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Influence of grinding methods on the preparation of ultra-clean coal from slime

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Abstract: This study discusses the technology of coal slime recovery for preparing ultra-clean coal (UCC). X-ray diffraction analysis (XRD) and scanning electron microscopy combined with energy dispersive spectroscopy (SEM-EDS) were used to analyze the characteristics of coal slime and to explore the types of minerals and embedded characteristics of coal slime. The particle interaction energy and contact angle analysis were used to determine the UCC preparation process through dry and wet grinding dissociation comparison and shear flocculation flotation tests, combined with zeta potential measurements. The results showed that the inorganic minerals in the slime were mainly kaolinite clay minerals, which were easy to mud in the pulp and needed grinding. The calculation results of the interaction energy indicated that the interaction force of dry-ground slime in the pulp was small, and flocculation was more likely to occur. The wet-ground product was subjected to three flotations to obtain UCC with an ash content of 0.95%.

Keywords: ultra-clean coal, grinding method, interaction energy, flotation

1. Introduction

Environmental issues are regarded as one of the three major challenges facing society in the 21st century (Langer, 2020; Mallapaty, 2020). This is related to changes in the living environment of human beings as well as global economic and geopolitical patterns(Al-Bayati et al., 2020; Bouglada et al., 2021). Coal is an essential source of economic growth for many countries. Ultra-clean coal (UCC) is a low-ash product with an ash content of less than 2% in coal, which can be prepared into various coal-based materials such as carbon fiber, high-grade activated carbon, and carbon black (Hao et al., 2020). The preparation of UCC is an important part of converting coal into carbon materials and realizing the transformation of fuel to raw materials, and is of great significance for developing new coal materials, increasing the added value of coal products, and promoting the transformation of coal from the fuel market to the raw material market (Ramudzwagi et al., 2020; Wang et al., 2019).

The dissociation of minerals and organic matter in coal is the primary task to realize the preparation of UCC. The methods of grinding dissociation include ball milling, rod grinding, high-pressure grinding, ultrasonic vibration-assisted grinding, stirred grinding, electrical disintegration, and high-voltage pulse technology among others (Abdelhaffez et al., 2022; Barma, 2019; Cheng et al., 2016; Ghadyani et al., 2018; Kizgut et al., 2010; Sahoo et al., 2011; Toraman and Katircioglu, 2011; Torres and Casali, 2009; Wei et al., 2022; Xie et al., 2015).

Through the preparation process of UCC, the fixed carbon content was further increased. Therefore, the preparation and industrial development of new carbon materials, such as graphene, graphite electrodes, and energy storage materials was gradually realized. Presently, the preparation of UCC includes physical and chemical methods, in addition to physical and chemical combined methods, and other preparation methods (Hao et al., 2020; Rahman et al., 2017; Wijaya and Zhang, 2011).

Using Taixi anthracite with ash content of 2.58% as raw material, acetic and propionic acids were added as regulators to the selective microbubble agglomeration flotation process to obtain UCC with an ash content of 0.65% to prepare the electrode materials(Dong et al., 2020). The flotation performance

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of the cyclonic static microbubble flotation column was greatly improved for fine low-rank coal through a turbulent mineralization mechanism and the high collision probability of microbubbles (Xing et al., 2017). Bokányi and Csöke (2003) crushed coal to less than 10 μm with a stirring ball mill to achieve mineral dissociation, after which the slurry was added to a foaming agent and a collecting agent, and the ash was obtained by flotation twice with a microbubble flotation column 2.85% UCC with an yield of 38%. Compared to experimental ball milling, high-pressure water jet pulverization has the advantages of highly combustible material recovery, high separation efficiency, and low energy consumption. Cui et al. (2007) and Cui et al. (2008) used a high-pressure water jet to dissociate a 3 mm coal sample to 2.71 μm and obtained UCC with an ash content of 1.05% to prepare an ultra-fine coal-water slurry.

Under a specific temperature and pressure, SiO_2 in coal reacts with alkali to form soluble salts and is removed, while kaolin clay minerals react with alkali to form products that are soluble in acid and insoluble in alkali (Mukherjee and Borthakur, 2001). Therefore, coal can be treated with alkali first and then with acid to remove the ash. Nabeel et al. (2009) achieved a deashing rate of more than 90% after a step-by-step treatment by changing the densities of $NaOH$-H_2SO_4.

Minerals such as phosphates and carbonates can be dissolved by HCl, and HF can react with minerals other than pyrite. Most of the reaction products are water-soluble, but some metal fluorides are insoluble in water (Steel et al., 2001; Steel and Patrick, 2003). Therefore, the product after HF treatment needs to be further leached by HNO_3 to remove fluoride deposits and pyrite sulfur (Hacifazlioglu, 2016). Furthermore, microwave irradiation pre-treatment has a positive effect on the leaching of minerals from coal with HF (Jorjani et al., 2011). Kizgut et al. (2006) crushed clean coal after gravity concentration to below 200 mesh and then used HNO_3 to reduce the ash content of the coal sample to between 0.15 and 0.41%; however, the content of N and O elements increased in the product. Jorjani et al. (2011) used a coal sample with an initial ash content of 8.31%, which was pretreated by microwave radiation, leached with HF solution, and then leached with HNO3 solution at a temperature of 65 °C for 1 h to obtain the ash content of the coal from 2.57% to 0.69%.

The chemical method of preparing UCC has a lower ash content but is costly. In the de-ashing process, high-priced acid and alkali chemicals are used, and a large amount of wastewater is generated, which must be purified before discharge. Therefore, a combined method consisting of physical separation and chemical cleaning has the potential to considerably reduce ash while producing less wastewater (Meshram et al., 2015). Hacifazlioglu (2016) separated coal samples with an ash content of approximately 7% by heavy media and then obtained UCC with an ash content of 0.82% by leaching with HF solution. Yang et al. (2019) used Taixi anthracite as a raw material to reduce the total amount of minerals with low reactivity, such as quartz and aluminosilicate, by flotation pretreatment and then reacting the minerals in the flotation clean coal with NaOH solution to obtain an ash content of 0.12% UCC.

Coal slime has high ash content, and direct sales not only affects economic benefits, but also wastes coal resources. This study aimed to prepare UCC from slime after underflow pressure filtration of heavy media, to promote the recycling of coal slime and improve the utilization value of coal slime. Proximate and ultimate analyses of the slime were performed to understand the characteristics of the primary slime. The types of minerals in the slime were analyzed using XRD and SEM-EDS. Though the Zeta potential and contact angle measurements of the coal slime sample after grinding; combined with the calculation of the interaction force of the coal particles and the comprehensive analysis of the contact angle, the dissociation characteristics of coal slime under different grinding conditions were studied. The grinding method was determined by wet grinding, and UCC was prepared by shear flocculation flotation.

2. Materials and methods

2.1. Materials

The slime sample was collected from a coal preparation plant located in Shanxi Province, China. Table 1. shows the proximate and ultimate analyses of primary coal slime. The ash content of the dry-basis coal slime was 35.80%, and the sulfur content was 0.96%, corresponding to low-sulfur coal. For the floating and sinking test analysis of -0.5 mm slime, the content of slime at +0.074 mm was 75.35%, ash content was 31.87%, and the content of slime at -0.074 mm was 24.65%, ash content was 47.33%. Therefore, to prepare UCC via flotation, it is necessary to remove high-ash components such as fine mud to reduce the influence of slime on the flotation effect. When the classification density was 1.4 g/cm^3, the slime ash content was 3.95%, and this sample was selected as the raw material for preparing UCC.

Table 1. Proximate and ultimate analysis of primary coal slime

M_{ad} (%)	A_d (%)	V_{daf} (%)	FC_{daf} (%)	C (%)	H (%)	O (%)	N (%)	S_t (%)
0.85	35.80	14.44	85.56	86.94	3.31	7.62	1.17	0.96

2.2. XRD test

XRD was used to analyze the types of minerals in the coal slime. Mineral composition analysis of the 0.125-0.5 mm coal slime of -1.4 g/cm^3 was conducted using a Bruker D8 Advance X-ray diffractometer. The XRD results were analyzed by MDI Jade 6.0 software, and semi-quantitative analysis of minerals by "easy Quantitative" option after linear fitting of XRD spectra.

2.3. SEM-EDS test

SEM-EDS was used to analyze the embedded characteristics of the minerals in the coal slime. The analysis of the embedded characteristics of the 0.125–0.5 mm coal slime of -1.4 g/cm^3 was conducted under backscattering conditions using a Phenom Pharos G1 scanning electron microscope system (Phenom, Netherlands).

2.4. Grinding and Flotation test

In this test, a vertical stirring ball mill with 5 mm and 8 mm alumina ceramic ball grinding media was used. The media filling rate was 40%, and the spindle speed of the mill was 300 r/min. The process flow for preparing UCC from slime is shown in Fig. 1.

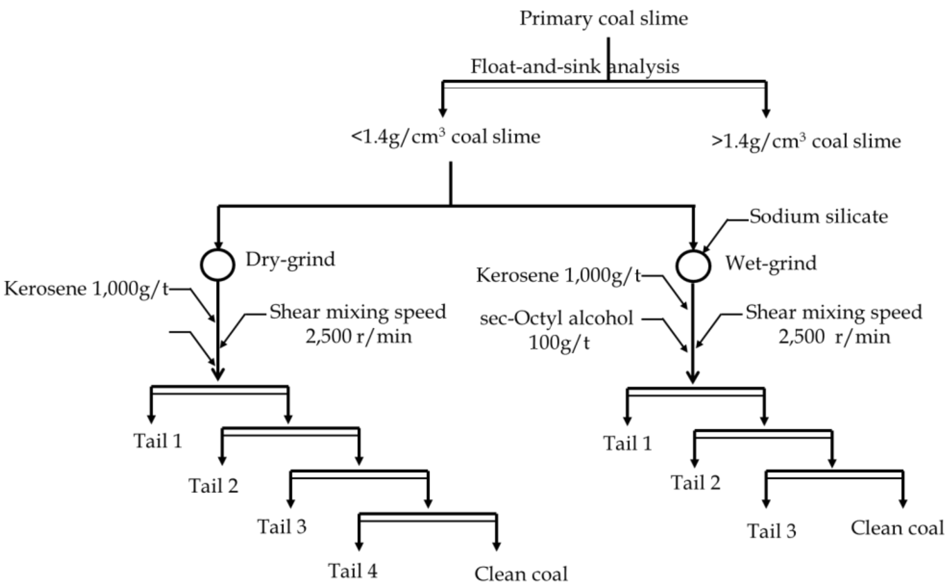

Fig. 1. The process flow of preparing ultra clean coal from slime

The dry-ground test was performed by grinding 600 g of -1.4 g/cm^3 slime for 2 h without adding any chemicals. In the wet grind-test, 600 g of -1.4 g/cm^3 coal slime and 1.8 L of water were added to the mill and ground for 2 h, and 5% water glass solution was added, the amount of water glass was 1,000 g/t. After grinding, 2 g each of the two groups of samples was added to anhydrous ethanol to wet, ultrasonically dispersed for 10 min, and then used to measure the particle size distribution with an Omega laser particle sizer. After grinding, the coal samples were subjected to shear flocculation and flotation, under the conditions listed in Table 2, to prepare UCC using an XFD-1 flotation machine.

Table 2. Test conditions of shear flocculation flotation

Test conditions	Reference values
Stirring speed (r/min)	2,500
Flocculation time (min)	20
kerosene (g/t)	1,000
Pulp concentration (g/L)	30
sec-octanol (g/t)	100
Flotation machine speed (r/min)	1,800
Aeration volume of flotation machine (m^3/h)	0.2

2.5. Zeta potential measurement and contact angle measurement

To explore the potential value change of the samples after grinding and the interaction between particles, 2 g of the samples after grinding was taken at different grinding times of 0.5, 1, 1.5, and 2 h. After removal, the sample was placed in 50 mL of water and stirred for 30 min using ultrasonic dispersion, and the supernatant was collected for measurement.

During the grinding operation, the samples produced a large amount of nascent surface during the process of decreasing the particle size. As the surface area "a" increases by "γda," this reversible surface work increases by "γda." The contact angle of the coal slime was measured using a JC2000 contact angle meter to compare the surface free energies of the samples under different grinding methods.

3. Results and discussion

3.1. Characterization

The XRD patterns are shown in Fig. 2; the minerals in the slime were mainly kaolinite and also contained a small amount of quartz and pyrite. Clay minerals are easily sludged in contact with water, and they coagulate heterogeneously with coal particles during the flotation process, thus making the surface of the coal particles hydrophilic. Fig. 2c shows the relative contents of kaolinite, dickite, and nacrite at 67.77%, 27.03%, and 5.20%, respectively.

As observed in Fig. 3, the dissemination size of minerals in coal slime was relatively fine (below 10 μm), and the dissemination size of different minerals was not uniform. The particle size of pyrite was

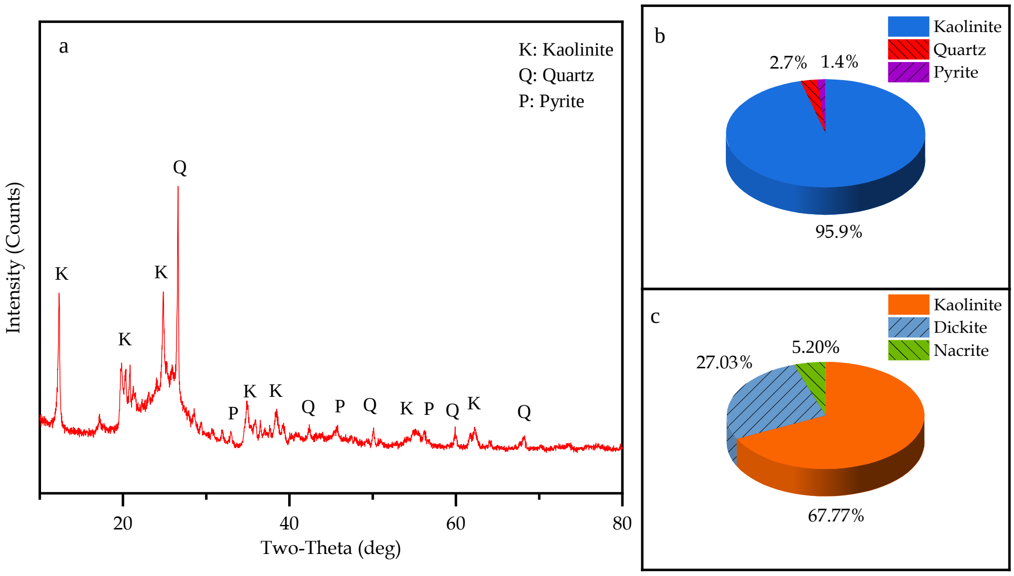

Fig. 2. X-Ray diffraction pattern of the coal slime

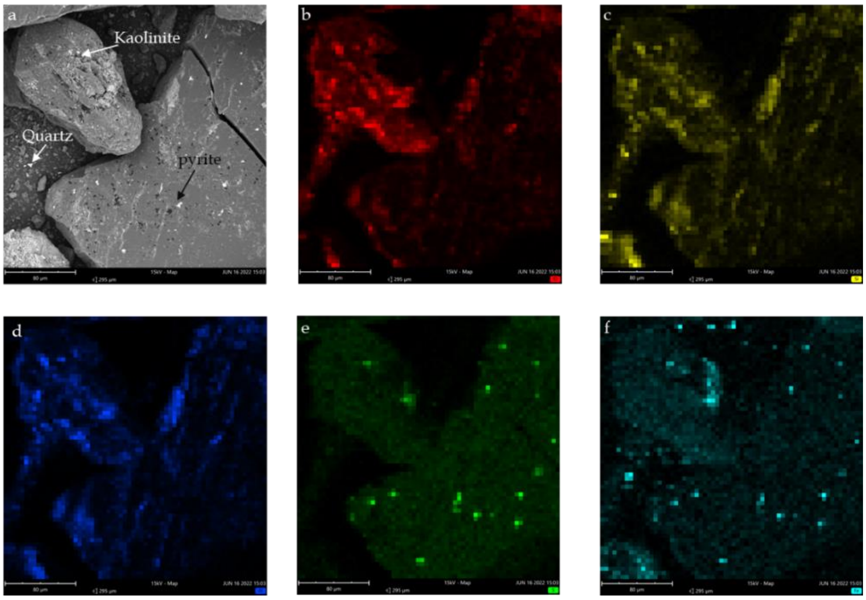

Fig. 3. Micro image and micro morphology of coal were observed using scanning electron microscopy combined with energy dispersive spectroscopy

relatively coarse at approximately 7 μm, followed by quartz particles at approximately 5 μm. However, quartz particles are prone to regional aggregation and become larger with a diameter of 10–15 μm. Kaolinite clay minerals had the smallest embedded particle size, ranging from 0 to 3 μm and were evenly dispersed in the coal in flocculent form. It is difficult for these clay minerals to float and deash. UCC can be obtained by removing high-ash fine mud.

3.2. Analysis of grinding results

The particle size distribution of coal slime with different grinding methods is shown in Fig. 4. The parameter characteristics of the coal slime particle size after grinding for 2 h are listed in Table 3. The particle sizes of the dry and wet grinders were normally distributed. After 2 h of dry grinding, the dry-ground particles were concentrated in the range of 3–7 μm, the value of D(4, 3) was 4.01 μm, and the differential distribution of dry-ground particles reached a peak value of 16% at a particle size of 4.50 μm. After wet-grinding for 2 h, the value of D(4, 3) was 3.79 μm. The cumulative distribution of particle sizes below 10 μm was 99.44%. Compared to dry grinding, wet grinding achieved a peak value of 12% at approximately 4 μm, and the cumulative distribution was approximately 60%. Wet-grind and dry-grind had little difference in grinding efficiency, and the coal slime particles could be ground to approximately 4 μm when the grinding time was 2 h.

4. Table 3. Parameter characteristics of coal slime particle size after grinding for 2 h

Grinding method	D(4, 3) (μm)	D(3, 2) (μm)	specific surface area (m^2/kg)
Dry-grind	4.01	3.32	3,533
Wet-grind	3.79	2.74	4,283

4.1. Calculation of total energy between particles

The variation in the zeta potential of the slime with grinding time under different grinding modes is shown in Fig. 5. The zeta potential value of the dry-ground sample was lower than that of the wet-ground sample for the same grinding time. The results showed that in the flotation process, the slime after dry grinding was more likely to agglomerate than that after wet grinding, which was not conducive to the separation of coal and inorganic minerals. When the grinding time was 2 h, the zeta potentials of the dry-and wet-ground samples were less than 25 mV.

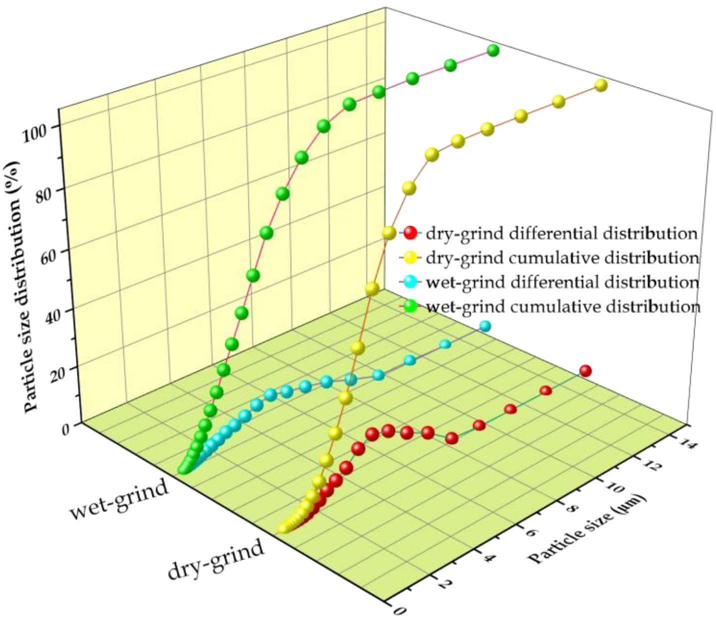

Fig. 4. Particle size distribution of slime with different grinding methods

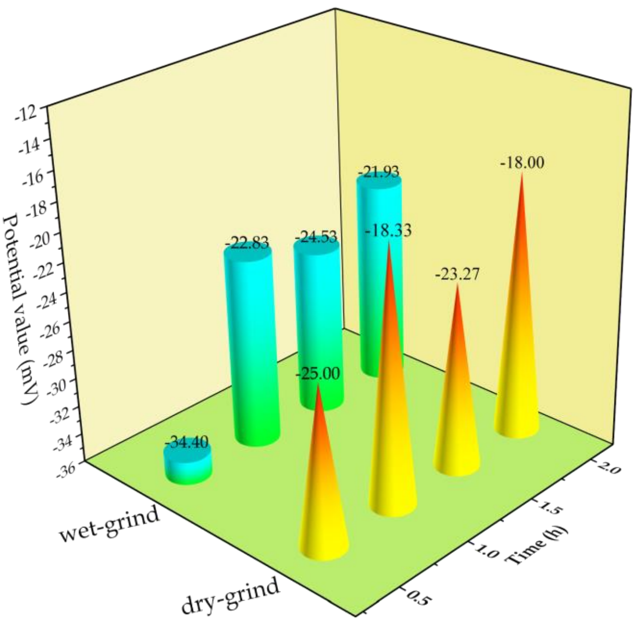

Fig. 5. Variation results of zeta potential of slime with grinding time under different grinding modes

The DLVO theory is based on the electrostatic energy and van der Waals energy between particles. When the particles are close to each other, the two opposite interaction energies determine the stability of the system. The extended DLVO (EDLVO) theory confirms that besides electrostatic energy and van der Waals energy, there is hydration or hydrophobic energy in the fine-particle flotation system (Yao et al., 2018; Yin and Wang, 2014). Eq. (1) describes the total energy between the slime particles. The electrostatic, van der Waals, and hydrophobic energies were calculated using Eq. (2)– (4).

$$V_{\mathrm{TED}} = V_{\mathrm{TD}} + V_{\mathrm{HA}} = V_E + V_W + V_{\mathrm{HA}} \tag{1}$$

$$V_E = 2\pi\varepsilon_\alpha R\psi_0^2 \, ln[1 + exp(-\kappa H)] \tag{2}$$

$$V_W = -\frac{AR}{12H} \tag{3}$$

$$V_{\mathrm{HA}} = \pi R h_0 V_{\mathrm{HA}}^0 \, exp(-\frac{H}{h_0}) \tag{4}$$

where V_E, V_W, V_{HA}, and V_{TD} are the electrostatic, van der Waals, hydrophobic, and DLVO energy (Yao et al., 2016); ψ_0: mineral surface potential; κ^{-1}: Debye length in units of nm (Hu et al., 2019). This value was calculated using Eq. (5). c is the ion volume molar concentration (mol/L); the solution was deionized water during the test, and the values of C_{H^+} and C_{OH^-} were 10^{-7} mol/L;

$$\kappa^{-1} = \frac{0.304}{\sqrt{c}} \tag{5}$$

ε_a: absolute dielectric constant of dispersion medium (Hu et al., 2019), 6.05 × 10^{-10} $C^{-2}J^{-1}m^{-1}$; H: interparticle interaction distance, nm; R: particle size, μm; A: Hamaker constant; the Hamaker constants of coal, kaolinite, quartz, and water were 6.07 × 10–20 J, 14.5 × 10–20 J, 7.5 × 10–20 J, and 4 × 10–20 J (Chen et al., 2020). The effective Hamaker constant for the interaction of ore particles in an aqueous medium was calculated using Eq. (6)– (7).

$$A_{131} = A_{11} + A_{33} - 2A_{13} \approx (\sqrt{A_{11}} - \sqrt{A_{33}})^2 \tag{6}$$

$$A_{132} = A_{12} + A_{33} - A_{13} - A_{23} \approx (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}}) \tag{7}$$

h_0: decay length, h_0 of coal was 10.3 nm (Xu and Yoon, 1990); V^0_{HA}: Hydrophobic interaction energy constant (Xu and Yoon, 1990); V^0_{HA} of coal was 1.247 mJ/m².

The interaction energies between the particles under different grinding conditions are shown in Fig. 6. The DLVO energies of the particles obtained using the different grinding methods were all positive. As the interaction distance between the particles decreased, the DLVO energy first increased and then decreased, and the energy barrier appeared at approximately 25 nm. On the right side of the energy barrier, electrostatic energy dominated, and on the left side of the energy barrier, the van der Waals energy gradually increased owing to the gradual shortening of the interaction distance between the particles, resulting in a decrease in the DLVO energy.

From Fig. 6a and b, the interaction energy between coal particles was the highest, the interaction energy between kaolinite and coal particles and the interaction energy between quartz and coal particles was smaller than that between coal particles. This indicates that coal, kaolinite, and quartz particles aggregated more easily than coal and coal particles. Therefore, the aggregation of coal, kaolinite, and quartz particles during the flotation process led to an increase in the ash content of UCC. The DLVO energy between the particles after wet grinding was greater than that after dry grinding. It was observed that the water glass increased the zeta potential of the ground sample at a higher level, and the electrostatic energy between the particles was larger.

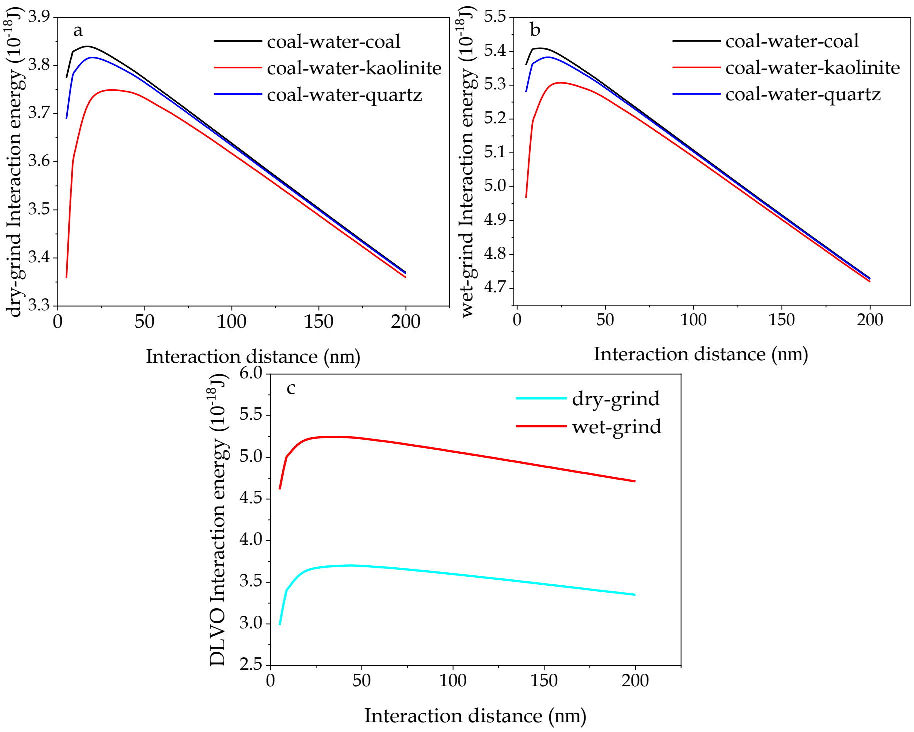

Fig. 6. Interaction energy between particles under different grinding conditions

The total energy change in the interaction between particles after the introduction of the hydrophobic interaction energy is shown in Fig. 7. As the interaction distance decreased, the hydrophobic interaction energy increased rapidly and the total energy between the particles became negative. This explains why agglomerate behavior easily occurs between particles, and the absolute value of the total action energy of dry-ground was larger than that of wet-ground. After dry grinding, the sample particles were more attractive to each other in the slurry, and flocculation was more likely to occur, which would aggravate the entrainment of fine mud during the flotation process, affecting UCC ash.

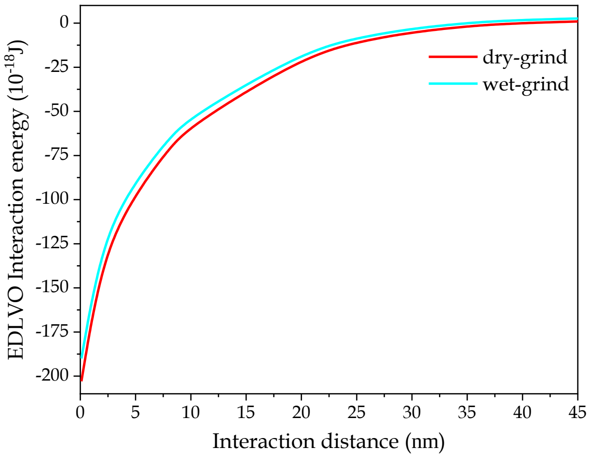

Fig. 7. Total energy of interaction between particles under different grinding modes

4.2. Calculation of interface free energy

As shown in Fig. 8, the contact angle value measured after 2 h of dry grinding and wet grinding was 82.23° and 80.23°, respectively. At the solid-liquid interface, the interfacial energy of the solid surface in the liquid phase can be calculated using the Dupre equation and Young's equation (Arkhipov et al., 2012; Thomas et al., 2008), as shown in Eq. (8)– (10).

$$\gamma_{s-g} = \gamma_{s-l} + \gamma_{l-g}\cos\theta \tag{8}$$

$$\Delta W = \gamma_{s_{-g}} + \gamma_{l_{-g}} - \gamma_{s-l} \tag{9}$$

$$\Delta W = \gamma_{l-g}(1+\cos\theta) = -\Delta G_{s-l} \tag{10}$$

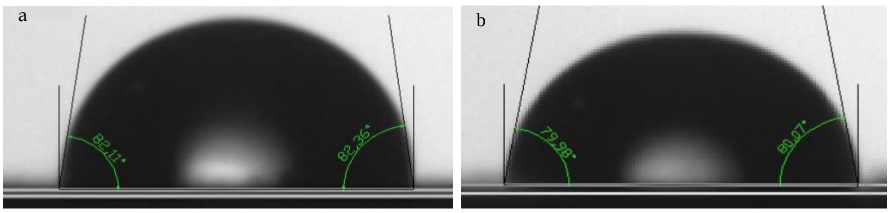

Fig. 8. Measurement of 2 h contact angle under different grinding modes

The calculation results are presented in Table 4. The interfacial free energy of the two grinding methods in the slurry was positive and that of the wet ground was slightly larger than that of the dry ground. The slime surface was more hydrophilic after wet grinding, whereas the surface of the slime after dry grinding was more hydrophobic. After dry-grinding, the hydrophobic force of slime in the slurry was stronger, and its agglomeration behavior was stronger than that of wet grinding; therefore, the entrainment of fine mud was more serious, which was also the cause of poor selectivity and high ash content in the flotation process of dry-ground products.

Table 4. Calculation results of interfacial free energy of different grinding methods

Grinding method	θ (°)	Specific surface area (m^2/kg)	$\Delta G_{s\text{-}l}$ (J)
Dry-grind	82.23	3.647	3.40
Wet-grind	80.03	4.283	3.89

4.3. Analysis of flotation results

The curves of the cumulative yield and cumulative ash content of UCC under different grinding methods are shown in Fig. 9. After dry grinding, the slime was floated four times to obtain UCC with an ash content of 1.93% and a yield of 25.27%. After wet grinding, the ash content of the slime was reduced to approximately 2% after one flotation, and this was achieved through three flotations to obtain UCC with an yield of 3.88% and ash content of 0.95%. This demonstrated that the water glass solution had a strong inhibitory effect on the gangue, and UCC with an ash content of less than 1% could be obtained after only three flotations. However, water glass also substantially inhibited the yield.

After wet grinding, the flocculation stirring speed was set to 2,700 rpm to obtain UCC with the maximum yield. The test results are presented in Table 5. After two flotations, UCC with a yield of 41.74% and an ash content of 1.90% was obtained.

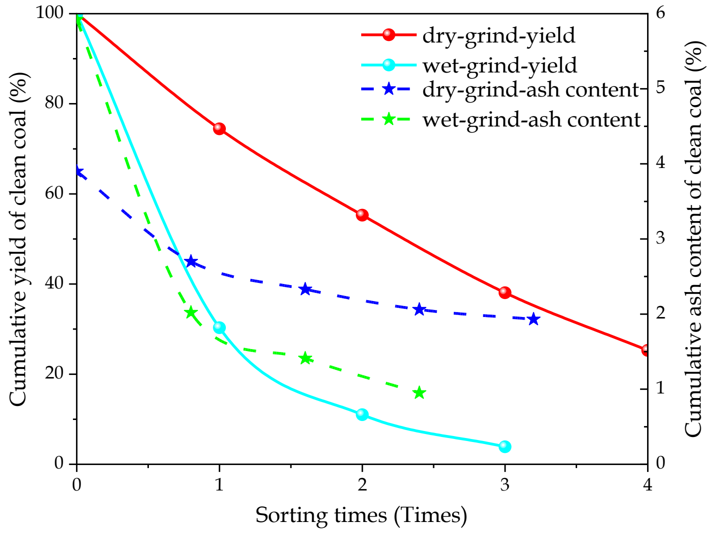

Fig. 9. The curve of cumulative yield and cumulative ash content of ultra-clean coal

Table 5. Test results of shear flocculation flotation for wet grinding under the best conditions

Product	Yield (%)	Ash (%)	Cumulative yield (%)	Cumulative yield (%)
Ultra-clean coal	41.74	1.90	41.74	1.90
Tail 1	17.32	3.10	59.06	2.26
Tail 2	40.94	10.53	100.00	5.64
Total	100.00	5.64		

5. Conclusions

This study discussed the technology of coal slime recovery for the preparation of UCC. Through the comparison of different grinding methods of slime, it was found that with a decrease in the interaction distance, the interaction energy between particles was negative, and the particles were prone to agglomeration. The interaction energy of the dry-ground slime was greater than that of the wet-ground slime; therefore, the dry-milled particles were more attracted to each other in the slurry and easier to agglomerate. Under the optimal flotation test conditions, the dry-ground slime yielded UCC with an ash content of 1.93% and yield of 25.27% after four flotations, whereas the wet-ground slime yielded UCC with an ash content of 1.90% and yield of 41.74% after two flotations. Therefore, the wet grinding method should be adopted in the grinding process, and an inhibitor should be added to the mill in a targeted manner. However, the addition of inhibitors resulted in a low yield of ultra-clean coal, and more efficient and selective inhibitors should be explored in the follow-up research.

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