WOOD RESEARCH
65 (6): 2020
865-876

doi.org/10.37763/wr.1336-4561/65.6.865876

PROPERTIES OF WATER STEAM-TREATED MAPLE WOOD (*ACER PSEUDOPLATANUS* L.)

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(Received April 2020)

ABSTRACT

The hydrothermal treatment of maple (*Acer pseudoplatanus* L.) wood by steam represents the modification method with the effective heat transfer, which can improve industrially significant properties of wood, i.e. its color, hydrophobicity and subsequently dimensional stability. The maple wood was modified by steam at 125°C during 8 hours, and at pressure of 0.18 MPa. The water contact angle of steam-treated maple wood increased from 44.9° (for untreated maple wood) to 55.3° (for steam-treated maple wood), and the stability of water drop on steam-treated maple wood surface increased. FTIR spectra show an increase in C=O and glycoside bonds concentration on the surface of steam-treated maple wood, but the concentration of C-O-C groups decreased. SEM micro photos confirmed the deformation and shrinking of maple wood cells due to steam treatment. XPS measurements confirmed, that the concentration of oxygen as well as C=O and C-O-O groups on the surface of steam-treated maple wood showed a slight decrease in comparison with pristine wood sample.

KEY WORDS: Maple wood, hydrothermal modification, contact angle, chemical changes.

INTRODUCTION

Wood is a cellular biomaterial with a complex multicomponent structure. The cell wall is mainly composed of cellulose, hemicelluloses, and lignin, and cellulose fibrils are joined with soft matrix, consisting of hemicelluloses and lignin (Sandberg et al. 2017). Wood modification is a term describing the application of either chemical, mechanical, physical, or biological methods to alter the properties of the wood (Bekhta et al. 2015, Bekhta et al. 2016, Hill et al. 2006). The modified wood should itself be nontoxic under service conditions, and furthermore, there should be no release of any toxic substances during service, or at end of life, following disposal or recycling of the modified wood (Sandberg et al. 2013). To modify wood, four main types of processes can be implemented: (1) chemical treatments, (2) thermo-hydro (TH) and thermo-hydro-mechanical (THM) treatments, (3) treatments based on biological processes, (4) physical treatment with the use of electromagnetic irradiation or plasma.

The thermophysical properties of wood have been widely studied in the literature (Akoshima and Baba 2006, González-Peña et. al 2009, Niemz et al. 2010, Nuopponen et al. 2005). The heat transfer in wood depends on the geometry of the wood sample as well as porosity, moisture content, and many other factors, e.g. thermal modification parameters (Kol 2009). Because wood is a hygroscopic material, it mostly contains water in the form of bonded water or free water. The amount of water has a profound effect on almost all properties of wood, including its thermal properties.

The water steam modification of wood at higher temperature is one of thermo-hydro treatments, and influences its final hydrophobicity (Akoshima and Baba 2006, Huges et al. 2015, Yin et al. 2011). This type of wood modification alters its chemical and physical properties (Altgen et al. 2016a, Altgen et al. 2016b, Gerardin 2016, Giebler 1983). The color of wood is one of the macroscopic features to identify individual tree species visually. Chromophores are molecules responsible for the color of wood, aromatic compounds absorbing light in the UV/VIS spectra present in the chemical components of wood such as lignin and extractive substances. Wood becomes darker when the steaming process is used to remove the undesirable color differences between light colored sapwood and dark colored heartwood. Also, the steaming changes the color of specific wood species into more or less bright shades or enhances the imitation of domestic to exotic tree species (Dzurenda and Dudiak 2020, Barański et al. 2017).

Chemical modification of wood using thermal pre-treatment represents frequently used method for preparation of wood with higher hydrophilicity or hydrophobicity (Sandermann and Augustin 1964, Nuoponnen et al. 2005, Giebler 1983, Adl-Zarrabi and Boström et al. 2004). Hydrophobization of wood by water steam were studied and FT-NIR spectroscopy was used to characterize this property for years. The effect and mechanisms of the water steam degradation process regarding to changes in the chemical structure have not been in details understood yet. FT-NIR spectroscopy was used for analysis of variations in 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 physical and chemical properties of maple wood and the chemical changes in the wood components. A further aim was to identify whether these changes correlate with surface properties as well as microscopic alterations of the selected wood species.

MATERIAL AND METHODS

The samples of maple wood (*Acer pseudoplatanus* L.) with dimensions 50×15×5 mm (Technical University in Zvolen, Slovakia) with the moisture content of 8% were pre-treated with water steam at these conditions: the temperature was 125°C, treatment time 8 hours and pressure 0.18 MPa. The physical and chemical changes were observed using measurements of water contact angles by contact angle meter, FTIR-ATR, XPS and SEM for all investigated maple wood samples.

Contact angles

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 angles measurements of maple wood with water were carried out using professional SEE (Surface Energy Evaluation) system completed with a web camera (Advex, Czech Republic) and necessary PC software. The measurements of contact angles were repeated on three different points of tangential surface on both sides of two samples and the arithmetic mean with measurement standard deviation has been taken into account.

Fourier transform infrared - Attenuated total reflectance (FTIR-ATR) spectroscopy

FTIR-ATR spectroscopy measurements were performed with the FTIR Nicolet 8700 spectrometer (Thermo Scientific, Madison, Wisconsin, USA) 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 (pristine and steamed) were recorded in micro ATR mode using the Continuum™ infrared microscope, which is an integral part of the Nicolet 8700 infrared spectroscope in the middle infrared region (4000-650 cm^{-1}). From each sample type, 20 spectra were taken at 10 different points on the surface of both sides (the locations were selected at random).

X-ray photoelectron spectroscopy (XPS)

The surface of pristine and steam-treated maple wood samples with dimensions 15×15×10 mm was irradiated by X-ray source. 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 for the C-H 1s peak in eV).

Scanning electron microscopy (SEM)

SEM method was used for investigating the maple wood morphology. Pristine maple wood and maple wood modified by steam were compared and discussed with results received by previously mentioned experimental techniques. SEM analysis was carried out using JSM 6400 Microscope (JEOL, Japan). The SEM samples taken from tangential surface of pristine and modified maple wood were sputter-coated (SCD 050, Baltec) with a thin Pt layer (4 nm).

RESULTS AND DISCUSSION

Water contact angle

The water contact angle (WCA) on the investigated pristine maple wood surface measured on 12 various places is relatively small and it is equal 44.9° (Tab. 1). After modification of maple wood with steam (T = 125°C, t = 8 hours, and p = 0.18 MPa) the value of WCA increased to value θ = 55.3° due to higher hydrophobicity of steam-treated maple wood surface.

Tab. 1: Water contact angle of pristine maple wood and steam-treated maple wood.

Pristine sample No.	WCA (°)	Steam-treated sample No.	WCA (°)
1	46.2	1	54.8
2	45.4	2	55.4
3	44.5	3	54.0
4	42.2	4	54.6
5	44.2	5	56.0
6	45.0	6	53.8
7	46.2	7	54.8
8	45.6	8	55.2
9	43.8	9	56.0
10	45.4	10	56.8
11	44.6	11	57.2
12	45.4	12	54.8
	Mean = 44.9		**Mean = 55.3**

The increase of WCA for steam-treated maple wood can be explained by chemical changes on/in steam modified maple wood. The hydrophilicity of the wood surface depends on the amount of polar oxygenic functional groups creating using modification of maple wood with steam. Kocaefe et al. (2008) investigated the effect of the heat treatment on the contact angle, consequently, on the wetting. They stated that heat treatment of North American white ash and soft maple increased the contact angle between wood and water indicating a decrease in wood wettability. This is in agreement with the results of this study.

The dependence of WCA versus time elapsed from drop deposition on pristine maple wood and steam-treated maple wood is summarized in Tab. 2. WCA of pristine maple wood (45.4°) diminished with time after water drop deposition, and after 20 s WCA decreased to 18.5°. After 30 seconds the WCA was non-measurable due to absorption of water drop into maple wood. In the case of steam-treated maple wood, the WCA decreased more slowly in comparison with untreated sample and after 300 seconds the value of WCA reached 14.2° without drop disappearance.

Tab. 2: Representative water contact angle of untreated and steam-treated maple wood versus time elapsed from drop deposition.

Pristine sample, time from drop deposition (s)	WCA (°)	Steam-treated sample, time from drop deposition (s)	WCA (°)
0	45.4	0	54.8
10	28.4	30	42.4
20	18.5	60	28.0
30	absorbed	120	15.6
60	absorbed	180	15.0
120	absorbed	300	14.2

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

FTIR-ATR spectroscopy

Fig. 1 illustrates the FTIR-ATR spectra of pristine maple wood (blue) and steam-treated maple wood (red) within the entire middle infrared region and Fig. 2 shows the FTIR-ATR spectra of pristine maple wood (blue) and steam-treated maple wood (red) in the local area of deformation vibrations.

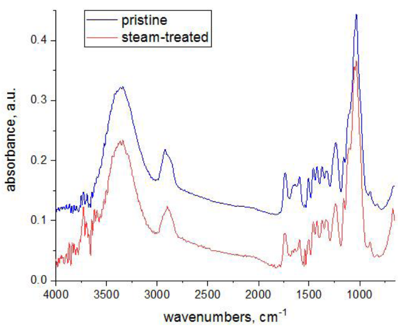


Fig. 1: FTIR-ATR spectra of pristine maple wood (blue) and steam-treated maple wood (red), the entire middle infrared region.

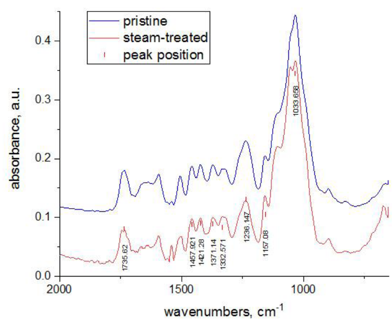


Fig. 2: FTIR-ATR spectra of pristine maple wood (blue) and steam-treated maple wood (red), area of deformation vibrations.

From FTIR-ATR spectra shown in Fig. 1 and Fig. 2 could be found three following regions: (1) C=O vibration region (1710-1697 cm^{-1}), maximum absorbance at about 1738-1726 cm^{-1}. (2) C-O-C bond region (1190-920 cm^{-1}), undifferentiated multi-peak band: 1160, 1110, 1056 and 1033 cm^{-1} and although this band is not very pronounced but is characteristic of cellulose, (3) band with a maximum at 896 cm^{-1} (β (1,4) glycoside bond) (Ciolacu et al. 2011).

The visual comparison of the measured FTIR-ATR spectra of pristine and steam-treated maple wood (Fig. 3 and Fig. 4) revealed that changes in the shape of the bands, respectively in their intensities, they are better visible in the area of deformation vibrations. The curves in Fig. 3 and Fig. 4 represents different samples from the same type of wood (pristine and steam treated maple wood). Therefore, to compare whether or not what chemical changes occur during the wood steaming process.

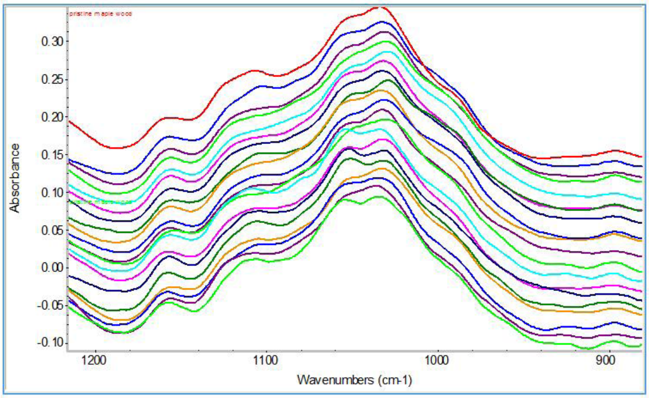


Fig. 3: FTIR-ATR spectra with C-O-C vibrations region, pristine maple wood.

Chemical changes of 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 different temperatures from 100°C to 240°C. They found that gradual degradation of amorphous share of cellulose was caused by high temperature, while 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 results showed that thermal welding of birch and maple woods degrades hemicelluloses and affects lignin polymer through depolymerisation.

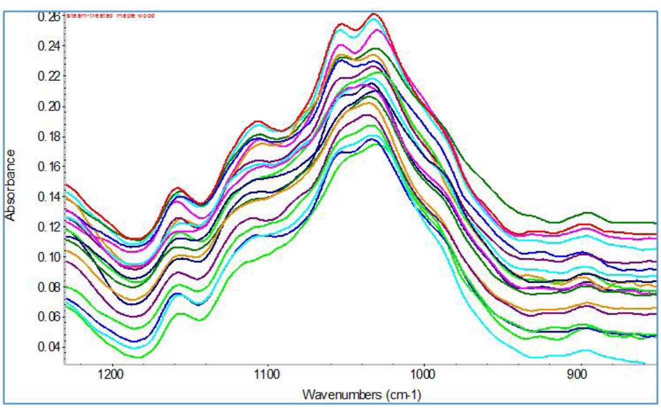


Fig. 4: FTIR-ATR spectra with C-O-C vibrations region, steam-treated maple wood.

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 changes in 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.

The values of the proportions of the individual areas in the carbonyl region are comparable for both the steamed and the pristine maple wood samples, but a greater variance in the data is observed for the steamed sample, which would indicate oxidation and degradation of the wood. The same trend was observed when comparing the area ratios (P (C-O-C)/P ($-CH_2$) with the variance in the values being lower for the steamed sample. There is a presumption that steaming caused "homogenization" of the sample in terms of its chemical composition (degradation of some bonds, or formation of oxidation products), whereas in the area of C-O-C bonds these changes are easier to observe (the band is not absorbance is higher than that of other bands). As far as the ratio of P (β1-4 glycosides)/P (CH_2) areas is concerned, in this case, comparing steamed wood with pristine one, it appears that the content of glycoside bonds after steaming slightly increased. On the base of average values of FTIR-ATR measurements summarized in Tab. 3 can be concluded: $[P(C=O)/P(CH_2)]_{pristine} : [P(C=O)/P(CH_2)]_{steamed} = 4.44 : 4.70$ (pristine compared with steamed maple wood); $[(P(C\text{-}O\text{-}C)/P(CH_2)]_{pristine} : [(P(C\text{-}O\text{-}C)/P(CH_2)]_{steamed} = 70.87 : 66.71$; $[P(\beta1\text{-}4)/P(CH_2)]_{pristine} : [P(\beta1\text{-}4)/P(CH_2)]_{steamed} = 0.37 : 0.50$, i.e. pristine maple wood contains lower concentration of C=O and (β1-4) glycosides groups, but higher concentration of O-C-O groups. If we summarize oxygen-functional groups for unmodified and steam-modified maple wood, we can compare the effect of wood treatment by steam on chemical composition: [P (C=O) + P (C-O-C)/P (CH2)]pristine : [P (C=O) + P (C-O-C)/P (CH2)]steamed = 75.31 : 71.41. It can be stated, that the amount of oxygenated functional groups of maple wood determined by the FTIR-ATR after steam treatment of maple wood slightly decreased (75.31 : 71.41).

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

Sample No.	Pristine sample			Steam-treated sample		
	$P(C=O)/P(CH_2)$	$(P(C\text{-}O\text{-}C)/P(CH_2)$	$P(\beta1\text{-}4)/P(CH_2)$	$P(C=O)/P(CH_2)$	$(P(C\text{-}O\text{-}C)/P(CH_2)$	$P(\beta1\text{-}4)/P(CH_2)$
1	4.34	78.18	0.44	4.86	63.02	0.45
2	4.20	66.21	0.25	4.71	63.86	0.67
3	4.71	67.60	0.40	5.06	71.03	0.46
4	4.49	81.53	0.29	3.72	94.18	0.69
5	4.98	69.31	0.25	6.56	61.05	0.34
6	4.67	65.51	0.26	4.29	53.95	0.54
7	4.31	81.18	0.43	2.21	44.05	0.27
8	4.91	68.01	0.40	5.45	75.81	0.55
9	3.79	66.15	0.74	4.64	59.56	0.32
10	4.01	65.02	0.25	5.54	80.56	0.71
Average	4.44	70.87	0.37	4.70	66.71	0.50

XPS measurements

XPS measurements of maple wood before and after modification with steam are included in Fig. 5.

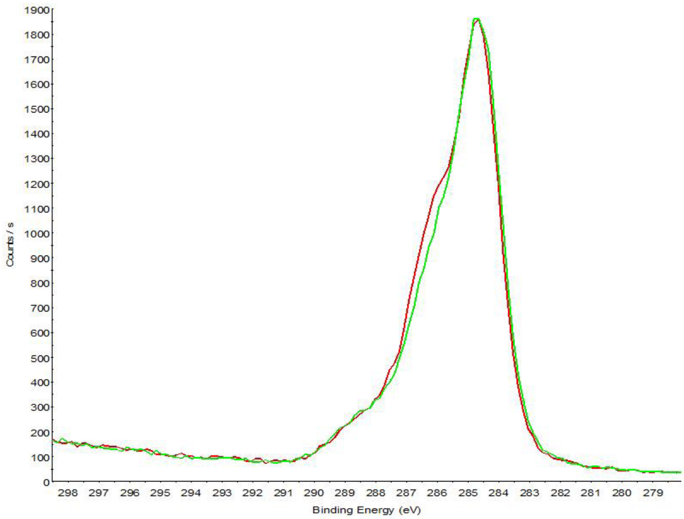


Fig. 5: XPS measurements for maple wood (red) and steam-treated (green) maple wood.

By Fig. 5, the content of C=O groups on the maple wood surface (red line) shows a slight decrease after treatment with steam (green line). The content of carbon (C1s) after steam treatment of maple wood increased slightly from 74.4 to 75.8 At.%, and in the same case we observed a slight decrease in concentration of oxygen from 23.8 to 22.5 At.% (Tab. 4).

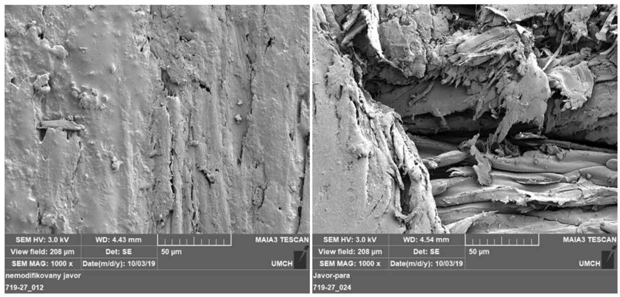
Tab. 4: XPS for pristine and steam-treated maple wood.

Element	Start BE	Peak BE	Pristine sample (At.%)	Steam-treated sample (At.%)
C1s	292.08	285.34	74.4	75.8
O1s	538.58	532.87	23.8	22.5
N1s	405.57	400.14	1.0	0.8
Si2p	106.89	102.20	0.6	0.7
Ca2p	353.05	347.39	0.1	0.1
S2p	171.87	168.21	0.1	0.1

This finding is related to the degradation of the wood during the steam treatment, as the amount of carbon on the surface of the maple wood increases, and the amount of oxygen decreases. The decrease in amount of oxygenic functional groups on the maple wood surface, because of its degradation, results in an increase in the wood's hydrophobicity and, consequently, a decrease in the values of water-contact angles. The decrease in amount of oxygen (O1s) measured by XPS was also confirmed by the results of FTIR-ATR measurements. These results are in agreement with the results of other authors who determined the chemical changes after hydrothermal treatment on other woods. Srinivas and Pandey (2012) also stated decrease in hydroxyl groups reduced the hygroscopic nature, resulting in increased dimensional stability of thermally modified rubberwood (*Hevea brasiliensis*) and silver oak (*Grevillea robusta*) wood. Geffert et al. (2019) examined chemical changes that occur from the hydrothermal treatment of oak (*Quercus robur* L.) wood through various steaming modes. Increase in temperature and extension of the steaming period primarily affected the holocellulose and extractives contents, and less so the contents of cellulose and lignin.

SEM microscopy

The SEM investigation of pristine maple wood and steam-treated maple wood is illustrated in Fig. 6a,b.



a) b)

Fig. 6: SEM micro photos of pristine (a) and steam-treated (b) maple wood surface (MAG = 1000 x).

Comparing the SEM micro photos A and B in Fig. 6, we can see, that due to effect of water steam the relief of pristine maple wood (A) surface changes, and becomes very rough with many irregularities (B). The above described phenomenon may be related to the steam removal of amorphous lignin from wood exposing the cellulosic fibres. These findings are in good agreement with similar studies (Kúdela et al. 2020) which showed that the thermal treatment induced changes to the beech wood surface morphology and these changes are observable as enhanced roughness. The strength of the adhesion between the surface area of wood and surface varnishes depends on the mechanisms occur between these two materials. Adhesion of different surface finishes to wood was evaluated by Slabejová et al. (2019), who confirmed that the adhesion of oil and wax surface finishes to native wood and to thermally modified wood was the same.

CONCLUSION

The influence of steam treatment on the properties of maple wood was investigated. The results and their analysis indicate that studied physical and chemical properties of treated maple wood were noticeably changed. Hydrophobicity of maple wood has increased after steam modification, resulting in increased dimensional stability of thermally modified wood. The increase of water contact angle on the treated maple wood surface was confirmed. The analyses of XPS and FTIR-ATR spectra provided essential information about chemical changes of wood components after steaming process. XPS and FTIR-ATR measurements results of steam-treated maple wood confirmed a slight decrease in oxygenic functional groups content as well as an increase in carbon content on the maple wood surface. SEM measurements allowed us to detect considerable changes in relief of steam-treated maple wood. Better understanding of physical and chemical changes might be helpful for further optimization of the steam treatment procedures in an industrial scale.

ACKNOWLEDGMENTS

This work was supported by Ministry of Education of the Slovak Republic and Slovak Academy of Sciences, project VEGA, Grant No.1/0570/17 and by the Slovak Research and Development Agency under contracts No. APVV-16-0177, APVV-17-0456 and APVV-18-0378. This publication is the result of the project implementation: Progressive research of performance properties of wood-based materials and products (LignoPro), ITMS 313011T720 supported by the Operational Programme Integrated Infrastructure (OPII) funded by the ERDF.

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