Effect of Hydrothermal Dehydration on the Slurry Ability of Lignite
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ABSTRACT: The high moisture content limits the large-scale utilization of lignite. Hydrothermal dehydration (HTD) has been confirmed as an effective method to improve the quality of lignite for further utilization. In this study, the effects of the changes in the lignite interface properties caused by the HTD modified final temperature on the slurry ability were investigated in the range of 160–200 °C. The results indicated that with the gradual rise of the HTD modified final temperature, the content of the carboxyl groups and phenolic hydroxyl groups on the surface of lignite decreased by 21.95 and 36.34%, respectively. In the meantime, the atomic ratio of oxygen/carbon, the content of equilibrium moisture, and the thickness of the hydrated film were reduced from 0.293, 14.63%, and 34.26 nm to 0.252, 9.43%, and 13.33 nm, respectively. Therefore, these changes of interfacial properties improved the slurry ability of lignite, with higher fixed-viscosity solid concentration, lower yield stress, increased pseudoplasticity, and gradually decreased static stability of the prepared lignite coal water slurry. Hydrothermal dehydration; slurry ability; oxygen-containing functional groups; hydrated film

1. INTRODUCTION
The proven reserves of lignite, about 130 billion tons, account for about 13% of the total coal in China.1−2 The clean and efficient utilization of lignite has attracted significant attention to meet the energy demand and achieve targeted emission reduction. However, due to the short formation time and low-grade metamorphism, lignite possesses high moisture, rich oxygen-containing functional groups, and well-developed pore structures,3 which limits the extensive application of lignite.4 The preparation of lignite into coal water slurry (CWS) for chemical conversion is one of the most widely used strategies of lignite. In order to reduce the inherent moisture content of lignite, improve the interfacial properties, and enhance the concentration of lignite CWS, lignite dehydration is necessary before the preparation of CWS.

Hydrothermal dehydration (HTD) is a widely used high-efficiency non-evaporative dehydration technology. Zhao et al.5 reported that hydrothermal dehydration could reduce the oxygen-containing functional groups and enhance the hydrophobicity of low-grade coal, thereby improving its slurry ability. Yu et al.6,7 used the wastewater generated from the lignite HTD process to prepare coal water slurry (or coal sludge slurry), which improved the slurry ability and enabled the energy recovery of the flammable substances in wastewater. Racovalis et al.8−12 studied the influence of temperature and residence time on the porosity of Australian lignite during HTD and found that the concentration of upgraded lignite CWS reached 64%. Allardice et al.13 investigated the changes in the form of moisture before and after the treatment of Victoria’s lignite by HTD. Mursito et al.14,15 reported that the coal rank of peat with an initial moisture content of 90% was enhanced by increasing the reaction temperature during HTD to 150−380 °C, and the fixed carbon content and calorific value were significantly increased. Therefore, HTD has been confirmed as an effective method to improve the quality of lignite for further utilization.

The research effort on HTD is mainly focused on the high-pressure condition at about 300 °C, which has the characteristics of high energy consumption and difficult waste heat utilization. On the other hand, the changes in the interfacial properties and slurry ability of the upgraded lignite at the low-temperature (below 200 °C) and low-pressure conditions have not been extensively studied. Therefore, in this study, the changes in the equilibrium moisture, oxygen-containing functional groups, and the thickness of the hydrated film of lignite before and after HTD at low-reaction-temperature conditions (160−200 °C) were studied. The influence of their changes on the fixed-viscosity solid concentration, rheology, and static stability of lignite CWS was investigated. Besides, a systematic quantitative method to evaluate the static stability of lignite CWS, combining the traditional standing observation method with an advanced Turbiscan LAB stability analyzer test, was also proposed.

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2. EXPERIMENTAL SECTION

2.1. Materials. The coal sample used in this study was Inner Monglia lignite. The lignite raw coal was crushed to <3 mm by using a jaw crusher. The crushed coal was subsequently triturated into two particle size distribution groups with the average particle sizes (volumetric) of 57.83 and 18.11 μm, ensuring that the particle was smaller than 300 μm.

2.2. HTD Experiments. HTD was conducted in a closed low-pressure reactor with the maximum working pressure, operating temperature, and stirring speed of 4 MPa, 350 °C, and 1000 rpm, respectively. The optimal mass ratio of the coarse and fine lignite samples was 8:2 based on the calculation of the packing efficiency evaluation index E developed by Tu.16 The evenly mixed lignite samples with the same amount of deionized water were placed in the reactor under a nitrogen atmosphere. The samples were heated at the final temperatures of 160, 170, 180, 190, and 200 °C at 500 rpm with a 60 min residence time, denoted as T1, T2, T3, T4, and T5, respectively. After the reaction, the reactor was rapidly cooled to room temperature using circulating cooling water, and the solid products were separated by vacuum filtration for further analysis and preparation of lignite CWS. For comparison, the coarse and fine lignite samples were also physically mixed in an 8:2 ratio for analysis, and the sample was denoted as T0.

2.3. Equilibrium Moisture Measurements. The equilibrium moisture is used to characterize the water holding capacity of lignite. In this study, it was expressed as the water content of the coal sample that maintained a constant weight at 25 °C and about 65% relative humidity.17

2.4. Measurement of Oxygen-Containing Functional Groups. The carboxyl and phenolic hydroxyl groups in lignite are the major active oxygen-containing functional groups. Chemical titrations18 were carried out to quantify their content.

2.5. Hydrated Film Thickness Measurement. In recent years, atomic force microscopy (AFM) has been used to study the surface properties and mineral interactions in solutions.19–21 In this study, a Bruker Dimension FastScan atomic force microscope was used in the contact mode to measure the force changes of the probe near the sample surface in the same KY33 solution and obtain the force–distance curve. Subsequently, the thickness of the hydrated film could be calculated from the method developed by XING.22 A silicon nitride cantilever with an elastic modulus of 0.12 N/m was used as the cantilever probe. In order to ensure the accuracy of the measurements, 9–12 test points were identified for each sample to measure the surface interaction forces, and each test point was subsequently measured for 150–200 test cycles. The average of the obtained results was reported as the final hydrated film thickness of the sample.

2.6. Preparation of Lignite CWS. The dry preparation technology was used to generate lignite CWS. The accurately weighed pre-treated lignite coal samples were mixed. Subsequently, deionized water and the dispersant were added. The mixture was operated at low-speed pre-kneading conditions by using a high-torque stirrer to make the coal fully wetted. Afterward, the mixture was stirred for 10 min at high speed (1000 rpm) to obtain the lignite CWS samples. The dosage of dispersant KY33 was 1.1 wt % based on dry lignite, which was prepared by the China University of Mining and Technology (Beijing). The solid concentration and viscosity of lignite CWS were measured by a automatic moisture meter (Sartorius MA35) and a rotational viscometer (NXS-4C), respectively. The solid concentration of the slurry with a shear rate of 100 s⁻¹ and an apparent viscosity of 1000 mPa·s was defined as the fixed-viscosity solid concentration of lignite CWS.23 The lignite CWS samples were divided into two groups, and the static stability was evaluated by observation and a Turbiscan LAB stability analyzer within 7 days.

2.7. Principle of Turbiscan LAB Stability Analysis. The stability of the sample is measured in a non-contact manner using the Turbiscan LAB Stability Analyzer (with a transmission light monitor and backward scattering optical detector) to determine the changes in the average particle size during settlement.24,25 As the light passes through the high-concentration opaque CWS sample, the spotlight of the backscattering light can be formed, and the light intensity of the middle bright part is analyzed by the Turbiscan LAB stability analyzer. The backscattered flux (BS) and dynamic
Turbiscan stability index (TSI) for evaluating the stability of lignite CWS can be determined by using eqs 1 and 3, respectively. The variation in the BS can reflect the migration of particles in the lignite CWS sample with time at a constant solid concentration of the CWS sample. The smaller the change in the BS value is, the more stable the lignite CWS.

Table 1. Characteristic Analysis of Lignite before and after HTD at Different Temperatures

| sample | proximate analysis (%) | Q_b,d (MJ/kg) | ultimate analysis (%) |
|--------|------------------------|----------------|-----------------------|
|        | M_eq  | A_d    | V_daf | FC_daf |        | C_daf | H_daf | N_daf | S_daf | O_daf | A_O/C |
| T0     | 14.63 | 21.74  | 45.58 | 54.42  | 19.18  | 66.15 | 4.30  | 1.21  | 2.46  | 25.88 | 0.293 |
| T1     | 9.88  | 20.73  | 44.70 | 55.30  | 20.32  | 68.10 | 3.85  | 1.11  | 1.40  | 25.54 | 0.281 |
| T2     | 9.75  | 20.64  | 44.57 | 55.43  | 20.58  | 68.85 | 3.66  | 1.12  | 1.44  | 24.92 | 0.271 |
| T3     | 9.70  | 20.60  | 43.51 | 56.49  | 20.62  | 68.99 | 3.58  | 1.10  | 1.48  | 24.85 | 0.270 |
| T4     | 9.55  | 20.50  | 43.48 | 56.52  | 20.64  | 69.17 | 3.65  | 1.12  | 1.42  | 24.64 | 0.267 |
| T5     | 9.43  | 20.56  | 43.36 | 56.64  | 20.76  | 70.20 | 3.62  | 1.12  | 1.46  | 23.60 | 0.252 |

Note: M, A, V, and FC refer to moisture, ash, volatile, and fixed carbon contents, respectively. Q_b refers to the bomb calorific value, and “eq” represents the equilibrium moisture. “d” and “daf” represent the dried basis and dried ash-free basis, respectively.

Figure 2. Relationship between A_O/C and the fixed-viscosity solid concentration.

Figure 3. Effect of the HTD modified final temperature on the oxygen-containing functional groups.

Turbiscan stability index (TSI) for evaluating the stability of lignite CWS can be determined by using eqs 1 and 3, respectively. The variation in the BS can reflect the migration of particles in the lignite CWS sample with time at a constant solid concentration of the CWS sample. The smaller the change in the BS value is, the more stable the lignite CWS.
sample is. The BS is inversely proportional to the square root of $l^*$, as shown in eq 1

$$\text{BS} = \frac{1}{\sqrt{l^*}}$$  \hspace{1cm} (1)

where $l^*$ represents the free path of the photon, which is the penetration distance of the photon in the dispersion. $l^*$ is defined in eq 2 according to Mie’s theory and is proportional to the average particle size $d$ of the particles, whereas it is inversely proportional to the volume concentration $\Phi$.

$$l^* = \frac{2d}{3\phi (1 - g)Q_s}$$ \hspace{1cm} (2)

where $g$ and $Q_s$ represent the parameters of the Mie theory.

The TSI is independent of the quantity of the sample in the measuring tube and calculated by eq 3 as

$$\text{TSI} = \sum_i \frac{\sum |\text{scan}_i(h) - \text{scan}_{i-1}(h)|}{H}$$ \hspace{1cm} (3)

where $h$ and $H$ represent the height of the scanning point and the total height of the lignite CWS sample, respectively. The $\text{scan}_i(h)$ represents the light intensity of the scanning point at a certain scanning duration, and $\text{scan}_{i-1}(h)$ is the light intensity of the scanning point at the last scanned interval.

### 3. RESULTS AND DISCUSSION

#### 3.1. Fixed-Viscosity Solid Concentration of Lignite CWS

The fixed-viscosity solid concentration of lignite CWS was measured at a shear rate of 100 s$^{-1}$ and an apparent viscosity of 1000 mPa·s.$^{23}$ The effect of the HTD modified final temperature on the fixed-viscosity solid concentration of lignite CWS is shown in Figure 1. As can be observed, the solid concentration enhanced from 50.71% (T0) to 53.27% (T5) with the increase in the temperature.

The interfacial characteristics of lignite samples were tested to ascertain the relationship with the solid concentration of lignite CWS under a fixed viscosity. Table 1 presents the analysis of the characteristics of lignite before and after HTD at different modified final temperatures. As the modified final temperature increased, the equilibrium moisture and volatile contents decreased, while the fixed carbon content and calorific value were observed to increase. As shown in the ultimate analysis, the carbon content element was increased, while the oxygen content decreased. These changes imply that HTD can enhance volatile decomposition and carbonization, thereby...

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**Table 2. Thickness of the Hydrated Film as a Function of the Modified Final Temperature**

| Sample | HTD modified final temperature/°C | Cantilever movement distance/nm | Cantilever deflection/nm | Hydrated film thickness/nm |
|--------|----------------------------------|--------------------------------|--------------------------|---------------------------|
| T1     | 160                              | 37.44                          | 12.48                    | 24.95                     |
| T2     | 170                              | 29.32                          | 6.60                     | 22.72                     |
| T3     | 180                              | 31.19                          | 9.08                     | 22.10                     |
| T4     | 190                              | 24.41                          | 6.50                     | 17.91                     |
| T5     | 200                              | 22.13                          | 8.80                     | 13.33                     |

**Figure 4.** Relationship between the total acidity and fixed-viscosity solid concentration.

**Figure 5.** Effect of the HTD modified final temperature on the hydrated film thickness.
reducing the reaction activity of lignite. Thus, the spontaneous combustion of lignite is inhibited, and the energy density is enhanced.\textsuperscript{26} The decrease of the equilibrium moisture content indicated that the water-holding capacity of the lignite after HTD was weakened, which is conducive to improving the fixed-viscosity solid concentration for more free water acting as the flow medium between lignite particles. The fixed-viscosity solid concentration of lignite CWS was enhanced by gradually decreasing the atomic ratio of oxygen to carbon (A\textsubscript{O}/C) (shown in Figure 2), which indicated that HTD promoted the preparation of high-concentration lignite CWS.

The variation in the acidic oxygen-containing functional groups after HTD is shown in Figure 3, which can also reflect the variant of the coal rank. The total acidity, as well as the content of the carboxyl and phenolic hydroxyl groups, decreased gradually with the increase in the HTD modified final temperature. Compared with raw coal, the modified final temperature reached 200 °C, and the carboxyl group content decreased from 1.64 to 1.28 mol/kg, a decrease of 21.95%. On the other hand, the phenolic hydroxyl group content decreased from 3.33 to 2.12 mol/kg, resulting in a decrease of about 36.34%. The removal of the carboxyl and phenolic hydroxyl groups indicated that the HTD temperature had a considerable effect on their decomposition, which implied an improvement in the degree of lignite modification and up-gradation of the coal rank.\textsuperscript{27} In order to study the influence of acidic oxygen-containing functional groups on the slurry ability of lignite, the relationship between the total acidity and fixed-viscosity solid concentration was investigated, as shown in Figure 4. It can be seen that the total acidity exhibited a significantly negative correlation with the fixed-viscosity solid concentration of upgraded lignite. Therefore, the removal of the hydrophilic acid oxygen-containing functional groups improved the interfacial hydrophobicity. As a result, a large extent of free...
water was produced as the flow medium between the lignite particles, improving the fixed-viscosity solid concentration.

The impact of HTD on the interfacial properties of lignite varied the thickness of the hydrated film, affecting the fixed-viscosity solid concentration of the upgraded lignite CWS. Therefore, the force–distance curves were obtained by measuring the variation in the interaction force as the AFM probe approached the surface of the sample in the dispersant solution, which allowed the determination of the thickness of the hydrated film, as shown in Table 2 and Figure 5. The hydrated film thickness of upgraded lignite was gradually decreased with the increase in the modified final temperature of HTD. When the modified final temperature reached 200 °C, the thickness of the hydrated film was determined to be 13.33 nm, while that of raw coal was 34.26 nm. The hydrophilic oxygen-containing functional groups (such as carboxyl and phenolic hydroxyl groups) on the surface of lignite were gradually stripped off, resulting in a strong-surface-hydrophobicity, weak-wettability, and thin hydrated film. The relationship between the thickness of the hydrated film and the fixed-viscosity solid concentration is shown in Figure 6. The fixed-viscosity solid concentration was noted to be negatively correlated with the hydrated film thickness. At a constant solid concentration, the thinner hydrated film requires less water, and the proportion of free water acting as a flow medium increases during the shearing process. Therefore, the frictional resistance between the coal particles decreases, which subsequently decreases the viscosity of lignite CWS and increases the fixed-viscosity solid concentration.

3.2. Rheological Characteristics of Lignite CWS. The rheological characteristics are an important performance index of CWS. As shown in Figure 7, the rheological curves with similar apparent viscosity values were selected to analyze the influence of the modified final temperature during HTD on the rheological characteristics of lignite CWS. It can be seen that lignite CWS exhibits a pseudoplastic fluid behavior with the characteristic “shear thinning” before and after HTD. In other words, the apparent viscosity was noted to decrease gradually on increasing the shear rate. Moreover, the apparent viscosity decreased significantly on enhancing the modified final temperature, indicating that HTD can enhance the pseudoplasticity of lignite CWS. To further understand the effects of temperature on the rheological characteristics of lignite CWS, the rheological curves were fitted by using the Herschel–Bulkley model shown in eq 4, and the fitting results are shown in Figure 8 and Table 3.

\[ \tau = \tau_y + K \gamma^n \]  

(4)

where \( \tau \) and \( \tau_y \) represent shear and yield stresses, respectively, \( K \) is the consistency coefficient, \( \gamma \) denotes the shear rate, and \( n \) represents the fluidity index.

The fitting degree (\( R^2 \)) of the rheological characteristics curve is observed to be close to 1, indicating that lignite CWS conforms to the Herschel–Bulkley model. The “\( n \)” value was less than 1 and gradually decreased on increasing the modified final temperature during HTD, which further confirmed the gradually increasing pseudo-plasticity of lignite CWS. The gradual reduction in the yield stress \( \tau_y \) is related to the removal of the surface oxygen-containing groups (such as carboxyl and phenolic hydroxyl groups) and the hydrated film thickness. The coal particles cannot form a spatial three-dimensional structure or be relatively weakened by the dipole hydrogen bonding of the polar groups. This weakens the static network structure of the slurry, minimizing the shear stress required to flow, thereby decreasing the yield stress. The internal moisture, oxygen contents, and the thickness of the hydrated film

Figure 8. Fitted rheological curves of lignite CWS as a function of the modified final temperature during HTD.

| HTD modified final temperature/°C | \( \tau_y/\text{Pa} \) | \( K \) | \( n \) | \( R^2 \) |
|----------------------------------|------------------|-------|-------|--------|
| raw coal                         | 18.52            | 2.16  | 0.8166| 0.99933|
| 160                              | 17.93            | 3.73  | 0.7118| 0.99973|
| 170                              | 16.85            | 4.71  | 0.6713| 0.99977|
| 180                              | 13.37            | 4.31  | 0.7081| 0.99940|
| 190                              | 12.01            | 4.57  | 0.6820| 0.99949|
| 200                              | 11.83            | 5.03  | 0.6663| 0.99993|

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reduced after HTD, resulting in a corresponding increase in the solid concentration of lignite CWS. The void ratio between the coal particles decreased, and the particles easily approached each other. It enabled the particles to overcome greater friction and interaction forces among themselves during the flow process, thus increasing the flow resistance, viscosity, and $K$ values. The enhanced solid concentration reduced the extent of free water in CWS, and the coal particles connected easily with each other to form a “coal–water” state (When coal particles agglomerate, part of free water is bound in coal particle aggregates, forming a composite coal–water structure). As the shear rate increased, this state was destroyed, and the free water flowed out. Therefore, the slurry viscosity was further reduced, indicating the reinforcement of pseudo-plasticity.

3.3. Static Stability of Lignite CWS. Stability is an important indicator for evaluating the quality of CWS. In addition, it plays a key role in the storage and long-distance transportation of CWS. The lignite CWS samples with similar apparent viscosity values were divided into two groups for stability analysis. The static observation method revealed a thin layer of soft sediment at the bottom of CWS within 7 days. No hard sedimentation was observed, with almost no water formed at the top. This indicated that lignite CWS was stable before and after HTD, but it was difficult to gain further

Figure 9. Backscattering spectra of lignite CWS with different HTD modified final temperatures.
be observed, the TSI value of the T0 CWS sample was the smallest with optimal static stability. The TSI value increased gradually with the modified final temperature, indicated that the stability of CWS decreased. The results show that the modified final temperature harmed the static stability of lignite CWS. This is mainly attributed to the destruction of the structural features of the three-dimensional network owing to the removal of the oxygen-containing functional groups on the lignite surface. Moreover, the thickness of the hydrated film was reduced, which deteriorated the mechanical and elastic resistances, thereby reducing the ability to prevent the particles from approaching each other. Therefore, the steric hindrance effect is weakened, and the static stability is relatively reduced. Besides, the TSI value of the TS CWS sample was observed to be the largest, by only 0.148, which indicated that the static stability of lignite CWS is better.

4. CONCLUSIONS

In this study, the effects of the change of lignite interface properties caused by the low-temperature HTD modified final temperature on the slurry ability were systematically analyzed. The following conclusions could be drawn:

(1) With the increase in the HTD modified final temperature, the contents of the carboxyl groups and phenolic hydroxyl groups on the surface of lignite decreased by 21.95 and 36.34%, respectively. The atomic ratio of oxygen/carbon, the content of equilibrium moisture, and the thickness of the hydrated film were reduced from 0.293, 14.63%, and 34.26 nm to 0.252, 9.43%, and 13.33 nm, respectively. These changes contributed to the increase in the fixed-viscosity solid concentration of lignite CWS.

(2) Lignite CWS is a pseudoplastic fluid and conforms to the Herschel–Bulkley model. With the increase of the HTD modified final temperature, the removal of carboxyl and phenolic hydroxyl groups weakened the static network structure, and thus, the yield stress is reduced. The increase in the solid concentration made the coal particles easily connect to form a “coal–water” state, and the pseudo-plasticity was gradually enhanced.

(3) The thickness of the hydrated film became thinner after HTD with less mechanical resistance and elastic resistance to prevent the particles from approaching each other; thus, the static hindrance effect was weakened, and the static stability of upgraded lignite CWS was relatively reduced.

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Notes
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