# Correlation Analysis between Mass Loss of Wood due to Thermal Modification and Equilibrium Moisture Content of Thermally Modified Wood

Yonggun Park,[a] Yeonjung Han,[b] Yong-Seok Choi,[a] Hyun-Mi Lee,[a] Sae-Min Yoon,[a] Hwanmyeong Yeo,[c,d] and Chang-Deuk Eom [b,*]

This study was conducted to determine the correlation between the mass loss (ML) of wood due to thermal modification and the equilibrium moisture content (EMC) of thermally modified wood. After thermal modification of larch lumber under various temperature and time conditions, ML according to treatment temperature and time was measured, and the (EMC) of the thermally modified wood was evaluated for each treatment condition. As the treatment temperature increased and the treatment time became longer, the ML of wood due to thermal modification increased. In addition, as the treatment temperature increased, the difference in EMC between the non-treated wood and the thermally modified wood tended to increase. Finally, a robust logarithmic correlation was observed between the ML due to thermal modification and the EMC of the thermally modified wood. These results suggest that the EMC of thermally modified wood can be predicted by simply measuring the weight of wood before and after thermal modification.

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*Contact information: a: Division of Wood Engineering, Department of Forest Products and Industry, National Institute of Forest Science, 57 Hoegi-ro, Dongdaemun-gu, Seoul 02455, Republic of Korea; b: Division of Wood Industry, Department of Forest Products and Industry, National Institute of Forest Science, 57 Hoegi-ro, Dongdaemun-gu, Seoul 02455, Republic of Korea; c: Department of Agriculture, Forestry and Bioresources (Department of Forest Sciences), Seoul National University, 1 Gwanak-ro, Gwanak-gu, Seoul 08826, Republic of Korea; d: Research Institute of Agriculture and Life Sciences, Seoul National University, 1 Gwanak-ro, Gwanak-gu, Seoul 08826, Republic of Korea;*
*[*]Corresponding author: willyeom@korea.kr*

## INTRODUCTION

Carbon neutrality has recently emerged as a major global concern. Carbon neutrality entails achieving a balance between carbon emissions and absorptions of carbon; that is, the net emissions of carbon dioxide should be zero. Several countries worldwide are establishing plans to reduce carbon dioxide emissions and enhance absorption to realize carbon neutrality.

Trees are the most important natural carbon absorption sources on Earth, as they can absorb carbon dioxide—the main cause of global warming—through photosynthesis, storing them in the form of carbohydrates. In addition, wood derived from trees is recognized as a representative of natural carbon storage. Therefore, many researchers are actively exploring ways to increase the utilization of wood and wood products to achieve carbon neutrality. However, as wood is an eco-friendly biodegradable material, its use is

often limited by its susceptibility to decay over the long term. Therefore, to extend the longevity of wood products for a long time, various methods, including physical (drying or thermal modification) and chemical methods (preservative-treatment or acetylation), are used (Lee *et al*. 2020; Lee and Kim 2020; Park *et al*. 2020; Ghani and Lee 2021; Park and Park 2021; Priadi *et al*. 2021).

Thermal modification of wood is a process that involves chemically altering the main components of the cell wall by applying high temperatures to the wood, resulting in changes in various physical properties (Militz 2002; Park *et al*. 2014). Since the early 2000s, many studies have been conducted on thermal modification of wood, mainly in Europe (Repellin and Guyonnet 2005; Esteves *et al*. 2008; Yoon *et al*. 2008; Yoon *et al*. 2009; Park *et al*. 2012; Park *et al*. 2014; Cho *et al*. 2015; Park *et al*. 2015; Kim 2016; Park *et al*. 2016; Chung *et al*. 2017; Kang *et al*. 2018; Kim *et al*. 2018; Lee and Lee 2018; Kang *et al*. 2019; Kim and Kim 2019; Cahyono *et al*. 2020; Park *et al*. 2020; Lee and Lee 2021; Schulz *et al*. 2021). Thermal modification of wood is generally performed in a temperature range of 160 to 260 °C when wood undergoes changes where hemicellulose is decomposed and the structure of lignin is denatured. At temperatures below this range, free water in the cell lumen and bound water and extractives in the cell wall are removed from the wood; however, almost no structural changes occur in the cell wall. At temperatures higher than this range, wood undergoes carbonization, leading to the decomposition of cellulose.

As reported in many previous studies, the most representative advantages of thermal modification of wood are the eco-friendly improvements in durability, dimensional stability, and enhanced hydrophobicity. Based on these characteristics, thermally modified wood has been widely utilized for applications in areas requiring durability and dimensional stability, such as in garden tools, fences, columns, outdoor storage cabinets, window frames, doors, decks, exteriors, and floorboards (Boonstra *et al*. 1998; Esteves and Pereira 2009; Kim 2016). Notably, the change in the properties of wood due to thermal modification increases with treatment temperature or treatment time; the greater the modification effect (Esteves and Pereira 2009; Park *et al*. 2014; Park *et al*. 2015; Park *et al*. 2016). Therefore, to produce thermally modified wood with targeted properties, it is essential to establish appropriate temperature and time conditions. Many companies have invested substantial time and resources in trial-and-error approaches to determine the appropriate temperature and time conditions, resulting in the production and sale of many products. However, limited research exists on predicting the properties of thermally modified wood based on conditions such as treatment temperature and time. Theoretical analysis and experimental verification of these aspects are essential to save time and energy in identifying the optimal conditions for thermally modified wood.

In the present study, a model was developed to predict changes in the properties of thermally modified wood to reduce the energy and time consumption required to produce thermally modified wood with the targeted properties. After thermal modification of the larch lumber under various temperature and time conditions, the specimen's mass loss (ML) under each treatment condition (temperature and time) was calculated and set as a variable representing the thermal modification condition. Furthermore, a variable representing the modification effect was established after evaluating the equilibrium moisture content (EMC) of the thermally modified wood for each treatment condition. The correlation between ML and EMC was analyzed, and the regression equation and coefficient of determination were derived.

## EXPERIMENTAL

### Thermal Modification of Wood

For thermal modification, 50 pieces of air-dried larch (*Larix kaempferi*) lumber without defects, such as cracks or splitting, were used. The size of the lumber was 50 mm (thickness) × 100 mm (width) × 2,000 mm (length). The average initial moisture content of the lumber used was approximately 16.5%.

The reactor for the wood thermal modification (Fig. 1C) was manufactured in a cylindrical shape with a diameter of 0.64 m, length of 2.2 m, and total volume of 0.7 $m^3$. An electric heater was installed on the reactor's wall to transfer heat to the wood specimens. The air temperature inside the reactor was set to reach the target temperature within 1 h after heating started, and atmospheric pressure was maintained inside the reactor. Additionally, a thermocouple was inserted into the center of the wood specimen to measure the temperature inside the wood during thermal modification.

To investigate the effect of treatment temperature on thermal modification, the process was conducted under five temperature conditions (130, 160, 190, 220, and 250 °C), and ten pieces of lumber were used for each temperature condition. In addition, to investigate the effect of treatment time on thermal modification, each lumber was divided into 6 pieces in the longitudinal direction to produce test specimens, each 300 mm long (Fig. 1A). Divided test specimens were thermally modified for 0 (non-treatment), 8, 16, 24, 36, and 48 h, respectively. Ten test specimens for the same temperature and time conditions were tied together using wire rope as shown in Fig. 1B to facilitate their removal from inside the reactor. Five bundles, excluding the non-treatment bundle, were placed in the order shown in Fig. 1D. Subsequently, thermal modification was initiated and the bundles were removed from the reactor according to the treatment time.

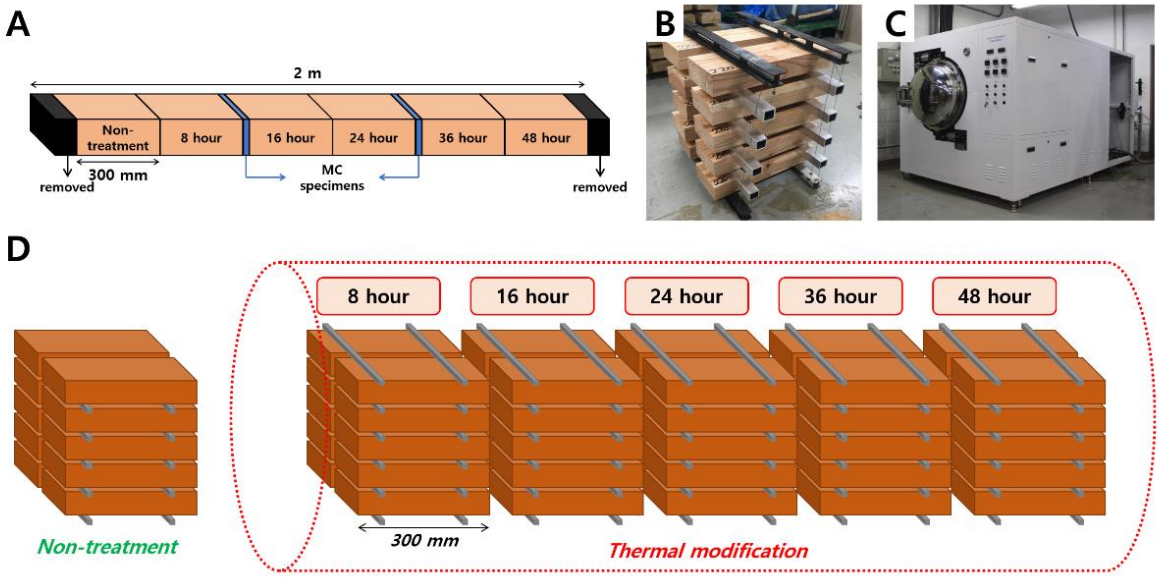



**Fig. 1.** Preparation of wood specimens for thermal modification. A. specimen preparation method for one lumber; B. specimen bundled with wire rope; C. thermal modification equipment; D. specimen bundles stacked in the reactor

### Establishing the Mass Balance of Thermally Modified Wood Depending on Treatment Conditions

Before thermal modification, the test specimens in an air-dried state had some water bound to the wood cell walls. When wood is exposed to a temperature higher than 100 °C, the water inside the wood evaporates, and when the temperature exceeds 160 °C, the main components of the wood cell wall begin to decompose by heat (Esteves and Pereira 2009; Park *et al*. 2017). Assuming that the amount of pure wood substance (the weight of completely dried wood) before thermal modification was 100%, the change in the mass balance of wood substances and water due to thermal modification can be analyzed. The oven-dried weight ($W'_o$) of the test specimen was estimated using Eq. 1, where, $MC_a$ is the average value of the two MC specimens produced from the lumber, and $W_a$ is the air-dried weight of the test specimen measured before thermal modification. The MC of the thermally modified wood was calculated using Eq. 2, using the weight ($W_h$) of test specimen immediately after thermal modification and oven-dry weight ($W_o$) of the test specimen. Furthermore, ML by the thermal modification was determined using Eq. 3, where $W'_o$ is the estimated oven-dry weight of the test specimen, and $W_o$ is the oven-dry weight of the test specimen.

$$W'_o = \frac{W_a}{(1+MC_a/100)} \quad \text{(Eq. 1)}$$

$$\text{MC} = \frac{W_h - W_o}{W_o} \times 100 \quad \text{(Eq. 2)}$$

$$\text{ML} = \frac{W'_o - W_o}{W_o} \times 100 \quad \text{(Eq. 3)}$$

### Establishing the Equilibrium Moisture Content of Thermally Modified Wood Depending on Treatment Conditions

To evaluate the difference in EMC for each treatment condition, a cube test specimen with a side length of 20 mm was extracted from the center of each test specimen, i.e., a total of 10 cube specimens were extracted from each treatment condition. The desorption EMC of the cube specimen was evaluated at 20 °C. All specimens were immersed in distilled water to obtain an MC higher than the fiber saturation point (FSP), which was assumed to be in a green state. After measuring in the green state, the cube specimens were placed in a thermo-hygrostat, and their weights were measured when they reached equilibrium under each relative humidity (RH) condition (90, 80, 65, 50, and 35%). When the weight of the cube specimen was measured every 8 h under each condition, if the rate of weight change was 0.2% or less, the specimen was considered to be in equilibrium.

## RESULTS AND DISCUSSION

### Changes in Temperature Inside the Wood During the Thermal Modification Process

The left side of Fig. 2 depicts the change in the temperature inside the wood and the air temperature in the reactor during the thermal modification process. The air temperature in the reactor reached the target temperature within 1 h, and the temperature inside the wood gradually increased. At the beginning of the process, the temperature of wood was measured slightly differently depending on the location in the reactor; however,

the difference decreased as the process progressed. All wood specimens were evenly thermally modified at the same temperature. In the graph, the sudden drop in the temperature of the wood was caused by the thermocouple being inserted into the wood, reaching the target temperature, and then being separated from the wood and left outside the reactor. Therefore, the internal temperature of the wood is valid only until the target time is reached, after which the value indicates the outside air temperature.

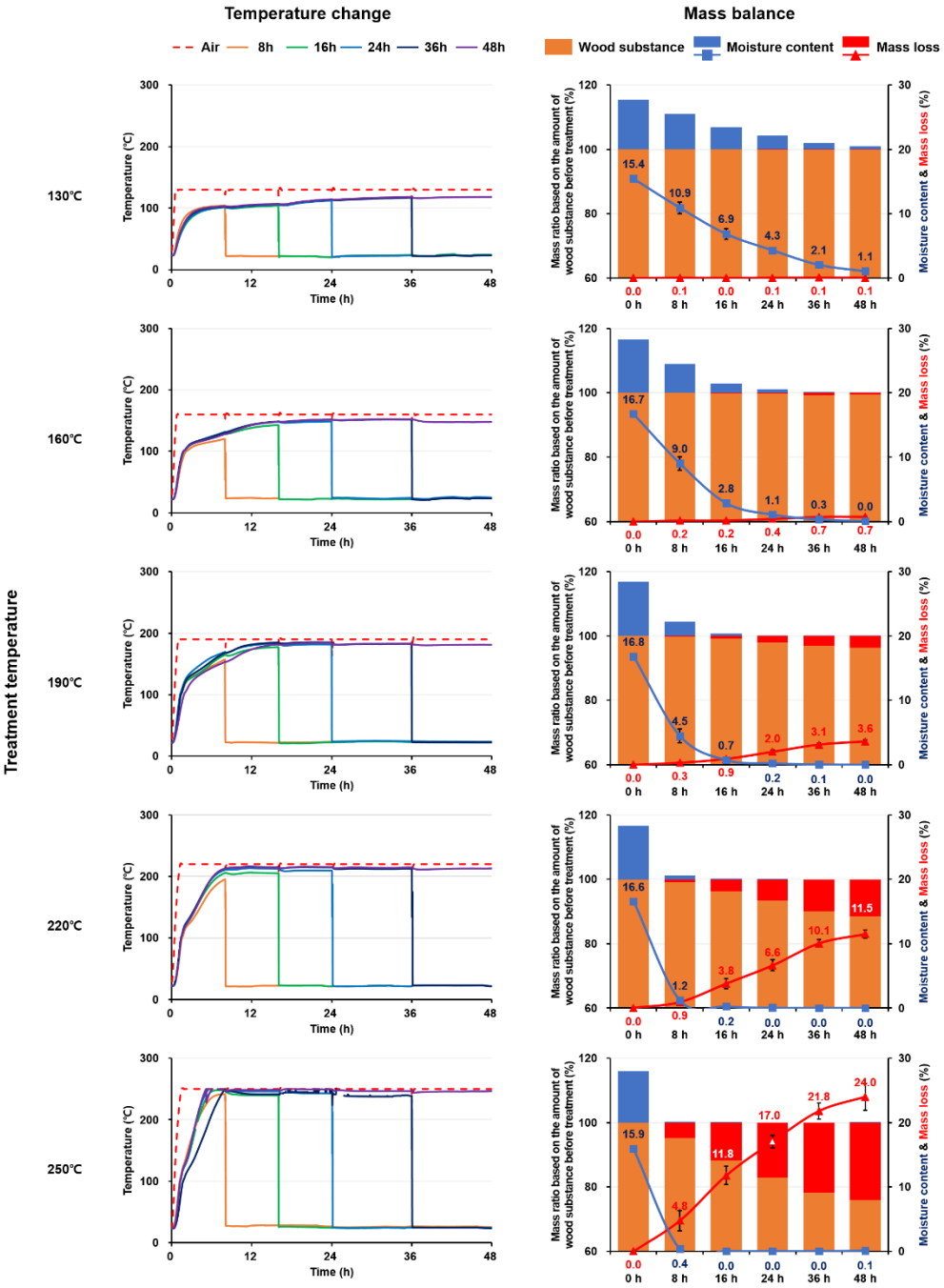



**Fig. 2.** Changes in air temperature (left) and in the mass balance of thermally modified wood (right)

## Evaluation of the Mass Balance of Thermally Modified Wood Depending on Treatment Conditions

The right side of Fig. 2 illustrates the proportion of moisture removal and ML through the weight decrease of wood specimens due to thermal modification, assuming that the mass of the pure wood substance before the process was 100%. When air-dried wood was exposed to temperatures exceeding 160 °C, the moisture inside the wood was removed, and simultaneously, a part of the main component of the wood was thermally decomposed, resulting in weight decrease. According to Park *et al*. (2017), exposing air-dried larch lumber to hot air at 250 °C for 18 hours (with approximately 10 h at a temperature exceeding 160 °C, the maximum internal wood temperature was 220 °C) resulted in significant hemicellulose decomposition and a transformation in the lignin structure. Consequently, an ML of 17.6% occurred, leading to a decrease in the density and strength of wood due to thermal modification.

In this study, during thermal modification at 130 °C, almost no ML occurred even when the wood specimen was treated for 48 h, suggesting that slight change occurs in wood properties at this temperature. In the case of the thermal modification at 160 °C for 48 h, an ML of 3.6% occurred, indicating some changes in wood properties. At a temperature of 190 °C, the ML was noticeably larger. Therefore, an increase in temperature induces changes in the chemical structure, leading to a substantial change in other wood properties. When thermal modification was performed at 250 °C for 48 h, an ML of approximately 24% occurred, indicating a quarter of the main wood components were decomposed and disappeared. Accordingly, the density and strength of the wood may have decreased (Esteves and Pereira 2009; Park *et al*. 2014, 2015, 2016).

## Evaluation of the Equilibrium Moisture Content of Thermally Modified Wood Depending on Treatment Conditions

Figure 3 shows the change in the desorbed EMC of wood that was thermally modified under each temperature condition. Larch wood that was thermally modified at 130 °C exhibited an insignificant difference in EMC according to treatment time under all RH conditions. At 160, 190, and 220 °C, the difference in EMC according to the treatment time was not large in the low RH range (35 to 65%); however, the difference increased significantly in the high RH range (80 to 95%). At 250 °C, the difference in EMC between thermally modified wood and non-treated wood was remarkably large in all RH conditions. Overall, the EMC of the thermally modified wood tended to increase as the temperature and time increased compared to that of the non-treated wood.

During the thermal modification of wood, the heat causes the main components of the wood cell wall to change, resulting in a decomposition of hemicellulose and a transformation in the chemical structure of lignin (Esteves and Pereira 2009; Park *et al*. 2017). Thermally modified wood tends to have a lower EMC (higher hydrophobicity) than non-treated wood because of the decrease in the number of hydroxyl groups (-OH) in the cell wall (Esteves and Pereira 2009; Park *et al*. 2015; Kim 2016; Park *et al*. 2016). In this study, the ML also increased as temperature and time increased, and the EMC tended to decrease accordingly. The difference in EMC between non-treated wood and thermally modified wood was not similar in all RH conditions and was larger at high RH (90%).

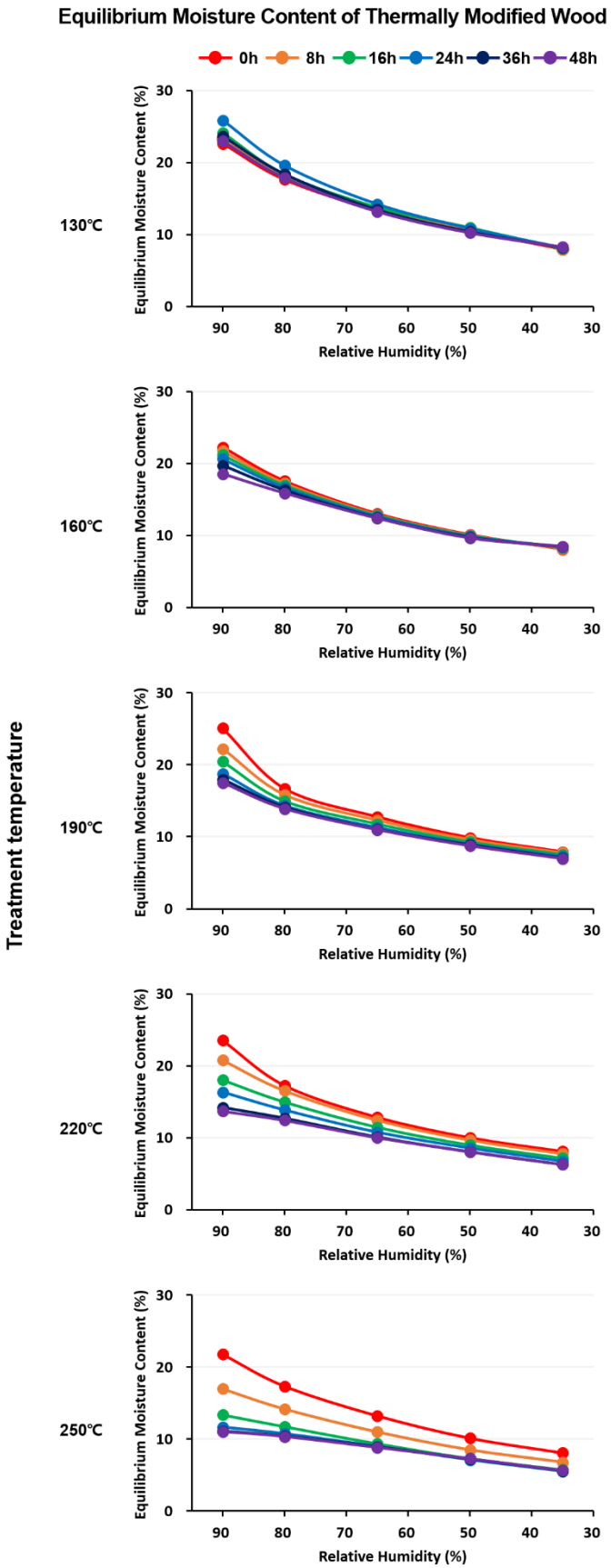


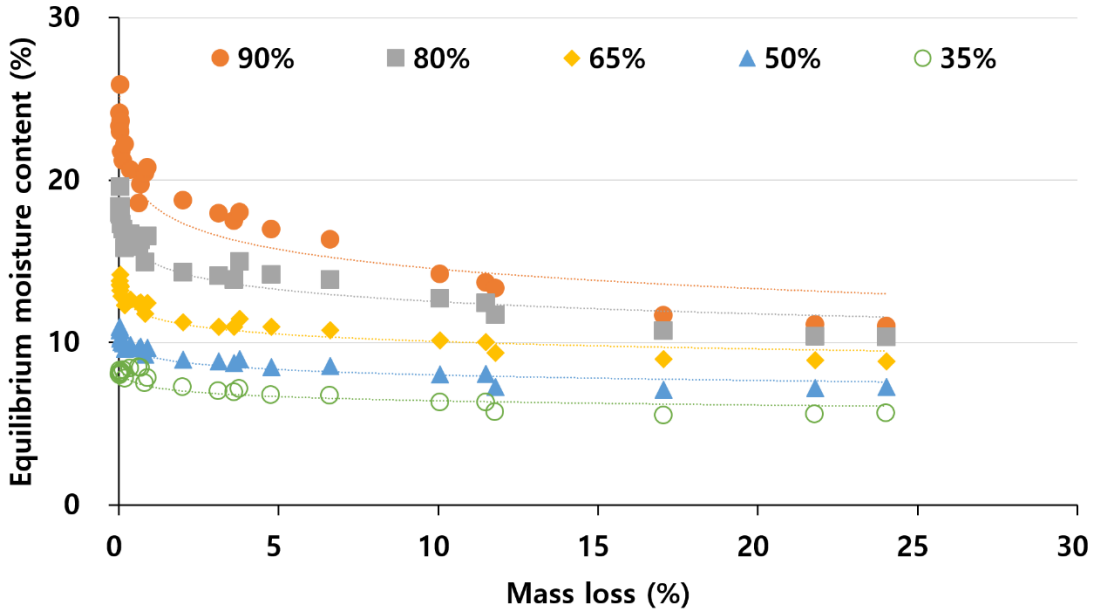

**Fig. 3.** Desorbed equilibrium moisture content of thermally modified wood

According to the classification of bound water types depending on the RH and MC, under conditions of high RH, it is affected by capillary condensed water (Kang *et al*. 2008; Yang 2013). Therefore, the lower EMC of wood that was thermally modified at high temperature compared to that of non-treated wood at high RH can be attributed to the absence of capillary condensed water. This absence is likely attributable to the negative effect of the structural change in the cell wall, induced by high temperatures, on capillary condensed water formation.

## Correlation Analysis Between Mass Loss and Equilibrium Moisture Content in Thermally Modified Wood

Thermal modification causes chemical changes in the constituent cells of wood, which manifest as ML. Some physical properties of wood are affected by these chemical changes, with EMC being a representative example. The experimental results of this study indicated that the higher the treatment temperature and the longer the treatment time for thermal modification, the greater the change in the physical properties of the wood. This result aligns with expectations based on several previous studies (Esteves and Pereira 2009; Park *et al*. 2012, 2014, 2015; Kim 2016; Park *et al*. 2018, 2020).

In addition, changes in the major chemical components of wood due to thermal modification exhibit a close relationship with the physical properties of thermally modified wood.




| Relative humidity | Regression equation | Coefficient of determination ($R^2$) |
|---|---|---|
| 90% | $-1.75\ \ln(x) + 18.56$ | 0.90 |
| 80% | $-1.08\ \ln(x) + 15.00$ | 0.90 |
| 65% | $-0.66\ \ln(x) + 11.59$ | 0.91 |
| 50% | $-0.48\ \ln(x) + 9.12$ | 0.91 |
| 35% | $-0.38\ \ln(x) + 7.28$ | 0.76 |

**Fig. 4.** Correlation between mass loss and equilibrium moisture content of the thermally modified wood

In the graph shown in Fig. 4, the *x*-axis is the ML for each treatment condition (right side of Fig. 2), which represents the chemical change in wood due to thermal modification, and the y-axis is the EMC for each treatment condition, which represents the change in physical properties of the thermally modified wood. Regression analysis of various models to derive the correlation between the ML and the EMC confirmed a robust log correlation. Except for the 35%RH condition, all $R^2$ values exceeded 0.9. In other words, the EMC of thermally modified wood can be predicted by simply measuring the weight of the wood before and after thermal modification. Furthermore, the application of this study to physical properties other than EMC may enable the prediction of these properties.

## CONCLUSIONS

1. Mass loss (ML) increases with increasing temperature and time of thermal treatment of wood.
2. As the temperature for thermal modification increased, the difference in equilibrium moisture content (EMC) between thermally modified wood and non-treated wood tended to increase.
3. The difference appeared considerably larger under high relative humidity (RH) conditions than under low RH conditions, which was attributed to structural changes in cells induced by thermal modification, which negatively affect the formation of capillary condensed water.
4. A robust logarithmic correlation was observed between the ML and EMC of thermally modified wood specimens.
5. The EMC of thermally modified wood can be predicted by simply measuring the weight of the wood sample before and after the thermal modification.
6. By applying this study to other physical properties, it may be possible to predict various properties of the wood following its pretreatment.

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