increased with the increase of diesel dosages. This was mainly caused by two reasons. On the one hand, with the increase of the concentration of collector in the pulp, the adsorption amount of collector on the surface of conjoint mineral gradually increased, which improved the hydrophobicity of the surface and finally entered the froth product, resulting in the increase of ash content of concentrate. On the other hand, a large number of bubbles will cause serious entrainment problems, so the ash content of froth products gradually increased. In addition, it can be seen from Fig. 6 that under the same diesel dosage, the yield of concentrate decreased and the ash content increased gradually with the increase of pulp concentration. This is because with the increase of pulp concentration, the content of fine gangue minerals in the pulp was also increasing rapidly, resulting in serious coating and competitive adsorption behavior (Li et al., 2021). The adsorption amount of diesel on the surface of coal decreased, resulting in a decrease in the yield of froth products. Therefore, in order to obtain higher clean coal yield and lower clean coal ash content, the pulp concentration of forward flotation was set at 50 g/dm^3, and the diesel dosage was 0.75 kg/Mg. Under this condition, the concentrate yield was 49.9% with 12.3% ash content, and the ash content of tailing was 76.9%. Therefore, the concentrate can be directly used as a clean coal product.

3.4. Reverse flotation results

3.4.1 Effect of DAH and dextrin dosages on reverse flotation

Effect of dextrin dosage on reject yield and ash content was presented as Fig.7. The pulp concentration and DAH dosage was 50 g/dm^3 and 1.0 kg/Mg. Tap water with pH 7.8 was used. It can be seen from Fig. 7 that selectivity of the reverse flotation becomes better in the presence of depressor dextrin. When dextrin was not added, the ash content of the reject was only about 60.4%, while when the amount of dextrin was 1.0 kg/Mg, the ash content increased to 76%, which increased by nearly 16%. It can also be found that with the increase of dextrin, the reject yield was continuously decreasing, because the cleaned coal particles was depressed by the dextrin and remained in the flotation cell to become cleaned coal. However, when the dextrin exceeds 1.0 kg/Mg, the ash content and yield of the reject tend to be stable. Therefore, for the reverse flotation of the coal sample, the optimal amount of dextrin is 1.0 kg/Mg.

Effect of DAH dosages on the reverse flotation of the underflow with different feed concentration were shown in Fig. 8. As shown in Fig. 8 that, the yield of the reject increased with the increase of DAH dosages. However, the ash content of the reject decreased gradually, which is due to the increasing concentration of collector in slurry, and the adsorption of collector on the surface of coal particles increased, which leads to the poor flotation selectivity. On the other hand, DAH has strong foaming ability. With the increase of DAH concentration in the pulp, the amount of foam in reverse flotation increased rapidly, which lead to serious mechanical entrainment. Because the main purpose of reverse flotation was to recover kaolinite from coal slime, the collector dosage and the pulp concentration were set at 1.0 kg/Mg and 50 g/dm³ respectively. Under this condition, the reject yield was 58.4% with 76% ash content, and the ash content of the concentrate was 33.6%.

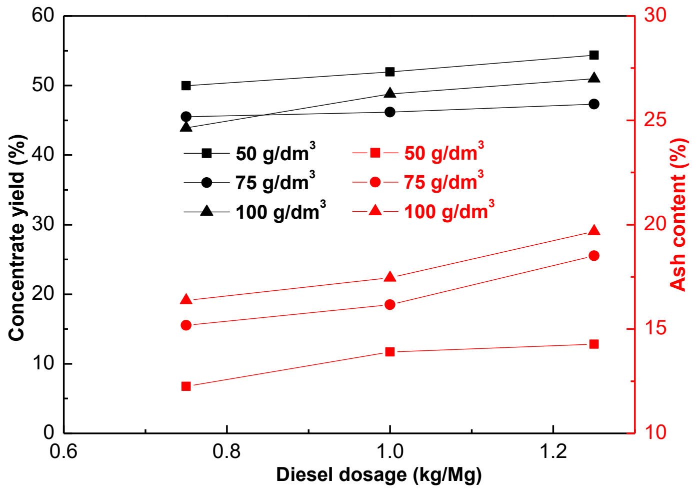

Fig. 6. Effect of diesel dosages on the forward flotation of the overflow

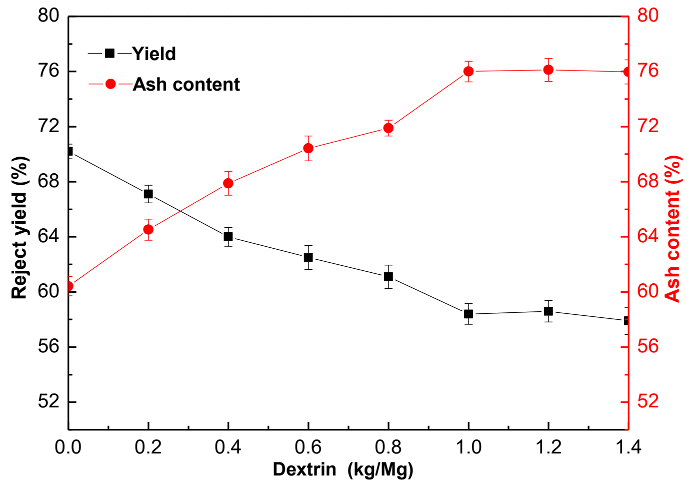

Fig. 7 Reject yield and ash content as a function of dextrin dosages(The data points represent the mean values (n = 3 ± standard deviation)

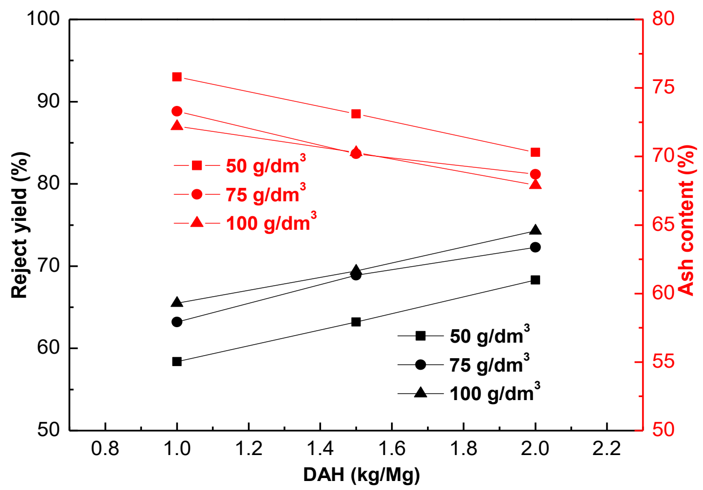

Fig. 8. Effect of DAH dosages on the reverse flotation of the underflow

3.4.2 Effect of pH on reverse flotation

The pH value of pulp is of great significance to the flotation process of minerals. According to the solution chemical balance of dodecylamine and the charging mechanism of silicate minerals, the surface charging properties of silicate minerals will change with the change of pulp pH. Effect of pH on reject yield and ash content was shown in Fig. 9. As can be seen from Fig. 9, with the increase of pulp pH value, the reject yield increased and the ash content decreased. It indicated that the selectivity of reverse flotation is better under acidic conditions, while the reverse flotation performance of DAH is seriously weakened under alkaline conditions.

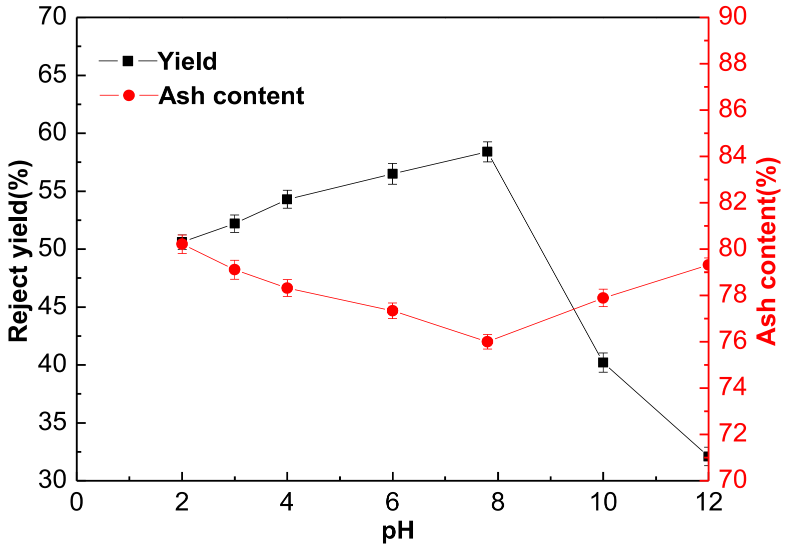

Fig.9 Reject yield and ash content as a function of pH

In order to reveal the influence mechanism of pH on coal slime reverse flotation, we calculated the concentration of each component of Dah in aqueous solution. As shown in Fig. 10, When the pH value is less than 9.2, dodecylamine mainly exists in the form of positively charged $C_{12}H_{25}NH_3^+$ component and a small amount of $(C_{12}H_{25}NH_3)_2^{2+}$cationic dimer in the pulp. In the process of coal reverse flotation, DAH is mainly adsorbed on the surface of kaolinite through electrostatic interaction, so the selectivity is better under acidic conditions. While in alkaline conditions, DAH mainly exists in the form of molecules and precipitation, which can not be adsorbed on the surface of kaolinite, resulting in the sharp decrease of reject yield.

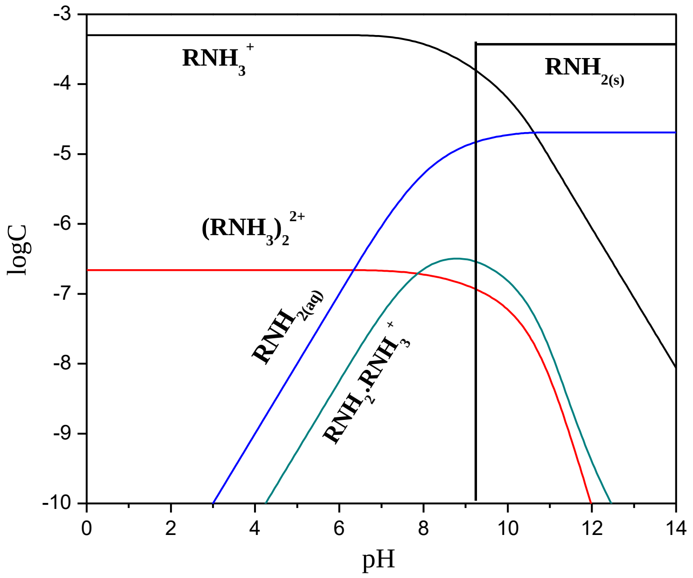

Fig.10 Species distribution diagram of DAH as function of pH (Concentration = 5×10^{-4} mol/L)

Reject obtained in the presence of 1.0 kg/Mg DAH, 1.0 kg/Mg dextrin, and 50 g/dm^3 pulp concentration, was used for XRF analysis. The XRF analysis result of the reject was presented in Table 4. As shown in Table 4 that the main chemical components in the reject were SiO_2 (40.13 wt%) and Al_2O_3 (32.54 wt%), which was close to the theoretical molar ratio of kaolinite. In most cases, coal series kaolinite is usually calcined before being used. After calcination, the content of SiO_2 and Al_2O_3 in the sample can reach 52.8% and 42.82% respectively, which can be used as raw materials for most industries Particles size distributions of the reject were given in Fig. 11. As shown in Fig. 11, the particle size of the reject was less than 5 μm, which can be used as an excellent industrial raw material.

Table 4 XRF analysis of the slime (wt%). Low content components were not listed

Compound	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	LOI
Content (%)	40.13	32.54	1.67	1.01	0.76	23.89

3.5. Preparation of coal water slurry

According to the flotation results in section 3.3 and section 3.4, the CWS contains reject with yield of 21.4% and ash content of 76.9% produced by forward flotation, and concentrate with yield of 23.84% and ash content of 33.6% produced by reverse flotation. The size distribution of the CWS was presented in Fig. 12. It can be seen in Fig. 12 that the particle size of CWS was very fine. On the one hand, the finer the particles in CWS, the easier it is to produce high concentration CWS, but on the other hand, it will also lead to the rapid increase of viscosity and poor fluidity of CWS. The existence of clay minerals in coal water slurry will also lead to the increase of its viscosity. In this experiment, kaolinite in coal slime used to make CWS has been successfully removed. In the process of increasing the concentration of coal water slurry, its viscosity was effectively reduced. Effect of shear rate on the apparent viscosity of CWS under different CWS concentration was presented in Fig. 13. It can be seen from Fig. 13 that with the increase of CWS concentration, the viscosity also increased gradually. When the shear rate was 100 s^{-1}, the CWS with 61% concentration and 967 mPa·s apparent viscosity was prepared.

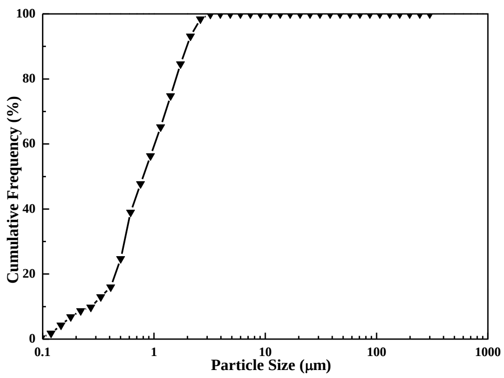

Fig. 11. Particles size distribution of the reject in the reverse flotation

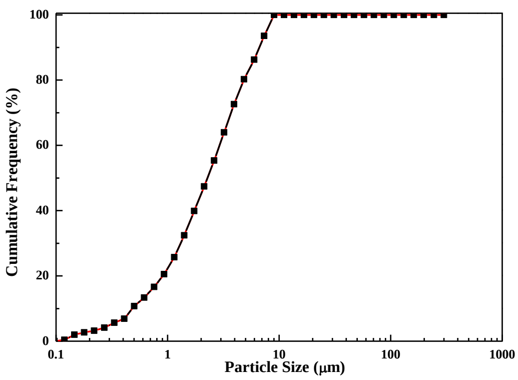

Fig. 12. Particles size distribution of the CWS

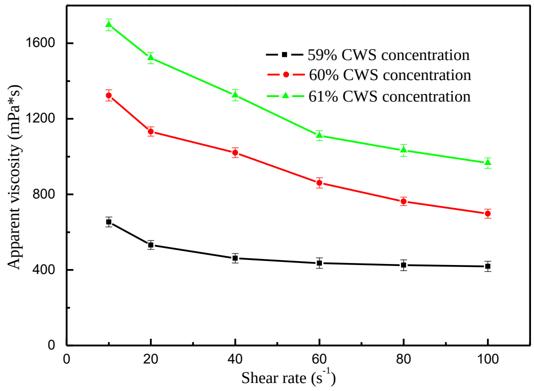

Fig. 13. Effect of shear rate on apparent viscosity of CWS in the presence of different concentration (The data points represent the mean values (n = 3) ± standard deviation.)

4. Conclusions

As a kind of solid waste in coal industry, coal slime has large output and strong pollution. A promising way of coal slime resource utilization was proposed in this paper.

The slime was mainly composed of the coal, kaolinite and quartz with a particle size of -100 μm. The results of hydrocyclone classification test show that most kaolinite minerals can be enriched in overflow when the feed pressure is 0.2 MPa by using 3# hydrocyclone. 21.3% clean coal with ash content of 12.3% and 33.46% kaolinite with particle size of -5 μm can be recovered from underflow and overflow of hydrocyclone by forward flotation and reverse flotation respectively. Coal water slurry with 61% concentration can be prepared from reject of forward flotation and concentrate of reverse flotation at shear rate of 100 s^{-1}. This study provides a new method for the comprehensive utilization of the coal slime, which has an important practical application value in clean and efficient utilization of coal.

Acknowledgments

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