



Effects of heat treatment on thermal properties of European beech wood

Łukasz Czajkowski¹ · Wiesław Olek¹ · Jerzy Weres²

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Abstract

Beech wood, due to its properties, is one of the most versatile and successfully used construction materials. The wood properties could even be improved, and among different wood modification processes, the thermal modification approach is usually considered as an environment-friendly technology based only on the heat and water application during wood treatment. Changes in material properties resulting from the thermal treatment of wood increase applicability of this material, but on the other hand, detailed knowledge of the modified properties is definitely necessary for the proper application of such materials to construction engineering. Unfortunately, credible data on thermal characteristics of thermally modified wood are usually provided in a very limited way, and there is no information on specific heat in particular. An original calorimetric method was used to determine the specific heat of untreated and thermally modified European beech wood (*Fagus sylvatica* L.). The inverse modeling was implemented to estimate the anisotropic thermal conductivity, and significant differences were found for the radial and tangential directions. The thermal modification highly influenced the increase in the thermal conductivity in the longitudinal direction. The validation procedure showed credibility of the applied methods, and it is clear that modeling of heat transfer in thermally modified wood leads to erroneous results when using thermal properties determined merely for untreated wood.

1 Introduction

Wood is widely used as a building material mainly due to its advantageous properties, i.e. low density and especially high ratio of strength to density (e.g., Bal 2016). The material is inexpensive, renewable and environmentally friendly (e.g., Ghosh et al. 2008). However, it is also characterized by low dimensional stability and susceptibility to decay. Different modification processes often improve the functional properties of wood. Nowadays, it is demanded that the modification products should be obtained with limited amount or even without the use of toxic chemicals (Sandberg and Kutnar 2016). Thermal modification is usually considered as an environmentally friendly, chemical-free technology of wood modification due to the application of only heat

and water during the treatment (Sandberg et al. 2017). The chemical composition and the ultrastructure of wood cell walls are seriously altered during the heat treatment (Olek and Bonarski 2014), resulting in changes of the material properties. It was commonly reported that thermally modified wood reveals increased dimensional stability due to the reduction in equilibrium moisture content. Such material is also characterized by improved resistance against fungi and insects (e.g., Sandberg and Kutnar 2016). The treatment has also some serious drawbacks as it negatively affects a majority of wood strength and stiffness properties as well as brittleness. However, Boonstra et al. (2007) claimed that thermally modified timber could be considered as a potential material for constructions with some reservations related to careful consideration of stresses in a construction. Therefore, the recommendations usually limit possible application of thermally modified wood to façade systems, terraces, windows, flooring, stairs etc. (Herrera et al. 2018; Sandberg and Kutnar 2016).

Some applications of the material require knowledge of thermal characteristics comprising specific heat, density and thermal conductivity. The data are essential for proper design of thermal envelopes of buildings. Moreover, the

✉ Łukasz Czajkowski
czajk@up.poznan.pl

¹ Faculty of Wood Technology, Poznań University of Life Sciences, ul. Wojska Polskiego 28, 60-637 Poznań, Poland

² Faculty of Informatics and Visual Communication, Collegium Da Vinci, ul. Gen. Tadeusza Kutrzeby 10, 61-719 Poznań, Poland

improved dimensional stability and darker color of thermally modified wood makes the material very attractive for flooring. Again, the possible alteration of thermal properties of thermally modified wood is crucial when considering floor-heating systems. Unfortunately, credible data on thermal properties of thermally modified wood are usually provided in a very limited way. The majority of studies report thermal conductivity reduction after the treatment and the extent of the decrease depends on time and temperature of the thermal modification (e.g., Kol and Sefil 2011; Olarescu et al. 2015; Pásztor et al. 2017). It was also depicted that the thermal conductivity reduction is related to wood density decrease after the modification (Kol and Sefil 2011). One can explain it by mass loss of wood during the treatment without a significant contraction of the modified elements. The opposite relation was found by Fu et al. (2018), and it was interpreted that the observed decrease in the thermal conductivity with increasing density was invoked by the alteration of the organization of crystalline cellulose at higher treatment temperature. Contrary to the reported data on the thermal conductivity, there is practically no information on specific heat of thermally modified wood as compared to untreated material.

The objective of the present study was to determine the density, specific heat, volumetric specific heat capacity and thermal conductivity of thermally modified and untreated European beech wood (*Fagus sylvatica* L.), which is highly important when considering application of such wood species to building engineering. The anisotropy of the thermal conductivity was also accounted for in the analysis.

2 Materials and methods

2.1 Thermal modification

The experimental material, i.e. European beech (*Fagus sylvatica* L.) wood, was obtained from sawn timber (50 mm thick boards). Timber without visible defects was selected and carefully kiln-dried in a laboratory dryer to the moisture content of ca. 8%. The dried and seasoned material was cut into oriented strips. Sets of three identical samples were cut from strips and then two of them were subjected to the thermal modification. The material moisture content of the samples prior to the modification was ca. 10%. The modification was made under laboratory conditions in the atmosphere of moist air. Each thermal modification process consisted of two phases. The initial phase of the modification was made in moist air only and ended when the material obtained the temperature of 130 °C. The main phase of the modification was carried out in the atmosphere of superheated steam. Two different target temperature values, i.e. 180 and 220 °C, were set during the main phase of the modification. The duration

of the main phase was 1 h. Majka et al. (2016) showed that duration of the main phase of the laboratory-based thermal modification has no significant influence on mass loss and hygroscopic properties. The target temperature values were kept constant during the main phase. Selection of the two temperature values for the main phase was made in order to obtain two different intensities of the treatment. After the modification, the material was cooled down, equilibrated, and finally dried to the oven dry state.

2.2 Specific heat determination

The measurements of the specific heat were taken with a water calorimeter, which was specially designed and constructed in order to account for specific heat of hygroscopic materials characterized by low density and resulting low heat capacity. The sheets of the investigated material were made in the shape of cylinders of a diameter of 85 mm and a height of 15 mm. The oven-dry material was placed in a heat-shrinkable membrane to prepare samples of cylindrical shape with a length of ca. 135 mm and diameter of ca. 75 mm. In order to control the temperature distribution in samples during the experiments and to measure the equilibrium temperature of the sample-calorimeter system, two type K thermocouples were also mounted in the samples. The positions of the thermocouples were at the center and the surface of each sample. The space of voids filled with air was minimized inside the samples. The amount of air being kept in the samples was minimized by maximizing contact of wood sheets and shrink warping with membrane. The detailed procedure of designing and constructing the calorimeter was reported by Czajkowski et al. (2016a).

It was assumed, during the calorimetric measurements, that a sample and sealing membrane were releasing heat while water and the inner cup of the calorimetric system were gaining it. The specific heat was calculated from the transformed form of the steady-state heat balance equation being used for designing the calorimetric system. The following formula (Eq. 1) was used to calculate the specific heat of the untreated and thermally modified beech woods:

$$c = \frac{(m_a \cdot c_a + m_{\text{H}_2\text{O}} \cdot c_{\text{H}_2\text{O}}) \cdot (t_e - t_{ic}) + m_m \cdot c_m \cdot (t_e - t_{is})}{m \cdot (t_{is} - t_e)} \quad (1)$$

where c ; J/(kg K)—specific heat of investigated material, c_a ; J/(kg K)—specific heat of aluminum, $c_{\text{H}_2\text{O}}$; J/(kg K)—specific heat of water, c_m ; J/(kg K)—specific heat of shrinking membrane, m ; kg—mass of investigated material (mass of sample), m_a ; kg—mass of aluminum (calorimeter inner cup with a stirring rod), $m_{\text{H}_2\text{O}}$; kg—mass of water, m_m ; kg—mass of shrinking membrane, t_e ; °C—equilibrium temperature of the calorimetric system, t_{ic} ; °C—initial temperature of the

calorimeter with water, t_{is} ; °C—initial temperature of the investigated material (sample).

2.3 Thermal conductivity identification

An inverse finite element analysis was applied to identify coefficient values of the heat transfer model. The mathematical model of the transient, three-dimensional, quasi-linear heat transfer problem in anisotropic materials was used as proposed by Czajkowski et al. (2016b). The model assumed the first kind boundary condition and the uniform initial distribution of temperature (the initial condition), and is given in its general form by the quasi-linear partial differential equation:

$$c\rho \frac{\partial t}{\partial \tau} = \nabla(k \nabla t), \quad (\mathbf{x}, \tau) \in \Omega \times (0, \tau_F] \quad (2)$$

with the initial condition

$$t(\mathbf{x}, 0) = t_0(\mathbf{x}), \quad (\mathbf{x}) \in \Omega \quad (3)$$

and the first kind boundary condition

$$t(\mathbf{x}, \tau) = t_s(\mathbf{x}), \quad (\mathbf{x}, \tau) \in \partial\Omega^I \times (0, \tau_F], \quad (4)$$

where: c ; J/(kg K)—specific heat, k ; W/(m K)—thermal conductivity, t ; °C—temperature, t_0 ; °C—initial temperature, t_s ; °C—temperature at the boundary, \mathbf{x} ; m—coordinates of a point in the orthocartesian system of coordinates, ρ ; kg/m³—density, τ ; s—time, Ω ; m³—domain of the body examined in the three-dimensional Euclidean space, τ_F ; s—final time of the heat transfer process, $\partial\Omega^I$; m²—boundary of the domain for the first kind boundary condition, ∇ —gradient operator.

The input data for the identification were obtained during transient heat transfer experiments, which were made for untreated and thermally modified (180 and 220 °C) beech wood. The experimental material was oven dried under laboratory conditions, and then cube-shaped samples were prepared. The dimensions of edges of the cubes were equal to 100 · 100 · 250 mm³ in the radial, tangential and longitudinal anatomical directions, respectively. Four thermocouples were mounted in selected locations of each sample. The locations of the mounted thermocouples are listed in Table 1 for each sample separately. Additional three thermocouples

were installed at surfaces of the samples in order to register temperature values at all faces of the samples.

Each transient heat transfer experiment consisted of two phases, i.e. individual samples were heated first to obtain the uniform distribution of temperature in the investigated material (the uniform initial condition), and then the cooling phase was started. The temperature changes in time were registered every 300 s in the cooling phase. The measured values were collected by a data acquisition system and then used as the input data for the thermal conductivity identification. An example of registered temperature values is depicted in Fig. 1. The other sets of empirical data registered during heat transfer processes were applied to validate the identified coefficients of the mathematical model of the process.

3 Results

It has already been depicted (e.g. Huang and Yan 1995, Monteau 2008, Czajkowski et al. 2016b) that the inverse analysis as applied for estimating coefficient values (here material properties) of the heat conduction problem given

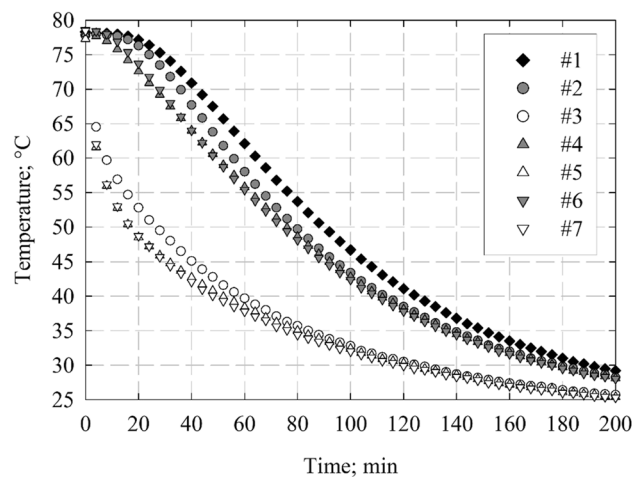


Fig. 1 Example of temperature changes with time as measured by thermocouples #1, #2, #3, #4, #5, #6 and #7 in the cooling phase of a transient heat transfer experiment (input data for thermal conductivity identification). Beech wood modified at a temperature of 180 °C

Table 1 Coordinates of the thermocouples locations in mm

Coordinates	Thermocouples location						
	#1	#2	#3	#4	#5	#6	#7
x_1	50	50	50	50	50	75	100
x_2	125	65	0	125	125	125	125
x_3	50	50	50	75	100	50	50

Thermocouples #3, #5 and #7 were mounted at three different faces of a sample, x_1 —tangential direction, x_2 —longitudinal direction, x_3 —radial direction

by Eqs. (2), (3), (4) has some serious limitations, i.e. simultaneous determination of specific heat and thermal conductivity results in finding the infinite number of pairs of the coefficients. Therefore, Kim et al. (2003) suggested taking measurements of the specific heat first, and then using the measured values for the identification of the thermal conductivity of anisotropic materials with the inverse modeling. The same approach was used in the present study.

The investigated material is characterized by low heat capacity, and it implies a release of a relatively small amount of heat during calorimetric measurements. Therefore, it is required to provide samples of relatively high mass (m), in this case ca. 400 g (Table 2). The measurements also required an assumption related to the minimum temperature increase of the calorimetric system (ΔT), which was found by Czajkowski et al. (2016a) as equal to 2 K. It implied setting the initial temperature (t_{is}) to ca. 90 °C and the equilibrium temperature of the calorimetric system (t_e) to ca. 20 °C (Table 2). The calorimetric measurements of specific heat were also accompanied by determining the oven dry density of the material, i.e. untreated and thermally modified beech wood. The results of the measurements of specific heat and density are presented in Table 2, and each value was a mean of 5 repetitions. As the research material was carefully selected and prepared, the applied calorimetric system enabled high repeatability of the experimental results, and the standard deviation for the specific heat measurements was lower than 4 J/(kg K).

The obtained results of the specific heat measurements for the oven-dry state are also presented in Table 2. The mean value observed for untreated beech wood was ca. 250 J/(kg K) higher as compared to the value reported by Sonderegger et al. (2011) at a wood moisture content of 0%. The difference was probably due to the fact that Sonderegger et al. (2011) gained the specific heat values from the measurements with a guarded hot plate apparatus, i.e. λ -Meter EP500, and recalculated the obtained specific heat values from the thermal diffusivity. Moreover, the data of the specific heat of European beech as obtained from calorimetric measurements are unavailable in the literature. In the case of thermally modified European beech wood, there are no reports on specific heat measurements with any experimental method. Therefore, it was not possible to compare

the obtained results to other data. However, the observed changes in the specific heat can be linked to alterations in wood density and ultrastructure. Wood density did practically not change after the treatment at a temperature of 180 °C, and it was accompanied by only a minor decrease in specific heat (Table 2). It can be explained by a very limited decomposition of wood components during the thermal modification at relatively mild conditions. However, thermal conductivity was much more sensitive to the modification at low temperature. The property significantly increased in the tangential and longitudinal anatomical directions, while a decrease was found in the radial direction (Table 3), which might be explained by the additional reorganization of paracrystalline cellulose as induced by heat treatment. Moreover, the high values of the longitudinal thermal conductivity after the modification are similar to the values reported for the wood substance in the same direction (Kollmann and Malmquist 1956), which can be explained by the decomposition of less ordered cell wall components and additional ordering of the crystalline cellulose (Olek and Bonarski 2014).

Fahlén and Salmén (2003) found changes in the cellulose ultrastructure caused by hydrothermal treatment and stated that the matrix polymers were enough softened to obtain rearrangement of cellulose, i.e. enlargement of the cellulose aggregates was noticed. Similarly, Tuong and Li (2010) found that heat treatment might result in additional ordering of quasi-crystalline or amorphous areas of cellulose due to rearrangement or reorientation of the polymer. The findings were supported by Olek and Bonarski (2014), who used the crystallographic texture to quantify wood ultrastructural changes as invoked by the

Table 3 Identified thermal conductivity of untreated and thermally modified beech wood

Treatment of wood	Thermal conductivity k ; W/(m K)			Objective function
	Tangential	Radial	Longitudinal	
Unmodified	0.1467	0.2361	0.4781	213.3
Modified at 180 °C	0.1622	0.1761	0.6386	108.0
Modified at 220 °C	0.1342	0.1505	0.7868	96.6

Table 2 Specific heat, density and volumetric specific heat capacity of untreated and thermally modified beech wood

Treatment of wood	Sample mass m ; kg	Sample initial temperature t_{is} ; °C	Equilibrium temperature of the calorimetric system t_e ; °C	Increase in temperature ΔT ; K	Specific heat c ; J/(kg K)	Density ρ ; kg/m ³	Volumetric specific heat capacity $c \rho$; MJ/(m ³ K)
Unmodified	0.4582	90.3	20.53	2.63	1383	691.7	0.9566
Modified at 180 °C	0.4562	90.0	19.51	2.58	1358	692.5	0.9404
Modified at 220 °C	0.3824	91.3	19.71	3.30	1440	617.0	0.8885

thermal modification. The dominating texture component being related to cellulose increases after the modification at 180 °C. Another observation was related to the increase in the texture index being an overall measure of the texture, i.e. the index increased from 0.76 (untreated wood) to 0.87 (thermally modified wood at 180 °C). The reported ultrastructural changes can account for the increase in the thermal conductivity in the tangential direction after the modification at 180 °C. However, the decrease in the property in the radial direction has another nature. European beech wood is characterized by a high content of rays exceeding the average content of 17% of the total xylem volume of hardwood species (Ressel 2007). Fengel and Wegener (1984) reported the rays content in European beech wood as high as 27%. The rays are anatomical elements favoring heat transfer in the radial direction in untreated wood. These anatomical elements are built of parenchyma, which is susceptible to thermal degradation even at relatively mild conditions of thermal modification, i.e. at 180 °C. It seems to be the most important reason for the significant decrease in the thermal conductivity in the radial direction after the treatment at 180 °C. The ratio of the radial to tangential thermal conductivity was reduced from $0.2361/0.1467 = 1.61$ (untreated wood) to $0.1761/0.1622 = 1.09$ (wood modified at 180 °C) (Table 3).

The increase in the treatment temperature to 220 °C affected the alteration of all investigated properties of beech wood. Density significantly decreased from 690 to ca. 620 kg/m^3 , and it was related to the decomposition of wood components resulting in disordering of the ultrastructure (Fahlén and Salmén 2003). The highest decrease in the thermal conductivity was observed for the radial direction as the property was reduced from 0.2361 W/(m K) (untreated wood) to 0.1505 W/(m K) (thermally modified wood at 220 °C) (Table 3). According to Olek and Bonarski (2014), the intensity of the texture component related to cellulose decreased at 220 °C. It clearly shows that even the durable and organized component of beech wood was affected by the decomposition. The decomposition of the ordered areas of the material treated at 220 °C was also depicted by the normalized texture index. The obtained value of the index was 0.62, i.e. it was below the values observed for the thermally

modified wood at 180 °C, and especially for untreated wood. These ultrastructural changes clearly depicted that the treatment at 220 °C induced decomposition of the wood ultrastructure even when compared to untreated material.

The thermal conductivity values identified for the tangential and radial anatomical directions were also compared to the only available study on the influence of the thermal modification on wood thermal conductivity, i.e. Kol and Sefil (2011) (Table 4). The measurements were taken with the use of the hot-wire method, which is affected by problems related to the determination of the amount of heat emitted by a heating element as well as to reduction in the contact resistance at the interface between a heating element and the examined material (Hammerschmidt and Sabuga 2000). Thus, in the case of measurements taken for wood, the hot wire method may be responsible for the high inaccuracy in thermal conductivity determination. This seems to correspond with the results reported by Kol and Sefil (2011), i.e. thermal conductivity data characterized by exceptionally low anisotropy for the tangential and radial anatomical directions as obtained for untreated and thermally modified beech wood at a temperature of 212 °C.

The quality of the calorimetric measurements and the thermal conductivity identification was validated by comparing results of transient heat transfer modeling to the empirical data of experiments that were not used for the identification of the thermal conductivity. The validation was made for the properties measured and identified in the present study, as well as for data available in the literature. Unfortunately, the full data on the thermal properties, i.e. specific heat and anisotropic thermal conductivity, are available for untreated beech wood only. Such sets of full data are not given for thermally modified wood. Although Kol and Sefil (2011) used the hot-wire method to determine thermal conductivity of the thermally modified beech wood, the provided data were limited to the radial and tangential directions only. Moreover, the results of specific heat measurements of thermally modified wood are not available. In contrast to that, Sonderegger et al. (2011) provided the full set of data for untreated European beech wood, and it consisted of the specific heat and thermal conductivity for all principal anatomical wood directions. Therefore, the

Table 4 Comparison of the thermal conductivity values for the tangential and radial anatomical directions. Results of the inverse identification vs. the hot wire measurements

Anatomical direction	Treatment temperature; °C			Source
	Untreated	180	220 ⁽¹⁾ /212 ⁽²⁾	
Tangential	0.1467	0.1622	0.1342	⁽¹⁾ Present study
	0.1824	0.1762	0.1556	⁽²⁾ Kol and Sefil (2011)
Radial	0.2361	0.1761	0.1505	⁽¹⁾ Present study
	0.1911	0.1818	0.1564	⁽²⁾ Kol and Sefil (2011)

(1) The highest modification temperature in the present study; (2) Modification temperature applied by Kol and Sefil (2011)

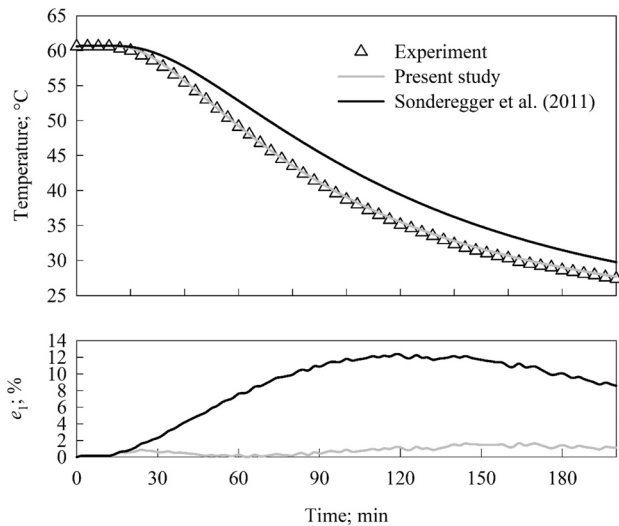


Fig. 2 Predicted temperature values as a function of time for measured and identified thermal properties and data reported by Sonderegger et al. (2011) compared to experimental data (upper plot), and the relative error e_1 of modeling (bottom plot). Beech wood thermally modified at 220 °C, thermocouple #1

validation was limited to the results obtained in the present study and the data available for untreated beech wood (e.g., Sonderegger et al. 2011). The analysis was also used to estimate the accuracy of temperature prediction in case of applying thermal properties of untreated wood to modeling transient heat transfer in the thermally modified wood.

An example of the performed validation is presented in Fig. 2, where the numerically obtained temperature changes with time were compared to the empirical data. The validation was quantified by two errors, which were introduced by Olek et al. (2003), i.e., the local in time relative error e_1 :

$$e_1(\mathbf{x}_i, \tau_j) = 100 \frac{|t_{\text{exp}}(\mathbf{x}_i, \tau_j) - t(\mathbf{x}_i, \tau_j)|}{t_{\text{exp}}(\mathbf{x}_i, \tau_j)},$$

$$i = 1, \dots, NS, \quad j = 1, \dots, NT \quad (5)$$

where t_{exp} ; °C—temperature, NS —number of thermocouples, NT —number of time intervals,

and the global in time relative error e_2 :

$$e_2(\mathbf{x}_i) = 100 \frac{\sqrt{\sum_{j=1}^{NT} [t_{\text{exp}}(\mathbf{x}_i, \tau_j) - t(\mathbf{x}_i, \tau_j)]^2}}{\sqrt{\sum_{j=1}^{NT} t_{\text{exp}}(\mathbf{x}_i, \tau_j)^2}}, \quad i = 1, \dots, NS. \quad (6)$$

The global in time relative error (e_2) was equal to 0.73% for measured and identified thermal properties (present study) and 7.78% for the thermal properties for untreated European beech wood as reported by Sonderegger et al. (2011). The analysis of the local in time relative error e_1

(bottom plot in Fig. 2) clearly showed that the maximum of the e_1 error was less than 1.7% for modeling with measured and identified thermal properties and above 12% for the properties measured for untreated wood. Therefore, it cannot be recommended to apply thermal properties as obtained for untreated wood to model heat transfer in the thermally modified wood.

4 Conclusion

1. The calorimetric method was effectively used for determining the specific heat of untreated and thermally modified beech wood. The applied experimental method ensured high accuracy of specific heat determination. A minor increase in the specific heat was found only for the thermally modified wood at a temperature of 220 °C.
2. The application of the inverse modeling to the thermal conductivity identification enabled us to account for anisotropy of the property. The significant differences in the thermal conductivity were found for the radial and tangential directions as the dominating ones in all wood elements being applied to building engineering. The observed anisotropy was preserved after the thermal modification.
3. The thermal modification highly influenced the increase in the thermal conductivity in the longitudinal direction (ca. 60%) and was related to alteration of the wood ultrastructure as induced by the heat treatment.
4. The validation procedure enabled quantification of the credibility of the applied experimental methods for determining thermal properties of wood. It was clearly presented that accurate modeling of heat transfer in thermally modified wood is not possible when using properties as determined for untreated wood.

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