

## Experimental study on freeze drying of Loy Yang lignite and inhibiting water re-adsorption of dried lignite



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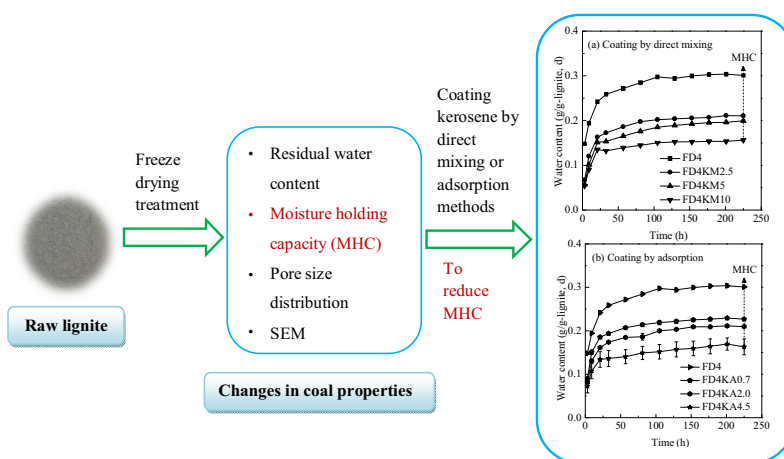
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### HIGHLIGHTS

- Lignite was dewatered using freeze drying (FD).
- MHC of FD treated sample was lower than that of raw lignite.
- Coating kerosene by adsorption or direct mixing methods can both decrease MHC.
- Adsorption method is better than direct-mixing method.
- MHC reduced from 0.3 (g/g-lignite,d) in FD4 to the lowest value 0.16 in FD4KA4.5.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Loy Yang lignite with a high water content was dewatered by freeze drying (FD). The drying kinetics of FD was calculated using thin-layer drying models given in the literature. Residual water content, re-adsorption or desorption behaviors, and pore size distributions of all of the samples were investigated. Furthermore, coating with different amounts of kerosene by direct mixing or adsorption methods to restrain water re-adsorption was also investigated. The results showed that FD dewatering contained three steps: a fast dewatering period (~2 h), followed by a reduced drying-rate period (2–3 h) and an apparently falling-rate period (>3 h). The Midilli-Kucuk model described the drying process perfectly and could be employed to predict residual water content in the sample at any time during the FD dewatering process. The moisture holding capacity (MHC) of the FD-treated samples was lower than that of raw lignite, which is because of the effect of the water–lignite bond strength and the diffusion adsorption or desorption force. Moreover, adding kerosene by either adsorption or direct-mixing methods can both

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decrease MHC because kerosene is coated on the surface and in the pores of the lignite. These prevent water re-adsorption. The adsorption method is better than the direct-mixing method because it consumes less kerosene.

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## 1. Introduction

Low-rank coal accounts for 45% of the world's coal reserves [1,2]. Lignite is a typical low-rank coal and is classified as a low grade fuel. Currently, lignite is primarily used for electricity generation, and it is believed that its utilization in other aspects will increase in the future because of its advantages over high-rank coal, such as abundance, easy access, low cost, and low sulfur content [3–5]. However, the greatest disadvantage of lignite is its high water content (ca. 30–65% on a wet-weight basis) [6], which causes serious application problems and inhibits its wide utilization. Lignite contains abundant oxygen-containing functional groups resulting in a hydrophilic character, which is the main reason for its high water content [5,6]. On the other hand, lignite is porous, leading to the easy transport of water, action of capillaries, and large volume for retaining water, which is also an important reason why lignite contains a high amount of water [5–7]. The high water content is responsible for low thermal efficiency, high power consumption, high transportation and storage costs, increased CO<sub>2</sub> emission, and great spontaneous combustion risk [8,9]. It is generally accepted that water retained in lignite can be divided into two major forms (i.e., freezable and non-freezable water) on the basis of the characteristic phase transition, which includes congelation and fusion [10,11]. The removal of water from lignite and restraint of water re-adsorption of dried lignite are necessary processes to increase its economic efficiency and widespread application in industry in an environmentally friendly and efficient manner and are receiving considerable attention. Various drying and upgrading technologies have been developed to dry lignite and to increase its calorific value. These can be grouped into two categories: evaporative drying (e.g., microwave-irradiation dewatering, fluidized-bed drying, and thermal heating) and non-evaporative drying (including hydrothermal treatment, mechanical thermal dewatering, solvent dewatering, and so on) [5].

Freeze drying (FD) is another alternative dehydration technique, which is widespread and plays an indispensable role among various drying techniques [12–14]. In general, the operation process of FD dewatering includes the following: (1) preparation of the material, (2) freezing step during which water in the material becomes frozen by low temperatures, (3) ice sublimation phase that is the transition directly from ice to gas at temperatures and pressures below the triple point of water, and (4) ultimate storage of the FD-treated materials [14–16]. For more than 65 years, FD was almost exclusively applied within the biological and pharmaceutical industries as a batch type operation. It has gradually shifted from batch to semi-continuous and even purely continuous operations with its utilization in the food industry, which faces large production demands. This significantly increases productivity [14]. In addition, FD can produce high-quality dried products, which are suitable for long-term storage and long-distance transportation [14,17]. This can solve the primary problem of lignite; it is mostly used at power stations located at or near a mine because of its high water content.

The major drawback of FD is that it is an expensive drying technique due to its low drying rate and high energy and capital costs generated by vacuum and refrigeration units [12–17]. Further modifications are required to make it more profitable. Researchers

have proposed several pathways to solve this challenging issue, for instance, atmospheric pressure lyophilization [12] and FD combined with microwave heating [13]. To our knowledge, so far no study appears in the literature of FD dewatering of low-rank coal. Usually, the utilization of one new technique starts with a laboratory scale investigation. The objective of this work is to present some fundamental results of a laboratory investigation of the dewatering of lignite using FD treatment, which may be useful for the evolution of FD and lignite dewatering techniques.

A study of drying kinetics has significant academic and practical meanings for the design of a lignite drying system. Thin-layer empirical equations are widely employed to investigate the drying process mainly because these empirical models do not need assumptions in mass diffusivity, geometry, and conductivity aspects [18–21]. Zhao et al. [19] investigated the effects of mineral matter on the drying kinetics of lignite using thin-layer models and argued that high-ash lignite reveals more promising drying kinetics with lower activation energy than does low-ash lignite. Pickles et al. [20] and Tahmasebi et al. [21] obtained the drying kinetics of low-rank coal in the microwave and using a superheated steam fluidized-bed.

Another problem related to water in lignite is that dewatered lignite easily re-adsorbs water, which has a negative effect on its storage and causes a loss of the drying effects. There are many factors that influence the water re-adsorption capacity of dried lignite, such as oxygen-containing functional groups, pore structure, the diffusion force between the internal water content in the sample and the water vapor in the external environment, and the storage environment. Shigehisa et al. [22] and Choi et al. [23] reported that the water re-adsorption content of dried lignite is reduced markedly by coating with asphalt. The asphalt was coated during coal-oil slurry dewatering, which mixed kerosene containing a small amount of asphalt with the raw lignite to produce the slurry. Although the kerosene was recovered by heating, there was still a certain amount of kerosene that remained in the pores and on the surface of lignite and the authors did not focus on this. Coating with different amounts of kerosene to prevent water re-adsorption is discussed in this study.

In the present work, Loy Yang (LY) lignite was dehydrated using an FD treatment. Nine different thin-layer drying models were employed to model the drying kinetics of LY lignite in FD and the one with the highest goodness of fit was found. Furthermore, the effects of FD treatment on water re-adsorption capacity were also studied. Of particular interest was the problem of how to restrain the water re-adsorption of the dewatered lignite by means of coating with different amounts of kerosene either by direct mixing or desiccator adsorption methods, which are simple, easy, and convenient.

## 2. Experimental

### 2.1. Sample

An Australian lignite (LY) was used as the coal sample. The lignite sample was ground to pass through an 840- $\mu$ m sieve for the experiments. The proximate and ultimate analyses are shown in Table 1.

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