



Drying characteristic and kinetics of Huolinhe lignite in nitrogen and methane atmospheres



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HIGHLIGHTS

- Drying characteristics, kinetics of lignite in methane were investigated.
- Drying process of HLH lignite follows random nucleation mechanism.
- Moisture re-adsorption properties were studied by static desiccator method.
- Moisture desorption and re-adsorption isotherms are irreversible.

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ABSTRACT

Lignite drying in methane atmosphere is a potential approach to utilize moisture in lignite as a feedstock for H₂ production via steam reforming of methane. In this paper, experiments on drying and moisture re-adsorption properties of Huolinhe lignite in nitrogen and methane were carried out in a fixed-bed apparatus and thermogravimetric analyzer to investigate the effect of drying temperature, particle size, moisture content, drying medium and sample weight on drying rates and drying dynamics. The drying kinetics and mechanism were studied by thermal analysis and thin layer models. The moisture of lignite decreases with increasing drying temperature, decreasing particle size and sample weight. The drying process follows random nucleation mechanism and Logarithmic model is the optimal thin layer model for drying process of Huolinhe lignite. The activation energy in methane is lower than that in nitrogen. Static adsorption method was used to analyze the equilibrium moisture contents (M_e) of dried coal sample. The results show that M_e decreases with the increase of drying temperature and M_e of dried coal is lower than that of raw coal. The moisture desorption and re-adsorption isotherms are irreversible.

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1. Introduction

Lignite and sub-bituminous coal are abundant, accounting for 53% of total coal resources in the world and 46% in China [1]. The high moisture content, low calorific value and spontaneous combustion of lignite affect its effective and economical utilization [2]. Drying, as the first and essential step in most utilization processes including pyrolysis, gasification and combustion, can improve the calorific value of lignite and utilization efficiency of downstream device and decrease transportation cost [3]. Various drying technologies based on evaporative or non-evaporative methods were developed to decrease the moisture while lignite upgrading. Drying in hot air or flue gas is a conventional approach for lignite drying, but it causes great risk of self-ignition and oxidation [4–6]. Application of superheated steam fluidized bed drying

technology in arid area is limited because of high requirements of quantity of water although the energy consumption is significantly lower than hot air drying process [7–12]. Besides, microwave drying has also been considered as a potential application method due to high heating rates, uniform heat supply, selective heating of moist areas, additional mechanisms of moisture transport due to internal evaporation of moisture and instant turn on or off capabilities [13–15]. However, among these drying technologies, most of the drained moisture is discarded without further utilization. Hydrogen, as a clean fuel and important feedstock in hydrogenation process, is mainly produced by steam reforming of methane (SRM) in industry [16–18], which results in the consumption of a large amount of water. Lignite contains moisture up to 60% by mass [12], if lignite is dried in methane atmosphere, the exhausted gases from drying process including methane and steam will be a potential feedstock for H₂ production via SRM. By this way, both the moisture in lignite and its latent heat in steam generated from drying can be fully recovered. It is helpful for the

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water supply in SRM process. However, almost no relative research was reported on lignite drying in methane atmosphere.

Studies on drying characteristic and kinetics using thermal analysis method and mathematical modeling of drying and moisture re-adsorption are important for design of dryer and downstream device [19–21]. To investigate coal drying behavior, moisture re-adsorption, pore structure of dried coal and combustion behavior, many fundamental researches have been carried out [22–26]. For example, effects of drying temperature, coal particle size, sample weight, gas flow rate on coal drying in nitrogen were considered; equations relating the initial drying rate and the parameters were developed [23–26]. The variation of the internal porous structure during drying and influence of temperature and heating time were investigated [27].

Drying models based on different mechanisms vary between theoretical, semi-theoretical and empirical models [10,28]. Usually, drying kinetics of lignite is often described by thin-layer empirical equations which are material dependence and developed according to experiment data, and it also needs no assumptions in geometric, mass diffusivity and conductivity aspects [29,30]. Since the amount of strongly held moisture in lignite is not significantly influenced by standard drying approaches, it may be the medium-sized water clusters which are reduced in number, indicating the irreversibility of pore structures [31]. Re-adsorption of dried coal was examined at constant relative humidity and temperature [2,32,33] and studied by empirical models of equilibrium moisture content of porous material [34].

To investigate drying and moisture re-adsorption characteristics, kinetics and mathematical modeling of Huolinhe (HLH) lignite in methane atmosphere, the effects of drying temperature, particle size and sample weight on drying of HLH lignite were studied on a lab-scale fixed-bed and thermogravimetric analyzer (TGA) and the results were compared with that in nitrogen. The thermal analysis and thin layer drying model were adopted to simulate the drying kinetics. The static desiccator method was used to analyze desorption and re-adsorption isotherms of moisture in raw and dried samples.

2. Experimental and modeling

2.1. Lignite samples

HLH lignite from Inner Mongolia of China was selected. Before experiment, the raw coal was pulverized and sieved in five particle ranges, including <100 mesh, 16–20 mesh, 10–12 mesh, 6–10 mesh, and >6 mesh, respectively. The proximate analysis of coal samples in different particle sizes and two raw coal samples (16–20 mesh) from different mines with different water content are presented in Table 1. Samples were sealed in plastic bag in nitrogen atmosphere and kept in refrigerator.

Table 1
Proximate analysis of Huolinhe lignite samples with different particle sizes and moisture contents.

Sample	M_{ad}	A_d	V_d	FC_d^a
>6 mesh	22.38	10.04	40.71	49.25
6–10 mesh	22.03	12.31	41.57	46.12
10–12 mesh	21.78	12.27	41.83	45.90
16–20 mesh	21.69	12.01	40.74	47.25
Lignite #1	15.50	11.33	40.27	48.40
Lignite #2	18.36	11.58	40.26	48.16

ad: air dry base; d: dry base.

^a By difference.

2.2. Experiment apparatus

2.2.1. Drying experiment

The drying experiments were conducted in a lab-scale fixed-bed apparatus as shown in Fig. 1. Coal sample with different mesh was first placed into the packed bed with quartz wool in vertical stainless-steel tube ($\varnothing 20 \times 3$ mm, 300 mm in length). Before each test, nitrogen with a flow rate of 90 ml/min was entrained into the drying tube to clean up the system at least 3 min. Pressure drop across the fixed bed was monitored continuously from pressure gauge so as to ensure there was no leak in the system. Lastly, lignite sample was quickly dried from room temperature to the desired drying temperature (40, 50, 60, 80, 100 and 120 °C) by a preheated furnace outside and held for different drying time in methane or nitrogen as dry media.

2.2.2. Moisture re-adsorption experiment

To investigate the effect of drying temperature and environmental temperature on re-adsorption characteristic of dried coal, static desiccator method was used to analyze the equilibrium moisture content (M_e) of two dried coal samples obtained by drying of lignite #2 at 40 °C and 120 °C in methane atmosphere, respectively. About 5 g dried coal sample was put into a weighing bottle placed in the desiccator. The desiccator was sealed and kept in a constant temperature filling with 300 mL saturated salt solution in consistent relative humidity (RH) for more than 8 days. The equilibrium moisture content (M_e) was calculated by the weight difference of sample, which was recorded before and after re-adsorption test. The RH of saturated salt solutions at 40 °C and 50 °C (from 11% to 97%), are listed in Table 2 [35].

2.3. Characterization of lignite drying process

2.3.1. TG–MS

The weight loss of HLH lignite during drying process was determined by a TGA (Mettler-Toledo TGA/SDTA851^e). The evolution of gases was detected through a coupled quadrupole mass spectrometer (GSD-301 T3 quadrupole, MS). About 20 mg coal sample was heated from 25 °C to 200 °C at a heating rate of 10 °C/min using N₂ or CH₄ as carrier gas with a flow rate of 90 ml/min.

2.3.2. FT-IR spectroscopy

Fourier transform infrared (FT-IR) spectroscopy of raw coal and dried coal samples obtained at different atmospheres were detected with EQUINOX55 spectrometer to confirm the changes during coal drying.

2.4. Drying kinetics

Moisture released with gas in the process of thermal drying of coals at low temperature. In all experiments, moisture content was obtained using Eq. (1):

$$M = (W_0 - W_e)/W_0 \quad (1)$$

where M is moisture content (g/(g dry coal)), W_0 is initial sample weight (g), W_e is dry coal weight (g). Moisture ratio was calculated using Eq. (2) based on experimental data:

$$MR = \frac{X - X_e}{X_0 - X_e} \quad (2)$$

where MR is moisture ratio, X is moisture content at any time t ; X_e is equilibrium moisture content, X_0 is initial moisture content of sample, air dry basis.

X_e is assumed to be zero at the end of drying process. Therefore, moisture ratio is expressed as:

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