



## Full Length Article

# Hydrothermal dewatering of a Chinese lignite and properties of the solid products



Junjie Liao<sup>a</sup>, Yi Fei<sup>b</sup>, Marc Marshall<sup>b</sup>, Alan L. Chaffee<sup>b,\*</sup>, Liping Chang<sup>a</sup>

<sup>a</sup> Key Laboratory of Coal Science and Technology, Ministry of Education and Shanxi Province, Taiyuan University of Technology, Taiyuan 030024, PR China

<sup>b</sup> School of Chemistry, Monash University, Clayton, Victoria 3800, Australia

## HIGHLIGHTS

- Lignite can be efficiently upgraded by hydrothermal dewatering (HTD) process.
- During HTD, organics and inorganics removed increase with HTD temperature.
- After HTD, the hydrophobicity of coal is higher and increases with HTD temperature.
- HTD coal has a higher spontaneous combustion tendency than the raw coal.

## ARTICLE INFO

### Article history:

Received 29 October 2015

Received in revised form 8 March 2016

Accepted 6 April 2016

### Keywords:

Lignite  
Hydrothermal dewatering  
Equilibrium moisture content  
Spontaneous combustion  
Wire basket method

## ABSTRACT

A lignite from Shengli mine in Chinese Inner Mongolia was upgraded by hydrothermal dewatering (HTD). The effects of the HTD process on the solid product yield and the removal rate of elements, organics and inorganics in this coal were determined. The moisture re-adsorption capacities and spontaneous combustion behaviors of coal products from the HTD process were studied, using the saturated salt solution and wire basket reactor methods respectively. FT-IR, XRD, CO<sub>2</sub> adsorption, mercury intrusion porosimetry (MIP) and helium pycnometry were carried out to characterize the physicochemical properties of samples. The results show that during the HTD process, organic matter was removed due to the loss of CH<sub>3</sub>, CH<sub>2</sub>, and O-containing functional groups, while the inorganic matter was mainly removed in the forms of Ca- and Fe-containing minerals. The amounts of both organic and inorganic matter removed increased with temperature. The equilibrium moisture contents of the hydrothermally dewatered coals at 30 °C in the relative humidity range 11–97% were lower than those of the raw coal. They decreased as the HTD temperature rose, probably due to the increase in the loss of hydrophilic O-containing functional groups and decrease in surface area with the increasing of HTD temperature. Moreover, the critical ignition temperature ( $T_{cr}$ ) of all the hydrothermally dewatered coals, as measured by wire basket spontaneous combustion experiments, was lower than that of raw coal.

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## 1. Introduction

China accounts for 67% of global coal growth to 2030 and remains the largest coal consumer [1]. With the rapid development of China in recent decades, much of its hard coal has been consumed to provide energy, so the efficient use of lignite is a very important and urgent topic. China has abundant lignite resources, with the proven lignite reserves being about 18.6 billion tons in 2008, 16% of the total Chinese coal reserves [2]. However, the high moisture content (25–65%) [3] in lignite lowers power plant efficiency, increases the capital costs and leads to higher CO<sub>2</sub>

emissions per unit of energy output, due to the larger size of lignite boilers required in electrical power plants compared with the hard coal fired boilers in the current state [4]. Moreover, due to the high moisture content, it is not economically feasible to transport lignite over long distances, which critically impacts on virtually every facet of its utilization including combustion, gasification and liquefaction [5]. Thus the moisture content of lignite should often be efficiently reduced before use. Techniques suggested for efficient moisture reduction from lignite include rotary drying [6,7], fluidized-bed drying [8,9], hot oil-immersion drying [10,11], microwave drying [12–14], hydrothermal dewatering (HTD) [15–20], and mechanical thermal expression (MTE) [21–26], some of which have been applied commercially. A particular advantage of HTD is that most of the water is removed as a liquid, saving the

\* Corresponding author.

E-mail address: [alan.chaffee@monash.edu](mailto:alan.chaffee@monash.edu) (A.L. Chaffee).

**Table 1**  
Relative humidity (RH) obtained at 30 °C with the saturated salt-water solutions used [37].

Salt	LiCl·H <sub>2</sub> O	MgCl <sub>2</sub> ·H <sub>2</sub> O	Mg(NO <sub>3</sub> ) <sub>2</sub> ·H <sub>2</sub> O	NaCl	K <sub>2</sub> SO <sub>4</sub>
RH (%)	11.28 ± 0.24	32.14 ± 0.14	51.40 ± 0.24	75.09 ± 0.11	97.0 ± 0.4

energy that in some other drying techniques has to be used to evaporate the water. Moreover, much of the water-soluble inorganic material will be leached out, leading to a decrease of inorganic content in the dried coal [17]. Disadvantages are that HTD has to be operated at high pressure and the waste water treatment may be a problem [5]. There are, however, two important properties of any dried coal that have not been investigated extensively for hydrothermally dried Chinese lignite. It is important to measure the moisture readsorption of dried lignite, because this will determine whether the dried lignite is suitable for storage and transport. It is also important to determine the tendency for spontaneous combustion of the dried lignite, as dried lignites in general do tend to combust spontaneously quite easily [27,28]. This tendency has long been associated with serious safety problems during storage and transport, and also leads to undesirable changes in the thermal and chemical properties of the dried lignites [29]. Most studies on HTD have focused on water-coal slurry preparation and slurry properties after HTD [30,31]. Few have considered the moisture readsorption and the spontaneous combustion behavior of the hydrothermally dewatered product, which are important subjects of this paper, along with determination of what parts of the coal are removed during HTD. As lignite reserves in Inner Mongolia constitute more than 75% of the total Chinese lignite reserves [32], it is an Inner Mongolian lignite, Shengli (SL) lignite, which is the subject of this paper.

## 2. Experimental

### 2.1. Determination of moisture content

The procedure was based on the Australian Standard [33]. About 5 g of as-received sample with particle size less than 0.25 mm was weighed and heated at 105 °C for 3 h in flowing N<sub>2</sub>. The sample was cooled in a desiccator containing silica gel and then weighed. The moisture content was calculated as described by the Australian Standard.

### 2.2. Hydrothermal processing of lignite

712.93 g of <3 mm Shengli raw coal (500.00 g db) and 1500 g distilled water were loaded into a 4 L autoclave. The autoclave was sealed, evacuated for 5 min to remove trapped air and then filled with N<sub>2</sub> to a pressure of about 0.25 MPa. Then it was heated to the desired temperature (290, 320 or 350 °C) in about 2 h, with stirring of the contents at 120 rpm, and was held at that temperature for 1 h with continued stirring. After overnight cooling, the slurry in the autoclave was filtered, washed with distilled water, and stored in a sealed container. The products prepared at 290, 320 and 350 °C are denoted HTD290, HTD320 and HTD350, respectively. The yield ( $Y_{solid}$ ) of the solid products was calculated by Eq. (1).

$$Y_{solid} = \frac{m_{HTD}}{m_{raw}} \times 100\% \quad (1)$$

where  $m_{raw}$  and  $m_{HTD}$  are the mass of the raw coal feed and the mass of solid products after HTD respectively, g db.

### 2.3. Equilibrium moisture content (EMC)

The EMC was measured by the desiccator method [34–36]. Samples of raw and hydrothermally dewatered coals put in a bucket which could be closed with a lid were equilibrated for two weeks in desiccators at 30 °C in a thermostatted cabinet, in the presence of saturated solutions of various salts to control the relative humidity. Table 1 shows the salt solutions used [37]. All the salts used were purchased ready for use except LiCl·H<sub>2</sub>O, which was made as described by the references [38,39].

The desiccators were evacuated before placing them in the cabinet to speed up the approach to equilibrium. After equilibration, the samples were dried in a vacuum oven for 24 h at 30 °C, and then subjected to a second and third EMC run under the same conditions as before. The EMC was calculated by Eq. (2).

$$EMC = \frac{m_2 - m_1}{m_1 - m_0} \times 100\% \quad (2)$$

where  $m_0$  was the weight of the dish, g;  $m_1$  was the weight of the dish and sample after drying, g;  $m_2$  was the weight of dish and sample after equilibration, g.

### 2.4. Spontaneous combustion

The wire basket method [40,41] was used to evaluate the spontaneous combustion behavior of the raw and hydrothermally dewatered coals, because the condition of the coal in such experiments, closely packed, is closer to the condition of the coal in large-scale transport and storage situations than in a thermogravimetric analysis experiment. The wire-basket method gives results similar to those found in practice [42]. A 2.5 cm cubic basket, made of 0.125 mm stainless steel mesh, was filled with coal, which was pressed down by a 250 g weight of 2.5 × 2.5 cm<sup>2</sup> square cross-section to ensure that the packing density was constant. Then, the basket was placed in an oven and three thermocouples were inserted, one into the center of the basket and the other two into both sides. Another thermocouple in the center of the oven measured the oven temperature. All four thermocouples were connected to a data logger. In a typical experiment, the oven was heated to a predetermined temperature and the readings of the basket thermocouples monitored. Either the temperature rose very rapidly after the readings of these thermocouples reached oven temperature, implying ignition had occurred, or the readings stabilized at near oven temperature when ignition did not occur. The parameter of interest derived from the temperature vs. time curve was the crossing point temperature, defined as that at which the standard deviation of the basket thermocouple readings was a minimum, corresponding to minimum heat conduction along the sample [40,41]. Repeated experiments with different oven temperatures were used to define the smallest temperature interval for which ignition occurred at the higher temperature of the interval and ignition did not occur at the lower temperature of the interval. The average of the crossing point temperatures at the limits of this smallest temperature interval defined above gave the critical temperature ( $T_{cr}$ ) of the sample. The activation energy and pre-exponential factor of the rate of temperature rise before ignition were calculated from Eq. (3) using the data for experiments below the ignition oven temperature [40]. A straight-line least-squares fit of  $\ln(\partial T/\partial t)_{T_p}$  vs  $1/T_p$  yields the kinetic constants,  $A$  and  $E$ .

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