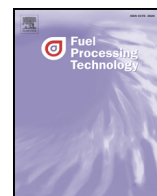




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Hydrothermal treatment of the blend of lignite and rapid hydrogasification char for preparing slurry fuels

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ABSTRACT

The char produced from the rapid hydrogasification (RH char) has a high carbon content, a low volatile matter content and a high calorific value, whereas lignite has high contents of oxygen and volatile matter and a low calorific value. These two carbonaceous fuels are complementary to each other for making CWS, but both of them are poor in slurryability. Hydrothermal treatment (HTT) is consequently used to improve the slurryability of the blend. HTT experiments were carried out in a stirring autoclave at 150–320 °C with a holding time of 30 min. Two types of feedstock were found to have some interactive effects during HTT on changing the elemental composition, acidic functional groups, particle size distribution, particle morphology and water holding capacity in or of the treated blend. The slurryability of the blend is appreciably improved by HTT, and the extent of improvement increases with the temperature increasing till 300 °C but not to a higher temperature. The reasons for the slurryability changed after HTT are discussed.

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1. Introduction

Lignite is a plentiful fossil fuel resource worldwide but has innately inferior qualities. The efficient utilization of lignite with low emissions for meeting the expanding energy demand of the future is a prevailing and challenging subject in the field. The preparation of lignite to coal-based water slurry fuels (CWS) for gasification has attracted considerable interest. However, lignite is not easy to make a commercially applicable CWS mainly due to its poor slurryability and a lower calorific value [1,2]. It is widely accepted that the abundance of hydrophilic groups and porosity in lignite causes more water to be immobile inside the micropores and between the particles, resulting in an unsatisfactory slurryability [3]. Many researchers have thereby devoted to improving the slurryability of lignite by various methods such as using coal blends [4–7], hydrothermal treatment [8–12], thermal treatment [13–15] and microwave radiation [16,17].

Blending lignite with another coal or carbonaceous fuel of good slurryability and high calorific value is a rational approach for manufacturing a better CWS. Wu et al. [6] prepared the slurry of a solid concentration as high as 71.3% by mixing lignite with petroleum coke, while the maximum solid concentration of the slurry obtained from lignite alone was only 46.7%. But there was a drawback that the addition of more petroleum coke in the blend worsened the dispersion stability of the slurry. Lv et al. [5] used the blends of lignite with a direct

coal liquefaction residue for making CWS. The blend with 1:1 weight ratio was the most suitable for producing a desired slurry. Liu et al. [4] studied the slurry characteristics by mixing a low rank coal with individual high rank coals. They observed that the maximum solid concentration was not simply the arithmetic means of those of two individual coals, depending on the coal blends.

Hydrothermal treatment (HTT) is perceived as a promising method for dewatering and upgrading of lignite, since the latent heat of steam may be recovered in the treatment system unlike the evaporative drying. Fei et al. [8] reported the hydrothermal dewatering of a Victorian lignite as a function of temperature (200–300 °C), and their results indicated that only when the hydrothermally processing temperature was elevated up to 250–300 °C were the content of equilibrium moisture and the content of acidic functional groups in lignite significantly lowered. Favas et al. [9] performed the hydrothermal dewatering of a Victorian lignite with varying processing conditions (temperature, reaction time, autoclave size and configuration, and slurry concentration). They revealed that of these variables, the temperature was the most critical factor affecting the intra-particle porosity of treated lignite, and they viewed the intra-particle porosity as a parameter representative of a maximum solid concentration of slurry. Inoue et al. [11] reported that the HTT enabled the solid coal concentration in a coal–oil slurry to rise from 28 to 40%. Fu and Wang [13] found that the apparent viscosities of all CWSs were reduced via the HTT of two Chinese lignites at 150–300 °C, and the enrichment of calcium in lignite could significantly affect the decarboxylation of lignite and the slurryability. In terms of the semi-quantitative determination of functional groups in

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the hydrothermally treated Inner Mongolia lignite with X-ray photoelectron spectroscopy, Yu et al. [12] concluded that both carboxyl and carbonyl groups decreased with the temperature increasing from 200 °C to 320 °C, whereas hydroxyl groups went through a maximum concentration. Interestingly, they found that compared to deionized water, the use of the filtrate (an organics-dissolved liquid waste) for preparing CWS increased the solid concentration at a fixed apparent viscosity and also improved the static dispersion stability.

In a previous paper [18], we investigated the slurryability of two rapid hydrogasification chars (RH char) that were the by-products produced from the rapid hydrogasification of two high-volatile bituminous coals in a pilot-scale entrained flow gasifier operated in ENN Group, Co. Ltd. This process was developed to produce synthetic natural gas and light aromatics. In the process design, the residual char was used to produce the hydrogen supplied for hydrogasification by the water slurry gasification. The RH chars were found to have an extremely poor slurryability, which was attributed to its highly rich micropores and capillary pores, low bulk density and high water holding capacity. However, the RH char has a higher carbon content and calorific value. Therefore, it can be considered as a complementary fuel to lignite for preparing CWS. In this work, we study the property changes of the blend of lignite and RH char via the HTT and the slurryability of the hydrothermally treated blends. We wish to present some new observations on the hydrothermal interactions between the RH char and lignite.

2. Experimental

2.1. Samples and hydrothermal treatment method

A Chinese lignite (YX lignite) mined from Yuxi district, Yunnan province and a RH char (YY-RH char) were used in this work. YY-RH char was the by-product produced from the hydrogasification of Yuyang bituminous coals (YY coal) in a pilot-scale entrained flow gasifier at the temperature of 850 °C, the pressure of 7 MPa and the residence time of about 10 s. The proximate analysis results of YY coal were 8.31% ash, 31.06% volatile matter, and 60.63% fixed carbon (all dry basis). The ultimate analysis results of YY coal were 77.85% carbon, 4.38% hydrogen, 0.95% nitrogen, and 1.32% sulfur (all dry ash-free basis). The char yield was estimated to be about 31.2% by the ash balance, close to the content of volatile matter.

HTT experiment was carried out in a stirring autoclave with a volume of 250 mL. In each run, about 50 g sample of lignite, RH char, or RH char/lignite blend and 150 g deionized water were added into the autoclave. After being completely sealed, the autoclave was heated at an average heating rate of 10 °C/min from room temperature to a predetermined temperature of 150–320 °C with a holding time of 30 min, at a fixed stirring speed 80 rpm. The maximum autogenous pressures were 0.4–11.3 MPa. The autoclave was cooled down to room temperature, and then the suspension was filtered using a Buchner funnel. Then the solid product was dried in electric vacuum oven at 65 °C, and the liquid product was collected in a plastic bottle and preserved in a refrigerator (−4 °C).

For brevity, the treated sample obtained from HTT of RH char/lignite blend at the blending ratio of 1/3 at a temperature of 300 °C is named as RHC:L-1:3-300, that from HTT of lignite alone at 150 °C is named as L-150, and the like.

2.2. Preparation and analysis of slurry

The slurry preparation method and the property measurement methods were described elsewhere [18]. For convenience, these methods are restated as follows. Approximately 40 g sample was accurately weighed and mixed with a predetermined proportion of distilled water in a glass container to prepare CWS with a desired solid loading, using 0.5% Naphthalene sulfonate formaldehyde (NSF) as a dispersant. The slurry was agitated as the rotating speed was increased gradually

from 0 rpm to 1200 rpm, and then the agitation was held at 1200 rpm for 20 min to sufficiently homogenize the slurry. In some cases stated specially, distilled water was replaced by the filtrate liquid waste for preparing slurry. For industrial coal gasification, CWS is generally required to meet a coal mass fraction of 55–70% with the apparent viscosity lower than 1200 mPa at a shear rate of 100 s^{−1} as well as to have good fluidity and storage stability. Hence, the following measurements of viscosity, fluidity and static stability of CWS are necessary.

Viscosity was measured with a rotating-type viscosimeter (model NXS-4C, Chengdu Instrument Factory, China). The readings of shear rate, shear stress and apparent viscosity were recorded automatically by an accessory computer. Apparent viscosity is an average of the viscosity values measured six times at the shear rate of 100 s^{−1}.

The fluidity of slurry was measured by an ocular estimation method [19] and the static stability was assessed by a glass rod penetration method [20]. At the moment when the slurry was prepared, about 40 mL of slurry was filled into a 50 mL graduated cylinder by slowly tilting the container. The fluidity was graded to A for a continuous flow, B for an intermittent flow, and C for no flow depending on the flow states. Then the suspension was covered with 5 mL of liquid paraffin to prevent moisture from evaporation and kept standing for two days at room temperature. The stability was characterized by two indexes, the liquid–solid stratification ratio (SR index) and the sediment hardness (SH index) [20]. After static storage, the suspension was stratified in some cases. The SR index is defined as the ratio of the height of upper layer to that of total suspension, represented in percentage. A zero value of SR index means no occurrence of stratification. The SH index was determined by the glass rod penetration test. Grade A refers to a very soft sediment where the glass rod could gravitationally sink to the bottom, B to a fairly soft sediment which the glass rod could penetrate through by hand push, and C to a hard sediment which the glass rod could not penetrate through by hand push.

2.3. Determination methods for acidic groups

The carboxyl groups and phenolic hydroxyl groups are the main acid groups in low rank coal. These acid groups were measured by an ion exchanging and chemical titration method [21,22]. In the determination of total acid, a weighed coal sample was allowed to react with an excess of barium hydroxide at a boiling point with reflux for 2 h. After the suspension being separated by filtration, a proper amount of hydrochloric acid was added to neutralize the residual barium hydroxide. Then, a sodium hydroxide solution was used to titrate the redundant hydrochloric acid. In the determination of carboxyl groups, a weighed coal sample was pickled with hydrochloric acid for 4 h to elute metal ions which may be combined with carboxyl groups. Then the sample was allowed to react with calcium acetate at a boiling point with reflux for 2 h. After filtration, a sodium hydroxide solution was used to titrate the acid in the liquid. Blank experiments were carried out in the same way without coal sample to eliminate the influences of possible contact with carbon dioxide in air and possible presence of carbonate in the barium hydroxide reagent. The amount of phenolic hydroxyl group was estimated by the difference of the amount of total acidity and carboxyl group.

2.4. Measurement method for water holding capacity

About 2 g sample was completely soaked into an excess amount of deionized water. The suspension was rested for 2 h to guarantee a sufficient adsorption of water onto the sample. Then the suspension was filtered using a Buchner funnel under a fixed condition of vacuum and filtration time to collect the wet filter cake. Immediately after the filtration, about 12 mg of the filter cake was transferred into a small corundum container for the thermogravimetric and differential scanning calorimetry analysis (METTLER TOLEDO TGA/DSC). The sample was heated in an argon gas flow (100 mL/min) at the rate of 5 °C/min from 20 °C to 120 °C with a holding time of 20 min. The mass and heat

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