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ASSESSMENT OF THE CHEMICAL CHANGE IN HEAT TREATED PINE WOOD BY NEAR INFRARED SPECTROSCOPY

Zuzana Vidholdová – Anna Sandak – Jakub Sandak

ABSTRACT

Fourier-transform near-infrared spectroscopy (FT-NIR) was used as none-destructive method to determinate changes in the chemical structure of heat-treated wood. For this purpose, pine sapwood (*Pinus sylvestris* L.) was treated at different temperatures (from 100 °C to 240 °C) and for three durations (1, 3 or 5 hours). The effects of chemical changes on the FT-NIR spectra are linked to absorbance changes of functional groups (–OH, –CH, –CO and $–CH_2$) of lignin, hemicelluloses and cellulose. Gradual degradation of amorphous portion of cellulose was caused by high temperature, while crystalline and semi-crystalline portions of cellulose seem to be less affected by the thermal treatment. The effect of various intensities of heat treatment on chemical changes of wood polymers varied depending on temperature and duration. Presentation of spectra in the form of the xylograms shows clear tendency of degradation kinetic. Evaluation of thermal stability of selected wood component and/or comparison of the influence of modification process parameters can be carried out.

Key words: pine, heat treatment, xylograms, FT-NIR.

INTRODUCTION

Heat-treated wood has become an established commercial product possessing a number of advantages over the natural wood. Heat-treated wood is considered an eco-friendly alternative to chemically impregnated wood materials. This treatment reduces the hydrophilic behaviour of the wood by modifying the chemical structure of its components (hemicelluloses, cellulose and lignin) which results in changes of their properties. Some previous studies (GÉRARDIN 2016, KUČEROVÁ *et al.* 2016, SANDBERG & KUTNAR 2016, ČABALOVÁ *et al.* 2014, REINPRECHT & VIDHOLDOVÁ 2011, HILL 2007, WELZBACHER *et al.* 2007) have reported that treatment temperature and its duration affect the chemical decomposition of wood. The most important positive effects of heat treatment of wood are: enhancement of resistance to biodegradation (ŠUŠTERŠIC *et al.* 2010, WELZBACHER & RAPP 2007, HAKKOU *et al.* 2006) improvement of the overall dimensional stability (VIITANIEMI *et al.* 1997, HILLIS 1984) and reduction of the heat transfer coefficient (MILITZ 2002). Heat treatment is lowering wood equilibrium moisture content (ALTGEN *et al.* 2016) and enhance the surface quality (PRIADI & HIZIROGLU 2013) in addition to bulk discoloration having attractive dark colour (TODOROVIC *et al.* 2012). High temperatures and long time of heat treatment decrease most of the mechanical properties of wood (YILDIZ *et al.* 2011). Thermal modification decreases the heat release rate and propensity for fire propagation in the flashover phase of some species (MARTINKA *et al.* 2016).

Fourier transform near-infrared spectroscopy (FT-NIR) is an efficient method for high-throughput non-destructive screening of chemical characteristics of different materials including the wood and wood based products. Energy of infrared light stimulates vibrations of -CO, -OH, -CH and -NH functional groups giving overtones and combination bands depending on the molecular structure, chemical composition or physical properties of the measured sample. A state of the art of the FT-NIR applications in wood and paper research has been published by TSUCHIKAWA & KOBORI (2015). Quality assessment of thermally treated wood by means of NIR was previously investigated by several researches (POPESCU *et al.* 2018, SANDAK *et al.* 2015, 2016, BÄCHLE *et al.* 2010, MEHROTRA *et al.* 2010, ESTEVES & PEREIRA 2008). NIR spectra can be pre-processes mathematically and evaluated by means of multivariate data analysis to obtain precise quantitative and qualitative information of physical-chemical nature of material.

In this study, the chemical changes due to heat treatment intensity were evaluated in pinewood by non-destructive FT-NIR spectroscopy. The chemical fingerprint of thermally modified wood is visualized by means of xylograms.

MATERIALS AND METHODS

Material and wood treatment

The defect-free pine sapwood (*Pinus sylvestris* L.) without cracks, knots or other growth inhomogeneity were used as experimental samples. The density in oven dry state ranged from 431 to 639 kg·m^{-3} with an average value of 506 kg·m^{-3}. Specimens were heat treated under atmospheric pressure in the laboratory heating oven (Memmert UFB 500, Germany) at Department of Mechanical Wood Technology, FWST at Technical University in Zvolen, as shown in Table 1.

The heat treatment started by putting the samples at ambient temperature in oven with subsequent increasing of the temperature and without forced air circulation. The period to reach expected temperature varied from 15 minutes (for 100 °C) up to 60 minutes (for 240 °C). Duration of the heat treatment at fixed temperature was 1, 3 or 5 hours. Extensively treated wood was prepared at the temperature 240 °C during 8 hours. At the end of each treatment, samples were cooled down in desiccators in dry environment.

Tab. 1 Thermal modification set-up, treatment parameters, set size.

Species	Dimensions (R × L × T) (mm)	Treatment temperature (°C)	Treatment duration (h)	Number of replica
Pine – sapwood	25 × 25 × 3	100	1	4
		150	3	
		160	5	
		200	8*	
		220		
		240		

Note: * The treatment duration of 8 hours was only used for preparation of extensively treated wood.

Wood degradation was monitored by measuring the mass loss (*ML*) and the CIE $L^*a^*b^*$ colour coordinates. Mass loss percentage was determined on the representative samples set by means of their dry-weight change before and after heat treatment, determined after oven drying at 103 ± 2 °C to constant weight.

Colour of heat treated samples expressed in CIE $L^*a^*b^*$ system was measured on samples conditioned at room temperature of 20 ± 2 °C and relative humidity of 60 ± 5 %.

Colour was measured using the Colour Reader CR-10 (Konica Minolta, Japan), with the illuminate type D65 light source and observer angle of 8 ° and approx. ϕ 8 mm measuring area.

FT-NIR measurements

The FT-NIR spectrometer (VECTOR 22-N) produced by Bruker Optics GmbH (Germany) equipped with a fibre-optic probe was used for spectra collection. FT-NIR measurements were performed in a climatic chamber (20 °C, 60 % relative humidity), on the radial face of samples. The spectral range was between 4000 cm^{-1} and 12 000 cm^{-1} (2500 nm - 833 nm) and the resolution was set to 8 cm^{-1}. Each spectrum was collected from 32 internal scans in the absorbance mode. Four measurements were performed on each sample and resulting spectra were averaged. All measurements and subsequent data evaluation were performed at Trees and Timber Institute CNR-IVALSA in San Michele all Adige (Italy).

Data evaluation

Opus QUANT 6.5 (Bruker), PLS toolbox (Eigenvector) and LabVIEW 17 (National Instruments) software packages were used for spectral pre-processing and data mining. For the needs of this research, different evaluation methods were applied on pre-processed data (Table 2). Spectral bands (Table 3) were assigned according to SCHWANNINGER *et al.* (2011).

Tab. 2 Applied methods during FT – NIR measurements and evaluation.

Sample presentation	Acquisition mode	Regression method	Spectral pre-treatment	Attributes
Intact, manual around the surface	absorbance	*PLS*	*EMSC* DT^{2nd} *SNV*	selected components

Abbreviations: *PLS* = Partial Last Squares, *EMSC* = Extended Multiplicative Scatter Correction, DT^{2nd} = second derivatives, *SNV* = Standard Normal Variate

Generation of xylogram

Dedicated software for creation of xylograms was developed in LabVIEW. The spectral pre-processing included computation of second derivatives for all treated samples. The degradation coefficient of thermal treatment $c_{deg.\,TT}$ was calculated according to modified formula which has been published in SANDAK *et al.* (2016) as equation (1).

$$c_{\mathrm{deg.}\,TT}(\lambda) = \frac{s_{MIN}(\lambda) - s_{TT}(\lambda)}{s_{MIN}(\lambda) - s_{MAX}(\lambda)} \tag{1}$$

where: s_{MIN} – the value of DT^{2nd} absorbance spectra of reference (untreated) wood at selected wavelength,
s_{TT} – the value of DT^{2nd} absorbance spectra of treated wood at selected wavelength,
s_{MAX} – the value of DT^{2nd} absorbance spectra of extensively treated wood (240 °C, 8 hours) at selected wavelength,
λ – the wavelength of the infrared light corresponding to the particular functional group.

The outer perimeter corresponds to $c_{deg.\,TT} = 0$ and indicates negligible changes to the NIR spectra. All the results plotted within the central part of the xylogram indicate significant changes to the NIR spectra and extensive degradation of the corresponding component/functional group. A value of $c_{deg.\,TT} = 1$ indicates a fully degraded chemical component. The expected degradation pattern is that the $c_{deg.\,TT}$ values gradually change from the outer to the inner part of the xylogram, following the acquired thermal treatment dose. It

was intended that the reference points were sorted according to wavenumber value but were not grouped according to chemical component and functional group.

RESULTS AND CONCLUSION

Various intensity of heat treatment leads to mass loss and colour change due to modification process. The *ML* of wood during treatment is a key characteristic and it is often used for expressing the changes in the treated wood properties. The *ML* reflects the heat treatment process intensity as are shown in Figure 1. The *ML* varied from 0.0 % (100 °C, 1 h) to 35.8 % (240 °C, 5 h). *ML* depended on the temperature and duration what is in accordance with the state-of-the-art knowledge. Similar results were reported by ESTEVES *et al.* (2008a), who determined that mass loss varies between 0.2 % (170 °C, 1 h) up to 12.0 % (200 °C, 12 h). It was found that different coupled process parameters had comparable *ML*, for example: *ML* of ~ 6 % can be achieved when treat wood in 200 °C for 5 h or in 220 °C for 1 h. It has to be mentioned that wood degradation is more intense in the presence of atmospheric air due to extensive oxidation reactions. Moreover, acetic acid produced in such process acts as an additional depolymerisation catalyst. It was previously reported that there is a higher content of acetic acid released during wood thermal treatment in the oxidizing environment (ESTEVES *et al.* 2008a, 2007, STAMM 1956).

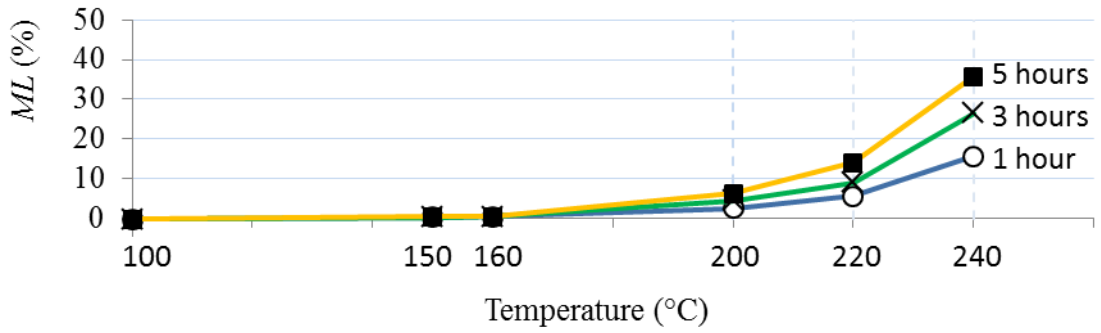


Fig. 1 Mass loss (%) of pine wood during its thermal treatment from 100 °C till 240 °C for 1, 3 and 5 hours.

The appearance and the average value of colour parameters (CIE L^*, *CIE* a^* and *CIE* b^*) of heat treated wood are shown in Figures 3, 4, 5 - parts II and III. The CIE L^* was the most sensitive parameter clearly related to the treatment intensity. There was a clear tendency of darkening with increasing of heat treatment temperature and time. In contrary, CIE a^* and CIE b^* parameters changed relatively slightly, when compare to CIE L^*. The same tendency of CIE L^*, CIE a^* and CIE b^* variations due to thermal treatment was reported by TOKER *et al.* (2016), KAMPERIDOU *et al.* (2013), AKSOU *et al.* (2011) and BEKHTA & NIEMZ (2003). Colour is an essential wood property for the final consumer. Particularly, it is the determining factor for the selection of a specific wood product for the decorative/visual function ESTEVES *et al.* (2008b).

Figure 2 presents the second derivative of an averaged near infrared absorbance spectra for thermally treated and reference (untreated) wood. The range of variations is limited only to spectral bands that can be interpreted and associated with well-defined functional groups, which are listed in table 3. Independently to applied thermal treatment (from 100 °C for 1 hour to 240 °C for 5 hours), the spectra present similar trends with typical broad vibration

bands associated to the chemical components of wood. Consequently this trend was figured upon their range, which was determined as the minimum (Figure 2 - green line) and maximum (Figure 2 - red line) value. There was clean tendency of decreasing or increasing of absorbance at various bands. These spectral changes caused by heat treatment were well corresponding to previous reports for heated pine (RIDLEY-ELLIS *et al.* 2014), larch (YANG *et al.* 2018) and spruce (POPESCU *et al.* 2018, BÄCHLE *et al.* 2010).

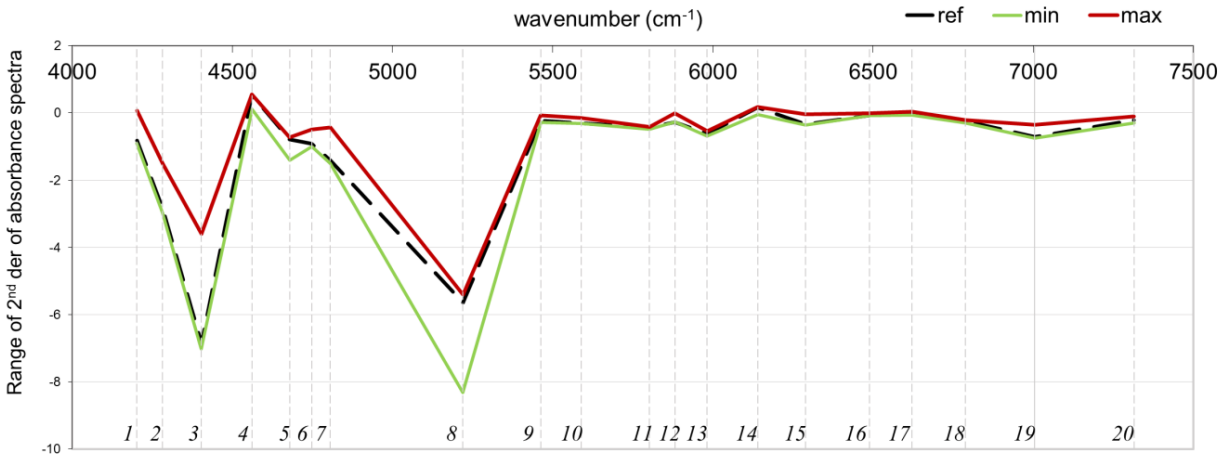


Fig. 2 The range of 2ND derivative of absorbance of FT-NIR spectra on thermally treated and reference samples (Note: used bands 1-20 correspond to components listed in Table 3).

The thermal modification of pine sapwood results in changes of the chemical composition of wood. Closer examination of the xylograms together with changes of absorbance peaks provides additional information regarding kinetics of chemical changes due to treatment temperature and duration (Figures 3, 4, 5 - parts IV. and V.). The most evident variations in NIR spectra, being consequence of changes in chemical composition of functional groups, were observed for the treatments with temperature over 200 °C. On the other hand, $c_{deg.\,TT}$ had small values when heating wood at 100 °C or other mild treatment temperatures (150 °C and 160 °C).

The absorption band at 4202 cm^{-1} (band 1 in Table 3 and Figures 3, 4, 5 - part V.) is assigned to the second overtone of –OH deformation of holocellulose. A gradual decrease of the absorbance occurred for all treatment durations; however, the changes were more intense for 3 and 5 hours heat treatment.

Hemicelluloses are polysaccharide with lower degree of polymerization than cellulose. The absorption bands present at the wavenumber 4403 cm^{-1} (3), 5882 cm^{-1} (12) and 5802 cm^{-1} (11) assigned to furanose/pyranose are due to $–CH_2$ stretching and deformation and –CH stretching. A shift in the peak position towards the higher wavelength region occurred with increase of the temperature. This confirms that the physical-chemical structure of the hemicelluloses changes rapidly and its content decreases with the temperature increase (YILDIZ & GÜMÜŞKAYA 2007, SANDAK *et al.* 2016).

In wood, cellulose has a strong interaction with water due to three hydroxyl groups attached to the glucopyranose ring. The absorption bands assigned to the first overtone of the fundamental –OH stretching mode were identified at wavenumber 4403 cm^{-1} (3), 4748 cm^{-1} (6), 6140 cm^{-1} (14), 6490 cm^{-1} (16), 6622 cm^{-1} (17), 6789 cm^{-1} (18). The absorption band at wavenumber 7005 cm^{-1} (19), assigned to -OH groups of amorphous regions of cellulose and water shows clear tendencies of its decrease with augmented temperature. The lower degradation intensity was observed for semi-crystalline (4806 cm^{-1}

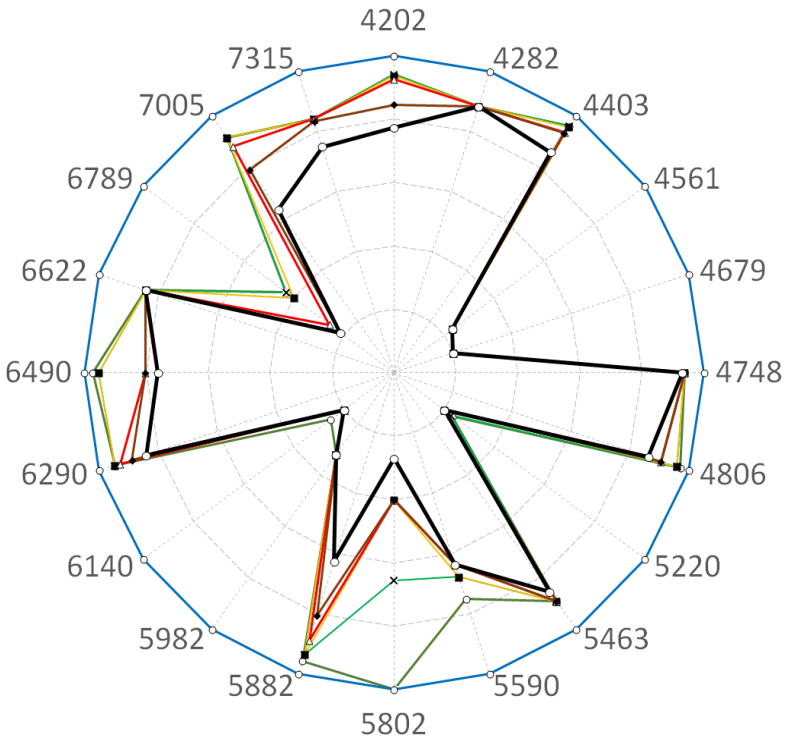
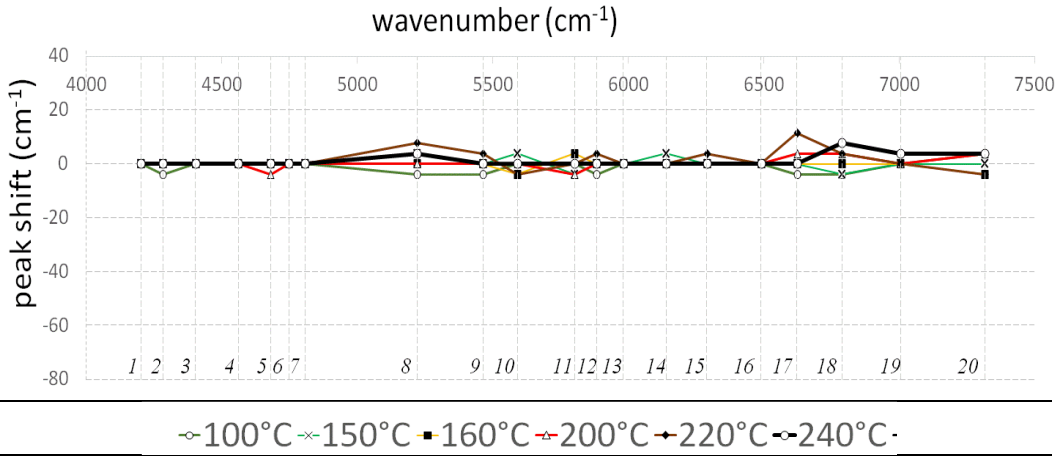
(7), 5463 cm^{-1} (9), 5590 cm^{-1} (10)) and crystalline (6290 cm^{-1} (15)), regions of cellulose. Similar tendency of degradation kinetic was recorded previously in KAČÍK *et al.* (2015) and SIVONEN *et al.* (2002).

Lignin in wood is chemically and physically bonded to cellulose and hemicelluloses forming a three-dimensional polymer complex that contains acetal, α-phenyl-β-ether, phenyl-β-glucosidic and hydrogen bonds. The absorbance of the functional groups associated to lignin (4561 cm^{-1} (4) and 5982 cm^{-1} (13)) as well as assigned to lignin and extractives at 4679 cm^{-1} (5) was reduced in all investigated treatment configurations indicating continue lignin degradation and/or condensation.

The sorption and desorption of water is an important phenomenon which highly affects mechanical and physical properties of wood (e.g. dimensional stability, shrinkage and swelling). The NIR absorption bands at 5220 cm^{-1} (8) and at 7005 cm^{-1} (19) are assigned to combination of –OH stretching and –OH bending vibration modes in water. As expected, clear changes in both absorbance bands (8 and 19) were observed when the treatment temperature increased from 100 °C to 240 °C.

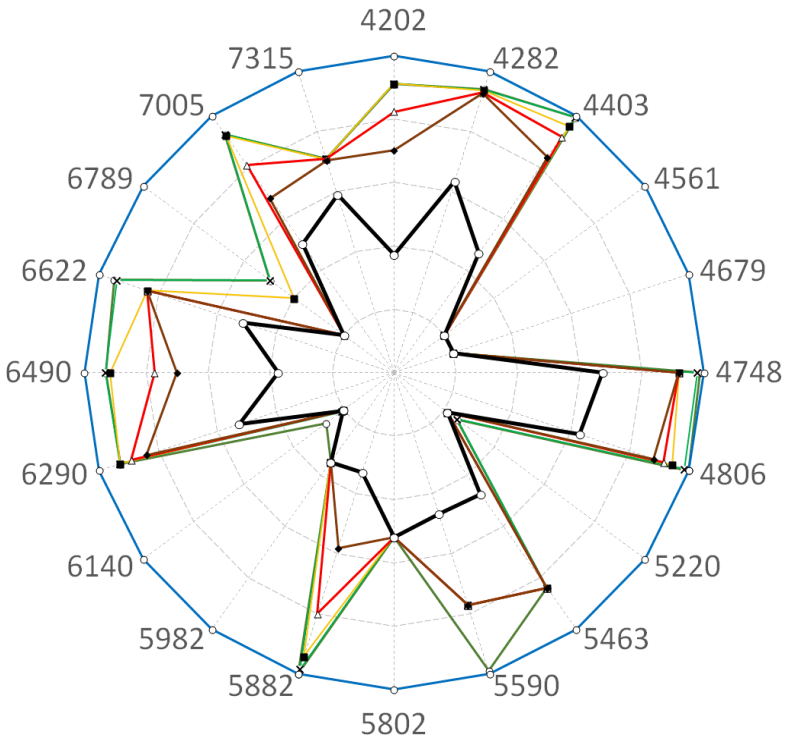
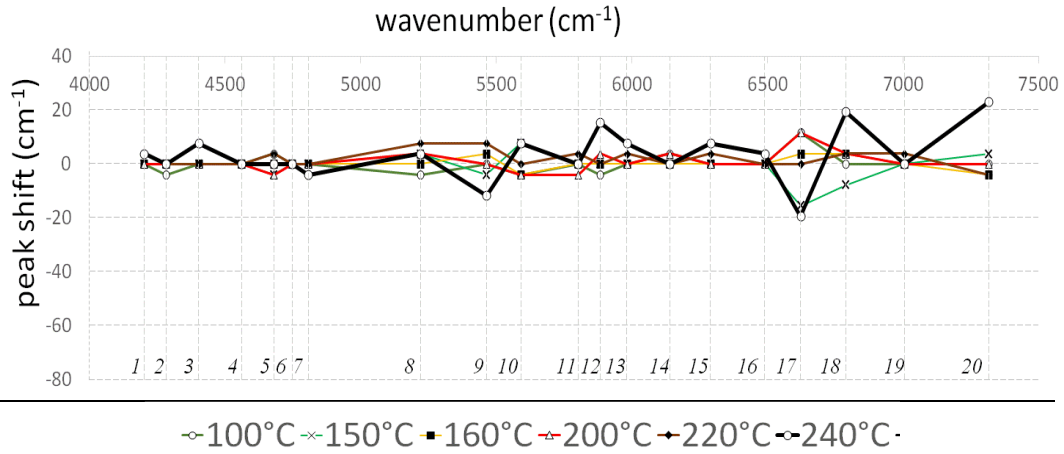
Tab. 3 Band assignments of selected wood components after thermal modification (according to SCHWANNINGER *et al.* 2011).

Band	Wavenumber (cm^{-1})	Chemical component; Bond vibration
1	4202	Holocellulose; O–H deformations (second overtone)
2	4282	Cellulose; C–H stretching, C–H_2 deformation
3	4403	Cellulose, hemicellulose; C–H_2 stretching, C–H_2 deformation,
4	4561	Lignin C–H stretching, C=O stretching
5	4679	Lignin/extractives C–H stretching, C=C stretching
6	4748	Cellulose; O–H deformation, O–H stretching
7	4806	Cellulose semicrystalline and crystalline regions, O–H stretching, C–H deformations
8	5220	Water; O–H stretching, O–H deformations
9	5463	Cellulose semicrystalline and crystalline regions; O–H stretching, C–O stretching (second overtone)
10	5590	Cellulose semicrystalline and crystalline regions; C–H stretching (first overtone)
11	5802	Hemicellulose (furanose/pyranose); C–H stretching (first overtone)
12	5882	Hemicellulose; C–H stretching (first overtone)
13	5982	Lignin; C–H stretching (first overtone)
14	6140	Cellulose; O–H, stretching (first overtone)
15	6290	cellulose crystalline regions; O–H stretching (first overtone)
16	6490	Cellulose; O–H stretching (first overtone)
17	6622	Cellulose; O–H stretching (first overtone)
18	6789	Cellulose; O–H stretching (first overtone)
19	7005	Amorphous cellulose/water; O–H stretching (first overtone)
20	7315	Cellulose; C–H stretching (first overtone), C–H deformations

		1 hour treatment					
I.		**100 °C**	**150 °C**	**160 °C**	**200 °C**	**220 °C**	**240 °C**
II.							
III.	L^*	82.13	79.08	73.84	57.91	43.82	32.75
	a^*	4.30	5.51	5.79	10.68	12.31	9.07
	b^*	20.86	23.16	23.67	25.77	20.00	12.18
IV.							
V.							

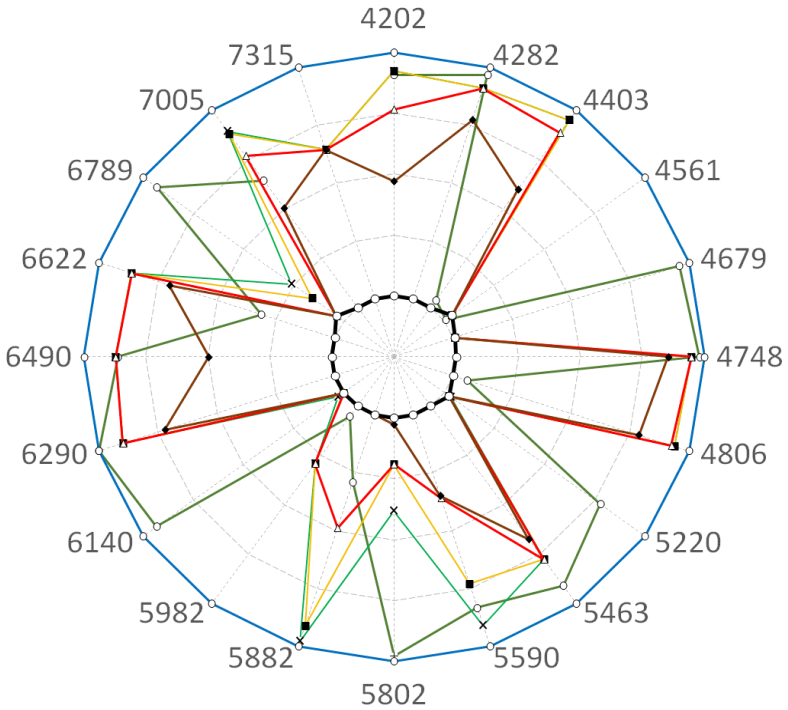
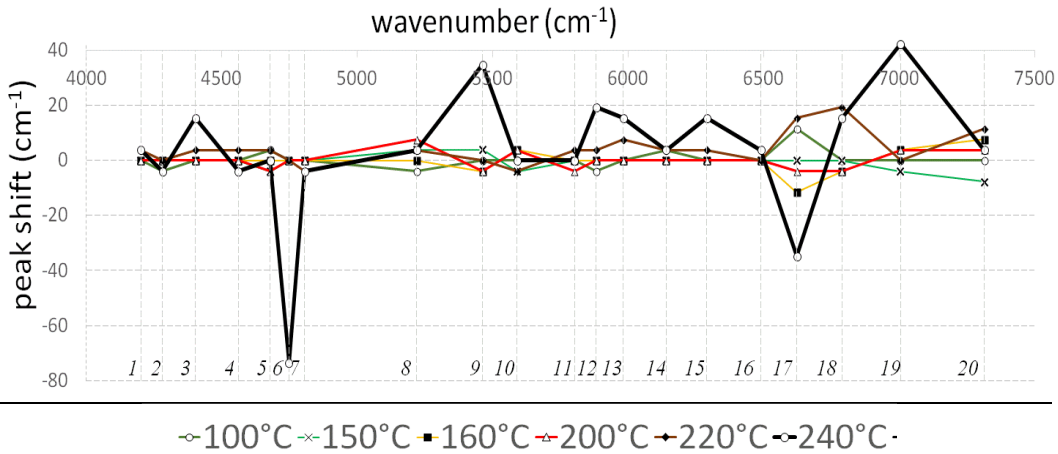
Note: I. *Temperature of heat treatment*, II. *Appearance of heat treated wood*, III. *Colour of heat treated wood* (colour coordinates after heat treatment – CIE L^*, CIE a^* and CIE b^*), IV. *Xylograms*, V. *Peak shift*

Fig. 3 Summary results for 1 hour heat treatment of pine sapwood.

		3 hours treatment					
I.		**100 °C**	**150 °C**	**160 °C**	**200 °C**	**220 °C**	**240 °C**
II.							
III.	L^*	81.82	75.09	73.81	47.45	34.61	30.26
	a^*	4.45	6.05	6.00	10.16	9.05	5.00
	b^*	22.11	24.73	23.97	22.13	13.27	5.36
IV.							
V.							

Note: I. *Temperature of heat treatment*, II. *Appearance of heat treated wood*, III. *Colour of heat treated wood* (colour coordinates after heat treatment – CIE L^*, CIE a^* and CIE b^*), IV. *Xylograms*, V. *Peak shift*

Fig. 4 Summary results for 3 hours heat treatment of pine sapwood.

		5 hours treatment					
I.		**100 °C**	**150 °C**	**160 °C**	**200 °C**	**220 °C**	**240 °C**
II.							
III.	L^*	80.66	75.92	73.65	44.71	31.59	28.31
	a^*	4.83	6.09	7.15	10.08	8.38	3.83
	b^*	21.72	25.46	25.52	20.68	10.97	1.21
IV.							
V.							

Note: I. *Temperature of heat treatment*, II. *Appearance of heat treated wood*, III. *Colour of heat treated wood* (colour coordinates after heat treatment – CIE L^*, CIE a^* and CIE b^*), IV. *Xylograms*, V. *Peak shift*

Fig. 5 Summary results for 5 hours heat treatment of pine sapwood.

CONCLUSSIONS

Treatment duration and temperature, beside of the oxygen concentration, are principal factors that affect wood chemical change and its final appearance due to thermal modification. The analyses of FT-NIR spectra provided essential information about chemical changes of wood components after that process. It was confirmed that heat-treatment of wood at elevated temperatures (over 200 °C) caused extensive destruction of hemicelluloses. Some extent of semi-crystalline cellulose and lignin degradation was also noticed but in a smaller degree. Therefore, the number of sorption sites available to link water with wood was dramatically reduced due to thermal treatment.

Profound understanding of chemical changes might be helpful for further optimization of the thermal treatment procedures at industrial scale. For that reason, xylograms are identified as a simple and illustrative method that might be highly suitable for visualization how thermal treatment effects on the chemical composition of wood. The same method may be implemented for studies on alternative modification/degradation processes of wood and other lignocellulosic materials.

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AUTHORS ADDRESS

Zuzana Vidholdová
Technical University in Zvolen
Faculty of Wood Sciences and Technology
Department of Mechanical Wood Technology
T. G. Masaryka 24
960 53 Zvolen
Slovak Republic
Corresponding author: zuzana.vidholdova@tuzvo.sk

Anna / Jakub Sandak
Trees and Timber Institute CNR-IVALSA
via Biasi 75
38010 San Michele all Adige
Italy
Corresponding author: anna.sandak@ivalsa.cnr.it

Jakub Sandak
InnoRenew CoE
Livade 6, 6310 Izola
Slovenia
e-mail: jakub.sandak@innorenew.eu

Jakub Sandak
University of Primorska
Andrej Marušič Institute
Muzejski trg 2
SI-6000 Koper
Slovenia