

SORPTION PROPERTIES OF WOOD IMPREGNATED WITH THE FIRE RETARDANT BURNBLOCK

SORPCIJSKE LASTNOSTI LESA, IMPREGNIRANEGA Z OGNJEZADRŽEVALNIM SREDSTVOM BURNBLOCK

Miha Humar^{1*}, Boštjan Lesar¹, Davor Kržišnik¹

UDK 630*843:630*812.7

Original scientific article / [Izvirni znanstveni članek](#)

Received / [Prispelo](#): 21. 12. 2021

Accepted / [Sprejeto](#): 14. 2. 2022

Abstract / [Izvleček](#)

Abstract: The sorption properties of wood have a characteristic influence on some of its properties, such as the mechanical properties and susceptibility to fungal decay. Moist wood is more susceptible to fungal decay, and wood is often impregnated in order to protect it from fungal decomposition, photodegradation or fire. In particular, inorganic salts affect the sorption properties of wood. For this purpose, the sorption properties of Norway spruce wood impregnated with Burnblock refractory (uptake 38 kg/m³) were investigated. The microscopic analysis confirmed the presence of crystals of this in the cell lumina of wood tissue. Sorption properties were determined using an instrument capable of dynamic vapour sorption (DVS) assessment. DVS analysis confirmed that the sorption properties of impregnated spruce wood are comparable to those of non-impregnated spruce wood. However, the higher hysteresis at higher relative humidity is probably due to the presence of crystals in the cell lumina.

Keywords: fire retardants, Burnblock, wood, sorption properties, laser confocal microscopy

Izvleček: Sorpcijske lastnosti lesa imajo značilen vpliv na nekatere njegove lastnosti. V največji meri vplivajo na mehanske lastnosti in dovzetnost lesa na glivni razkroj. Vlažnejši les je bolj dovzeten za glivni razkroj, zato ga pred glivnim razkrojem, fotodegradacijo ali gorenjem pogosto impregniramo. Predvsem anorganske soli vplivajo na sorpcijske lastnosti lesa. S tem namenom smo preiskali sorpcijske lastnosti smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock (navzem 38 kg/m³). Mikroskopska analiza je potrdila prisotnost kristalov sredstva v celičnih lumnih lesnega tkiva. Sorpcijske lastnosti smo določili z opremo, ki vrednoti dinamično sorpcijo vodne pare (DVS). DVS analiza je potrdila, da so sorpcijske lastnosti impregniranega smrekovega lesa primerljive s sorpcijskimi lastnostmi neimpregnirane smrekovine. Večja histereza pri višjih relativnih zračnih vlažnostih je verjetno posledica prisotnosti kristalov anorganskih soli, ki so se izločili v celičnih lumnih po impregnaciji.

Ključne besede: ognjezadrževalna sredstva, Burnblock, les, sorpcijske lastnosti, laserska konfokalna mikroskopija

1 INTRODUCTION

1 UVOD

Wood is hygroscopic due to its specific chemical composition and large internal surface area. Therefore, the moisture content of wood oscillates depending on the varying climatic conditions. Under stable conditions, wood reaches hygroscopic equilibrium or equilibrium moisture content (EMC). The interactions between wood and water have been studied scientifically for more than a century (Engelund et al., 2013). The moisture content

of wood has a significant effect on some relevant properties, especially the mechanical properties (Gerhards, 1982) and service life of wood used outdoors (Meyer et al., 2016). Fungi can decompose wood if the moisture content is above a certain limit. The moisture content of wood must be high enough to promote the flow path for the reaction products of the enzymes, but low enough to prevent waterlogging. A wide variety of data on this is available in the literature. In the first set of data, it is indicated that the MC limits for fungal decay de-

¹ Univerza v Ljubljani, Biotehniška fakulteta, Jamnikarjeva 101, 1000 Ljubljana, SLO

* e-mail: miha.humar@bf.uni-lj.si

pend predominantly on the fungal species. For example, Schmidt (2006) reported that the minimum MC of wood was 25% for *Coniophora puteana* and *Serpula lacrymans* and 30% for *Fibroporia vaillantii* and *Gloeophyllum trabeum*. However, recent data suggest that the limiting moisture content for fungal growth depends on the wood species and fungal species studied. For example, the limiting moisture content for *C. puteana* growth on thermally modified Scots pine (*Pinus sylvestris* L.) sapwood is 12.1%, while the limiting moisture content for fungal decay on the same species of wood is 24.4% (Meyer et al., 2016).

Similar to wood-decaying fungi, MC has a significant effect on the growth and development of sap stain fungi. Sap stain fungi are mainly associated with *Ascomycetes* and *Fungi imperfecti*, and are characterised by the pigmentation of the hyphae walls, which leads to discolouration of the wood. Suitable conditions for the growth and reproduction of the various mould and sap stain fungi vary. Some thrive at relatively low air relative humidity (RH = 75%), while most fungi require higher RH levels and consequently higher wood MC for optimal growth. Different building materials have different susceptibilities to mould growth (Isaksson et al., 2010).

The relationship between the EMC and RH is expressed by sorption isotherms obtained by progressive equilibration in the adsorption or desorption process. Differences in hygroscopic and sorption isotherms result from the wood species, chemical composition of the wood, the amount of microcracks in the cell walls, density, possible hydrothermal and chemical treatment, and stress conditions (Hartley et al., 1992; Willems, 2018).

Sorption isotherms can be divided into three regions. The first represents the EMC from an absolutely dry state to the equilibrium state reached at RH between 20% and 30% (Mitchell, 2018). In this interval, the adsorption of water molecules continues gradually until the outer surface of the cell wall is completely covered by a water monolayer. The wood MC changes more rapidly in the upper part of the region, but slows as it approaches a dry state (Lesar et al., 2009). The second region begins when the first layer is saturated. The adsorption of water molecules on the first layer and the resulting formation of additional layers is a feature of

this region, and the isotherms here are quasi-linear (Mangel, 2000). In the third region, capillary condensation of water occurs in microcapillaries. Water molecules form large groups, while the bonds between hydroxyl groups and the first layer of water molecules become weaker, and thus the water molecules can move in clusters (Khali & Rawat, 2000). The water concentration in this region is sufficient for liquid water to form in the pores by capillary condensation, so the microcapillary water forms a continuous phase. In the third hygroscopic region, sorption properties are also influenced by low-molecular secondary heartwood compounds such as polyphenols (flavonoids, lignans, tannins), biocides (boric acids) and fire retardants (Blahovec & Yanniotis, 2008).

As mentioned earlier, the hygroscopic properties of wood can be affected by various treatment processes, like the use of wood biocides and fire retardants. Wood impregnated with various inorganic salts is usually more hygroscopic than untreated wood, especially at high RH. The increase in EMC of such wood depends on the chemicals used, retention, and wood species (White & Dietenberger, 2010). The EMC of impregnated wood and the effects of preservative retention on the equilibrium point are still unknown. High EMC is problematic because it promotes leaching of active ingredients, corrosion of metals, and the creation of favourable conditions for the growth of fungi and especially moulds, and presents difficulties in surface treatment and gluing of moist wood (Lesar et al., 2009). In this study, the sorption properties of wood impregnated with the fire retardant Burnblock were investigated throughout the hygroscopic range during the adsorption and desorption process.

2 MATERIAL AND METHODS

2 MATERIALI IN METODE

The analysis was carried out on wood treated with fire-retardant, and specifically on Norway spruce (*Picea abies* (L.) Karst.) planks treated with Burnblock (Burnblock, København, Denmark) in a commercial impregnation plant using the full cell impregnation method. Five planks were delivered. The cross-section of the planks was approximately 23 mm × 100 mm and length 200 mm. The retention of Burnblock was 38 kg/m³. Burnblock is made



Figure 1. Cross-section of spruce wood plank, used for microscopic and sorption analysis.

Slika 1. Prečni prerez deske, uporabljene za mikroskopsko in sorpcijsko analizo.

of ingredients that can be found in nature and are considered environmentally friendly. Treated wood is biodegradable and has no adverse environmental effects (Medved et al., 2019). Five parallel samples were conditioned at laboratory conditions (21 °C; RH 65%), then measured and weighted. The nominal density of the wood was then calculated.

Microscopic analysis was performed on cross-sections of the treated wood. The outer 6 mm of the wood that was fully impregnated with the fire retardant was analysed. Microscopic analysis was performed using a confocal laser scanning microscope (Olympus OLS50-BSW, Tokyo, Japan) and a digital microscope (Olympus DSX1000, Tokyo, Japan). The surface was planed with a stainless steel blade. The MC of the wood was approximately 12%.

Dynamic water vapour sorption of treated and native (i.e. reference, non-treated) samples was performed using a gravimetric dynamic sorption analyser (DVS Intrinsic, Surface Measurement Systems Ltd., London, UK). Samples were ground and homogenised into fractions smaller than 1 mm prior to analysis using a SM 2000 mill (Retsch GmbH, Haan, Germany) and a perforated sieve with a perforation of 1 mm (Conidur™). The ground samples were conditioned at 20 °C and $1 \pm 1\%$ RH. A small amount of the ground sample (≈ 400 mg) was used. The measurement was performed at a constant temperature of 25 ± 0.2 °C. A total of two sorption and desorption cycles were measured from 0% RH to 95% RH, and vice versa.

3 RESULTS AND DISCUSSION

3 REZULTATI IN RAZPRAVA

The cross-sections of the wood planks indicate the typical structure of Norway spruce wood. The annual rings are about 2 mm to 3 mm wide (Figure 1). The anatomical structure (Figure 2) shows the gradual transition between earlywood and latewood cells. The respective resin canals are bordered by 8 to 12 or more thick-walled epithelial cells (Wagenführ, 2014). The density of the air-dry planks examined was 420 kg/m^3 (st. dev. 17 kg/m^3). This is in line with the data in the literature (Gryc et al., 2011; Humar, 2013).

As seen from microscopic analysis (Figure 3, Figure 4), Burnblock crystals are seen in the cell lumina. The presence of the crystals was confirmed using two independent microscopy techniques, confocal laser scanning microscopy and digital microscopy. The presence of the crystals in the cell lumina is not surprising, as the retention of Burnblock and other fire-retardants is higher than the retention of wood preservatives. For example, the retention of typical copper-ethanol wood preservatives is about 20 kg/m^3 (Nordic Wood Preservation Council 2021) (for in-ground use), while the retention of classical CCA barely reaches 12 kg/m^3 (Willeitner, 2001). The crystals in the cell lumina are rather significant. It can be assumed that they were at least partially damaged during cutting.

In the graphs (Figure 5), the sorption curves of the untreated and treated spruce wood are plotted. As can be seen, both the untreated and

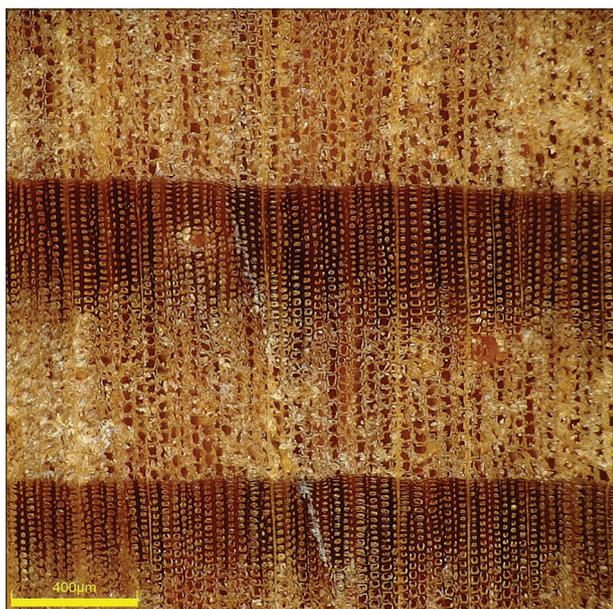


Figure 2. Annual ring of spruce wood plank
Slika 2. Branika v lesu smrekove deske

Burnblock-treated wood show typical sorption isotherms of type II. The differences between the EMC at 95% RH of untreated and treated wood are negligible. For example, in the first sorption cycle, the EMC of untreated spruce wood (23.09%) is slightly higher than the EMC of Burnblock-treated

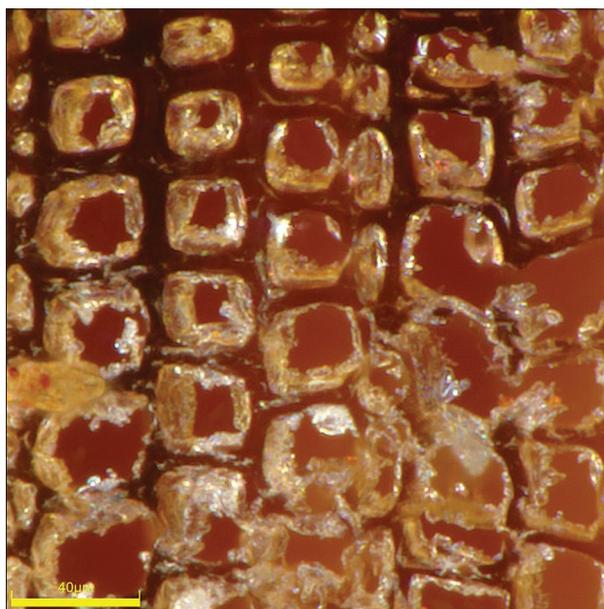


Figure 3. Cell lumina of all cells are filled with crystals of the fire retardant Burnblock. Microscopy was performed with a digital microscope. Colours are not always representative.

Slika 3. Mikroskopska slika, ki prikazuje zapoljenost vseh celičnih lumnov z ognjezadrževalnim sredstvom Burnblock. Slika je posneta z digitalnim mikroskopom. Barve niso vedno reprezentativne.



Figure 4. Cell lumina filled with crystals of the fire retardant Burnblock. The image was obtained with confocal scanning laser microscope (field of view $128 \mu\text{m} \times 128 \mu\text{m}$). Colours are not always representative.

Slika 4. Celični lumni, zapolnjeni s kristali ognjezadrževalnega sredstva Burnblock. Slika je bila posneta s konfokalnim laserskim vrstičnim mikroskopom (vidno polje = $128 \mu\text{m} \times 128 \mu\text{m}$). Barve niso vedno reprezentativne.

wood (22.79%). However, in the second sorption cycle, the EMC of Burnblock-treated wood was slightly higher (23.70%) than that of untreated spruce wood (22.47%). Normally, the second EMC at 95% RH for lignocellulosic materials is lower than the first (Glass et al., 2018), but in this case it was slightly higher for the Burnblock-treated sample.

As DVS analysis was performed in controlled conditions it enables a reliable comparison, but statistical analysis was not performed due to low number of measurements (Glass et al., 2018).

The interpretation of the sorption curves is that the surfaces of the analysed wood samples are more polar than water molecules, and therefore

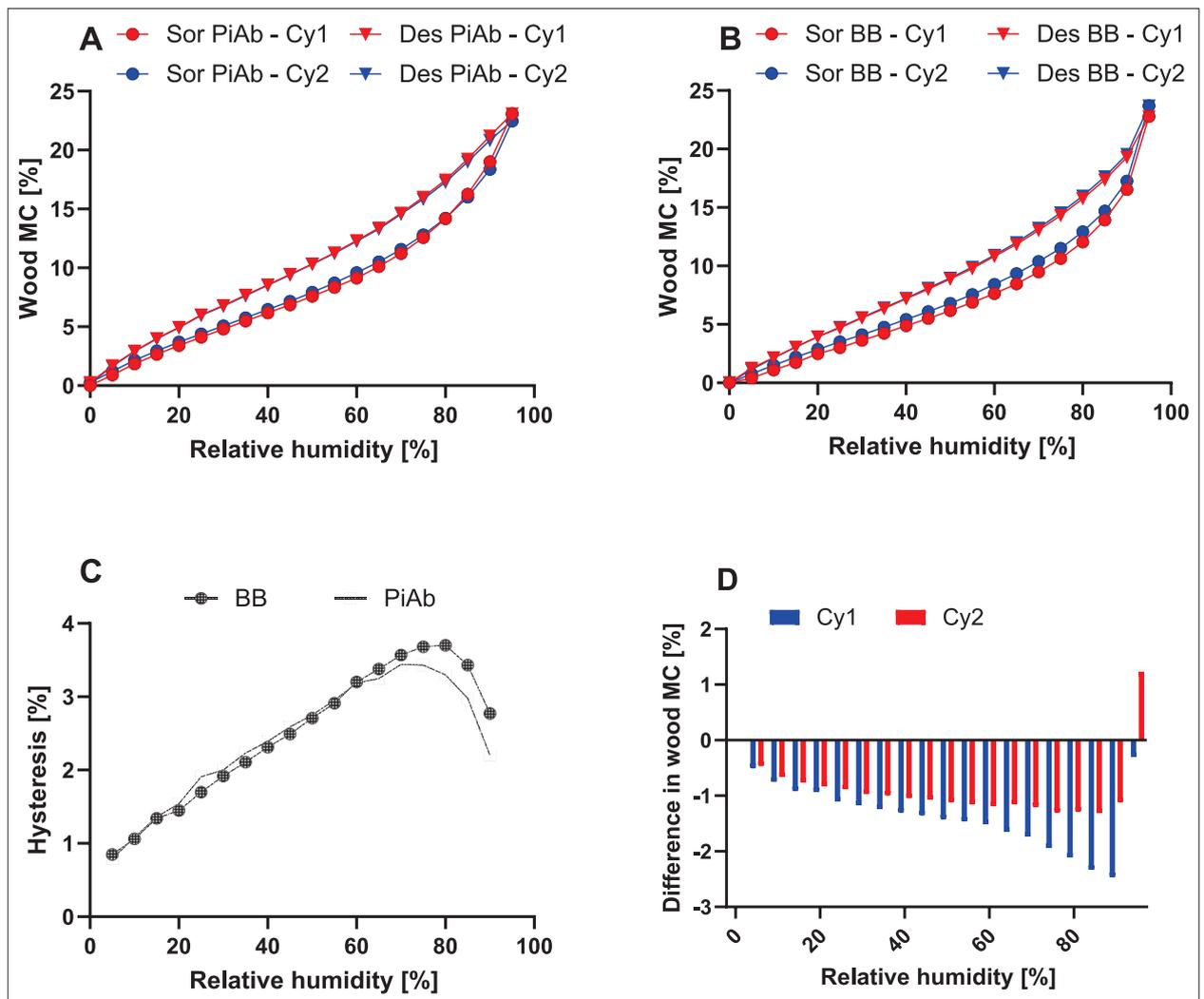


Figure 5. Results of the sorption analysis of the (A) reference Norway spruce wood (PiAb), (B) Burnblock-impregnated Norway spruce wood (BB) in two sorption and desorption cycles. In graph (C), hysteresis is plotted. In contrast, in graph (D) differences in equilibrium wood moisture content between treated and untreated spruce in the first and second sorption cycles are presented. Negative values indicate that the MC of treated wood was lower than that of untreated wood.

Slika 5. Rezultati sorpcijske analize (A) referenčne smrekovine (PiAb), (B) smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock v dveh sorpcijskih in desorpcijskih ciklih. Slika C prikazuje histerezo, slika D pa razliko v vlažnosti med impregnirano in neimpregnirano smrekovino v prvem in drugem sorpcijskem ciklu. Negativne vrednosti nakazujejo, da je bila vlažnost impregniranega lesa nižja od vlažnosti neimpregniranega lesa.

show increased water uptake at low RH (0 to 10%). Once a single (mono-)layer of water has formed, additional adsorption increasingly resembles the condensation of water. At high RH, i.e. above 70%, adsorption is enhanced due to the presence of tiny surface pores (mesopores, with pore diameters of 2 to 50 nm). These attract water molecules on more than one side, i.e. by capillary condensation. This leads to hysteresis in this humidity region caused by the reluctant release of the adsorbed water (Mangel, 2000).

As the hysteresis between the sorption and desorption curves for Burnblock-treated wood increases at the higher sorption range (Figure 5), this indicates that there are more condensation sites present in the Burnblock-treated wood than in the reference spruce wood. This can be ascribed to the presence of crystals in cell lumina, as clearly seen from microscopic analysis (Figure 4).

4 CONCLUSIONS

4 ZAKLJUČKI

The sorption properties of Burnblock-treated wood are comparable to those of untreated wood, while the moisture content of Burnblock-treated wood is comparable to that of untreated Norway spruce. The only difference can be found in the hysteresis between the sorption and desorption curves at higher relative humidities. This can be ascribed to the presence of the crystals of Burnblock in the wood cell lumina. It can be thus presumed that the Burnblock-treated wood with retentions up to 38 kg/m³, exhibits the same susceptibility towards staining fungi.

5 SUMMARY

5 POVZETEK

Sorpcijske lastnosti lesa imajo značilen vpliv na nekatere lastnosti lesa. V največji meri vplivajo na mehanske lastnosti in dovtetnost lesa na glivni razkroj. Vlažnejši les je bolj dovteten za glivni razkroj. Mejna vrednost za glivni razkroj je tri do pet odstotnih točk pod točko nasičenja celičnih sten. Po drugi strani dovtetnost lesa na pojav gliv plesni in gliv modrivk pogosto opišemo s kritično relativno zračno vlažnostjo, pri kateri se pojavijo plesni. Za večino lesnih vrst ta meja znaša okoli 75 %. V prime-

ru bolj higroskopnih lesnih vrst je ta meja lahko tudi nižja. Za zaščito pred glivnim razkrojem, fotodegradacijo ali gorenjem les pogosto impregniramo. Predvsem anorganske soli vplivajo na sorpcijske lastnosti lesa. To je še posebej značilno za les, impregniran z borovo kislino, boraksom ali natrijevim kloridom. Zelo higroskopna so tudi nekatera ognjezadrževalna sredstva. S tem namenom smo preiskali sorpcijske lastnosti smrekovine, impregnirane z ognjezadrževalnim sredstvom Burnblock (navzem 38 kg/m³). Burnblock sodi med okolju prijazne rešitve, brez znanih negativnih vplivov na okolje. Impregniran les smo preiskali z dvema mikroskopskima tehnikama (konfokalna laserska vrstična mikroskopija in digitalna mikroskopija).

Mikroskopska analiza je potrdila prisotnost kristalov v celičnih lumnih. Sorpcijske lastnosti smo določili z opremo, ki omogoča dinamično sorpcijo vodne pare (DVS). Les smo zmlili in homogenizirali ter ga izpostavili dvema cikloma navlaževanja in sušenja v območju med 0 % in 95 % relativne zračne vlažnosti pri 25 °C. Rezultati so pokazali, da so sorpcijske lastnosti lesa, obdelanega z Burnblockom, primerljive s sorpcijskimi lastnostmi neobdelanega lesa smreke. Vsebnost vlage v lesu, impregniranim z Burnblockom, je primerljiva z vsebnostjo vlage neobdelane smrekovine. Edina razlika je v histerezi med sorpcijskimi in desorpcijskimi krivuljami pri višji relativni vlažnosti. To lahko pripišemo prisotnosti kristalov v lumnih lesnih celic. Tako lahko domnevamo, da je les, obdelan z Burnblock, z navzemom do 38 kg/m³, primerljivo dovteten za delovanje gliv modrivk in plesni kot neimpregniran les.

ACKNOWLEDGEMENT

ZAHVALA

The authors acknowledge the financial support of the Slovenian Research Agency (ARRS) within the research program P4-0015 (Wood and lignocellulosic composites) and the infrastructural centre (IC LES PST 0481-09). Part of the published research was also supported by a project financed within the joint program of the Ministry of Agriculture, Forestry and Food and Slovenian Research Agency (V4-2017).

REFERENCES

LITERATURA

- Blahovec, J., & Yanniotis, S. (2008). Gab generalized equation for sorption phenomena. *Food and Bioprocess Technology*, 1(1), 82–90. DOI: <https://doi.org/10.1007/s11947-007-0012-3>
- Engelund, E. T., Thygesen, L. G., Svensson, S., & Hill, C. A. S. (2013). A critical discussion of the physics of wood-water interactions. *Wood Science and Technology*, 47(1), 141–161. DOI: <https://doi.org/10.1007/s00226-012-0514-7>
- Gerhards, C. C. (1982). Effect of Moisture Content and Temperature on the Mechanical Properties of Wood: An Analysis of Immediate Effects. *Wood and Fiber*, 14(1), 4–36.
- Glass, S. V., Boardman, C. R., Thybring, E. E., & Zelinka, S. L. (2018). Quantifying and reducing errors in equilibrium moisture content measurements with dynamic vapor sorption (DVS) experiments. *Wood Science and Technology*, 52(4), 909–927. DOI: <https://doi.org/10.1007/s00226-018-1007-0>
- Gryc, V., Vavrčík, H., & Horn, K. (2011). Density of juvenile and mature wood of selected coniferous species. *Journal of Forest Science*, 57(3), 123–130. DOI: <https://doi.org/10.17221/18/2010-jfs>
- Hartley, I. D., Kamke, F. A., & Peemoeller, H. (1992). Cluster theory for water sorption in wood. *Wood Science and Technology*, 26(2), 83–99. DOI: <https://doi.org/10.1007/BF00194465>
- Humar, M. (2013). Influence of Norway spruce and European larch heartwood ring-width on extractive content and durability. *Drvena Industrija*, 64(2). DOI: <https://doi.org/10.5552/drind.2013.1244>
- Isaksson, T., Thelandersson, S., Ekstrand-Tobin, A., & Johansson, P. (2010). Critical conditions for onset of mould growth under varying climate conditions. *Building and Environment*, 45(7), 1712–1721. DOI: <https://doi.org/10.1016/j.buildenv.2010.01.023>
- Khali, D. P., & Rawat, S. P. S. (2000). Clustering of water molecules during adsorption of water in brown rot decayed and undecayed wood blocks of *Pinus sylvestris*. *Holz Als Roh-Und Werkstoff*, 58(5), 340–341. DOI: <https://doi.org/10.1007/s001070050441>
- Lesar, B., Gorišek, Ž., & Humar, M. (2009). Sorption properties of wood impregnated with boron compounds, sodium chloride and glucose. *Drying Technology*, 27(1). DOI: <https://doi.org/10.1080/07373930802565947>
- Mangel, A. (2000). Identifying physical and chemical phenomena with gravimetric water sorption analysis. *Journal of Thermal Analysis and Calorimetry*, 62(2), 529–537. DOI: <https://doi.org/10.1023/A:1010183407622>
- Medved, S., Jones, D., Faelled, P., Pirs, D., Humar, M., & Lesar, B. (2019). Investigation of fire-retardant additive on particleboard properties. *Proceedings of the International Panel Products Symposium 2019*, 141–148.
- Meyer, L., Brischke, C., Treu, A., & Larsson-Brelid, P. (2016). Critical moisture conditions for fungal decay of modified wood by basidiomycetes as detected by pile tests. *Holzforschung*, 70(4), 331–339. DOI: <https://doi.org/10.1515/hf-2015-0046>
- Mitchell, P. H. (2018). Calculating the equilibrium moisture content for wood based on humidity measurements. *BioResources*, 13(1), 171–175. DOI: <https://doi.org/10.15376/biores.13.1.171-175>
- Nordic Wood Preservation Council. (2021). List no 99.
- Schmidt, O. (2006). Wood and tree fungi: Biology, damage, protection, and use. In *Wood and Tree Fungi: Biology, Damage, Protection, and Use*. Springer-Verlag Berlin Heidelberg. DOI: <https://doi.org/10.1007/3-540-32139-X>
- Wagenführ, R. (2014). *Holzatlas*. 4th Edition. In IAWA Journal. Fachbuchverlag.
- White, R. H., & Dietenberger, M. A. (2010). Fire safety of wood construction. In *Wood Handbook—Wood as an engineering material* (pp. 1–22). U.S.D.A. Forest service, Forest products Laboratory. <http://www.fpl.fs.fed.us/documnts/fplgtr/fplgtr113/fplgtr113.htm>
- Willeitner, H. (2001). Current national approaches to defining retentions in use. *COST E22*.
- Willems, W. (2018). Hygroscopic wood moisture: single and dimerized water molecules at hydroxyl-pair sites? *Wood Science and Technology*, 52(3), 777–791. DOI: <https://doi.org/10.1007/s00226-018-0998-x>

Humar, M., Lesar, B., & Kržišnik, D.: Sorpcijske lastnosti lesa, impregniranega z ognjezadrževalnim sredstvom Burnblock