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Studying the Impact of Heat Treatments and Distance from Pith on the Sorption Behavior of Tree of Heaven Wood (*Ailanthus altissima* (Mill.) Swingle)

Fanni Fodor ¹, Lukas Emmerich ^{2,3}, Norbert Horváth ¹, Róbert Németh ^{1,*} and Tamás Hofmann ^{4,*}

¹ Institute of Wood Technology and Technical Sciences, Faculty of Wood Engineering and Creative Industries, University of Sopron, Bajcsy-Zsilinszky St. 4, H-9400 Sopron, Hungary; fodor.fanni@uni-sopron.hu (F.F.); horvath.norbert@uni-sopron.hu (N.H.)

² Team Wood-Based Industries, Centre of Forest and Wood Industry (FBV), Wald und Holz NRW, Carlsau Str. 91a, D-59939 Olsberg, Germany; lukas.emmerich@wald-und-holz.nrw.de

³ Wood Biology and Wood Products, Faculty of Forest Sciences and Forest Ecology, University of Goettingen, Bûsgenweg 4, D-37077 Göttingen, Germany

⁴ Institute of Environmental Protection and Nature Conservation, Faculty of Forestry, University of Sopron, Bajcsy-Zsilinszky St. 4, H-9400 Sopron, Hungary

* Correspondence: nemeth.robert@uni-sopron.hu (R.N.); hofmann.tamas@uni-sopron.hu (T.H.)

Abstract: The application of tree of heaven (*Ailanthus altissima* (Mill.) Swingle) is constrained by its poor durability and dimensional stability. Despite exhibiting promising physical and mechanical properties comparable to ash wood (*Fraxinus excelsior* L.), it is regarded as an invasive species and receives limited attention in wood property enhancement research. This study subjected tree of heaven to heat treatment at 180 °C and 200 °C to investigate its sorption characteristics using dynamic vapor sorption tests. The results revealed a 13% reduction in equilibrium moisture content at 95% relative humidity and 25 °C after thermal modification at 180 °C and a 25% reduction after thermal modification at 200 °C. Increasing the treatment temperature lowered the moisture content ratio to 0.76 and shortened the conditioning time by up to 10%. The highest hysteresis, ranging from 3.39% to 3.88%, was observed at 70% relative humidity.

Keywords: thermal modification; ailanthus; tree of heaven; dynamic vapor sorption; moisture sorption; hysteresis



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1. Introduction

Water vapor sorption is a critical property of wood, influencing dimensional stability, mechanical strength, durability, heat capacity, thermal conductivity, and corrosiveness [1].

Sorption isotherms depict the equilibrium moisture content (EMC) of wood relative to relative humidity (RH) at a specific temperature (T). An absorption isotherm shows an increase in EMC from an oven-dry state close to the cell wall saturation as RH increases. Conversely, a desorption isotherm illustrates drying to a 0% EMC (oven-dry state). Both isotherms are influenced by previous RH exposure, with scanning curves representing isotherms collected along varying RH paths. Desorption curves begin from almost cell wall saturation at 100% RH, while scanning desorption isotherms initiate from 95% RH [2].

Dynamic vapor sorption (DVS) analyzers expose small wood samples to controlled streams of dry and saturated air, regulated by mass flow controllers. Precisely controlling temperature and RH, these instruments measure wood sample mass to rapidly and accurately generate sorption isotherms, replacing labor-intensive manual equilibration of the moisture conditions along wooden specimens at a corresponding RH level [1].

DVS data are commonly fitted with the Parallel Exponential Kinetics (PEK) model; however, this model does not capture the actual form of the sorption curve and the physics of the sorption process, so it is not physically meaningful [1].

Wood normally shows an absorption isotherm with an IUPAC type II (sigmoidal) shape, and it can be characterized with the knowledge of the sorption sites and absorption of hydroxyl groups [3–5]. They decline due to heat treatment, so the moisture absorption isotherm of heat-treated wood changes accordingly [6,7].

While many papers still use water vapor sorption isotherm models (e.g., Guggenheim Anderson DeBoer, Dent, Hailwood-Horrobin), it has been discussed whether those may correctly capture thermodynamic quantities [8]. They may provide a good fit to the DVS data, but, on the other hand, they do not yield and consider physically meaningful parameters of the wood cell wall [1].

The present research focused on the wood samples from tree of heaven (*Ailanthus altissima* (Mill.) Swingle), which is native to China, but can be found all over the world, except for Antarctica. In 2019, it was reported that there were 7142 ha covered by tree of heaven in Europe [9]. This wood species is commonly utilized as firewood rather than for wooden products (material use) due to its susceptibility to fungal decay, insect damage, blue stain, cracks, and warping. Its limited durability and dimensional stability have traditionally constrained its application, specifically in outdoor applications, while it demonstrates promising physical, mechanical, and product-related properties [10–17], comparable to ash wood (*Fraxinus excelsior* L.) [12,13,18–20]. Recent studies have extensively reviewed its potential utilization [15,21,22].

Recognized for its rapid growth, short felling time, and invasive nature, tree of heaven tree spreads easily and exhibits high reproducibility. However, obtaining high-quality logs and utilizing the available wood amount remains challenging [23]. The wood typically features an average annual ring width of 4–5 mm, with potential widths extending to 17 mm [24].

Various wood modification techniques, such as heat treatment [25,26], heat treatment in oil [27], impregnation modification [28], and alkali treatment [29], have been explored with promising outcomes, enhancing the resistance to moisture and microorganisms, and improving mechanical strength while reducing hygroscopicity [30,31].

According to the pertinent literature, heat treatment reduces accessible hydroxyl groups, equilibrium moisture content (EMC), hysteresis, and moisture sorption rates [7,32–34]. Heat-treated wood consistently exhibits a lower EMC compared to untreated wood under similar climatic conditions [35]. Given the chemical variations between annual rings closer to and farther from the pith, moisture-related properties may vary accordingly [36]. Extensive literature reviews were published recently on the hygroscopicity of heat-treated wood [7,37]. Thus, thermal modification of wood from tree of heaven wood could open up its application for outdoor furniture, paneling, decking, poles, and even façade claddings [38,39].

This study conducted dynamic vapor sorption tests on various parts of tree of heaven wood to investigate the impact of thermal modification at 180 °C and 200 °C on its moisture-related properties.

2. Materials and Methods

2.1. Wood Material

The tested wood material was from a 13-year-old tree of heaven log with a breast height diameter of 13 cm, originating from the Botanical Garden of Sopron (Sopron, Hungary). The middle, quarter-sawn board was taken from it, a part of it was spared as a control, and the other part was taken for heat treatment. The width of the annual rings were 7mm on average. The juvenile and mature wood was used as well [40].

2.2. Heat-Treatment

The wood was heat-treated at the University of Sopron (Hungary) in an insulated chamber, having an internal volume of 0.4 m³. In this equipment, internal air heating is applied with two pairs of ribbed, U-shaped electric heating wires with a power of 750 W each. These are separated from the heat-treatment area by a steel plate which is located approximately 10–15 cm from the back wall. The air circulation is provided by two pieces

of aluminum fans with a diameter of 23 cm, placed above the heater. The temperature was set by a PT100 thermometer and a Siemens control unit. The removal of decomposition products and gases was ensured by the pressure difference [40].

The experimental thermal treatments were carried out under atmospheric conditions, in an open system, without added steam or injected water. These “dry heat treatments” were performed with schedules at 180 °C and 200 °C temperatures and 10 h durations. These temperatures indicate the temperature of the atmosphere in the chamber, not the wood itself. During the treatment, the drying chamber was heated to 100 °C in the first 5 h, then it was heated to 130 °C for 7 h. In the next phase, the temperature was set to 180 or 200 °C in one hour, and it was held for 10 h. Then, it was cooled back to 20 °C in 28 h [40].

Following heat treatment, the equilibrium moisture content (EMC) and density were determined at a normal climate (20 °C, 65% RH). The average EMC was 13.6% for untreated tree of heaven wood. It was reduced by heat treatment at 180 °C to 7.2%, and at 200 °C to 4.8%. The average density of tree of heaven wood was 641 kg/m³, which decreased by thermal modifications at 180 °C to 604 kg/m³, and at 200 °C to 591 kg/m³ [19].

Three samples were taken from each investigated annual ring from each material with dimensions of 7 × 20 × 30 mm (rad × tan × lon) [40] (Figure 1).



Figure 1. Sampling from different annual rings of the tested wood material starting with “1” at the pith and increasing numbers to the outer sections of the tree. Cross section of untreated tree of heaven wood. Annual rings in brackets were analyzed by dynamic vapor sorption tests.

2.3. Dynamic Vapor Sorption Tests

Samples were taken from the 5th, 9th, and 13th annual rings of control and heat-treated wood, which were milled in a cutting mill (Retsch SM 2000, Retsch GmbH, Haan, Germany). The particles were passed through a 2 mm mesh screen in order to homogenize the material sample.

Circa 20 mg of wood particles were placed on a sample holder of the DVS apparatus (DVS Advantage, Surface Measurement Systems, London, UK) and sorption isotherms were recorded. The temperature was constant at 25 °C and the nitrogen flow was 200 sccm (sccm = standard cubic centimeter at 0 °C).

First, the samples were dried at 0% RH to a constant weight, until the mass change of the specimen per minute (dm/dt) was less than $0.002\% \text{ min}^{-1}$ over a period of 10 min. Following this, RH levels were incrementally increased (10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, and 95%) to generate an absorption curve.

After this, the scanning desorption curve was established by decreasing the RH to 0% RH stepwise in the reverse order. A window of 10 min was used to calculate the dm/dt and each RH was maintained until the dm/dt was $<0.002\% \text{ min}^{-1}$ for >10 min. The moisture content (MC, %) was determined by dividing the mass of absorbed water by the dry wood mass, using the sample mass at the end of each RH step. The moisture content ratio (MC_R) was calculated by relating the treated MC to the corresponding reference MC at each RH step.

3. Results and Discussion

Both untreated and heat-treated wood showed typical for wood sigmoidal shaped sorption curves. The moisture uptake of wood was faster in the initial phase, then it slowed down until reaching the EMC at 95% RH. It has a logarithmic function [32]. By gradually

increasing the relative humidity, the moisture content increases, which is attributed to the abundant hydroxyl groups and cell wall swelling. In the case of heat-treated samples, the EMC decreased further as a result of increased treatment temperature during the modification process, which occurred independent of the RH level observed. By that, the sorption isotherm become flatter and showed an even more linear slope in case of thermal modifications at elevated temperature (Figure 2). The latter can be attributed to heat-induced degradation, which reduces the hemicellulose fraction and by that the availability of free sorption sites (OH groups), and increases the percentage amount of crystalline cellulose fractions related to the samples mass. Consequently, the number of accessible hydroxyl groups is reduced by the thermal modification, leading to a lower moisture content levels and sorption processes itself. The cell wall also becomes stiffer as chemical changes and degradation take place in lignin which may further and irreversibly affect the EMC at certain RH levels [41]. The EMC of the untreated samples was always higher compared to heat-treated wood. These results are in accordance with the previous findings dealing with the moisture-related properties of TMT tree of heaven wood [25,26].

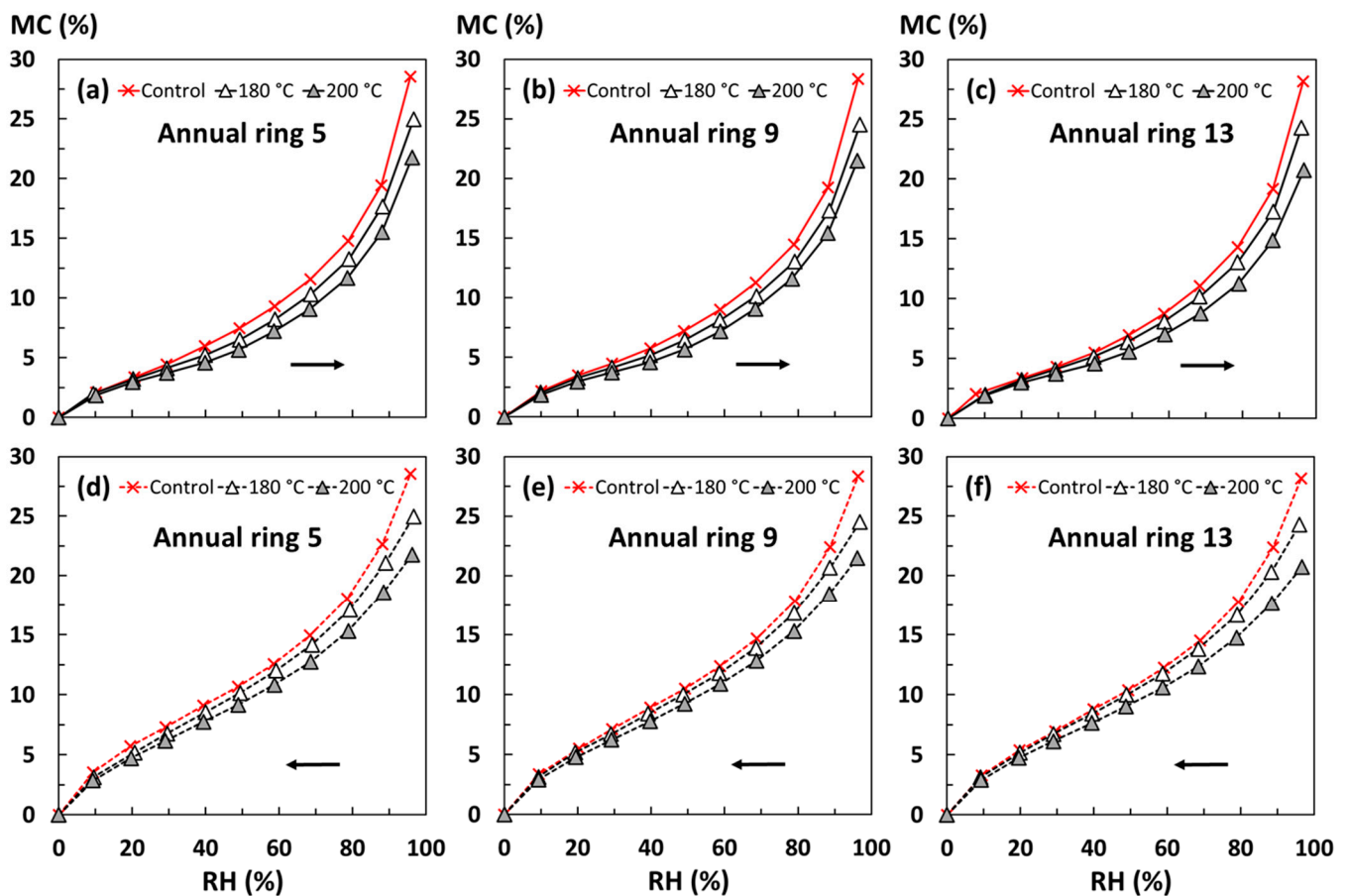


Figure 2. Comparison of the moisture content values of untreated (red), heat-treated at 180 °C (white triangle), and heat-treated at 200 °C (grey triangle) wood from 0 to 95% relative humidity. Samples taken from the 5th (a,d), 9th (b,e), and 13th (c,f) annual ring are compared as well. Absorption (a–c) and desorption (d–f) results are compared, showing the direction of moisture change with a black arrow.

Below 80% relative humidity, the EMC increment and decrement of the tested materials varied between 0 and 4% at every RH step. After heat treatment, these values were lower compared to those of untreated wood, but only by less than 1%. Above 80% relative humidity, a considerable jump in EMC was found, ranging from 4 to 9% at every RH step.

After heat treatment, these values were even lower compared to untreated wood, by a maximum of 3.12%.

The EMC, which resulted from exposure of tree of heaven wood to a 95% RH moisture regime was slightly higher than reported in the literature [42]. On average, it decreased from 28.50% to 24.76% (a 13% reduction) at a treatment temperature of 180 °C, and to 21.50% (a 25% reduction) at 200 °C (Figure 2).

Since wood with fewer annual rings has a higher ratio of juvenile wood, a greater moisture content, and a different chemical composition, samples with fewer annual rings exhibited higher EMCs and FSPs in all cases (Figure 3, Table 1).

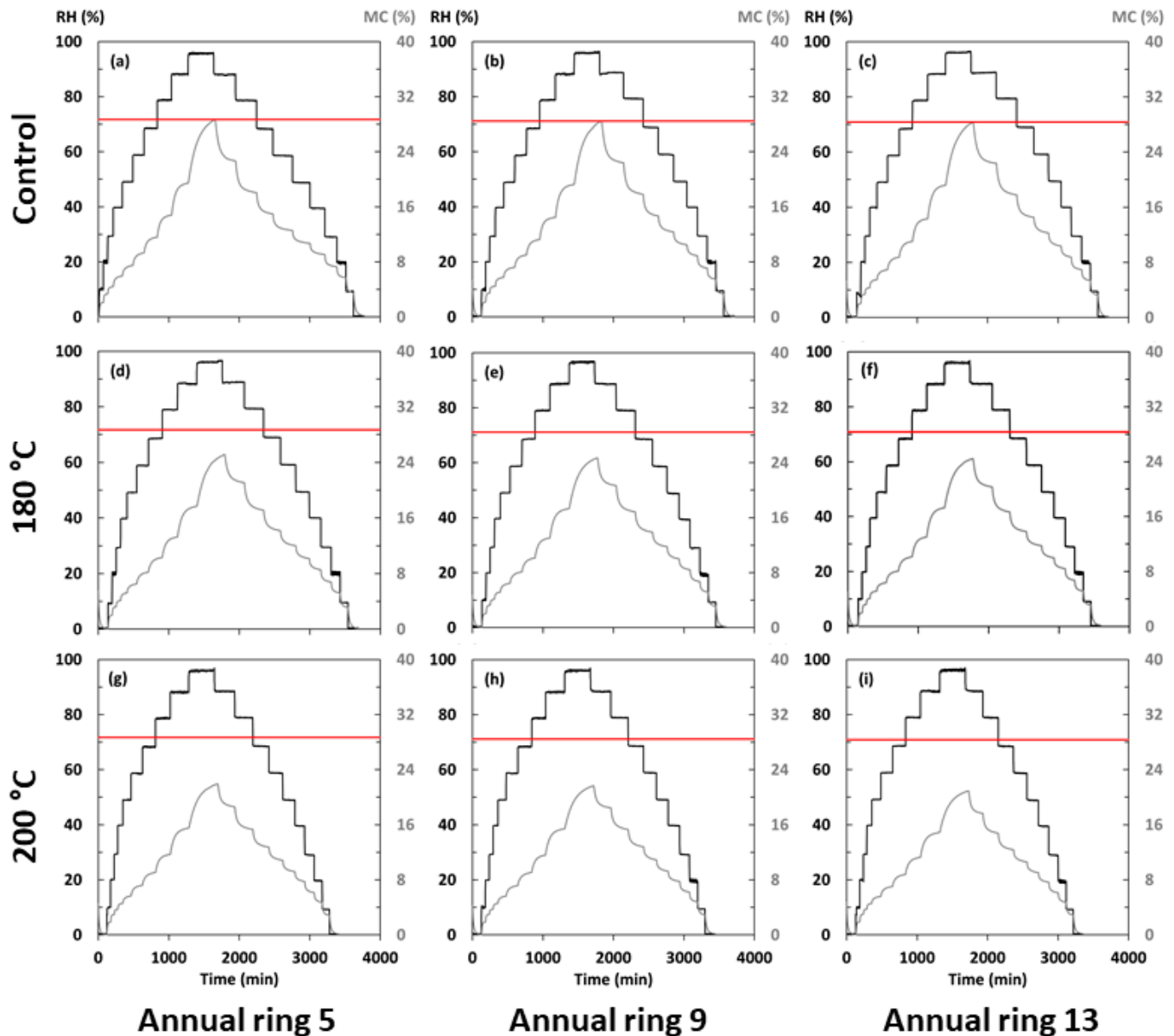


Figure 3. Moisture content (%) development (grey curve) under stepwise changes in relative humidity (%) (black curve) depending on the time of exposure in a DVS apparatus. Control (a–c), heat-treated at 180 °C (d–f) and heat-treated at 200 °C (g–i) samples were taken from the 5th, 9th, and 13th annual ring, and the moisture content at the 95% RH of the corresponding control samples is expressed by the continuous red line.

Table 1. Comparison of DVS data between untreated (control) and heat-treated (180 °C, 200 °C) samples, which were taken from the 5th, 9th, and 13th annual ring: maximum equilibrium moisture content ($EMC_{95\%RH}$), time to reach equilibrium moisture content ($t_{95\%RH}$), time to reach half value of the equilibrium moisture content ($t_{EMC/2}$), time required to reach 10% moisture content ($t_{10\%MC}$).

Temperature	Annual Ring	$EMC_{95\%RH}$ (%)	$t_{95\%RH}$ (min)	$t_{EMC/2}$ (min)	$t_{10\%MC}$ (min)	Total Running Time (min)
Control	5	28.69	1656	922	661	3774
	9	28.46	1830	1077	787	3712
	13	28.35	1792	1081	776	3711
180	5	25.14	1791	977	804	3689
	9	24.70	1771	954	826	3596
	13	24.44	1774	970	849	3592
200	5	21.92	1692	872	831	3411
	9	21.67	1717	896	860	3441
	13	20.92	1724	887	865	3349

The conditioning time, the time needed to reach a given EMC at a given RH, was reduced by the heat treatments (Table 1), and it was shorter when a higher treatment temperature was applied, which corresponds to related research [30]. The total conditioning time decreased by a maximum of 10%. Samples originating from annual rings closer to the pith took longer times to be conditioned compared to those that were farther from the pith.

It should be noted that the initial dry mass and density, which may influence the moisture content of the wood, will always be lower for heat-treated samples due to heat-induced degradation processes, which furthermore lead to chemical and physical changes of the cell wall structure.

The peak in absolute hysteresis generally occurs around 75% RH [43], aligning with the findings seen in Figure 4. The hysteresis increased gradually at each RH step, reaching its maximum at around 70% RH. After that, it began to decrease. This phenomenon is due to the cell wall matrices, which try to relax and go back to the state they were before during adsorption. This is harder as the treatment temperature goes above 140 °C, and the changes become irreversible [44]. The highest hysteresis value was 3.39–3.52% for untreated tree of heaven wood, and higher after heat treatment (3.60–3.88%), with ranging values depending on the annual ring origin and treatment temperature.

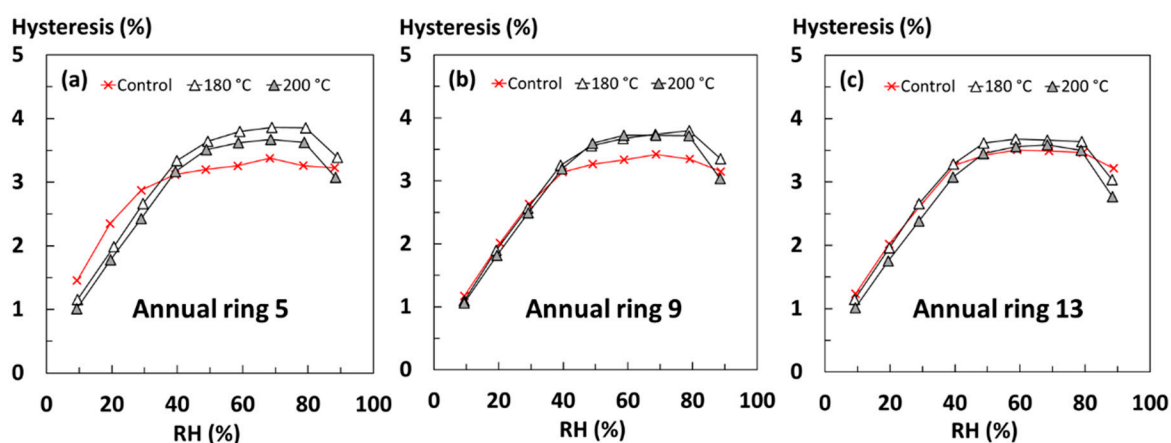


Figure 4. Comparison of the hysteresis values of untreated (red), heat-treated at 180 °C (white triangle), and heat-treated at 200 °C (grey triangle) wood from 10 to 95% relative humidity. Samples taken from the 5th (a), 9th (b), and 13th (c) annual ring are compared as well.

As a result of the EMC reduction following heat treatment, the moisture content ratios were below 1 and decreased as a result of increasing the treatment temperature during

the modification process (Figure 5). For the 5th and 9th annual rings, a minimum in the absorption curve and a maximum in the desorption curve were observed at 50–60% RH. These moisture content ratio values were lower in the 5th annual ring. The 13th annual ring exhibited no peaks, only a continuous reduction during both adsorption and desorption, reaching a minimum value at 90% RH. The lowest moisture content ratio, ranging from 0.76 to 0.78, was recorded for the 200 °C treatment, indicating the highest EMC reduction compared to untreated wood.

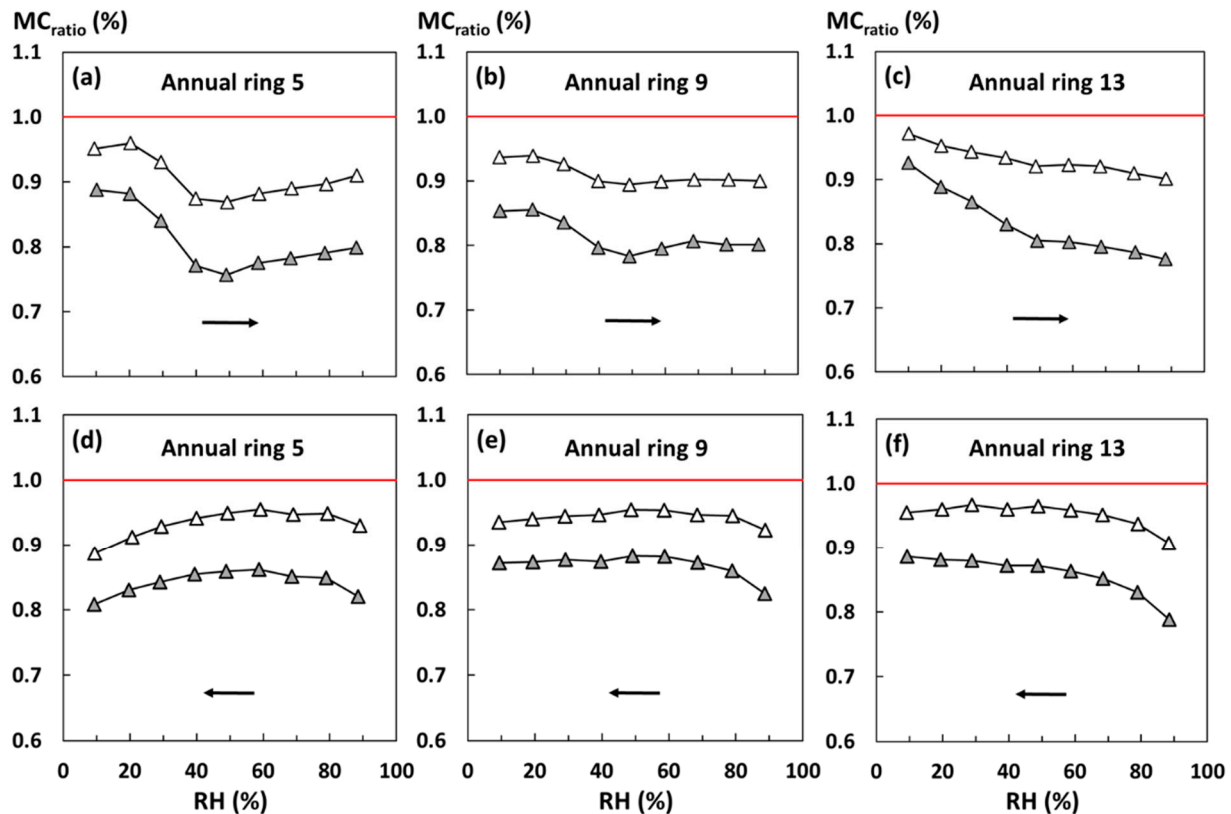


Figure 5. Diagrams comparing the moisture content ratios (MC_{ratio}) of heat-treated tree of heaven wood. Two treatment temperatures are compared: 180 °C (white triangles) and 200 °C (grey triangles). Samples were taken from the 5th, 9th, and 13th annual ring. Adsorption (a–c) and desorption (d–f) results are compared, showing the direction of moisture change with a black arrow.

The moisture content values of the annual rings showed no remarkable differences, and the average air-dry density was likewise unaffected by the annual rings, aligning with previous research findings. Comparing the treatment temperatures, the anti-swelling efficiency increased by 19–26% at 180 °C, and it was even higher, increasing by 32–44%, at 200 °C [19,40].

DVS parameters could indicate the intensity of thermal modification and the THT material quality [32]. A realistic model describing the sorption isotherm and sorption hysteresis loop observed for water vapor interactions with wood has not yet been defined [37].

4. Conclusions

Heat treatment was carried out on tree of heaven wood of Hungarian origin. The treatment temperatures were 180 and 200 °C with 10 h long durations. Dynamic vapor sorption tests were executed for sorption isotherm analysis. The samples were taken from different parts (annual rings) of the wood.

Corresponding to the literature, the heat-treatment at 180 °C and 200 °C resulted in a reduction in equilibrium moisture content. The EMC at 95% RH and 25 °C decreased by 13% as a result of the 180 °C thermal modification and 25% after modification at 200 °C.

Comparing the samples from different annual rings, those that were taken from parts closer to the pith tended to higher EMCs at a given RH than those that lay farther from the pith. Nevertheless, the impact of the sample position, expressed as distance from the pith, showed quite small impacts on the resulting EMC, while the dominant impact originated from the treatment temperature of the thermal modification. By increasing the treatment temperature, the sorption isotherm became flatter and the moisture content ratio was reduced. The conditioning or equilibrium time was shorter for samples treated at a higher temperature, and for samples originating farther from the pith. The treatment temperature of 200 °C influenced the moisture sorption-related characteristics more compared to 180 °C. At 70% relative humidity, the highest hysteresis was exhibited.

In the future, sorption isotherm model analysis could be used to further evaluate the presented results. Durability tests should be performed to assess the ability of this wood species to withstand exposure to biological organisms and weathering after undergoing a thermal modification process.

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