

PENETRATION OF WOOD PRESERVATIVES INTO THERMALLY MODIFIED BIRCH AND PINE WOOD

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Abstract:

The objective of the present study was to investigate the interaction between Cu-containing preservatives and birch (*Betula* spp.) and pine (*Pinus sylvestris* L.) wood, modified at a relatively mild temperatures (150 – 180°C). The disposition of wood to absorb water was evaluated by capillary absorption (CA) tests through the specimens' tangential and radial surface. Changes in wood drying characteristics due to thermal modification (TM) were evaluated by monitoring wood moisture dynamics after impregnation. In order to assess the capacity of wood to absorb preservatives, a vacuum/pressure process was used to impregnate small specimens for which uniform saturation into the entire volume can easily be reached. Quantitative determination of copper Cu content in the specimens was performed by using atomic absorption spectroscopy (AAS). The fixation of the absorbed Cu was evaluated by subjecting the specimens to leaching procedures according to EN 84 and assessing the ratio of retained Cu in the specimens. The CA test showed deceleration of capillary absorption in TM birch wood through both surfaces, with similar absorption rates regardless of treatment temperatures. A significant increase in the absorption rate through the tangential surface was recorded for TM pine wood and the increase was greater for specimens treated at higher temperatures. The results of moisture content monitoring showed a similar reduction in the drying rate due to thermal modification regardless of species. Comparing wood of one species with similar densities, less preservative was absorbed by TM wood. However, the results of AAS showed that, in comparison with unmodified wood, 10% (birch) and 25% (pine) more Cu per one gram of wood was introduced during impregnation. Nevertheless, TM also resulted in higher Cu leaching rates for both species.

Key words: thermal modification; impregnation; capillary absorption; drying dynamics.

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INTRODUCTION

Recently, the legislation concerning the usage of wood preservation formulations has been changed, leading to alteration in the most widely used wood protection practices. Because of concern regarding the preservatives' effect on the environment, the focus on Cu-based preservatives has increased and impregnation with Cu-based formulations has become the main preservation method in use (Freeman and McIntyre 2008). However, the service properties of wood materials impregnated with these formulations is often unsatisfactory. Another alternative for enhancement of wood bio-resistance is thermal modification (TM). It is well established that thermal treatment improves wood bio-resistance. However, adequate wood protection against biodegradation is ensured only by wood treatment at temperatures that cause significant reduction of wood mechanical strength (Kamdern *et al.* 2002; Metsä-Kortelainen and Viitanen 2010; Candelier *et al.* 2017). Consequently, the application area of TM wood is restricted due to declined strength properties.

Different complex wood treatment procedures are proposed for overcoming deficiencies of conventional modification methods. Among them, wood thermal modification in combination with various pre- or post-treatment methods has been intensively studied (Wang *et al.* 2013; Baysal *et al.* 2014; Salman *et al.* 2016; Turkoglu 2016).

Knowledge regarding changes in the interaction between wood and impregnation solutions caused by wood transformation during TM is mandatory for designing effective combined processes involving wood TM followed by impregnation. Permeability is an important material property related to preservative impregnation. The primary fluid flow in wood takes place through wood anatomical elements of larger diameter such as tracheids, vessels and rays, which are interconnected by pits (Yin *et al.* 2015). However, wood permeability is largely affected by pit pore size, distribution, and interconnectivity, as pores are the main obstruction for fluid flow. Some fluid can also move through the cell wall's capillary system, but generally it is believed that most of the solution passes through the pit system (Murmanis and Chudnoff 1979). Horizontally aligned rays are the main route for liquid transport during impregnation in the transverse direction (Lehringer *et al.* 2009).

It was found that TM facilitates impregnation of pine wood because of pit deaspiration due to modification (Ahmed *et al.* 2013). On the other hand, it was observed that even kiln-drying improves pine wood permeability by provoking ray cell collapse and formation of interstitial spaces, resulting in the creation of secondary flow paths (Ahmed *et al.* 2012). Moreover, liquid flow path can be changed also during impregnation because of cell wall damage, as a consequence of the impregnation process (Olsson *et al.* 2001). In turn, the finding that micro-pores in cell walls relate to the degree of cross-linking and condensation of lignin (Kojiro *et al.* 2010) implies that the liquid flow through the cell wall capillary system may be changed as a consequence of lignin transformation during TM (Weiland and Guyonnet 2003; Boonstra and Tjeerdsma 2006; Windeisen and Wegener 2008).

Preservative fixation in wood is another important aspect of impregnation concerning treatment efficiency for ensuring long-term durability to the treated material. The main factors affecting fixation are preservative formulation, wood properties, and treatment and post-treatment conditions (Tascioglu *et al.* 2008; Yu *et al.* 2009; Lee and Cooper 2010; Bahmani *et al.* 2015). Understanding the relations between them might help to find the best parameters for a combined treatment.

This study is part of a project that is aimed at improving wood service properties by combining thermal treatment and impregnation with Cu-organic preservative. It is expected that, by applying the proposed combined treatment process, a material with decent mechanical strength and meeting the requirements of the use class 3 according to the EN 335-1 standard will be obtained. The objective of the present study was to investigate the interaction between TM wood treated at relatively mild temperatures (150 – 180°C) and water, as well as Cu-containing preservatives. In the study two wood species, birch (*Betula* spp.) and pine (*Pinus sylvestris* L.), were used.

MATERIALS AND METHODS

For the research, kiln-dried boards of birch (*Betula* spp.) and pine (*Pinus sylvestris* L.) wood were obtained from a commercial sawmill. The modification was carried out in a multifunctional wood modification pilot device produced by Wood Treatment Technology (WTT) (Denmark). The boards (25×100×700mm) were thermally modified (TM) in a water vapour medium under elevated pressure (0.6 – 0.8MPa depending on the treatment temperature) for 1h at the peak temperature. Each of the wood species was treated at three peak temperatures: birch wood at 150, 160 and 170°C, and pine wood at 160, 170 and 180°C. Unmodified and TM boards were conditioned (RH 65±5%; 20±2°C) for at least two weeks before preparing specimens for further experiments.

Capillary water absorption (CA) through radial (Rad) and tangential (Tg) surfaces was tested using cubic specimens (20×20×20mm) with the annual ring and grain orientations strictly parallel to the edges. The fluid flow in water uptake through the radial surface takes place in the wood's tangential direction, and

through the tangential surface – in radial direction, respectively. Specimens were sealed with waterproof coating on four edges, leaving uncoated two opposite faces, one of which was the testing surface (radial or tangential) for CA evaluation. After conditioning (RH 65±5%; 20±2°C) to constant mass, specimens were installed into a frame that restricted water evaporation from the container and fixed the specimens in a position in which the contact surface was 2±0.2mm under the water level. The container was filled with distilled water the level of which was monitored and adjusted every day. After certain time periods, specimens were removed from the water, excess water was blotted with a tissue and specimen weight was recorded with an accuracy of 0.0002g. The experiment was performed in a room with controlled RH (65±2%) at 20±2°C. Ten replicates were used for the CA experiment through each surface.

The drying test was carried out with unmodified and TM specimens only modified at one temperature for each species: 160°C for birch and 170°C for pine. For the test, specimens measuring 20×20×100mm with straight grain and growth rings oriented parallel to one edge were used. To prevent drying through end-grain, the end-grain surfaces were sealed with waterproof coating. Two impregnation solutions were used for the test: distilled water and 1% (m/v) water solution of commercial Cu-containing biocide. Impregnation was performed by applying vacuum (1.7 – 1.8kPa) for 30min before and 20min after the impregnation liquid was admitted into the beaker with the specimens, and after release of vacuum maintaining the specimens in the solution for one extra hour. After extracting the specimens from the impregnation liquid, they were blotted with a tissue, weighed and exposed to drying in a controlled environment (RH 40%, 20°C) with mass control after definite periods of time. The liquid content was calculated as the ratio to the dry mass of the specimen.

The absorption of preservative solution by unmodified and TM wood was evaluated by impregnation of specimens in a vacuum (1kPa for 30min)/pressure (0.8MPa for 60min) process. To ensure complete liquid penetration, small (20(Rad) × 20(Tg) × 5(L) mm) specimens were used in this test. Water solutions of commercial Cu-containing preservative with three concentrations (0.50, 0.85, 1.20%) were impregnated into unmodified and TM wood. The preservative uptake was calculated from the specimen mass recorded before and after impregnation, as well as subsequent drying. For specimen dry density calculation, dimensions of dry specimens were measured with an accuracy of 0.02mm. Ten replicates were used per each preservative concentration. After measuring the data for density determination, each specimen was split diagonally in half. One specimen half was used to evaluate Cu content in impregnated specimens. The second half was subjected to a leaching procedure according to EN 84. The analysis of Cu content in impregnated specimens before and after leaching was performed by using atomic absorption spectroscopy (AAS). In the scope of the present study, Cu content was determined only for specimens impregnated with the preservative solution of the highest (1.2%) concentration.

RESULTS AND DISCUSSION

Capillary absorption

Because of the inherited anatomical wood structure, liquid flow in wood is highly anisotropic and the flow rate in the longitudinal direction is much greater than in the lateral directions (Banks 1973; Siau 1984). However, exactly lateral movement of fluid is pivotal for wood impregnation treatments, as normally the ratio of the board transverse sections area is rather small and the liquid preservative penetration through it is of secondary importance. For evaluation of thermal treatment effect on wood liquid uptake characteristics, CA tests through wood radial and tangential surfaces were performed and the results are presented in Fig. 1.

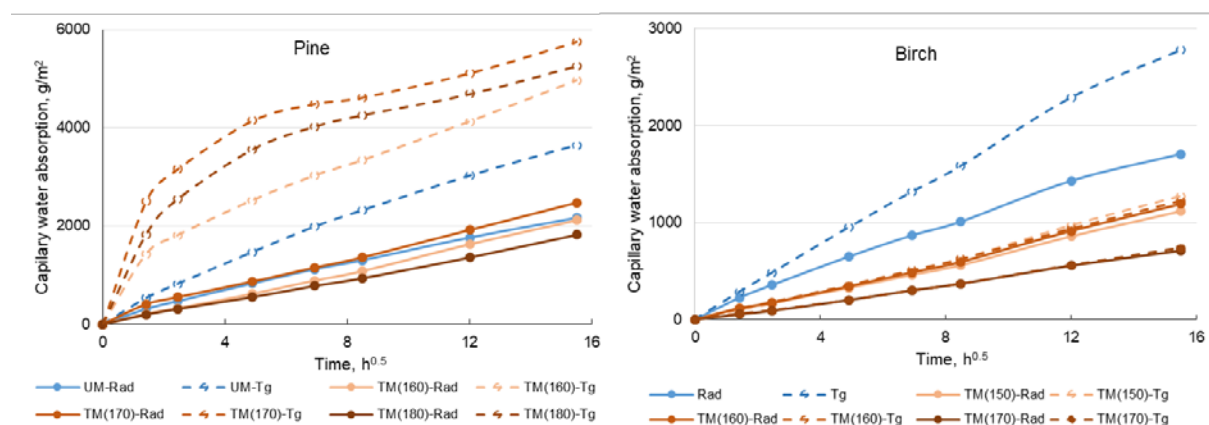


Fig. 1.

Capillary water absorption of unmodified (UM) and thermally modified (TM) birch and pine wood through radial (Rad) and tangential (Tg) surfaces.

As it was expected, for unmodified wood of both species faster water absorption was observed through the tangential surface in the radial direction. It is in good agreement with findings that rays are the main lateral path for liquid movement in wood and transport between adjacent cells takes place through pits (Olsson *et al.* 2001; Lehringer *et al.* 2009; Ahmed *et al.* 2012). However, in an experiment with water absorption monitoring by means of neutron radiography, a conflicting result was observed of slower water uptake in pine wood in radial direction than in tangential direction (Sedighi-Gilani *et al.* 2012). The predominant route for liquid transport through the radial surface (in tangential direction) goes through pits located on the wood cells' radial walls. Comparing unmodified wood of pine and birch, faster water uptake was detected for pine through both wood surfaces. It may be explained by differences in wood anatomical structure with generally poorer penetrability of rays in hardwoods, hindering radial flows, and longitudinal fibres with scanty pit structure compared with softwood tracheids being the crucial obstacle influencing the tangential flow (Siau 1984). Moreover, for pine wood greater water absorption rate anisotropy was found, with twice as much water absorbed through the tangential surface, while only 50% more water was absorbed through the tangential surface in birch wood during equal periods of time.

Table 1

Capillary water absorption and absorption anisotropy after 24 h and 240 h of unmodified (UM) and thermally modified (TM) birch and pine wood through the radial and tangential surfaces

	Regime	Surface	24 h		240 h	
			Capillary water absorption, g/m ²	Anisotropy	Capillary water absorption, g/m ²	Anisotropy
Birch	UM	Rad	649 (146)	1.5	1708 (256)	1.6
		Tg	949 (261)		2782 (863)	
	TM-150	Rad	295 (37)	1.1	1062 (87)	1.2
		Tg	339 (41)		1272 (151)	
	TM-160	Rad	342 (71)	1.0	1191 (204)	1.0
		Tg	351 (62)		1222 (181)	
	TM-170	Rad	201 (35)	1.0	710 (120)	1.0
		Tg	199 (35)		735 (107)	
Pine	UM	Rad	734 (190)	2.0	1961 (337)	1.9
		Tg	1469 (324)		3642 (518)	
	TM-160	Rad	594 (104)	4.3	1899 (426)	2.6
		Tg	2524 (743)		4964 (552)	
	TM-170	Rad	982 (268)	4.2	2736 (387)	2.1
		Tg	4145 (997)		5752 (1083)	
	TM-180	Rad	555 (171)	6.4	1743 (541)	3.0
		Tg	3562 (809)		5256 (891)	

Standard deviations in parentheses; Rad – radial surface, Tg – tangential surface.

The CA test results of TM wood demonstrate that the rate of the wood's lateral water uptake is changed in dissimilar pattern for pine and birch wood due to thermal treatment. For birch wood, significant deceleration of water uptake was observed for both studied surfaces. Besides that, less decrease of absorption rate was recorded for liquid movement through the radial wood surface, which resulted in almost equal uptake in both directions for TM birch wood (Table 1). From these results it is obvious that TM makes birch wood more isotropic regarding lateral water absorption rate. Moreover, quite similar CA rates were detected for TM birch specimens regardless of treatment temperatures. Regarding TM pine wood, converse effect of heat treatment on water uptake through the radial and tangential surfaces was observed. Little changes in comparison with unmodified wood were observed for water uptake through the TM wood's radial surfaces regardless of the modification temperature, whereas significant increase in the absorption rate especially during the initial period of the CA test was detected for TM pine water uptake through the tangential surfaces. Besides that, higher modification temperatures had provoked greater increase in absorption rates. Therefore, regarding water uptake rates, significantly greater anisotropy was observed for TM pine than for unmodified pine, with especially high anisotropy at the outset of the capillary absorption. A similar trend of increased radial permeability of *Pinus radiata* caused by high-temperature drying has been reported by Booker (1990). The author attributes the observed permeability increase to liquid flow through radial resin canals and collapse of thin ray parenchyma cells which create interstitial spaces along which relatively free movement of liquid can take place. Increase in water uptake due to TM of pine sapwood has been observed also by Metsä-Kortelainen *et al.* (2006). Concerning opposite trends in changes of wood CA

due to TM for pine and birch woods, Johansson *et al.* (2006) have reported about similar effects of TM on the alteration of wood longitudinal CA, with absorption increase for pine and decrease for birch wood. However, contradicting results have also been reported with substantially less absorption in floating and submersion tests detected for TM pine in comparison with unmodified pine (Van Acker *et al.* 2015).

Drying

High drying rate is a favourable characteristic for wood materials that can come in contact with liquid water during their service life. On the other hand, it was found that prolonged time of wood wetness after impregnation is advisable for Cu fixation (Yu *et al.* 2009; Humar and Lesar 2009). The results of the impregnation-drying test are shown in Fig. 2.

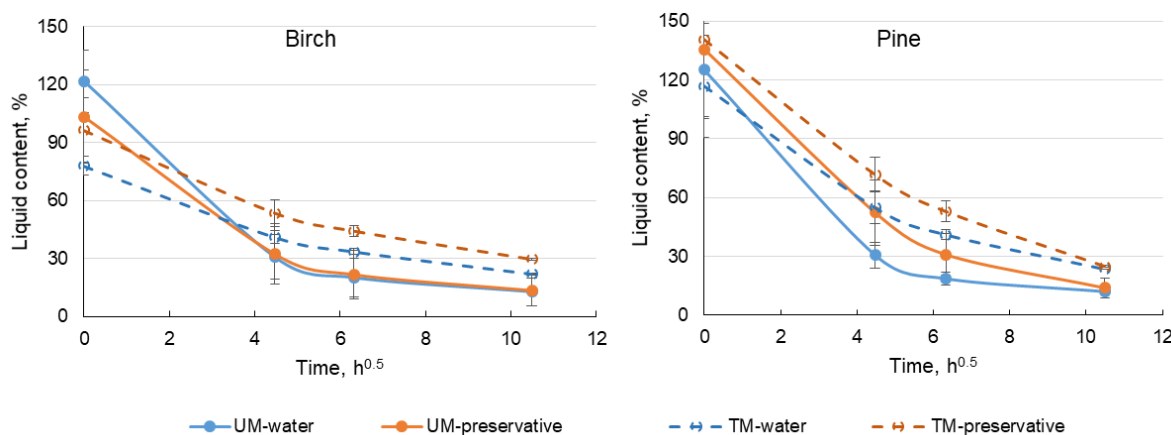


Fig. 2.

Drying dynamics of unmodified (UM) and thermally modified (TM) birch and pine wood after impregnation with water and copper-containing preservative solution.

As bigger specimens were used for the impregnation-drying test, no full saturation was reached for all specimens during the impregnation schedule used. However, the uptake of water is in accordance with the results obtained in the CA test, with less liquid absorbed by TM birch and more absorbed by TM pine compared with their unmodified counterparts. Focusing on the absorbed amounts depending on the liquids used for the impregnation, water or preservative solution, no substantial differences were found for either of the tested wood types. The results regarding the wood drying rate show that the release of moisture from specimens impregnated with preservative solution proceeds slower than from specimens impregnated with water. Such a trend was observed for both unmodified and TM wood. Moreover, drying of TM wood was found to be substantially slower than that of unmodified wood regardless of the impregnation liquid. For TM wood, wetness above the fibre saturation point implying liquid water presence in cells was recorded at specimen examination after drying for 40 hours. The moisture content recorded after drying for 40 hours was substantially above the fibre saturation point of TM wood, which implies liquid presence in cells. Considering aforementioned improvement in Cu fixation with prolonged wood contact time with the preservative solution, slower drying rates of TM wood could be beneficial for Cu fixation into wood. More detailed experiments regarding Cu fixation depending on the wetness time after impregnation as well as impact of temperature on this process have been started.

Absorption of preservative solution

A similar trend was observed for all wood types regarding specimen capacity to absorb aqueous preservative solutions depending on the wood density. The gravimetrically determined amount of the absorbed liquid into the small specimens during impregnation showed that less uptake (g/g) of preservative solutions is characteristic of wood with higher density (Fig. 3). Focusing on unmodified and TM wood with similar densities, somewhat less liquid uptake was detected for the TM specimens. Less absorption of liquid in TM wood may be related to lower wood cell wall density of TM wood (Zauer *et al.* 2013), which inevitably results in less space available for liquid uptake. Another reason could be the formation of some closed cavities within cell walls due to wood structural transformations caused by wood treatment at high temperature (Borrega and Kärenlampi 2011).

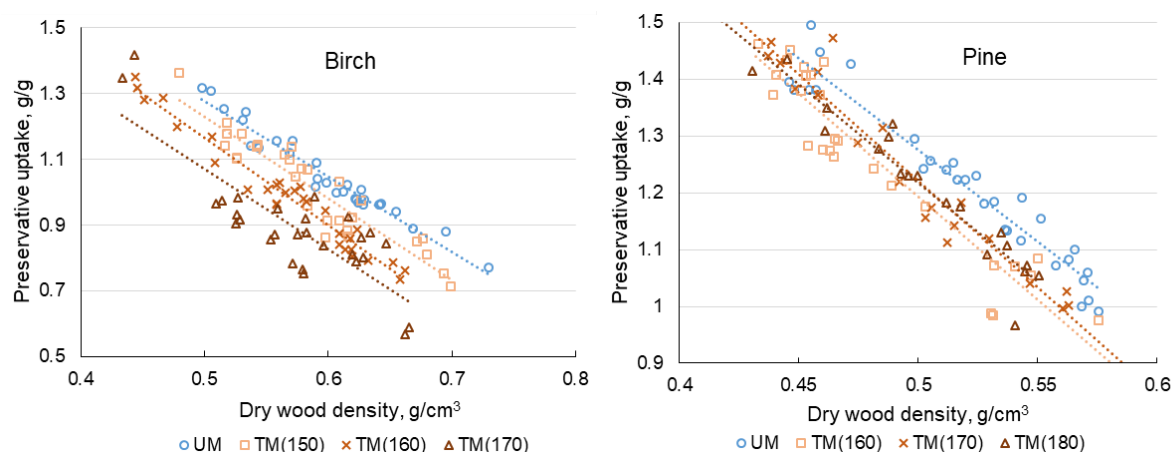


Fig. 3.

Copper-containing preservative uptake of unmodified (UM) and thermally modified (TM) birch and pine wood depending on wood density.

In addition to that, no dependence of the absorbed preservative amount was detected for impregnation with preservative solutions of different concentrations (results not presented). This finding agrees with the results reported by Humar and Lesar (2009), who found that the concentration of the Cu-containing preservative solutions did not influence the uptake of preservative during the first day of impregnation, but increased uptake was observed for longer impregnation periods.

Table 2

Copper (Cu) content in unmodified (UM) and thermally modified (TM) birch and pine wood after impregnation with Cu-containing preservative and leaching

	Regime	Cu content after impregnation, mg/g	Cu content after leaching, mg/g	Retained Cu after leaching, %
Birch	UM	1.43 (0.25)	0.99 (0.21)	69
	TM-150	1.30 (0.24)	0.62 (0.12)	48
	TM-160	1.60 (0.40)	0.56 (0.09)	35
	TM-170	1.52 (0.58)	0.63 (0.13)	41
Pine	UM	1.79 (0.18)	1.51 (0.18)	84
	TM-160	2.25 (0.44)	1.50 (0.21)	67
	TM-170	2.23 (0.49)	1.33 (0.29)	60
	TM-180	2.24 (0.55)	1.14 (0.24)	51

Standard deviations in parentheses.

AAS results of Cu content per gram of wood after the impregnation test show on average more Cu in TM wood than in unmodified wood (Table 2). For birch wood, the difference is about 10%, and for pine wood – 25%. However, due to rather big deviations among the data, no statistically significant difference ($p > 0.05$) was found between TM and unmodified wood. Comparing the two wood species, more Cu was detected in the pine wood, which agrees with lower densities and consequently higher uptake of preservative solution. Moreover, more intense leaching of Cu was detected for the birch wood and especially for TM birch wood, from which more than 50% of Cu was leached. Regarding unmodified wood, 31% loss of the absorbed Cu due to leaching was detected for birch wood, in comparison with 16% for pine wood. A similar trend was observed in other researches as well, in which higher leaching rates for hardwood in comparison with softwood were detected (Radivojevic and Cooper 2010; Temiz *et al.* 2014). In the present research, higher uptake in combination with a lower leaching rate resulted in about two times higher Cu content per gram of wood after leaching in TM pine wood, and for 50% higher in unmodified pine wood in comparison with corresponding birch wood. However, in general, less Cu per gram of wood was detected in TM wood than in unmodified wood after leaching, despite higher Cu uptake by TM wood during impregnation. The exception was TM pine modified at 160°C, for which similar Cu content as in unmodified pine wood was detected in leached specimens.

Based on the results of the present study, further experiments have been designed and are now in progress.

CONCLUSIONS

From the present study, it was concluded that the thermal modification of wood significantly affects several properties related to preservative impregnation processes, and the change patterns differ for birch and pine wood.

1. For birch wood, thermal modification causes decrease in the water uptake rate through both the tangential and the radial surface. In addition to that, larger changes through the tangential surface lead to decrease in anisotropy of the wood's transverse absorption rates. For pine wood, thermal modification slightly affects the rate of water uptake through the radial surface. However, it significantly increases the rate of water uptake through the tangential surface, with higher modification temperatures causing greater increase, which, contrary to birch wood, results in increased anisotropy.

2. Thermal modification reduces the rate of drying for both birch and pine wood, which might be beneficial for preservative fixation in thermally modified wood.

3. Preservative uptake in wood strongly depends on wood density for both unmodified and thermally modified wood, with a trend of larger liquid uptake for lower density. Comparing wood of one species with similar densities, less preservative was absorbed by TM wood.

4. In general, thermal modification results in higher Cu absorption, as well as higher leaching rates, for both pine and birch wood. However, because of higher Cu absorption and lower Cu leaching, preservative impregnation is more effective for both unmodified and thermally modified pine wood in comparison with birch wood.

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