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Response of hygroscopicity to heat treatment and its relation to durability of thermally modified wood

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HIGHLIGHTS

- Hailwood-Horrobin model calculates moisture adsorption of thermally modified wood.
- The m_0 is a critical property of moisture adsorption for thermally modified wood.
- A correlation between m_0 and durability of thermally modified wood was found.
- An m_0-based method is proposed to evaluate durability of thermally modified wood.

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ABSTRACT

In an attempt to study the effect of heat treatment on hygroscopicity and durability of wood, Poplar (*Populus* spp.) wood was thermally modified using five different temperatures between 170 °C and 210 °C, for a fixed duration of 3 h. Moisture adsorption behavior and the resistance to soft rot fungi were investigated thereafter. Based on the Hailwood-Horrobin sorption model, the amount of available sorption sites within specimens for the different groups of Poplar wood were calculated from the model's m_0 parameter. Chemical analysis of the changes in wood components induced by heat treatment allows for a comparison between the easily obtained m_0 and the results of time-consuming wood decay tests. The proposed m_0-based method for highly efficient evaluation and prediction of durability of thermally modified wood could optimize future research on the mechanisms of heat treatment processes.

1. Introduction

Wood is a renewable lignocellulosic building material with a high strength-to-weight ratio. Compared to other building materials, wood is relatively easy to work with, and the energy requirements for processing are low. With increasing eco-awareness around the world, wood products are increasingly utilized in indoor and outdoor applications. When wood is exposed to the environment, however, moisture adsorption or desorption will occur, depending on the relative humidity (RH), because of wood hygroscopicity. That is, the moisture content (MC) of wood will change in response to the variation in moisture in the surrounding environment until reaching equilibrium moisture content (EMC) [1,2]. Therefore, wood is susceptible to deformations and decay, especially in an environment with a frequent high-low variation in moisture [3].

Hemicellulose is the most hydrophilic and thermally labile wood component, cellulose is the other main wood polymer that also provides accessible moisture sorption sites, which starts to degrade at high temperatures [3]. In the past two decades, low oxygen heat treatment processes at 160–240 °C, also known as thermal modification, have been widely researched and successfully industrialized to improve the performance of wood and/or bamboo-based products [3–7]. In general, the heat treatment process can effectively decrease wood hygroscopicity by thermally degrading hemicellulose significantly, and sometimes cellulose at higher temperature than 200 °C [3,4,6]. And the problem of dimensional stability for solid wood products, induced by the change in MC below the fiber saturation point, can be partially resolved [8,9].

Another main benefit of the heat treatment process is that wood durability can be improved [4,10,11]. To optimize the heat treatment process, the response of various wood properties, including hygroscopicity and durability, should be evaluated [4,6]. However,

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the wood decay test is well-known for being a time-consuming procedure that will run for 12 weeks in a laboratory environment and many years in field test [12]. Tjeerdsma et al. [13] pointed out that there is a probable cross-linking reaction in lignin during the heat treatment process, which may be induced by the thermal degradation of hemicellulose, and these reactions could suppress the expansion of micropores in the cell walls when water adsorption occurs. Phuong et al. [14] further suggested that there is a relation between lower hygroscopicity and the physical amount of passage for fungal invasion, thus, a decline in hygroscopicity would have a positive impact on eliminating fungal attacks on wood. Therefore, the change in hygroscopicity for thermally modified wood should be a comprehensive indicator of the responses of chemical components and durability of wood to the heat treatment process, and could serve as an index of heat process effectiveness.

It is generally acknowledged that the adsorption isotherm of wood is a IUPAC type II (sigmoidal) shape, and the sorption sites within wood are the main determinant of moisture adsorption [2]. There are some quick methods that can determine the amount of accessible hydroxyl (–OH) groups within wood, such as hydrogen-deuterium exchange by NMR and DVS [14,15]. As Rautkari et al. [15] reported that, however, there is a poor relation between the amount of accessible hydroxyl groups and the EMC of thermally modified wood. Altgen et al. [16] recently cited this finding in their work. According to Berthold et al. [17], the carboxylic (–COOH) groups are also important sorption sites within wood polymer constituents. The decline in carboxylic groups during the heat treatment process [18] may explain the above finding by Rautkari et al. [15].

Hailwood-Horrobin (H-H) sorption theory was originally developed to analyze the adsorption part of the isotherm [19]. Over the past two decades, the H-H theory has been successfully applied to study the hygroscopicity of modified wood by chemical modification and heat treatment processes [20–24]. The H-H theory suggests that the water within porous hygroscopic materials with a type II adsorption isotherm can consist of both a monolayer and a multilayer of water adsorbed by sorption sites. The equation of the H-H model is as follows [2]:

$$m = \frac{m_0 K_1 K_2 h}{1 + K_1 K_2 h} + \frac{m_0 K_2 h}{1 - K_2 h} \quad (1)$$

where m is EMC or MC at equilibrium with a particular RH and temperature, h is the relative vapor pressure that can be easily obtained from the value of RH (=100 h). K_1, and K_2 are two equilibrium constants related to the adsorption isotherm of the specimen, and m_0 is a critical property related to the hygroscopicity of materials, which indicates the MC corresponding to all available sorption sites in wood are completely occupied by water molecules with a monolayer style [2]. Therefore, the main objective of this study was to apply the H-H theory/model to analyze the moisture adsorption behavior of thermally modified wood, and determine their values of m_0 (total sorption sites). It also aimed to establish a correlation between the m_0 and the durability of thermally modified wood based on the chemical analysis of wood components.

2. Material and methods

2.1. Samples and heat treatment process

Fifteen commercial flat-sawn boards of Poplar wood (*Populus* spp.) at kiln-dried MC (=10–12%) levels were used in this study. The average oven-dried density of these selected Poplar wood boards was 400 kg/m^3. To achieve good comparison of moisture adsorption and durability properties across the different groups, six end-matched wood samples, measuring 30 mm × 50 mm × 250 mm (Radial × Tangential × Longitudinal direction), were carefully cut and machined in the clear area of each board. There were 15 samples in each of the six different groups, which were randomly assigned as a reference to the following five different heat treatment processes at 170 °C, 180 °C, 190 °C, 200 °C, and 210 °C, for a fixed duration of 3 h. Based on the Thermowood process [3] and past research by Li et al. [1], heat treatment experiments were conducted in a small modified laboratory high-temperature drying kiln with superheated vapor. The other process parameters, such as heating and cooling rate, were also fixed in the 5 heat treatment experiments, and Fig. 1 shows the heat treatment schedule at a temperature of 190 °C.

Following the heat treatment process, each sample was first planed and cut into two moisture adsorption specimens of 25 × 50 × 10 (R × T × L) mm^3 and two wood durability specimens of 10 × 5 × 100 (R × T × L) mm^3. The remaining wood was ground with a mill to a homogeneous powder (60–80 mesh) for chemical analysis.

2.2. Durability

At high MC levels, such as in outdoor applications, all three main chemical components of wood (lignin, hemicelluloses, and cellulose) can be attacked by soft-rot fungi [25]. Meanwhile, the environment used for the wood decay test with soft-rot fungi is closer to what might be expected in outdoor applications of wood products [3]. European pre-standard ENV 807 [26] for the resistance of wood to soft-rot fungi was applied to evaluate the durability of different groups of Poplar wood. The soil used in the wood decay test was a composite mixture of soil from household waste and soil with a high activity of soft-rot fungi (mainly *Daldinia concentrica*) and tunneling bacteria, at a ratio of 2:3. It had a pH of 7.7 and a water holding capacity (WHC) of 125%. In the decay test, the MC of the soil was set at about 95% of its WHC and periodically monitored, deionised water was used to adjust the MC.

2.3. Chemical analysis

The lignin content was determined from acid-insoluble Klason lignin involving hydrolysis with 72% sulfuric acid, and cellulose and hemicellulose content were calculated using HPLC analysis to determine the recovered sugar content according to method NREL/TP-510-42618 [27,28]. Two replicates for each heat treatment group were analyzed.

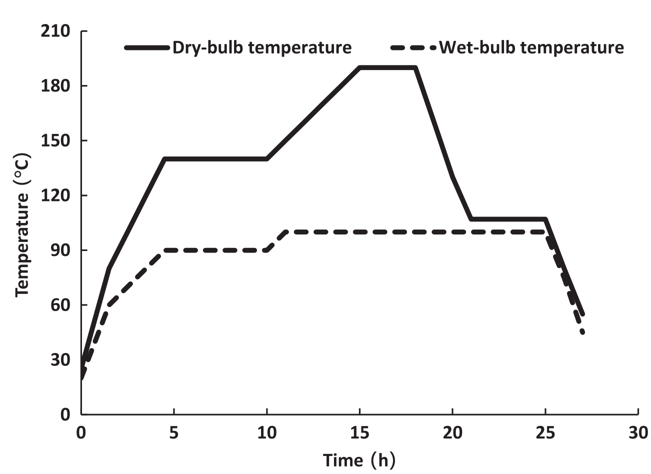

Fig. 1. Schedule for the heat treatment process at a temperature of 190 °C.