



Full Length Article

Experimental study of the physicochemical structure and moisture readsorption characteristics of Zhaotong lignite after hydrothermal and thermal upgrading



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HIGHLIGHTS

- Typical Chinese lignite was upgraded hydrothermally and thermally to study the moisture readsorption characteristic.
- Different upgrading method has different function for the removal of oxygen-containing functional groups in lignite.
- Evolution mechanisms of pore structure by temperature and pressure during upgrading were obtained.
- A moisture readsorption model was established with changes of the physical and chemical structure.

ARTICLE INFO

Article history:

Received 20 April 2016

Received in revised form 19 June 2016

Accepted 26 July 2016

Keywords:

Lignite

Hydrothermally

Thermally

Oxygen-containing functional groups

Pore structure

Moisture readsorption

ABSTRACT

Hydrothermal and thermal upgrading techniques are promising coal drying methods that enable the utilization of low rank coals. In this study, Zhaotong (ZT) lignite from the Yunnan province of China was hydrothermally upgraded at 150–300 °C and thermally upgraded at 200–500 °C; then, the correlation between the changes in the physical and chemical structures and the moisture readsorption characteristics were investigated. The maturity of lignite was improved by the upgrading treatment. The main hydrophilic oxygen-containing groups, including hydroxyl and carboxyl, were effectively removed as the upgrading temperature increased. In addition, the removal of hydroxyl groups was more remarkable during hydrothermal upgrading. Irreversible changes occurred in the pore structure of the treated lignites; the specific surface area and total pore volume first increased and then decreased with hydrothermal upgrading, whereas the specific surface area and total pore volume continually increased with thermal upgrading as the temperature increased. In addition, the gel-like structure of the lignite experienced violent shrinkages and collapses. The moisture readsorption performance was effectively inhibited under the synergy effects of the physicochemical structure such that the moisture readsorption ratio (MRR) and moisture holding capacity (MHC) decreased continuously with the increasing upgrading temperature. An MHC model built through multiple linear regressions proved suitable to describe the moisture readsorption of the upgraded lignites.

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1. Introduction

Lignite, a low-rank coal, has relatively low market prices and abundant reserves, composing approximately 13% of the gross coal reserves in China [1–3]. However, high transportation costs and storage difficulties resulting from the high moisture content, high spontaneous combustion tendency and low calorific value greatly restrict the utilization of these reserves, which are mainly used

for pithead power plants [3–6]. Therefore, upgrading lignite to reduce its moisture content is necessary to increase lignite utilization.

In general, dewatering and upgrading methods can be divided into evaporative drying and non-evaporative drying methods according to the difference in the removal of the water within the coal [7]. Hydrothermal treatment and thermal upgrading are current promising upgrading techniques for lignite; hydrothermal upgrading is a non-evaporative method that not only saves the latent heat of vaporization but also filters out the water-soluble inorganic material [8–10]. Thermal upgrading, an evaporative drying method, refers to the process in which solid fuels decompose

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mildly in an inert atmosphere while being heated, which can greatly reduce the moisture and volatile contents and also remove some volatile toxic heavy metals contained in low-rank coals [1,11,12]. Compared to thermal upgrading, hydrothermal upgrading can provide higher energy efficiency, but the former can produce more coproducts, such as upgraded coal, gases and tar. In the process of hydrothermal and thermal upgrading, the physical and chemical structures of lignite will remarkably change, together with a significant decrease in the moisture content with an increase in the upgrading temperature [13–17].

However, the upgraded lignite will adsorb water again if exposed to a damp environment because of its intricate gel-like structure [18]. Moisture readsorption can increase the spontaneous combustion tendency and lower the upgrading effect; therefore, it is necessary to study the moisture readsorption behavior of upgraded coal to restrict moisture readsorption. Some research [19–21] has indicated that surface oxygen-containing groups play key roles in the moisture readsorption of upgraded lignite, especially hydroxyl and carboxyl groups; these groups have a high affinity for water molecules under the influence of hydrogen-bond interaction [21] and are removed to a great extent during upgrading treatment, leading to a significant decrease in moisture readsorption, as reported by Yu [20]. Additionally, some studies [5,22,23] have reported that moisture readsorption by upgraded coal is closely related to the pore structure. During the upgrading treatment, the pores of brown coal collapse and shrinkage as moisture is removed [22]; cross-linking reactions could seal the pores, including collapsed pores, to restrict the ability of moisture readsorption [22,23]. Similar results were also obtained when asphalt was added to lignite upgrading, which covered fine pores and inhibited the reaction of the hydrophilic functional groups with moisture and oxygen in air to result in a decrease of the moisture readsorption ratio (MRR) [5,11].

The moisture holding capacity (MHC, equilibrium moisture content at 96% relative humidity and 30 °C) of lignite is considered a suitable index to assess the maximum moisture adsorption ability of upgraded lignites and the degree of coalification to some extent [24,25]. Wang et al. [21] studied the effect of inherent oxygen-containing groups on the MHC of thermally upgraded lignites and observed a positive correlation between the MHC value and the content of oxygen-containing groups. Kaji et al. [26] indicated that both oxygen-containing groups and pore structure of lignite were key factors in determining the MHC and found that the decomposition of oxygen-containing groups and the plugging of small pores resulting from volatile matter condensation made the surfaces hydrophobic during pyrolysis.

The previous work mostly focus on the description of changes in physical and chemical structures of lignite treated by a single upgrading method, only qualitative analysis have been reported for the correlation of the changes in physical or chemical structure with the moisture re-adsorption characteristic of upgrade lignite, especially, which may be not suitable for both upgrading methods. Therefore, it is considerably necessary to deeply understand and reveal the evolution mechanism of physicochemical structure and moisture re-adsorption characteristics during hydrothermal and thermal upgrading for dewatering and upgrading of the lignite.

This study was designed to compare the different changes in the physicochemical structure and moisture readsorption characteristics of the lignite due to hydrothermal and thermal upgrading and to establish a moisture readsorption model in order to describe the moisture readsorption mechanism basis on the influence of the chemical and physical structures on moisture readsorption. The Zhaotong (ZT) coalfield, with a total area of 140 km² and 8 billion tons of available reserves, lies in the northeast of Yunnan, China. In this work, ZT lignite was treated by hydrothermal and thermal upgrading before analysis of its preliminary characteristics; then,

a range of techniques was applied to analyze its oxygen-containing groups and pore structure. Moreover, the moisture readsorption performance of the upgraded lignite was investigated in an environment with constant humidity and temperature. Additionally, a mathematical model was developed for the MHC of the upgraded lignite with physical and chemical structural changes.

2. Experimental

2.1. Material

The lignite sample was air dried, ground, and sieved to obtain a 0.075–0.1 mm particle size. A TGA2000 instrument (Las Navas Corp., Spain) was used to perform the proximate analysis. The carbon, hydrogen, nitrogen, and sulfur contents were determined using a Vario Micro Cube (Elementar Corp., Germany). A Parr-6300 bomb calorimeter (Parr Instrument Corp., USA) was applied to measure the heating value.

2.2. Upgrading treatment

2.2.1. Hydrothermal upgrading

The hydrothermal upgrading experiment was performed using the CJF-0.5 autoclave (Dalian Tongda Corp., China) shown in Fig. 1. In each experiment, 25 g of the lignite samples and 125 g of deionized water were required in the reactor. Thereafter, the autoclave was first subjected to 2 MPa of N₂ pressure to transfer any residual gas and subsequently it was sealed for 2 h to check for air leakage; then, the pressure was reduced to 0.2 MPa to maintain the N₂ reaction atmosphere. The autoclave was heated at a heating rate at 5 °C/min and maintained for 30 min when the pre-set temperature was reached. In the next step, the entire apparatus was naturally cooled down to room temperature. The treated lignite was separated from the coal-water mixture using a vacuum pump and then collected. The hydrothermally upgraded lignite at different temperatures of 150, 200, 250, and 300 °C was numbered as H150, H200, H250, and H300, respectively.

During hydrothermal upgrading, the pressure inside the autoclave exponentially increased and reached approximately 8.7 MPa at 300 °C, as shown in Fig. 1. The measured pressure value with 0.2 MPa of initial pressure was slightly greater than the theoretical value [27] but was almost consistent in the variation trend, which indicates that this system had high reliability.

2.2.2. Thermal upgrading

The thermal upgrading of the lignite was performed in a horizontal tube furnace, as shown in Fig. 2. In this experiment, the quartz tube was constantly rinsed with N₂ at a flow rate of 1.5 L/

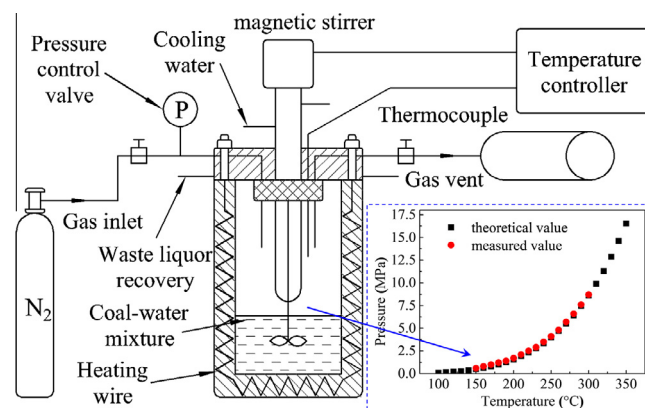


Fig. 1. Schematic diagram of the hydrothermal upgrading experimental apparatus.

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