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THE EFFECT OF THERMAL TREATMENT WITH SATURATED WATER STEAM ON THE PROPERTIES OF BIRCH WOOD

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ABSTRACT

Industrially important properties of wood can be changed due to the modification of birch wood (*Betula pendula* L.) after the process of thermal treatment with saturated water steam. The birch wood was modified by saturated water steam at 125 ± 2.5 °C for 8 hours and at a pressure of 0.18 MPa. The water contact angle of steam-treated birch wood increased from 42.1° (for untreated birch wood) to 52.4° (for steam-treated birch wood), and the stability of water drop on steam-treated birch wood surface increased. FTIR spectra showed an increase in C=O and glycoside bonds concentration on the surface of steam-treated birch wood, but the concentration of C–O–C groups decreased. XPS measurements confirmed that the concentration of oxygen as well as C=O and C–O–O groups on the surface of steam-treated birch wood showed an increment in comparison with the native wood sample. SEM micro photos confirmed the minor changes of birch wood cells due to the action of saturated water steam modification.

Key words: birch wood, thermal treatment, saturated steam, contact angle, chemical changes

INTRODUCTION

Wood is a cellular biomaterial with a complex multi-component structure (SANDBERG *et al.* 2017). The cell wall is composed of cellulose, hemicelluloses, and lignin, and cellulose fibrils ~~are~~ joined with a soft matrix, consisting of hemicelluloses and lignin. The treatment of wood using various modification methods, e.g., corona discharge or low temperature plasma, can change the chemical and physical properties of wood (BEKHTA *et al.* 2015, BEKHTA *et al.* 2016, HILL *et al.* 2006). In the steam treating process of wood, steam alters the chemical and physical properties of wood (SANDBERG *et al.* 2013). The treatment with water steam represents a hydrothermal method of modification (AKOSHIMA and BABA 2006, HUGES *et al.* 2015, YIN *et al.* 2011). This method can improve the dimensional stability of wood as well as the colour of wood (DUDÍK *et al.* 2021). The heat and water steam modification of wood (ALTGEN *et al.* 2016a, ALTGEN *et al.* 2016b, GERARDIN 2016, GIEBELER 1983, LIN *et al.* 2017) results in chemical changes influencing its hydrophobicity. Hydrophilization and/or hydrophobization of wood by water steam were studied by authors who reported the properties of wood after steam treatment, and FTIR spectroscopy was used for analysis. The effect and mechanisms of the water steam degradation process regarding changes in the chemical structure have not been understood in detail (DZURENDA and DUDIAK 2020, BARAŃSKI *et al.* 2017). In recent years, FTIR spectroscopy has been used for

analysis of variations in the chemical structure of wood treated with steam. NUOPPONEN *et al.* detected the effect of heat treatment on the behaviour of softwood extractives (SANDERMANN and AUGUSTIN 1964, NUOPONNEN *et al.* 2005, GIEBELER 1983, ADL-ZARRABI and BOSTRŐM *et al.* 2004). FT-NIR spectroscopy was used for the analysis of variations in the chemical structure of wood treated with heat (VIDHOLDOVÁ *et al.* 2019).

The aim of this study was to investigate the effects of water steam-treatment process on the chemical changes of wood components. A further aim was to identify whether these changes correlate with the surface properties of selected wood.

MATERIALS AND METHODS

The samples of birch wood (*Betula pendula* L.) (Technical University in Zvolen, Slovakia) were pre-treated in an autoclave by saturated water steam under these conditions: the temperature of 125 °C, treatment time 8 hours and a pressure of 0.18 MPa. The final value of the moisture content of test pieces with dimensions of 50×15×5 mm was 8 %. The surface of test pieces was sanded by the grit paper P180.

The physical and chemical changes were observed using measurements of water contact angles (WCA) by contact angle meter, Fourier Transform Infrared - Attenuated Total Reflectance (ATR-FTIR), X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) for all investigated birch wood samples.

Contact angle

The drops of testing liquid (re-distilled water, V = 20 μl) were placed on the wood surface with a micropipette (Biohit, Finland), and the stable value of contact angle, due to penetration of water into wood, was determined. The contact angle measurements of birch wood with water were carried out using the professional Surface Energy Evaluation (SEE) system completed with a web camera (Advex, Czech Republic) and necessary PC software. The measurements of contact angle were repeated 12 times and the arithmetic mean with measurement standard deviation has been considered.

ATR-FTIR spectroscopy

FTIR spectroscopy measurements were performed with the FTIR NICOLET 8700 spectrometer (Thermo Scientific, UK) using a single bounce ATR accessory equipped with a Ge crystal. For each measurement, the spectral resolution was 2 cm^{-1} and 64 scans were performed. The infrared spectra of wood samples (native and steamed) were recorded in micro-ATR mode using the CONTINUUM™ infrared microscope, which is an integral part of the NICOLET 8700™ infrared spectroscope (ThermoScientific, Madison, WI, USA) in the middle infrared region (4000-650 cm^{-1}). From each sample type, 20 spectra were taken at different points – 10 from both sides (the locations were selected at random).

X-ray photoelectron spectroscopy

XPS spectra were recorded using a VG Scientific ESCALAB 250 (Thermo Fisher Scientific Inc., UK) device equipped with a micro-focused, monochromatic Al K_α X-ray source (1486.6 eV) and a magnetic lens which increases the electron acceptance angle and hence the sensitivity. The spectra were acquired in the constant analyser energy mode, with pass energies of 150 and 20 eV for the survey and narrow regions, respectively. The Avantage software, version 2.2, was used for digital acquisition and data processing. Spectral calibration was performed by setting the main C1s peak at 285 eV (binding energy (BE) for the C-H 1s peak in eV). A more detailed analysis of the XPS spectra of the steam

treated wood reveals the formation of different oxygen-containing functional groups XPS spectra of the carbon C1s region can be observed at binding energy 289.0, 287.9 and 286.6 eV and indicate the existence of carboxyl, carbonyl and alkoxy groups. The incorporation of oxygen into wood can take place while the substrate is shortly after steam treatment.

SEM microscopy

SEM method was used for investigating the birch wood morphology. Native and modified birch wood by steam were compared and discussed with results received by other experimental techniques. SEM analysis was carried out using JSM 6400 Microscope (JEOL, Japan). The specimens were sputter-coated (SCD 050, BALTEC) with a thin Pt layer (4 nm).

RESULTS AND DISCUSSION

Water contact angle

The water contact angle on the investigated native birch wood surface was relatively small and it is equal 41.6° (Table 1). After modification of birch wood with saturated water steam (T = 125 °C, t = 8 hours, and p = 0.18 MPa) the value of WCA increased to the value θ = 52.8° due to higher hydrophobicity of steam-treated birch wood surface. An increase in WCA for birch wood is related to the chemical changes after wood modification by saturated water steam. The hydrophilicity of the birch wood surface is related to the amount of oxygen-based polar functional groups created after birch wood treatment with water steam.

Tab. 1 Water contact angle of native birch wood and water steam-treated birch wood.

Native wood sample No.	WCA (°)	Steam-treated wood sample No.	WCA (°)
1	42.1	1	52.4
2	41.4	2	53.4
3	41.7	3	52.6
4	42.4	4	52.0
5	41.2	5	52.2
6	40.0	6	52.8
7	42.2	7	53.2
8	41.6	8	53.4
9	41.8	9	52.8
10	41.4	10	52.4
11	41.6	11	53.2
12	42.1	12	52.7
	Mean = **41.6**		Mean = **52.8**

The dependence of WCA of native birch wood treated by water steam versus the time that passed since the drop was placed, is illustrated in Table 2. WCA of native birch wood (42.1°) diminished with time after water drop deposition, and after 20 seconds WCA decreased to 19.8°. After 30 s the WCA was non-measurable due to the complete absorption of water drop into birch wood. In the case of steam-treated birch wood, the WCA decreased more slowly in comparison with the untreated sample and after 300 seconds the value of WCA reached 17.2° without drop absorption.

The results obtained by KÚDELA *et al.* (2020) confirmed that the thermal treatment of beech wood (*Fagus sylvatica* L.) significantly improved this wood surface resistance to wetting by water. The time necessary for the complete soaking of the drop into the substrate was one order of magnitude longer than in untreated wood.

Tab. 2 Water contact angle of native birch wood and modified by water steam versus time elapsed from drop location.

Native wood, time from drop location (s)	WCA (°)	Sample treated by steam, time from drop location (s)	WCA (°)
0	42.1	0	52.4
10	26.4	30	44.4
20	19.8	60	28.4
30	absorbed	120	20.6
60	absorbed	180	18.4
120	absorbed	300	17.2

FTIR spectroscopy

Figure 1 illustrates the FTIR spectra of native birch wood (blue) and water steam-treated birch wood (red), the entire middle infrared region shows the area of deformation vibrations. There are important following regions: C=O vibration region (1710-1697 cm^{-1}) with a maximum absorbance at about 1738-1726 cm^{-1}, undifferentiated multi-peak band: 1160, 1110, 1056 and 1033 cm^{-1}, C–O–C bond region (1190-920 cm^{-1}), and band with a maximum at 896 cm^{-1} (β (1,4) glycoside bond) (CIOLACU *et al.* 2011).

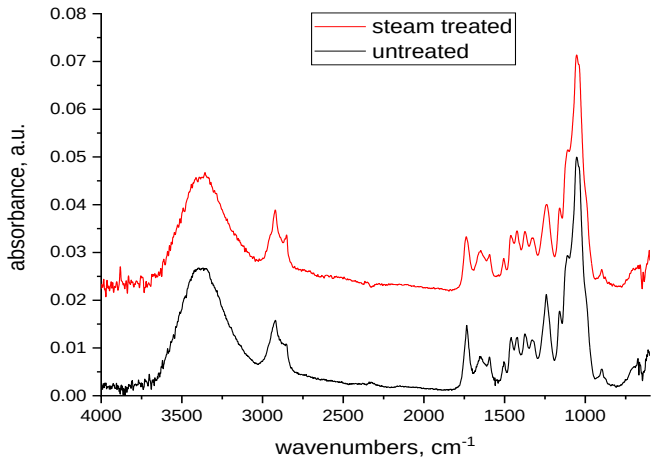

Fig. 1 FTIR spectra of native birch wood (blue) and steam-treated birch wood (red), in the middle infrared region.

The visual comparison of the measured FTIR spectra of native and steam-treated birch wood (Figure 2 and Figure 3) shows that changes in the shape of the bands, or in their intensities are better visible in the area of deformation vibrations.

Due to the chemical changes generated during the wood water steaming process, it can be concluded that three regions were taken from this region: region C=O vibrations with maximum absorbance at ca 1738-1726 cm^{-1}, the second region is the C–O–C bond region (between 1190-920 cm^{-1}, undifferentiated multi-peak band: 1160, 1110, 1056, and 1033 cm^{-1}) as well as the band with a maximum at 896 cm^{-1} (β (1,4) glycoside bond).

Chemical changes owing to wood under hydrothermal treatment are confirmed and described by many authors. The research of VIDHOLDOVÁ *et al.* (2019) investigated heat-treated pine sapwood (*Pinus sylvestris* L.) at the different temperatures from 100 °C to 240 °C. They found that gradual degradation of the amorphous share of cellulose was caused by high temperature, while the crystalline and semi-crystalline share of cellulose were less affected by the thermal treatment. Thermochemical changes during heat loading up to 550 °C were investigated by BELLEVILLE *et al.* (2013) in two hardwood species: sugar maple

(*Acer saccharum*) and yellow birch (*Betula alleghaniensis*). ATR-FTIR and XPS spectroscopy were used, and the results showed that hemicelluloses were degraded by thermal treatment of birch and maple wood and lignin polymer is affected through depolymerisation.

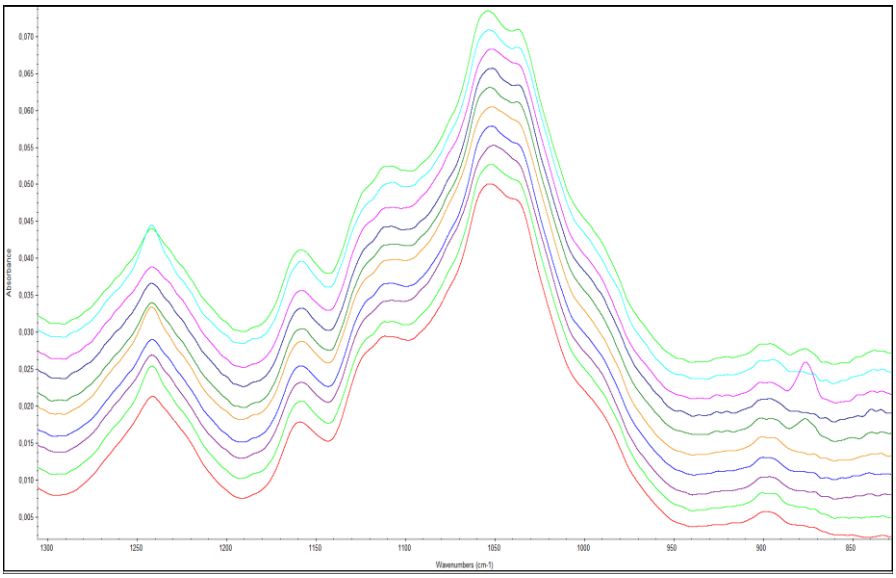

Fig. 2 FTIR spectra with C–O–C vibrations region, native birch wood.

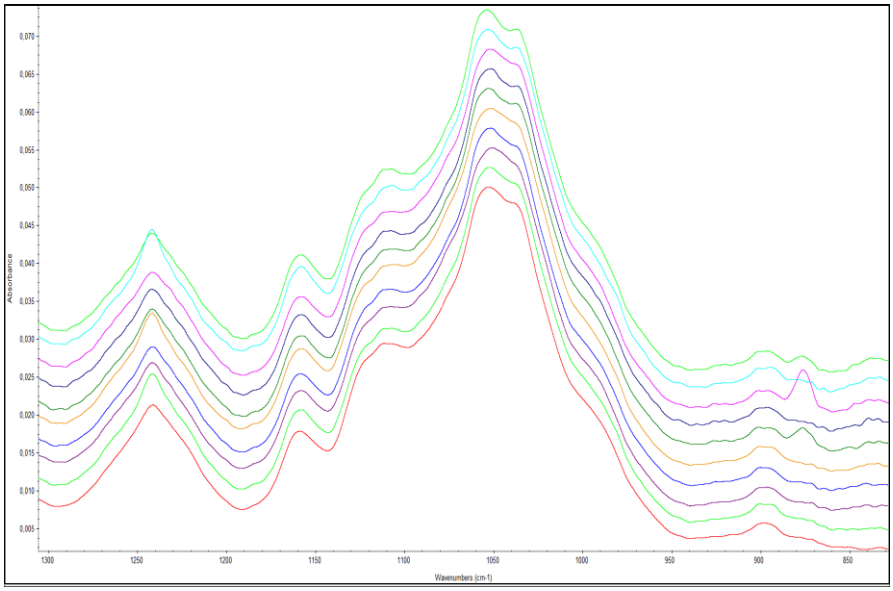

Fig. 3 FTIR spectra with C–O–C vibrations region, water steam-treated birch wood.

Three selected regions mentioned above were compared to the CH_2 bond region (band in the range 1440-1396 cm^{-1}), which was chosen as the "internal" standard, as there is a presumption that spectrum changes in this region are negligible compared to the changes in the other regions (CIOLACU *et al.* 2011, MULLER *et al.* 2009). Comparison of the area ratios in the regions was performed by manual calculation for each spectrum separately. An attempt was also made to construct a "chemical map of the surface" from individual spectra using the "series" routine, but the predictive value of this map is mainly to confirm the homogeneity of the samples. The results of FTIR measurements introducing changes in the

ratio of oxygenic functional groups and (β 1-4) glycosides to non-polar groups for native birch wood and steam-treated birch wood determined by FTIR are summarized in Table 3.

Tab. 3 Changes in ratio of oxygenic functional groups and (β 1-4) glycosides to non-polar groups for native birch wood and steam-treated birch wood determined by FTIR.

Sample No.	Native sample			Steam-treated sample		
	P(C=O)/ P(CH_2) 1732 cm^{-1}	(P(C–O–C)/ P(CH_2) 1037 cm^{-1}	P(β1-4)/ P(CH_2) 896 cm^{-1}	P(C=O)/ P(CH_2) 1732 cm^{-1}	(P(C–O–C)/ P(CH_2) 1037 cm^{-1}	P(β1-4)/ P(CH_2) 896 cm^{-1}
1	11.676	108.518	0.851	9.194	97.516	0.761
2	8.563	100.092	0.923	9.998	98.887	0.676
3	9.661	109.774	0.773	13.254	83.973	0.504
4	13.932	110.698	0.773	10.060	98.073	0.804
5	17.851	98.850	0.675	6.690	101.245	1.197
6	6.967	103.470	0.659	8.086	91.606	0.661
7	6.716	101.715	0.956	8.559	98.813	0.608
8	10.425	104.773	0.748	5.716	75.095	0.628
9	16.215	101.484	0.389	5.781	56.791	0.471
10	9.566	118.166	0.878	7.795	97.516	0.761

The values of the proportions of the individual areas in the carbonyl region are comparable for the water steam-treated sample as well as for the native birch wood, but a higher level of wood oxidation and degradation can be assumed in the steamed sample. This result ~~was~~ obtained for (P (C–O–C)/P (–CH_2) area ratios and the variance in the values is lower for the steamed sample. In the area of C–O–C bonds, the changes are easier to observe. The ratio of P (β1-4 glycosides)/P (CH_2) slightly decreased comparing the steamed wood with native one. Based on the FTIR results presented in Table 3, it can be concluded: P (C=O)/P (CH_2) = 11.16 : 8.51 (native compared with steam-treated birch wood); (P (C–O–C)/P (CH_2) = 105.76 : 90.0; P(β1-4)/P (CH_2) = 0.71 : 0.76, i.e. native birch wood contains higher amount of C=O and (β1-4) glycosides groups as well as higher amount of O–C–O groups. If we summarize oxygen-functional groups for unmodified and steam-modified birch wood, we can compare the effect of wood treatment by steam on the wood chemical composition: P (C=O) + P (C–O–C)/P (CH_2) = 116.92 : 98.51. It can be stated that the amount of oxygenated functional groups of birch wood determined by the FTIR after steam treatment of birch wood decreased.

XPS spectroscopy

XPS measurements of birch wood before and after modification with steam are presented in Figure 4. The content of C=O groups on the birch wood surface (black line) decreased after the treatment with steam (green line).

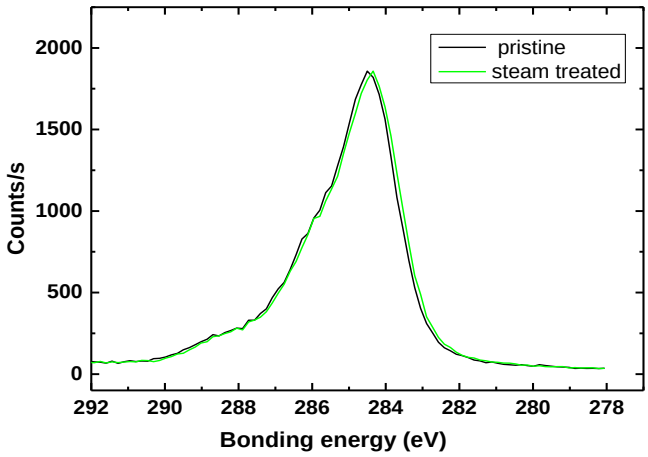

Fig. 4 XPS measurements for native (black) and steam-modified (green) birch wood.

The content of carbon (C1s) after the steam treatment of birch wood increased from 74.2 to 76.0 At.%, and the amount of oxygen in the same case decreased from 23.6 to 22.6 At.% (Table 4). The content of nitrogen also slightly decreased from 1.1 to 0.9 At.%. This finding is related to the degradation of the birch wood during the water steam treatment, as the amount of carbon on the surface of the birch wood increases, and the amount of oxygen, on the other hand, decreases. Because of its degradation, a decrease in the amount of oxygenic functional groups on the birch wood surface results in an increase in the wood's hydrophobicity and, consequently, in a decrease in the values of water-contact angles. A decrease in the amount of oxygen (O1s) measured by XPS was also confirmed by the results of FTIR measurements. These results agree with the results of other authors who determined the chemical changes after hydrothermal treatment on other woods. SRINIVAS and PANDEY (2012) also stated that a decrease in hydroxyl groups reduced the hygroscopic nature, resulting in increased dimensional stability of thermally modified rubber wood (*Hevea brasiliensis*) and silver oak (*Grevillea robusta*) wood. GEFFERT *et al.* (2019) examined chemical changes that result from the hydrothermal treatment of oak (*Quercus robur* L.) wood through various steaming modes. An increase in temperature and extension of the steaming period primarily affected the holocellulose and extractives contents, and less the contents of cellulose and lignin.

Tab. 4 XPS for native and water steam modified birch wood.

Element	Start BE	Peak BE	Native sample (At.%)	Water steam-treated sample (At.%)
C1s	292.08	285.34	74.2	76.0
O1s	538.58	532.87	23.6	22.6
N1s	405.57	400.14	1.1	0.9
Si2p	106.89	102.20	0.5	0.6
Ca2p	353.05	347.39	0.1	0.1
S2p	171.87	168.21	0.1	0.1

SEM microscopy

The SEM investigation of native birch wood unmodified and steam-modified wood is shown in Figure 5A and Figure 5B. Prepared scans confirmed that the effect of water steam on the birch wood is practically not observable.

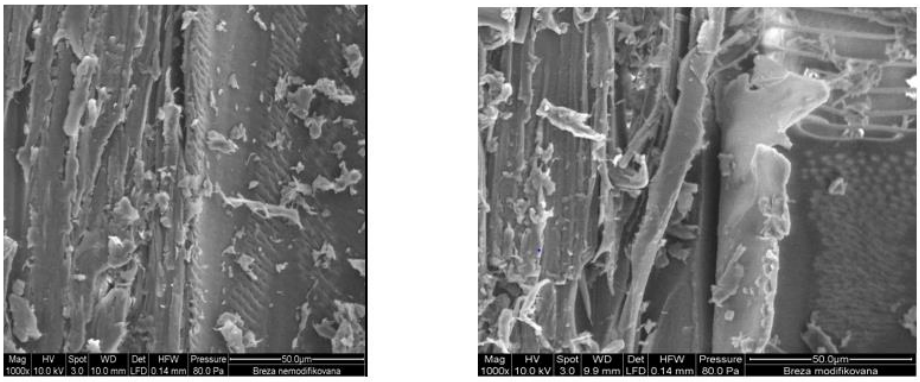

Fig. 5 SEM micro photos of unmodified birch wood (a) and birch wood modified by water steam (b).

Detailed analysis of changes that occurred in the microstructure of birch wood after hydrothermal treatment was carried out by BIZIKS *et al.* (2013). Test pieces were placed in an autoclave, which ensured the thermal treatment in a water vapour medium at the modification temperatures of 140, 160 and 180 °C. They stated that the integrity of wood morphological structure begins to break up after the treatment at 180 °C. Only minor changes in the sizes of all morphological elements (libriform, vessels, rays, annual rings) were found after the treatment at 140 °C.

CONCLUSION

The effect of saturated water steam modification on the surface properties of birch wood was investigated. Hydrophobicity of birch wood after water steam modification was found to increase. A decrease in the absorption rate of water drops was confirmed on the steam-treated birch wood surface. XPS and FTIR measurements of water steam-treated birch wood confirmed a decrease in oxygenic functional groups content as well as an increase in carbon content on the birch wood surface. SEM measurements confirmed minor changes in the morphology of birch wood structures after modification with water steam.

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