Physicochem. Probl. Miner. Process., 61(2), 2025, 204061 | Physicochemical Problems of Mineral Processing

http://www.journalssystem.com/ppmp | ISSN 1643-1049

Experimental study on enhanced flotation of low rank coal by high efficiency collector

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Abstract: Flotation of low-rank coal presents challenges due to its low surface hydrophobicity. The poor flotation behavior of low-rank coal is mainly caused by the high content of functional oxygen groups present on the coal surface. A high-efficiency flotation collector (XZ-1) was developed to address the problem that low-rank coal is difficult to be recovered by flotation with conventional non-polar oil collector. Results showed that when the dosage of XZ-1 was 300g/t, the yield of flotation concentrate is 60.99% and the ash content is 12.08%. Compared with the use of conventional collector diesel (when the dosage of reagent is 500g/t, the yield of flotation concentrate is 56.03% and the ash content is 12.42%). The flotation results showed that the flotation of low-rank coal in the presence of XZ-1 contributed to better flotation performance than that obtained by diesel, which indicated that XZ-1 can be more effective collector than diesel to float low-rank coal. The contact angle, Fourier transform infrared spectroscopy test and X-ray photoelectron spectroscopy analysis demonstrated that XZ-1 collector can significantly improve the hydrophobicity of low-rank coal, resulting in a great improvement of flotation efficiency. Therefore, XZ-1 could increase the residual carbon recovery and reduce the flotation collector dosage, which has a good application prospect.

Keywords: low rank coal flotation, high-efficient collector, contact angle

1. Introduction

Coal is an important non-renewable fossil energy and occupies an important position in the world energy (Ni et al., 2016). China is rich in coal resources. Low-rank coal has become more and more important in social development. Its dominant position in China's energy consumption structure is difficult to replace in the short term (Xi et al., 2017).

It is generally known that low-rank coals are the most difficulty to float. Low-rank coal is a porous material. The oxygen-containing functional groups .hydroxyl, carboxyl, carbonyl, etc.) on the surface of low-rank coal can better contact with water and react with water molecules to form hydrogen bonds, so that more water is stored in the pores (Stanczyk et al., 2021; Xi et al., 2014; Xi et al., 2019). And because of the high oxygen content and poor hydrophobicity on the surface of low-rank coal, it is difficult to float. In addition, low rank coal has large water content, high impurity content and low calorific value. If it is directly burned, not only the energy consumption is large, but also the amount of flue gas generated during the combustion process is more than that of high rank coal, which greatly limits the mining value of low-cost coal (Sahoo et al., 2016). In addition, if low-rank coal is not fully utilized, it will not only cause waste of resources, but also cause environmental pollution around the mining area (Chen et al, 2018). Therefore, the upgrading, processing and comprehensive utilization of low rank coal with large storage capacity plays a very important role in alleviating the increasingly urgent energy pressure and reducing energy consumption in China (Zhang et al., 2025).

Flotation is one of the most effective methods for separating fine-grained low-rank coal slime (Cebeci, 2002; Xia et al., 2013). However, due to the low degree of metamorphism of low-rank coal, the surface is rich in a large number of oxygen-containing functional groups, resulting in poor hydrophobicity (Zhu

DOI: 10.37190/ppmp/204061

et al., 2020). Traditional non-polar hydrocarbon oil collectors such as kerosene and diesel are difficult to effectively improve the hydrophobicity of the surface of low-rank coal, resulting in unsatisfactory flotation effect and thus plaguing the flotation upgrading of low-rank coal slime (Dey, 2012; Xia, 2016; Zhang et al., 2020). Therefore, in order to improve the flotation efficiency of low rank coal, it is necessary to find a highly selective collector that can efficiently adsorb organic matter in coal and enhance its wetting effect with minerals to improve the flotation efficiency of low rank coal (Zhang et al., 2021). Xia et al, in their study on the flotation of oxidized coal, demonstrated that biodiesel, which contains a higher concentration of fatty acids, was a more effective collector than diesel. Peng et al proposed that the enhanced performance of a composite collector, comprising TX-100 and diesel, in oxidized coal flotation was due to TX-100's ability to emulsify the diesel, thereby rendering the hydrophilic surface hydrophobic through "head-on" adsorption. Additionally, Zhang et al introduced the cationic surfactant DTAB into diesel as a combined agent to improve the collection efficiency of low-rank coal.

In this investigation, XZ-1 was used t as high-efficiency collector for flotation tests and diesel was also used to be compared. In order to overcome many problems such as low-rank coal oxygen-containing functional groups, high oxidation degree, and poor floatability. The mechanism of high-efficiency collector on the flotation of low-rank coal slime was studied to solve the problems of low clean coal yield, high ash content, and large reagent consumption in the flotation process of low-rank coal in coal preparation plants.

2. Materials and methods

2.1. Experimental materials

The coal sample was collected from Inner Mongolia in China. The raw coal was crushed and ground to less than 0.5 mm and then used in the flotation experiment. According to the national standard 'Industrial analysis method of coal' (GB/T212-2008), the industrial analysis of the test coal samples was carried out. According to the national standard " coal element analysis method " (GB /T476-2001), the element analysis of the test coal sample was carried out by using the element analyzer. The results are shown in table 1-2.

Table 1. The proximate analysis of coal sample

M_{ad} (%)	A_{ad} (%)	V_{ad} (%)	FC_{daf} (%)
2.15	21.95	25.91	49.99

M_{ad}: the moisture content in the coal on an air-dried (ad) basis; A_{ad}: the ash content in the coal on an air-dried (ad) basis; V_{daf}: the volatile content in the coal on dry ash-free (daf) basis; FC_{daf}: the fixed carbon content in the coal on dry ash-free (daf) basis.

Table 2. Relative contents of chemical elements on coal surface

Element	C_{daf} (%)	O_{daf} (%)	N_{daf} (%)	H_{daf} (%)	$S_{t,\ d}$ (%)
Content	82.86	7.98	1.61	4.82	2.1

According to the proximate analysis in Table 1, the ash content of the coal sample was 21.95%, the moisture content (M_{ad}) was 2.15% and the air-drying volatile matter (V_{ad}) was 23.91%. The fixed carbon (FC_{ad}) content was 51.99%. According to the ultimate analysis, the carbon content was 82.86%, while the oxygen content was 7.98%. Indicating that the coal still contains high carbonaceous components and is a low metamorphic coal sample with high separation value.

2.2. Flotation and analysis method

2.2.1. Raw coal flotation employing various reagents

Batch experiments were to compare the low-rank coal flotation performance under various chemicals additions. As for the control group, the dosages of diesel oil were 200, 300, and 500 g/t, and meanwhile sec-octyl alcohols usage was 50、75、125 g/t. The dosage of collector (XZ-1) and frother in the experimental group was the same as that in the control group. A 1.5L flotation cell with aeration of 0.35m^3/h was used. The speed of impeller was 1900 r/min and the pulp density was 60g/L. The experimental procedures were as follow: At room temperature 26 degrees Celsius, 90 g of raw coal and water were firstly transferred into the flotation cell and followed by a pre-wetting treatment for 3 min. Then, diesel oil was injected immediately and the solution conditioning lasted for 2min. Later, pulp was conditioned for another 30 s after frother was added and then turn on the aeration. The experimental group is the same.

The concentrate and tailings were collected, dried and weighed at last. The flotation concentrate product was collected until there was no froth product left in the flotation cell. The concentrate es and tailings were filtered and dried in an oven at 80°C to a constant weight; later, ash measurement was conducted.

The test instruments include: RK/FD type 1.5L single tank flotation machine (spindle speed 0~2000r/min, scraper speed 0~30r/min), WS-6130 intelligent integrated muffle furnace, electronic balance, type analytical balance (precision 0.0001g), DHG-9623A blast drying oven, beaker, pinhole syringe, porcelain boat.

2.3. Analysis method

2.3.1. Contact angle measurement

The contact angle refers to the angle between the tangent of the gas-liquid interface at the intersection of gas, liquid and solid through the liquid and the solid-liquid interface. Before the test, the coal samples treated with different reagent ratio were pressed on a tableting machine to form coal pieces with a thickness of about 2 mm, and the pressure of the tablet press was set to 20 MPa. Contact angle measurement apparatus (JC2000D1, Shanghai Zhongchen Digital Technic Apparatus Co., Ltd., Shanghai, China) was used for measuring the contact angle. Using the free sessile drop method, contact angles on coal samples were measured in distilled water at a room temperature of 26C. A drop of DI water (about 0.016 mL) was placed on the surface of the sample, and the images of the water drop on the surfaces were taken by computer software once the droplet had stabilized. Each sample was measured five times and the average value was reported.

2.3.2. Surface tension

In this investigation, we employed the Surface Tensiometer to measure the surface tension of solution under various concentrations of SCG. A platinum ring with diameter of 19.69 mm was used. The trigger tension of this method is 0.5 mN/m. The ring was vertically inserted into the solution and then lifted to determine the air–liquid interface tension. The ring was washed by deionized water with ethanol, followed by burning to remove remaining organic before testing. Treble parallel tests were conducted to ensure the accuracy.

2.3.3. FTIR test

In this study, Fourier transform infrared spectroscopy was used to analyze the changes of oxygen-containing functional groups after pretreatment of low-rank coal with different collectors. Low-rank coal samples were pretreated with different collectors, and stirred at a constant temperature in a magnetic stirrer for an appropriate amount of time, so that the collector can achieve full adhesion on low-rank coal samples. For conventional conditions (only diesel oil), the dosage of collector is 200 g/t. For the XZ-1, the dosage was also kept as 200 g/t as a control test. Then filter paper was used to filter and dried at a low temperature of 40 °C, and then an appropriate amount of low-rank coal samples was ground to a coal sample with a particle size of < 0.045 mm. About 1 mg and about 150 mg KBr were mixed to press the mixture into transparent flakes at a pressure of 20 MPa. Finally, it was put into the FTIR instrument for analysis. The resolution of the instrument is 4 cm^{-1}, and the wavenumber accuracy is less than 0.1 cm^{-1}. The absorbance range is 4000 ~ 500cm^{-1}.

2.3.4. XPS Analysis

XPS was used to analyze the changes of surface elements and functional groups of low rank coal samples after different collector treatments. X-ray photoelectron spectroscopy (XPS) is a widely used surface analysis technique. It can measure the elemental composition of the material surface and provide the chemical state information of each element. The XPS spectrum data can be used for qualitative and quantitative analysis of the sample, thus revealing the composition and content of different components in the sample. The chemical state of each component, the chemical structure of atoms and molecules, and the combination of chemical bonds can also be analyzed separately. In this study, XPS was used to analyze the changes of oxygen-containing functional groups on the surface of low rank coal after the action of different collectors. In the XPS test, the test coal sample should be pretreated first. First, the test coal sample is mixed with different collectors at a dosage of 3.0 kg/t, and then stirred at room temperature on a magnetic stirrer for 30 min. After filtration, it is dried in an electric blast drying oven at 313 K for 6 h. Finally, XPS was used to narrowly scan the elemental carbon of low-rank coal samples after the action of different collectors. XPS uses parameter settings; the radiation source is monochromatic AlKα, and the spot size is 500 µm. The pressures of the analysis chamber and the preparation chamber were 5.0 × 1010 and 7.0 × 1010 mbar, respectively. The wide scanning energy is 50.0 eV (resolution = 1.0 eV). The narrow scanning energy is 20.0eV (resolution = 0.05eV).

2.3.5. Zeta potential measurements

Zeta potentials were determined using Brookhaven. Zeta plus zeta meter (USA). The mineral samples were ground to 0–5 Am in an agate mortar. The suspensions containing solids 0.05% (mass fraction) were conditioned in a beaker for 15 min and pH was measured at room temperature (26℃).

3. Results and discussion

3.1. Flotation test results

Diesel and XZ-1 were used to compare the flotation effects of low-rank coal. The total dosage of collector was 200, 300, 500g/t, and the results are shown in Fig. 1. It can be seen from Fig. 1 that the yield of XZ-1 is obviously higher than that of diesel. When the dosage is 500g/t, the use of XZ-1 is 14.73 percentage points higher than that of diesel, and the combustible recovery is 15.76 percentage points higher. When the dosage is 200g/T, the yield of XZ-1 is high and the ash content of clean coal is low.

Therefore, the flotation effect of XZ-1 is better than that of diesel oil, which can improve the flotation effect of low-rank coal to a certain extent.

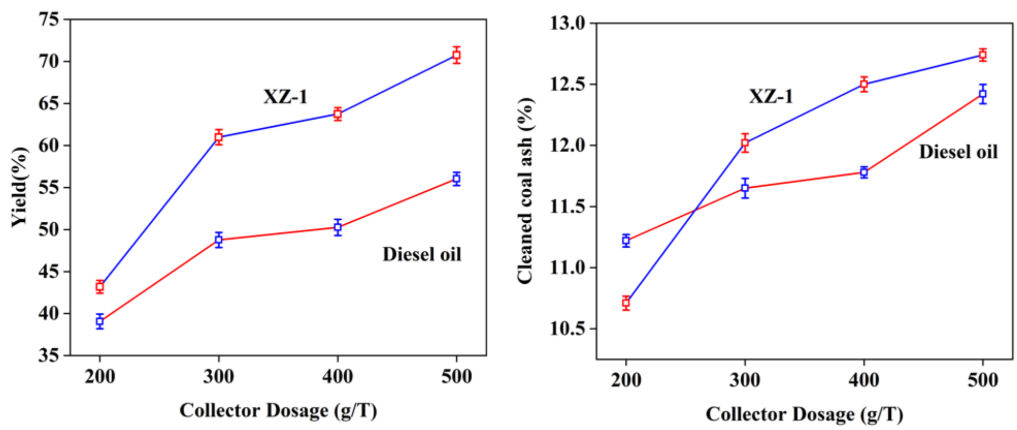

Fig. 1. Clean coal yield and ash content

3.2. Improvement of the low-rank coal surface hydrophobicity

3.2.1. Contact angle

Contact angle is the most intuitionistic index to measure the hydrophobicity of a mineral surface, and can also indicate a mineral's floatability difference. The larger the contact angle, the better the hydrophobicity and floatability of the mineral surface (Zhou et al., 2018). The floatability of the mineral is defined as the difference between the number 1 and the wettability, expressed as: Floatability=1-cos(θ). According to Eq.(1), the wettability and floatability of coal particles treated with different proportions of chemicals can be calculated and analyzed. In this study, the DSA100 optical contact angle measuring instrument was used to measure the contact angle of coal samples with different blending ratios. For conventional conditions (only diesel oil), the collector and dosage were 200g/t diesel oil. For XZ-1, the equivalent kerosene dosage was also kept as 200 g/t as a control test. The test results are shown in Fig. 2.

As shown in Fig. 2, the contact angles without the treatment using collectors is 52.95◦, The contact angle of low-rank coal is increased when coal particles were treated with diesel oil and XZ-1. The contact angle after diesel treatment is 53.58◦, and the contact angle after XZ-1 treatment is 59.53◦. The use of compound collectors produced a greater contact angle for low-rank coal than that of diesel oil only. It is revealed that both diesel oil and XZ-1 can improve the hydrophobicity of the low-rank coal surface. But the XZ-1 increased by a larger margin.

$$cos\theta = (\gamma_{SG} - \gamma_{SL})/\gamma_{LG} \quad (1)$$

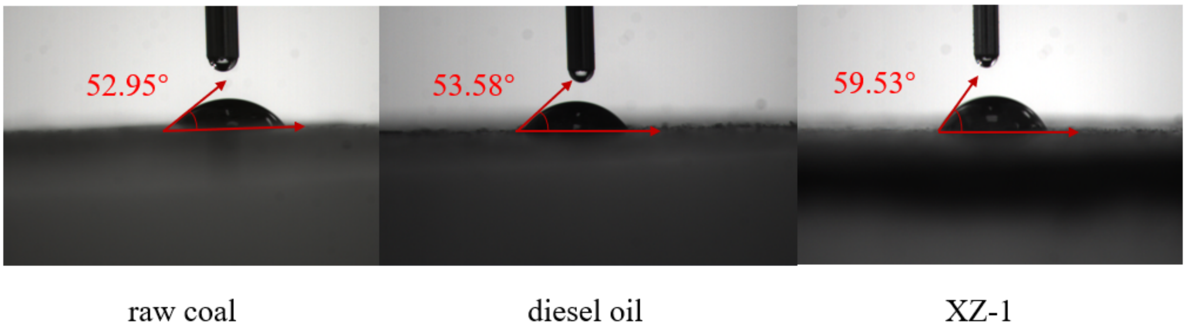

Fig. 2. Contact angle under the action of different collectors

3.2.2. Surface tension

The influence of collector on foam characteristics is mainly analyzed from the surface tension of the solution, foaming ability, foam stability, bubble size distribution and so on. By studying the influence of the type and concentration of the combined collector on the surface tension of the solution system, it is helpful to analyze its foam characteristics and flotation effect. Under the natural pulp pH value, the surface tension changes of the high-efficiency collector XZ-1 and diesel oil at different reagent concentrations (mol/L) were investigated. The results are shown in Fig. 3.

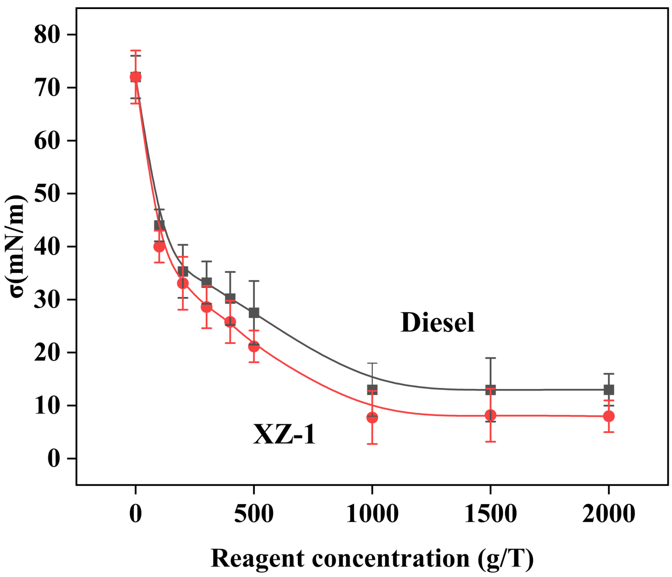

Fig. 3. Surface tension under different concentrations

From Fig. 3, it can be seen that under the two reagent systems, with the increase of collector concentration, the ability to reduce the surface tension of the solution gradually increases. When the collector concentration reaches the critical micelle concentration, the surface tension of the solution no longer changes with the increase of the collector concentration. In addition, under the same concentration conditions, the surface tensions of the two collector solutions are different, and the surface tension of XZ-1 solution is smaller than that of diesel solution. The smaller the surface tension of the solution under the action of the collector, the stronger the foam performance of the agent. It can be seen that under the same conditions, the foaming and foam stability of XZ-1 are the most significant.

3.2.3. FTIR results

The FTIR spectrum of low-rank coal is presented in Fig. 4. A prominent absorbance peak around 3350 cm⁻¹ corresponds to the –OH group, while the peak at 1605 cm⁻¹ is attributed to the COOH group. Absorption peaks between 1150 cm⁻¹ and 1380 cm⁻¹ are indicative of C-O and O-H bonds in phenoxy groups. These results suggest that subbituminous coal contains a significant number of oxygen-containing functional groups on its surface. The presence of these functional groups reduces the coal's hydrophobicity, thereby making flotation more challenging (Zhu et al., 2020).

The chemical structure of low rank coal mainly determines its wettability, but its surface can be modified by doping different collectors. The changes of surface functional groups of low rank coal after diesel and XZ-1 treatment were analyzed by FTIR. The results are shown in Fig. 4.

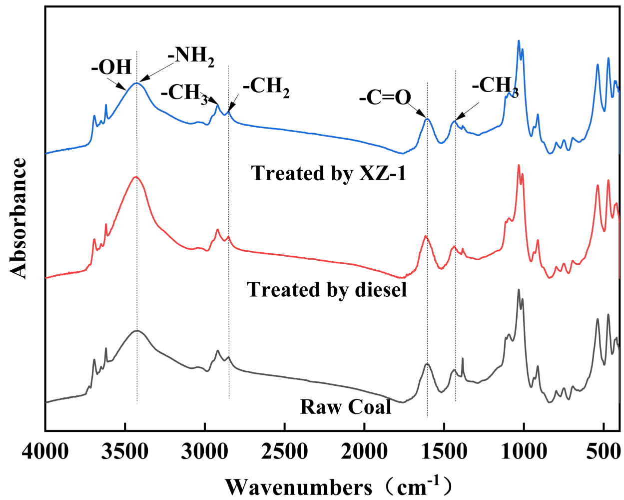

Fig. 4. Functional groups on low rank coal treated by diesel and XZ-1

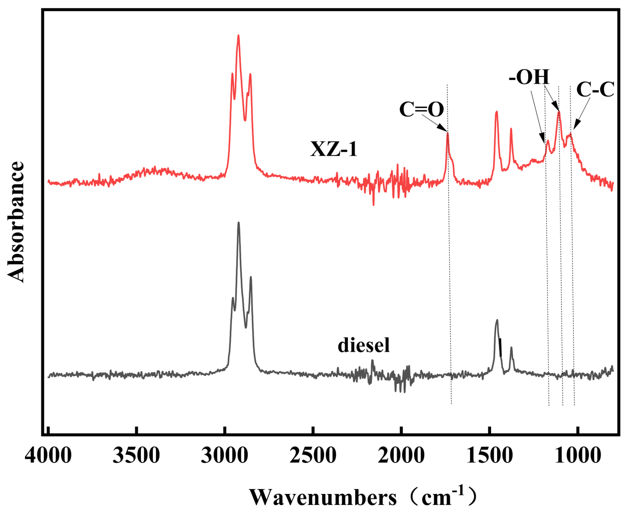

Fig. 5. Functional groups on diesel oil and XZ-1

As shown in Fig. 4, the presence of diesel oil and XZ-1 on the surface of low-rank coal reduces the intensity of the absorption band around 3350 cm^{-1}. Additionally, the abundant polar hydrophilic groups on the surface of low-rank coal hinder interactions with the non-polar diesel oil. In contrast, the hydroxyl and carboxyl groups on the surface of XZ-1 facilitate the formation of hydrogen bonds with the polar groups on the coal's surface. When the polar groups of XZ-1 interact with those on the low-rank coal surface, the non-polar ends of XZ-1 are exposed, thereby enhancing the hydrophobicity of the coal's surface.

For low-rank coal, the addition of XZ-1 significantly increases the content of hydrophobic functional groups, such as $-CH_3$ and $-CH_2$, while effectively reducing the presence of hydrophilic functional groups, including −OH, −C=O, and −COOH. This finding is consistent with the results obtained from XPS, contact angle measurements, and flotation tests (Chen et al., 2024).

3.2.4. XPS analysis

The C1s fitting results of different collectors under 300g/t pretreatment are shown in the Fig. 6. The binding energies of C-C/ C-H and C = O groups are 284.6 and 286.6 eV, respectively. C-C/C-H groups are hydrophobic groups, and C=O is hydrophilic group. The relative contents of three groups were calculated according to the fitting peak area. The higher the content of hydrophobic functional groups, the better the flotation effect, and the greater the effect of collector on the flotation performance of low rank coal. On the contrary, the higher the content of hydrophilic functional groups, the worse the flotation effect.

The hydrophobic group content of low rank coal treated by collector was 77.88%,75.70% and 72.74%, respectively, corresponding to; XZ-1>diesel> coal sample. It can be seen that the content of hydrophobic functional groups of C-C/C-H is the highest when XZ-1 is added in the flotation process, which is 5.14% higher than that of raw coal, which is consistent with the flotation results.

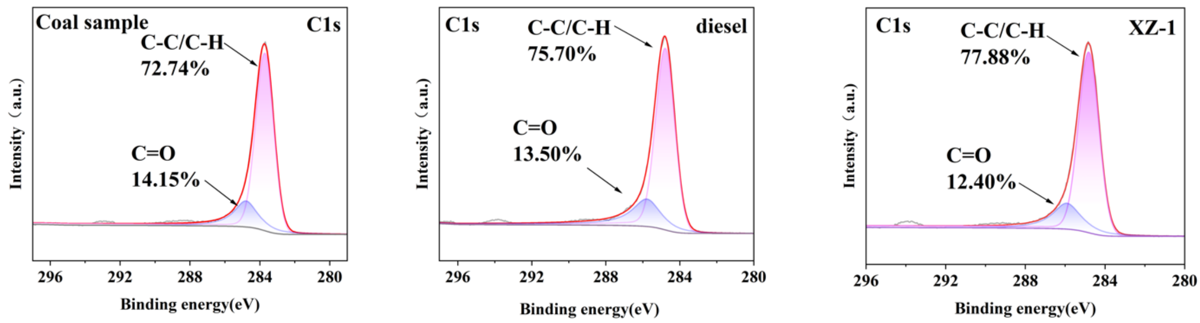

Fig. 6. XPS results for samples

The hydrophilic group C=O is the main oxygen-containing functional group. The contents of hydrophilic groups in XZ-1, diesel and raw coal were 12.40%, 13.50% and 14.15%, respectively. The content of hydrophilic group of C=O decreased by 1.75% and 0.65%, respectively, compared with the raw coal. Therefore, XZ-1 has a greater effect on the surface hydrophobicity of low rank coal.

Compared with raw coal, the content of C-C/C-H hydrophobic functional groups on the surface of low rank coal particles after XZ-1 treatment is higher than that of diesel oil, while the content of hydrophilic functional group C=O is lower. Therefore, it is verified that the oxygen-containing groups of XZ-1 are effectively adsorbed on the surface of low rank coal particles through van der Waals force and other effects, thereby improving the hydrophobicity of raw coal.

3.2.5. Zeta potential measurements

The zeta potential of the suspension of low-rank coal treated with diesel oil and XZ-1 was measured to further study the interaction between the collector and the coal sample. The absorption of XZ-1 increased the absolute value of the potential of low rank coal, from 22.50 mV to 26.8 mV. The zeta potential of low-rank coal treated with diesel is -23.2mV. This can be attributed to the hydrogen bond between the oxygen-containing molecular groups in XZ-1 and the oxygen-containing functional groups on the coal surface, resulting in a larger three-dimensional structure to store charge, thereby significantly increasing electronegativity.

4. Conclusions

In this paper, the influence of high-efficiency collector on the flotation effect of low-rank coal, as well as its wettability and adsorption difference on the surface of low-rank coal, was studied through the experimental exploration and characterization analysis of low-rank coal flotation. The main conclusions of this paper are as follows:

(1) Flotation experiments showed that when XZ-1 and diesel oil were used, XZ-1 achieved a higher flotation yield and significantly improved the flotation performance of low-rank coal.

(2) The contact angle between raw coal and water is 52.95°. After treatment with diesel oil, the contact angle increases to 53.58°, suggesting that diesel oil improves the hydrophobicity of the coal surface, but with limited effect. When treated with XZ-1, the contact angle rises to 59.53°. This increase is due to XZ-1 forming hydrogen bonds with oxygen-containing groups on the coal surface, while its hydrophobic carbon chain extends outward, significantly enhancing the coal's hydrophobicity.

(3) Pretreating low-rank coal with different collectors increases the content of hydrophobic groups (C-C, C-H) and decreases the content of hydrophilic groups (C-O, COOH). The levels of C-C and C-H on the coal surface are higher with XZ-1 than with diesel oil, which aligns with the flotation results. XZ-1 adsorbs its hydrophilic functional groups onto the coal surface's hydrophilic sites through hydrogen bonds, while the hydrophobic end is exposed to the bubbles. This significantly increases the hydrophobic functional groups on the coal surface, reduces adhesion time, and enhances its hydrophobicity.

Acknowledgement

The authors are thankful for the National Natural Science Foundation of China (Project No. 52174264).

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