



Static stability and rheological behavior of lignite char–water mixture

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ABSTRACT

In this study, lignite char–water mixture (LCWM) was prepared with char from lignite pyrolysis at different temperatures. Sodium lignosulfonate (SL), naphthalenesulfonate formaldehyde (NSF) or the mixture of SL and NSF was used as dispersant in the preparation of LCWM. The effect of dispersant on fluidity and stability of LCWM was investigated at the fixed char loading of 60 wt.%. The results showed that the mixture of SL and NSF as dispersant can effectively decrease the apparent viscosity to less than 400 mPa s and enhance the fluidity of LCWM. The optimum dispersant content for four lignite char samples was examined and the adsorption behaviors of SL, NSF and their mixture on the char surface were also discussed. Meanwhile, the static stability of LCWM was investigated with sodium salt of carboxymethyl cellulose as stabilizer.

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1. Introduction

Coal–water mixture (CWM), including 60–70% coal, 29–39% water and about 1% additives, as one of coal utilization methods, has been extended to many fields, from traditional coal gasification to combustion instead of crude oil for its low cost and less pollution to environment. The feedstocks for preparing slurry have been enlarged from traditional bituminous coal to petroleum coke [1] and lignite [2]. However, the high moisture content and large amount of hydrophilic groups in lignite make it difficult to prepare desirable CWM. To enhance the slurryability of CWM from lignite, some treatment methods such as thermal or chemical modification have been proposed to decrease the amount of hydrophilic groups and moisture content of lignite [3,4].

Lignite pyrolysis, as a thermal treatment method to upgrade lignite, has been widely investigated to enhance the value of lignite and to extend the field of lignite application. Through low temperature pyrolysis, lignite can be converted into liquid, gas and solid products. The tar in liquid product can be further processed for liquid fuel or chemicals; the gas can be used as town gas, heating gas or synthesis gas, etc., and the solid product, lignite char, could be used for combustion in power station, gasification, and other uses.

Lignite char shows different properties from its original lignite, such as low moisture, generally high heating value, low oxygen content, and may be used in different manners [5–7]. Kaji et al. [8] investigated the lignite char from pyrolysis at 400 °C and found

that the oxygen content decreases and the hydrophobic property increases significantly. Ye et al. [9] also found that the oxygen content of lignite char predominantly decreases in pyrolysis at 300–600 °C. The lignite char with lower oxygen than lignite is preferable for preparing lignite char–water mixture (LCWM).

Similarly as in CWM preparation, the property of additives is one of the most important factors to affect the performance of LCWM. It is well known that sodium lignosulfonate (SL) has satisfied static stability and naphthalenesulfonate formaldehyde (NSF) has good fluidity for preparing CWM, and appropriate amount of sodium salt of carboxymethyl cellulose (CMC-Na) is efficient to enhance the static stability of CWM. In this paper, SL, NSF and their mixture were selected as dispersants to investigate the effect of dispersant on LCWM performances. Additionally, CMC-Na was used as stabilizer to improve the static stability of LCWM.

2. Experimental

2.1. Materials

The lignite char samples were obtained from pyrolysis of Huolinghe lignite at different temperature with solid heat carrier technology [10]. In the process, the dried lignite with the particle size less than 6 mm was mixed with high temperature char in a mechanical mixer and moved into a reactor where the lignite was pyrolyzed. The lignite char obtained at pyrolysis temperature of 450 °C, 510 °C, 540 °C, and 600 °C is expressed as C450, C510, C540, and C600, respectively. The proximate and ultimate analyses of the char samples are listed in Table 1.

Two commercial anion dispersants SL and NSF were purchased from Anyang Additive Factory in Henan province of China.

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Table 1
Proximate and ultimate analyses of lignite and char samples.

Sample ^a	Proximate analysis (wt.%)			Ultimate analysis (wt.%) (daf)				
	<i>M</i> _{ad}	<i>A</i> _d	<i>V</i> _{daf}	C	H	N	S	O ^{**}
Lignite	11.84	9.26	38.78	66.57	5.10	1.77	0.50	26.06
C450	4.99	14.28	18.10	81.67	3.09	2.06	0.38	12.80
C510	5.63	14.24	16.94	81.45	2.95	2.12	0.39	13.09
C540	5.37	14.66	15.11	83.16	2.88	2.11	0.33	11.52
C600	5.48	15.63	11.85	85.42	2.54	2.00	0.32	9.72

^a Char sample C450 means that the char was obtained from lignite pyrolysis at 450 °C.

^{**} By difference.

2.2. Chemical structure analyses

FT-IR spectroscopy of EQUINOX55 was used to analyze the functional groups of char samples and dispersants with the KBr-pellet technique. The spectra were recorded within a scanning range of 4000–400 cm⁻¹.

The content of hydrophilic functional groups was measured by chemical titration. The total acidity of char samples was measured as follows: (1) the acidity functional groups on the char samples were neutralized with excessive amount of Ba(OH)₂; (2) a certain amount of HCl was added to the system and neutralize the residual Ba(OH)₂; (3) NaOH was used to titrate the overdosed HCl. The total acidity was calculated by the volume of NaOH that neutralizes the residual HCl. Carboxyl was measured by ion exchange method with calcium acetate. In the process, Ca²⁺ dissociated from calcium acetate interacts with carboxyl in the char sample and forms sediment. The carboxyl was determined by the volume of NaOH that neutralizes the H⁺ which was exchanged from char sample in suspensions. Phenolic hydroxyl was obtained from the difference of acidity and carboxyl.

2.3. Preparation of LCWM

Before preparation of LCWM, the char was crushed to a particle size below 250 μm and sieved into three size fractions: 250–150 μm, 150–74 μm and below 74 μm. The char with a particle size distribution according to Alfred distribution model [11] was gradually added into the desired additives solution under low speed agitation of 250 rpm, and then stirred at 1000 rpm for 10 min to ensure well mixing of dispersant solution with char sample. During preparation of LCWM, the temperature was maintained at 25–30 °C.

2.4. Determination of apparent viscosity and rheology of LCWM

The apparent viscosity of LCWM was measured by NDJ-79 rotation viscosimeter with an approximate shear rate of 344 s⁻¹ at 25–30 °C. The slurryability and rheology of LCWM were determined by NXS-11A rotation viscosimeter at 25–30 °C. The slurryability of LCWM was defined as the char loadings in LCWM at the apparent viscosity of nearly 1200 mPa s and the shear rate of 28.28 s⁻¹ [12]. The rheological behavior, according to Ostwald-de-Waele equation [13], was expressed as follows:

$$\tau = K\dot{\gamma}^n$$

$$\eta = K\dot{\gamma}^{n-1}$$

where τ is shear rate, η is apparent viscosity, K is the rheological constant and n is the rheological exponential. For a Newtonian fluid, $n = 1$, for non-Newtonian fluids, $n < 1$ (pseudoplastic) or $n > 1$ (dilatant).

2.5. Adsorption measurement

The adsorption of dispersant on lignite char was measured by the change of dispersant concentration in the solution before and after adsorption [14]. Twenty-five milliliters of solution with different concentrations of dispersant was transferred to a series of 100 ml conical flasks, each containing 0.5 g char sample. The suspension was allowed to equilibrate for more than 40 h with intermittently manual shaking at 25 °C. Then the suspension was centrifuged at 1500 rpm for 2.5 h so that the char sample can be sufficiently separated from the suspension. The suspension was analyzed to determine the dispersant concentration by the absorbance at 278 nm for SL and 287 nm for NSF using predetermined calibration curves on a UV-Vis spectrophotometer (HP-8453). The adsorption was calculated from the initial and final dispersant concentration in the solution.

3. Results and discussion

3.1. Structure characteristics of dispersants and samples

FT-IR spectra as an efficient method to analyze the functional groups of dispersants and samples have been reported by others [14,15]. Fig. 1a shows the infrared spectra of lignite and its chars from different temperature. The characteristic peaks include 3400 cm⁻¹ (Ar-OH), 2800 cm⁻¹ and 2900 cm⁻¹ (C-H stretching in methyl and methylene), 1400–1600 cm⁻¹ (the stretching vibration of aromatic rings) and 1000–1200 cm⁻¹ (C-O-C). The results revealed that the characteristic peak intensities of the oxygen-containing functional groups, such as carboxyl, hydroxyl and ether, decrease greatly when lignite converted into chars. Meanwhile, the aromatic rings on the surface of chars show strong polycondensation after pyrolysis of lignite. Fig. 1b shows the infrared spectra of two dispersants used in this work. The spectra of dispersants show characteristic absorption peaks of Ar-OH at 3500 cm⁻¹, -SO₃- at 900–1000 cm⁻¹ and 625–700 cm⁻¹. The wavenumber at 700–900 cm⁻¹ belongs to bend vibration of branched chain on phenyl. Compared with SL, NSF shows relative larger absorption peak intensities at 3500 cm⁻¹, 1100 cm⁻¹, 700–900 cm⁻¹, indicating that NSF has higher content of hydrophilic groups and more complex branched structures than SL. Because the infrared spectra can only qualitatively describe the hydrophilic functional groups of samples, the quantitative analysis of the functional groups was done by chemical analysis method. Table 2 shows the major hydrophilic functional groups including carboxyl and hydroxyl in lignite and chars. The results showed the similar tendency with those in infrared spectra analysis.

3.2. Adsorption behavior of dispersant on char

Excessive dispersant can increase apparent viscosity and is unbeneficial to other properties of LCWM as well as the cost of

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