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Róbert Németh, Christian Hansmann, Holger Militz, Miklós Bak, Mátyás Báder

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Thermal modification affects the dynamic vapor sorption of tree of heaven wood (*Ailanthus altissima, Mill.*)

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ABSTRACT
The utilization of tree of heaven wood is hindered by its low durability and dimensional stability. Although it has promising physical and mechanical properties similar to ash wood, it is considered as an invasive wood species, and it is rarely the base of wood property improvement research. In this study, heat treatment was carried out on this species at 180°C and 200°C to evaluate the sorption characteristics using dynamic vapor sorption tests. This way, the wood-water relationships, the rate of sorption, the reduction in moisture content could be analyzed and assumptions could be made on the change of moisture-related properties.

INTRODUCTION
Water vapor sorption is one of the most important characteristics of wood, as it affects many of its properties like dimensional stability, mechanical properties, durability, heat capacity, thermal conductivity, and corrosion (Zelinka et al. 2021).
When drawing a (water vapor) sorption isotherm, the equilibrium moisture content (EMC) of wood can be observed as a function of relative humidity (RH) at a given temperature (T). There is an absorption isotherm, where the EMC increases from oven-dry state to fiber saturation point (FSP) by increasing the humidity. There is also a desorption isotherm, where the wood is dried to 0% EMC. The sorption isotherm and the EMC depend also on the previous RH history. Scanning curves are those isotherms, which have been collected along different RH path history. Here, the desorption curves start from a fully water-saturated state with 100% RH. Those isotherms, which start from 95% RH, are scanning desorption isotherms (Fredriksson and Thybring 2018).
In dynamic vapor sorption (DVS) analyzers, small wood samples are exposed to dry and saturated streams of air, the RH of which is controlled by mass flow controllers. As the temperature and RH are tightly controlled, the mass data of the wood sample gives the sorption isotherms precisely and faster and with less labor than measuring the mass manually after equilibrating the samples at each RH level (Zelinka et al. 2021).
The base of this research was tree of heaven wood (*Ailanthus altissima* MILL. SWINGLE). It is usually used as firewood, as it is prone to fungal decay, insect damage, blue stain, cracks and warps. Although its low durability and dimensional stability hinders its usage, it has promising physical, mechanical and product-related properties (bonding, coating, manufacturing). Its colour and properties are comparable to ash wood (*Fraxinus excelsior*). Extensive literature review has been carried out on the utilization potential of this wood species recently (Terzopoulou et al. 2023).
It is considered to be an invasive species, it has a short felling time, and logs of high-quality are rare. It is fast-growing, it spreads easily and has high reproducibility. The utilization of the existing wood amount is a task to be resolved (Bartha 2020).

Using wood modification techniques, its resistance to moisture and microorganisms could be improved. In the scientific literature on the modification of this wood species, heat treatment (Barboutis and Kamperidou 2019), heat treatment in oil (Bak and Németh 2015), impregnation modification (Miao et al. 2014) were carried out with promising results like higher mechanical strength and lower hygroscopicity.

In this research, dynamic vapor sorption tests were carried out on different parts of heat-treated tree of heaven wood (*Ailanthus altissima*) to see how does thermal modification at 180° and 200° affect the wood-water relations in this underutilized wood species with low dimensional stability and durability. Based on relevant literature, heat-treatment will probably decrease the equilibrium moisture content, the hysteresis, and the rate of moisture sorption compared to untreated wood (Humar et al. 2020; Jiang et al. 2024). As the chemical components are different in annual rings closer to the pith compared to the outer part of the wood, the results may also vary in moisture-related properties (Akgul and Tozluoglu 2009).

MATERIALS AND METHODS

Material

One tree of heaven log was acquired from the Botanical Garden of Sopron (Hungary). The middle boards were taken from them, and they were cut to three pieces. These pieces were sawn to 30 by 30 mm sticks. The sticks from the middle part were marked as control, and the sticks from the sides were heat-treated (Gyuricsek 2015).

Heat-treatment

The samples were heat-treated at the University of Sopron (Hungary) in an insulated chamber, having an internal volume of 0.4 m^3. 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 diameter of 23cm, 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 (Gyuricsek 2015).

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 hours duration. 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 hours, then it was heated to 130°C for 7 hours. In the next phase, the temperature was set to 180 or 200°C in one hour, and it was held for 10 hours. Then, it was cooled back to 20°C in 28 hours (Gyuricsek 2015).

After heat treatment, the equilibrium moisture content (EMC) and density were determined at 20°C temperature and 65% relative humidity. The average EMC decreased from 13.6% to 7.2% for the 180°C treatment, and to 4.8% for the 200°C treatment. The average density decreased from 641kg/m^3 to 604 kg/m^3 for the 180°C treatment, and to 591 kg/m^3 for the 200°C treatment (Gyuricsek et al. 2014).

Samples of 7 × 20 × 30 mm (thickness × width × length) were produced from the one control and two heat-treated pieces from each annual ring corresponding to the anatomical directions (Gyuricsek 2015).

Dynamic Vapor Sorption tests

For DVS measurements, small-sized untreated and treated wood specimens from the 5th, 9th and 13th annual rings were milled in a cutting mill (RETSCH SM 2000, Retsch GmbH, Haan, Germany) to pass through a 2 mm mesh screen. Sorption isotherms were recorded in a DVS apparatus (DVS Advantage, Surface Measurement Systems, London, UK). Approximately 20 mg of wood particles were placed on a sample holder of the DVS and sorption isotherms were recorded at a constant temperature of 25 °C and a nitrogen flow of 200 sccm (sccm = standard cubic centimeter at 0 °C). The samples were first dried at 0 % RH until the mass change of the specimen per minute (dm/dt) was < 0.002 % min^{-1} over a period of 10 min.

Afterwards, the RH was increased stepwise in the following sequence: 10, 20, 30, 40, 50, 60, 70, 80, 90, and 95 % RH (absorption curve), which was followed by a decrease to 0 % RH in the reverse order (scanning desorption curve). A window of 10 min was used to calculate the dm/dt and each RH was maintained until the dm/dt was < 0.002 % min^{-1} for > 10 min. The moisture content (MC, in %) was calculated by relating the mass of water to the dry mass of the wood samples, using the sample mass at the end of each RH step. The MC_R ratio was calculated by relating the MC_R of the modified sample to the corresponding reference MC at each RH step.

RESULTS AND DISCUSSION

The sorption isotherms and hysteresis can be observed in Figure 1 and Figure 2. According to our results, heat treatment did decrease the moisture sorption of tree of heaven wood.

By increasing the treatment temperature, the EMC decreased more, and the sorption isotherm became flatter. This can be associated with heat-induced degradation, where the hemicellulose fraction decreases and cellulose crystallinity increases, which reduces the amount of accessible hydroxyl groups, which lead to lower moisture content.

The peak in absolute hysteresis is usually around 75% RH (Vahtikari et al. 2017), which corresponds to our results. The highest hysteresis was found at about 70% RH, which was lower for the control samples (3.39 - 3.52%) than for the heat-treated samples (3.60 - 3.88%).

The fiber saturation point of tree of heaven was slightly higher than related literature (Kržišnik et al. 2020). It decreased on average from 28.33% to 24.57% at 180°C, and to 21.31% at 200°C treatment temperature.

As the wood with less annual rings has higher juvenile wood ratio, higher moisture content and different chemical composition, the samples with less annual rings had higher EMC in every case. For example, the fiber saturation point of samples from the 5th annual ring was 28.54%, 24.94% and 21.74% with no treatment, treatment at 180°C, and 200°C, respectively. The FSP of samples in the 9th and 13th annual rings was lower, less than 28.32%, 24.51% and 21.48% with no treatment, treatment at 180°C, and 200°C, respectively.

Consequently to the EMC reduction after heat-treatment, the moisture content ratios were below 1, and decreased with elevated temperature (Figure 3). In the case of the 5th and 9th annual ring, there is a minimum in the adsorption curve and a maximum in the desorption curve at 50-60% RH. These moisture content ratio values are lower in the 5th annual ring. The 13th annual ring did not show any peak, just continuous reduction during both adsorption and desorption, with a minimum value at 90% RH. The lowest moisture content ratio was found for treatment 200°C having the highest reduction in EMC compared to untreated wood.

Corresponding to related research on this material, there was no significant difference between the moisture content values of the annual rings, and the average airdry density was also not influenced by the annual rings. On the other hand, the anti-swelling efficiency improved, it was 19-26% for 180°C treatment and 32-44% for 200°C treatment (Gyuricsek et al. 2014; Gyuricsek 2015).

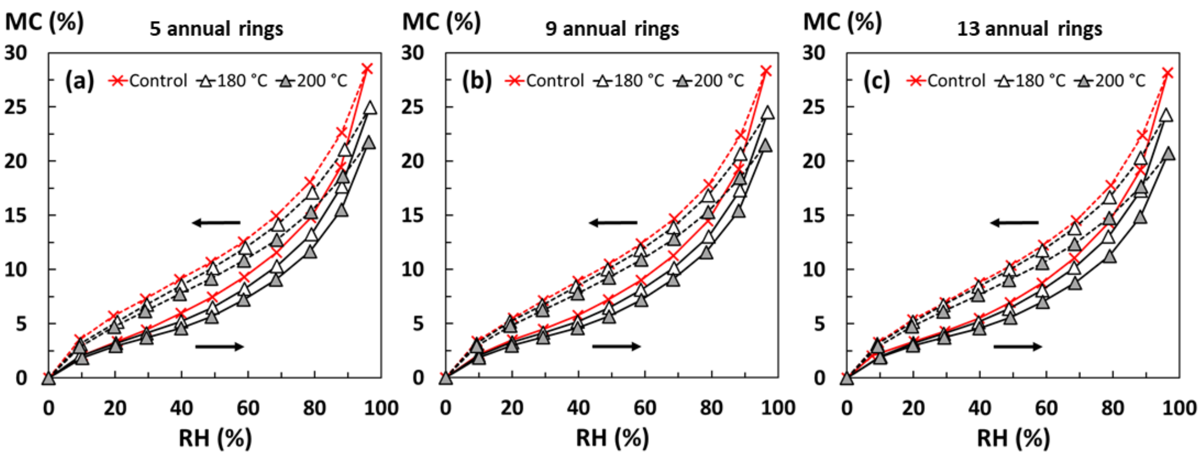

Figure 1: Sorption isotherms of untreated (Control) and heat-treated tree of heaven wood at 180°C and 200°C. Samples were taken from annual rings 5, 9 and 13. The change of moisture content (MC) is plotted in the function of relative humidity (RH).

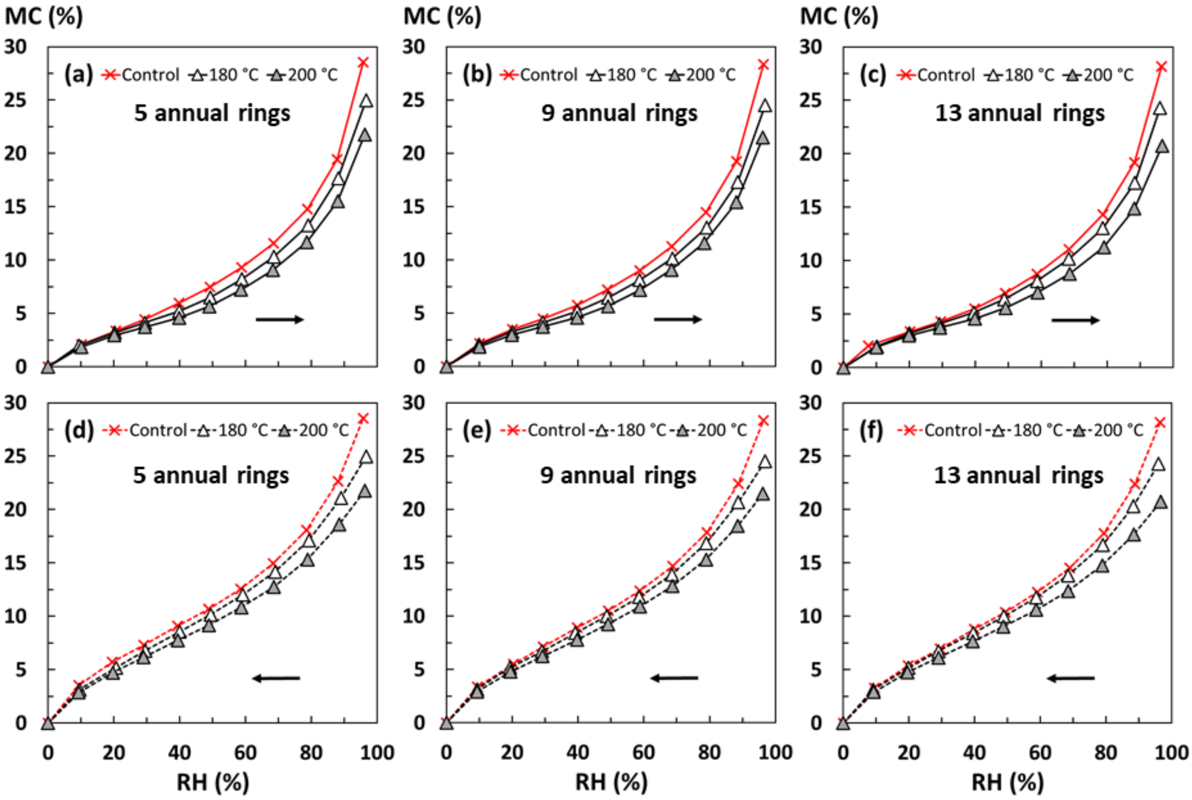

Figure 2: Adsorption and desorption curves of untreated (Control) and heat-treated tree of heaven wood at 180°C and 200°C. Samples were taken from annual rings 5, 9 and 13. The change of moisture content (MC) is plotted in the function of relative humidity (RH).

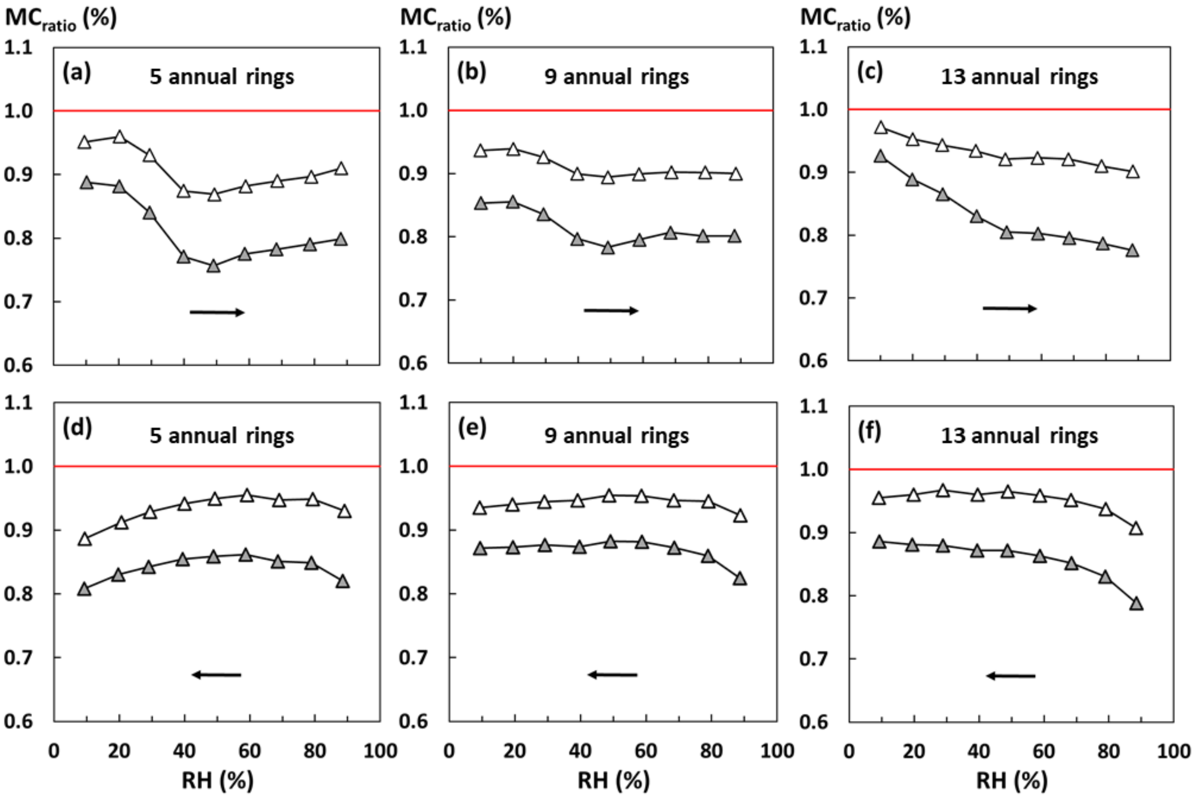

Figure 3: Moisture content ratios (MC_{ratio}) of heat-treated tree of heaven wood at 180°C (white triangles) and 200°C (grey triangles). Samples were taken from annual rings 5, 9 and 13. The arrows indicate the direction of moisture change, the upper row shows the adsorption results and the bottom row the desorption results.

CONCLUSIONS

The sorption isotherms of untreated and heat-treated tree of heaven wood were analyzed, taking into account which annual rings were they taken from. Heat-treatment resulted in the reduction of EMC, flatter sorption isotherm, and lower moisture content ratio. Samples from annual rings closer to the pith had higher EMC. Treating at 200°C had higher impact on the sorption properties than 180°C. Highest hysteresis was found at 70% RH. In the future, durability tests could be carried out to see how this species can withstand the exposure to biological organisms and weathering after heat treatment.

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REFERENCES

Akgul M, Tozluoglu A (2009) Some Chemical and Morphological Properties of Juvenile Woods from Beech (Fagus orientalis L.) and Pine (Pinus nigra A.) Plantations. Trends in Applied Sciences Research 4:116–125. https://doi.org/10.17311/tasr.2009.116.125

Bak M, Németh R (2015) Crack formation during Oil-Heat-Treatment in relation with the initial moisture content. In: Proceedings of the 8th European Conference on Wood Modification. Helsinki, pp 334–337

Barboutis I, Kamperidou V (2019) Impact of Heat Treatment on the Quality of Tree-of-Heaven Wood. Drvna industrija 70:351–358. https://doi.org/10.5552/drvind.2019.1842

Bartha D (2020) Black List. Invasive tree and shrub species of Hungary. University of Sopron Press, Sopron

Fredriksson M, Thybring EE (2018) Scanning or desorption isotherms? Characterising sorption hysteresis of wood. Cellulose 25:4477–4485. https://doi.org/10.1007/s10570-018-1898-9

Gyuricsek T (2015) Bálványfa fatestének komplex faanyagtudományi vizsgálatai (Complex analysis of xylem of Tree of Heaven). Bachelor thesis, University of West Hungary

Gyuricsek T, Horváth N, Németh R (2014) Effect of heat treatments on selected properties of Tree-of-Heaven (Ailanthus altissima). In: Proceedings of the “IAWS Plenary Meeting 2014 - Sopron (Hungary) - Vienna (Austria)” - Eco-Efficient Resource Wood with Special Focus on Hardwoods. Sopron & Vienna, 2014. (14) 15-18th September. University of West Hungary Press, Sopron, pp 63–64

Humar M, Repič R, Kržišnik D, et al (2020) Quality Control of Thermally Modified Timber Using Dynamic Vapor Sorption (DVS) Analysis. Forests 11:666. https://doi.org/10.3390/f11060666

Jiang X, Van den Bulcke J, De Ligne L, Van Acker J (2024) Biological durability and moisture dynamics of untreated and thermally modified poplar. Eur J Wood Prod. https://doi.org/10.1007/s00107-023-02033-3

Kržišnik D, Humar M, Maks M (2020) The dynamic water vapour sorption behaviour of the invasive alien species in Slovenia. In: 9th Hardwood Proceedings Part I. With Special Focus on “An underutilized Resource: Hardwood Oriented Research.” University of Sopron Press, Sopron, pp 149–152

Miao X, Chen H, Lang Q, et al (2014) Characterization of Ailanthus altissima Veneer Modified by Urea-formaldehyde Pre-polymer with Compression Drying. BioResources 9:5928–5939. https://doi.org/10.15376/biores.9.4.5928-5939

Terzopoulou P, Kamperidou V, Barboutis I (2023) Utilization Potential of Tree-of-Heaven Species Biomass—A Review. Applied Sciences 13:9185. https://doi.org/10.3390/app13169185

Vahtikari K, Rautkari L, Noponen T, et al (2017) The influence of extractives on the sorption characteristics of Scots pine (Pinus sylvestris L.). J Mater Sci 52:10840–10852. https://doi.org/10.1007/s10853-017-1278-0

Zelinka S, Thybring EE, Glass S (2021) Interpreting Dynamic Vapor Sorption (DVS) Measurements: Why Wood Science Needs to Hit the Reset Button. In: Proceedings of the World Conference on Timber Engineering. Santiago, p EPFT0101