Effect of NaNO$_2$ Reductant Pretreatment on Low-Rank Coal Flotation

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ABSTRACT: As the reserves of high-quality coal resources in China are decreasing, it is imperative to improve the processing and comprehensive utilization of low-rank coal. In this study, NaNO$_2$ was used for the flotation pretreatment test of the low-rank coal obtained from Majialiang, and the mechanism was discussed by contact angle analysis, zeta potential measurements, and XPS peak fitting analysis. The results showed that when the dosage of NaNO$_2$ was 2000 g/t and the pretreatment time was 5 min, the flotation effect was the best, the ash contents of concentrate ash and tailings and the combustible recovery were 17.15, 37.12, and 42.23%, respectively; the combustible recovery increased by 12%. The contact angle, surface functional group content, and zeta potential measurements showed that with the change of NaNO$_2$ dosage, the content of the hydrophobic functional group and the zeta potential value were consistent with the change of combustible recovery. The increase of hydrophobic functional groups can effectively enhance the hydrophobic interaction on the surface of the coal, which is conducive to the combination of collector and coal, and improve the efficiency of the collector. The NaNO$_2$ pretreatment test can promote flotation efficiency, and the addition of reductant is an effective method for the flotation efficiency of low-rank coal in reducing oxygen-containing functional groups on the surface of low-rank coal to improve the poor floatability. In this study, the method of chemical pretreatment is put forward to provide a new idea for slime flotation.

1. INTRODUCTION

Low-rank coal is a kind of coal in the early stage of coal formation, and due to its concentrated distribution and large reserves, it has great potential for separation, quality improvement, and comprehensive utilization. After decades of massive consumption, the reserves of high-quality coal resources decreased. Upgrading and processing low-rank coal can not only improve the economic benefits of coal preparation plants and reduce the waste of coal resources but also alleviate the pressure of energy supply, which is of great significance to energy security and environmental protection.

There are two main reasons for the poor floatability of low-rank coal:

(1) Coal surface oxidation. Coal surface oxidation is the fundamental cause of low-rank coal flotation difficulty. The oxygen-containing functional groups on the surface of low-rank coal combine with water to form hydrogen bonds and mask the hydrophobic part of the coal surface, which makes its poor floatability.

(2) Large porosity. The surface of the coal is rough, and there are a lot of pores and fissures, which makes the consumption of the collector to increase.

After direct oxidation of coal particles or coal oxygen adsorption, oxygen-containing functional groups such as −OH, −C=O, and −COOH that are easy to form hydrogen bonds with water molecules are formed on the coal surface. The coverage of the hydrophobic organic matter by the hydration layer reduces the floatability of coal slime. A comprehensive analysis of the internal molecular structure and surface oxygen-containing functional groups of low-rank coal and a targeted treatment method are effective ways to improve the floatability of low-rank coal.

Froth flotation that is based on the adherence of hydrophobic particles to air bubbles and the collection of particle-laden bubbles has been widely used to separate value commodities into concentrates from wastes in coal and mineral processing. Conventional hydrocarbon oil collectors have low activity, which makes it difficult to achieve low-rank coal flotation quality...
and improve slime flotation efficiency. However, the existence of oxygen-containing functional groups hinders the direct contact between the reagent and the hydrophobic part of coal particles, which not only reduces the flotation efficiency but also increases the reagent consumption. Therefore, it is necessary to select a new reagent for coal surface modification pretreatment before flotation.

The combination of sodium sulfite can separate a variety of metal minerals. The combination of sodium hypochlorite and sodium pyrosulfite can obtain good activation results in cyanide tailings. Fe(II) is a common reductant that has the characteristics of nontoxicity, low cost, strong applicability, and quite strong coagulation ability. The flotation recovery of coal-pyrite can be improved by ferrous ion under pH < 6.

In chemical reactions, these agents are defined as reductants that can reduce the oxygen-containing functional groups and the oxygen content in organic matter. Theoretically, this can solve the problem of oxygen-containing functional groups and improve flotation efficiency.

In this study, the reductant NaNO₃ was used for the pretreatment of coal samples, to explore the optimal pretreatment conditions such as the dosage of agents, pretreatment time, pH, etc., and mesoscopic analysis of the hydrophobic mechanism of NaNO₂ and zeta potential change. The method of chemical pretreatment is put forward to provide a new idea for slime flotation.

2. MATERIALS AND METHODS

2.1. Materials. The low-rank coal sample used in this study was obtained from the Majialiang Coal Preparation Plant in China. After being screened to −0.5 mm, the coal was mixed, reduced, and sealed for preservation.

The mineral composition of the Majialiang coal with a density of +1.6 g/cm³ was analyzed using a MiniFlex 600 X-ray diffractometer (XRD). The ash content of coal was analyzed using an ARL PERFORM X-ray fluorescence (XRF) spectrometer.

Using an ASAP 2460 3.01 automatic specific surface and porosity analyzer, nitrogen adsorption–desorption method was adopted. During the test, the coal sample was degassed first at 120 °C for 6 h and then measured at −195.85 °C in nitrogen. Also, BET and BJH methods were used to analyze the specific surface area and pore characteristics of coal.

2.2. Methods. 2.2.1. Flotation Experiments. The flow sheet for the flotation experiments is shown in Figure 1. An XFD III 1.5 L single-cell flotation machine (mixing speed, 1800 r/min; charging airflow rate, 0.25 m³/(m²-min)) and a magnetic stirrer were used for the flotation experiments, and the pulp concentration was 100 g/L.

In all the flotation experiments, 150 g of low-rank coal (~0.5 mm) was used. First, the coal sample was prewetted with 150 mL of tap water (the pH was 5, 7, 9, 11, or natural pH 7.6) for 1 min and then pretreated for a fixed time (0, 3, 5, 10, or 15 min). The mixing speed of all the experiments was 750 r/min. Finally, the pulp was transferred to the flotation cell for the flotation experiments.

The collector of the flotation used kerosene, and the dosage was 2000 g/t. The dosage of the foaming agent (octanol) should be kept at 200 g/t. After mixing, collector and sec-octanol were added at the intervals of 120 and 60 s, respectively. The flow speed was 1 L/min, and the foam flotation concentrate was collected, and the bubble blowing time was 3 min.

2.2.2. Flotation Index. The concentrate yield γc (%) reflects the degree recovery of the cleaned coal.

\[ \gamma_c = \frac{m_c}{m_i} \times 100\% \]  \hspace{1cm} (1)

where \( m_c \) is the quality of the concentrate product (%) and \( m_i \) is the quality of the raw coal (%).

The combustible recovery ε (%) reflects the degree recovery of the concentrate products relative to the raw coal.

\[ \epsilon = \frac{\gamma_c (100 - A_i)}{100 - A_c} \times 100\% \]  \hspace{1cm} (2)

where \( A_i \) is the ash content of the concentrate product (%) and \( A_c \) is the ash content of the raw coal (%).

2.2.3. X-ray Photoelectron Spectroscopy. XPS analysis of the coal sample was tested by a Thermo Scientific K-Alpha X-ray photoelectron spectrometer to analyze the surface oxygen-containing functional groups.

2.2.4. Contact Angle Measurements. To determine the wettability of the coal, the contact angle of the coal sample was measured using a DSA100 contact angle measuring instrument. In this measurement, the coal sample (~0.074 mm) was pressed into the sheets under 10 MPa, and then the DI water (laboratory homemade) was dropped onto the coal surface to observe the contact angle that was averaged after multiple measurements.

2.2.5. Zeta Potential Measurements. The zeta potential of the coal sample (~0.074 mm) was measured by a JSM94 microelectrophoresis apparatus. The DI water was used to prepare a suspension of 0.1% mass fraction, and the coal was oscillated with an oscillator for 15 min. The zeta potential of the coal sample in NaNO₃ solution at different concentration gradients was measured after the coal was measured six times.

3. RESULTS AND DISCUSSION

3.1. Coal Characterization. Table 1 presents the particle size analysis of the coal sample as a function of particle size. The

| dominant size/mm | weight/% | ash/% |
|------------------|----------|------|
| 0.5−0.25         | 30.07    | 30.60  |
| 0.25−0.125       | 19.38    | 29.93  |
| 0.125−0.074      | 11.80    | 29.05  |
| 0.074−0.045      | 10.15    | 26.89  |
| 0.045−0            | 28.60    | 27.66  |
| Total             | 100.00   | 29.07  |

Figure 1. Flow sheet for flotation experiments.
The dominant size is 0.5–0.25 mm, −0.045 mm, and the yield is nearly 60%. The ash content of the raw coal is 29.07%.

The results of proximate analysis and ultimate analysis of −1.6 g/cm³ coal sample are shown in Table 2. The coal sample had a higher moisture and volatile content, lower fixed carbon, and oxygen content as high as 13.12%. The contents of hydrogen and nitrogen in the Majialiang coal were higher, indicating that the aromatic degree of the skeleton structure was low and a low degree of coalification.

3.2. Surface Oxygen-Containing Functional Groups. Figure 2 shows the C1s peak fitting diagram of XPS for the coal sample, and Table 3 presents the content and form of the functional groups.

According to the results of XPS analysis, the surface of low-rank coal contains many oxygen-containing functional groups, and the contents of the oxygen groups C−O, C=O, and O=C−O are 10.86, 2.76, and 2.88%, respectively.

Due to its strong polarity and hydration, oxygen-containing functional groups are easy to form hydrogen bond with water molecules and form a highly hydrophilic hydration layer on the coal surface, reducing the contact probability between the collector and the coal surface.

3.3. Physical Property Analysis of Coal. Table 4 presents the ash composition of Majialiang low-rank coal. The main elements were Si, Al, Ca, and Fe, accounting for as much as 94.59%.

Figure 3 shows the XRD spectrum of +1.6 g/cm³ coal. Also, noncarbon mineral elements mainly exist in the form of quartz, kaolinite, calcite, pyrite, and muscovite, which can form hydrogen bond adsorption with water and form hydration film composed of multilayer water molecules, affecting the flotation efficiency.\(^{11,12}\)

Figure 4a shows the BET nitrogen adsorption−desorption curve for the coal sample. The adsorption isotherm has a type H4 hysteresis ring, which belongs to type IV adsorption isotherm, indicating that there are narrow fissure holes on the surface of the coal.

Figure 4b is the differential diagram of the BJH pore size distribution, reflecting the characteristics of pore size distribution, and Table 5 presents the pore structure parameters of the coal sample. There are many pores in the coal sample, which micropores volume is small. Also, the average pore diameter is about 8.80 nm.

Figure 5 shows the SEM image of the coal, reflecting the surface morphology of the coal. The gray part of the image is the coal with a certain electrical conductivity, and the white part of the image is the nonconductive minerals. Figure 5 shows the mineral distribution on the surface of the coal, and many fine minerals are attached to the surface of the coal and the folds on the surface, rich, and rough.

Due to the existence of pores and large specific surface area on the coal surface, small collector molecules or anions were formed in adsorption for reducing the efficiency of the collector. These microchemical behaviors on the hydrophilic surface may be important factors that affect the flotation of refractory minerals.\(^{13}\) A large number of lump and fragment minerals are attached to the coal surface, which cover the hydrophobic part of the surface of the coal. Also, minerals with strong hydrophilicity, such as quartz, enhance the hydrophilicity of the coal and further reduce the flotation efficiency of the coal.

3.4. Pretreatment Test with NaNO₂. 3.4.1. Effect of Dosage. Under the 3 min pretreatment time process with tap water pH, the influence of NaNO₂ dosage on the flotation recovery was investigated, and the results are shown in Figure 6.

With the increase of the NaNO₂ reagent dosage, the flotation indexes such as concentrate yield, tailings ash, and combustible.

Table 2. Proximate and Ultimate Analyses of the Coal Sample

| Mₐd | Aₐd | Vₐdaf | FCₐ | Cₐd | Hₐd | Nₐd | Oₐd | Sₐd |
|-----|-----|-------|-----|-----|-----|-----|-----|-----|
| 3.82 | 10.80 | 33.24 | 59.54 | 70.17 | 4.13 | 1.32 | 13.12 | 0.46 |

Figure 2. C1s peak fitting diagram of XPS for the coal sample.

Table 3. Content Analysis of Functional Groups in the Coal Sample

| binding energy (eV) | group type | relative content (%) |
|---------------------|------------|---------------------|
| 284.8 | C−C/C−H | 83.50 |
| 286.1 | C−O−C/C−OH | 10.86 |
| 287.5 | C=O | 2.76 |
| 289.0 | O=C−O | 2.88 |

Table 4. Chemical Analysis of the Coal Sample

| element | SiO₂ | Al₂O₃ | CaO | Fe₂O₃ | TiO₂ | SO₃ | MgO | K₂O | Na₂O | P₂O₅ | Total |
|---------|------|-------|-----|-------|------|-----|-----|-----|------|------|-------|
| result  | 44.52 | 36.20 | 9.45 | 4.42  | 2.25 | 0.82 | 0.75 | 0.47 | 0.24  | 0.23  | 99.35 |

Figure 3. Mineralogical analysis of the coal sample.
recovery showed an increase—decrease trend. When the dosage of NaNO$_2$ is 2000 g/t, the flotation indexes reach the peak value, the concentrate ash, tailings ash, and combustible recovery were obtained as 17.09, 37.03, and 42.00%, respectively. Also, the combustible recovery increased by 11.94%. When the dosage of NaNO$_2$ exceeds 2000 g/t, the ash content of the concentrate decreases and the recovery rate of the combustible material decreases as well.

3.4.2. Effect of Pretreatment Time. Under the condition of a NaNO$_2$ reagent dosage of 2000 g/t, the influence of the pretreatment time on the flotation recovery was explored, and the results are shown in Figure 7.

With the increase of the pretreatment time, the concentrate yield and the combustible recovery showed an increasing and then declining trend. When the pretreatment time was 5 min, the ash contents of the concentrate and tailing were 17.15 and 37.12%, respectively. Also, the combustible recovery reached an extreme value of 42.23%. Compared with no pretreatment, the ash content of the tailings increased by 2.38% and the combustible recovery increased by 1.06%.

When the reaction time was more than 5 min, the concentrate ash decreased with the increase of time, and the combustible recovery descended too. It shows that the pretreatment time has a positive effect on the flotation, and the appropriate enough pretreatment time can make the efficiency of flotation to improve.

3.4.3. Effect of Pretreatment pH. Under the conditions of a NaNO$_2$ reagent dosage of 2000 g/t and a pretreatment time of 5 min, the influence of pH on the flotation recovery was explored. The results are shown in Figure 8.

As seen from Figure 8, the change of soaking pH affected the flotation index, but the effect was not significant, and pH = 7.6 is the result of the flotation test under tap water conditions. With the increase of pH, the combustible recovery and tailings ash showed a falling—rising trend.

At pH = 7, the lower ion concentration in the solution reduced the effect of NaNO$_2$ on the surface electrical properties of the Majialiang coal. When the solution environment is acidic or alkaline, due to the ion concentration increase of OH$^-$ or H$^+$, the effect of ions is enhanced and the combustible recovery increases. The pH of tap water is about 7.6, and the combustible recovery is 41.20%. The results indicated that the pH value of the solution showed no significant effect on the pretreatment effect of NaNO$_2$, and the pH cannot be adjusted, considering the limitation of the test cost and operation complexity.
Among the above three conditions, the dosage of NaNO\textsubscript{2} has the greatest influence on the test results, while the pretreatment time should be appropriate, and the pH had little influence. A NaNO\textsubscript{2} dosage of 2000 g/t and a pretreatment time of 5 min, using tap water for pretreatment, can obtain the best flotation test effect.

4. MECHANISM ANALYSIS

4.1. Hydrophobic Analysis. To explore the coal surface hydrophobicity with changes in the amount of NaNO\textsubscript{2}, the contact angle and XPS analyses were performed. The greater the contact angle, the better the hydrophobicity of the coal, which means that the content of hydrophobic functional groups increases laterally. The change trend of the contact angle with NaNO\textsubscript{2} dosage is shown in Figure 9.

The variation trend of the contact angle is similar to that of the combustible recovery. On the surface of Majialiang coal, NaNO\textsubscript{2} can be capable of chemical reactions that reduce oxygen-containing groups to hydrophobic groups, making the coal more hydrophobic and increasing the contact probability between the collector and the coal.
XPS was used to characterize the coal samples treated at different dosages of NaNO₂. After peak processing, the proportion of surface groups was calculated. Figure 10 shows the ratio of surface hydrophobic functional group content to surface functional group content.

As shown in Figure 10, the content of hydrophobic functional groups first increased and then decreased with the increase of reagent dosage. It is consistent with the trend of combustible recovery.

The increase of the hydrophobic functional group content can effectively enhance the hydrophobic interaction on the surface of the coal, which is conducive to the contact between the oil collector and the coal surface, improve the collection efficiency, and then ameliorate the flotation effect. The proportion of oxygen-containing functional groups on the surface decreases, indicating that the addition of NaNO₂ leads to the redox reaction with oxygen-containing functional groups on the coal surface, as shown in Figure 11.

\[
(C_nH_m)_p CH_2COOH \xrightleftharpoons{NaNO_2} (C_nH_m)_p CH_2CHO \\
NaNO_2 \xrightleftharpoons{C_nH_m}_p CH_2CH_2OH \xrightarrow{NaNO_2} (C_nH_m)_p CH_2CH_3 \\
(C_nH_m)_p CH_2COO(CH_2) \xrightarrow{NaNO_2} (C_nH_m)_p CH_2(C = O) \\
(CH_2) \xrightarrow{NaNO_2} (C_nH_m)_p CH_2CH_2(CH_2) \\
\]

Due to the different polarities of the oxygen-containing group, its hydrophilicity is also different. The more electronegative the oxygen-containing functional groups are, the more polar and hydrophilic they are. Through the electronegativity of the oxygen-containing group, the hydrophilicity of the oxygen-containing group can be obtained. The hydrophilic magnitude of the oxygen-containing group is as follows: \( \text{−COOH} > \text{−OH} > \text{−C−O−C} > \text{−C=C} \).

Figure 12 shows the ratio of oxygen-containing functional groups. When the dosage of NaNO₂ reaches 2000 g/t, \( \text{−COOH} \) is transformed into \( \text{−C=C} \), which is relatively not hydrophilic, so that the hydrophilicity decreases. Also, the adsorption probability of the collector is improved, making it easier for the collector to collect the coal samples.

### 4.2. Zeta Potential Measurements

The zeta potential provides a measure of the surface charge characteristics of the coal particle solution across the diffuse part of the electrical double layer at the surface. The change of the zeta potential of the coal sample is shown in Figure 13. The introduction of ions into the pulp can directly affect the surface potential of coal particles and bubbles.

Zeta potential refers to the potential difference between the continuous phase and the fluid stable layer (sliding layer) on the surface of the attached coal particles and reflects the changes of electrical properties on the surface of coal particles. With the addition of NaNO₂, the zeta potential of the coal sample first decreased and then increased, and the electronegativity reached the maximum at 2000 g/t. The trend is consistent with the combustible recovery.

\( \text{NO}_2^- \) is formed after NaNO₂ is dissolved in water, enhancing the electronegativity of the coal pulp. According to the Derjaguin–Landau–Verwey–Overbeek theory, some negative ions, such as NO₂⁻, are adsorbed on a particle surface, making the zeta potential of coal particles to decrease. The greater the change in zeta potential, the stronger the adsorption. Evidently, NO₂⁻ can be adsorbed on the coal surface, causing the zeta potential of the coal sample to decrease.

\[
(C_nH_m)_p CH_2COOH \xrightarrow{NaNO_2} (C_nH_m)_p CH_2CHO \iff (C_nH_m)_p CH_2CH_3 \\
(C_nH_m)_p CH_2COO(CH_2) \iff (C_nH_m)_p CH_2(C = O) \\
(C_nH_m)_p CH_2CH_2(CH_2) \iff (C_nH_m)_p CH_2CH_3 \\
\]

Figure 11. Schematic diagram of the functional group reaction.

Figure 10. Effect of NaNO₂ dosage on the hydrophobic functional group content.

Figure 11. Schematic diagram of the functional group reaction.

Figure 12. The ratio of oxygen-containing functional groups.

Figure 13. Effect of reagent dosage on the zeta potential.
The interfacial tension and the energy barrier of the surface between kerosene and mineral particles are reduced and needed to overcome when interacting, and it is easier to adhere to the particle surface.

5. CONCLUSIONS

(1) The experimental studies prove that the method of reductant treating oxygen-containing functional groups is feasible in the field of coal flotation. Under the conditions of a NaNO₂ reagent dosage of 2000 g/t and a pretreatment time of 5 min, the flotation experiments were carried out after the pretreatment test. The ash content of tailings was 37.12%, increased by 1.5%, and the combustible recovery was 42.23%, increased by 8%. The flotation effect was significantly improved.

(2) The surface hydrophobicity of the Majialiang coal treated with NaNO₂ solution was improved. The increase of hydrophobic functional groups can effectively enhance the hydrophobic interaction on the surface of the coal and improve the flotation efficiency.

(3) The anions with a small radius in NaNO₂ solution can easily reach the surface of coal particles, act on the electrical layer of the coal surface, change the electrical properties of coal particles, and improve the flotation efficiency.

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