Color and FTIR Analysis of Chemical Changes in Beech Wood (*Fagus sylvatica* L.) after Light Steaming and Heat Treatment in Two Different Environments

Maria Cristina Timar, Anca Varodi,* Murat Hacibektasoglu, and Mihaela Campean

Color and chemical changes were investigated in beech wood (*Fagus sylvatica* L.) following light steaming and further heat treatment for 2.5 h at 200 °C by two techniques (industrial ThermoWood *versus* a laboratory procedure in the presence of air). Colour changes were evaluated in the CIE Lab system, while Fourier transform infrared spectroscopy with attenuated total reflection (FTIR-ATR) investigation was employed to highlight and compare the associated chemical changes. Light steaming caused only minor chemical changes (limited hydrolysis of hemicelluloses) not ready detectable by FTIR. In contrast, heat treatments caused visible changes in the FTIR spectra, especially in the region 1800 to 1500 cm^{-1}, consisting mainly in a clear decrease of the absorption band for conjugated carbonyls at 1643 cm^{-1} and a slight apparent increase of the absorption bands for lignin at 1506 and 1595 cm^{-1}. A significant variation of the ratios of relevant absorption bands indicated complex chemical changes, including hydrolytic, oxidative, and condensation reactions. FTIR ratios and the mass loss values in the two heat treatments relate, both indicating a more advanced modification in the case of the ThermoWood process.

Keywords: Beech wood; Thermal treatments; Steaming; Heat treatment; Color; Chemical changes; FTIR

Contact information: Department of Wood processing and Wood products design, Faculty of Wood Engineering, Transilvania University of Brasov, Str. Universitatii 1, 500168 Brasov, Romania;
** Corresponding author: anca.varodi@unitbv.ro*

INTRODUCTION

Both steaming and heat treatment are thermal treatment processes that modify the color of wood in a pleasant way while also improving other wood properties. These changes are related to specific chemical properties of different intensity and complexity, which may involve the main and the secondary chemical components of wood in direct relation to the actual treatment conditions (temperature, time, and treating environment). Analyzing property changes in relation to chemical changes under different treatment conditions is, therefore, important for a better understanding the processes involved.

Spectroscopic methods, such as color measurements in the CIE Lab system and FTIR, are valuable modern research tools, as they are rapid, versatile, and non-destructive. FTIR is often employed to reveal chemical changes in the structure of organic materials, including wood (Tolvaj and Faix 1995; Colom *et al*. 2003; Pandey 2005; Pandey and Vuorinen 2008; Candelier *et al.* 2016; Cheng *et al.* 2016). Reliable qualitative and semi-quantitative information on the chemical changes in the composition of wood and the structure of wood main components can be obtained by comparatively analyzing FTIR spectra of control and treated wood (Tdjeersma and Militz 2005; Pandey and Vuorinen 2008; Emandi *et al.* 2011; Chen *et al.* 2012, 2014; Li *et al.* 2015; Candelier *et al.* 2016).

The limit of detection of FTIR is about 5%, which means that extractives and their modifications in various processes are not usually detected (Tolvaj *et al.* 2013), due to their low amount in the common wood species, including beech (Fengel and Wegener 1984; Wagenführ 2000). According to Wagenführ (2000), the content of organic-solvent-soluble extractives for beech wood is about 1.5 to 1.9%, while water extractives are around 1.9%.

Beech wood (*Fagus sylvatica* L.) is an important wood resource in Romania and is being extensively used for the fabrication of interior furniture and other interior design elements (*e.g.*, staircases). Lately, these have been made mostly from solid wood panels resulting from longitudinal finger-jointing and lateral gluing of wooden strips. The beech wood employed for such applications is frequently steamed before drying to open the pores and to equalize the moisture content within the boards. This reduces the drying time and the risk of checking during drying, and it also leads to a more uniform, darker, and exquisite color of wood, making it more desirable for the furniture market, where color is an important aesthetics aspect. More recently, there is interest in producing solid wood panels from heat-treated beech for special outdoor and humid indoor applications.

During steaming, wood is subjected to saturated wet steam, usually at a temperature of 100 °C. This procedure results in a pleasant color change of wood and better drying because the wooden tissues are softened. Thus, they become more elastic, allowing an easier migration of water through the capillaries. In addition, the internal growth stresses, which exist in wood, are partly attenuated during the steaming process, which diminishes the checking risk during drying. In addition to these positive effects, steaming also inhibits biological attack (fungi, insects), contributing to the sterilization of wood before it is introduced to the drying kiln. Dimensional stabilization (swelling tendency reduction after drying) is also possible if the steaming is performed in autoclaves at high pressure (Lohmann 1998; Trübswetter 2006). The main requirement for correct steaming (intense and uniform coloring) is the high initial moisture content of wood (50 to 60%). The steaming temperature and duration are chosen as function of the desired degree of color darkening and the wood species (Varga and van der Zee 2008; Todaro *et al.* 2012; Tolvaj *et al.* 2012).

To obtain a pale shade of red color in beech wood, light steaming is performed with temperatures below 90 °C and a duration of 2 to 5 h (Trübswetter 2006). For a pronounced darkening into a dark brick-red color, a temperature close to 100 °C is applied, with 15 to 20 h direct steaming and 24 to 48 h with indirect steaming (Kollmann 1962; Marinescu 1980). Steaming at higher temperatures (125 to 130 °C) for intense coloration is also possible (Dzurenda 2013), but the mechanical strength can decrease significantly, increasing the checking risk during drying (Trübswetter 2006).

Chemical changes during steaming depend on the treatment conditions. Steam acts by dissolving or altering (oxidizing, decomposing) extractives, which are responsible for the natural color of wood and play an important role in color changes following steaming due to the conjugated chemical double bonds systems. Therefore, wood species with higher content in water extractives (*e.g.,* black locust) will undergo more important color changes by steaming (Tolvaj and Molnar 2006; Varga and van der Zee 2008). Other chemical components, such as hemicelluloses are involved in hydrolytic processes. According to Kosikova *et al.* (1999), the steaming of wood at 135 and 150 °C is accompanied with cleavage of lignin-polysaccharide complexes by organic acids released from hemicelluloses, causing changes of physical and chemical properties, while further treatment (drying at 120 °C) lead to formation of secondary lignin-carbohydrate linkages.

More advanced color and related chemical structural changes (*e.g.*, Tdjeersma and Militz 2005; Hill 2006; Windeisen *et al.* 2007; González-Peña *et al.* 2009; Niemz *et al.* 2010; Esteves *et al.* 2013; Yildiz *et al.* 2013; Tolvaj *et al.* 2014; Fabiyi and Ogunleye 2015; Cheng *et al.* 2016) occur by heat treatment of wood at high temperatures (180 to 240 °C) in controlled inert atmosphere, steam, vacuum, fumes with low/no content of oxygen to avoid burning. Chemical changes following exposure of wood (including beech) to elevated temperatures have been extensively studied (*e.g.*, Militz 2002; Tdjeersma and Militz 2005; Windeisen *et al.* 2007; González-Peña *et al.* 2009; Esteves *et al.* 2013; Yildiz *et al.* 2013; Tolvaj *et al.* 2014; Fabiyi and Ogunleye 2015; Cheng *et al.* 2016). However, there are further questions that need to be answered to regarding the chemical mechanism involved in relation to the different behavior of wood species, influence of treating conditions, especially composition of treating atmosphere. In a recent review Candelier *et al.* (2016) stated as key message that: "*an efficient use of thermal treatment of wood requires a depth understanding of the chemical modifications induced*".

Appealing color changes, dimensional stabilization, and the improved durability to biological factors are the most important and valuable improvement aspects envisaged in the heat treatment of beech wood, a non-durable wood species with high swelling and shrinking coefficients (Gonzales-Pena and Hale 2009; Altinok *et al.* 2010; Chaouch *et al.* 2010; Kaymakci and Akyildiz 2011; Wetzig *et al.* 2011; Mitani and Barboutis 2012; Todorovic *et al.* 2012; Ferrari *et al.* 2013; Olarescu *et al.* 2014).

Hydrolytic and oxidative processes are considered to mainly contribute to the formation of chromophores during heat treatments. The chemical changes leading to color changes in heat-treated wood are complex and not completely understood yet, but they are related to degradation products of hemicelluloses, lignin, and certain extractives (Fengel and Wegener 1984; Sundquist 2004; Chen *et al.* 2012). Chen *et al.* (2014) showed that color changes of wood flour following heating (24 h at 120 °C) were different in terms of chromaticity and intensity for non-extracted and extracted samples, as well as for delignified samples, which actually demonstrates the participative and specific role of main and secondary wood components in color change. Formation of extensive conjugated structures, such as unsaturated ketones and quinones due to the cleavage of lignin units has been shown by FTIR (Chen *et al.* 2012).

Successive chemical reactions occur in wood during heat treatments, and these determine evolving changes detectable by FTIR spectra throughout the whole process. Cleavage of acetyl groups of the hemicelluloses, leading to formation of acetic acid, is followed by hydrolysis of hemicelluloses in the acidic conditions created. Further dehydration reactions of hemicelluloses (Gerardin 2015) leading to formation of furfural and hydroxyl-methyl-furfural, which may undergo further degradation (*e.g.*, to levulinic and formic acids), contribute to the decrease of free hydroxyl groups. Esterification reactions (Tdjeersma and Militz 2005) and oxidative processes of lignin and carbohydrates (Esteves *et al.* 2013) also occur later in the process. This mechanism is observed in FTIR spectra by an initial decrease of carbonyl absorption band at ~1730 cm^{-1} followed by its increase later in the process. A clear increase of the ratio of absorption bands of un-conjugated (~1730 cm^{-1}) and conjugated (~1650 cm^{-1}) carbonyl groups (A1730/A1650) was observed, being assigned to condensation of lignin and carboxylation of polysaccharides (González-Peña *et al.* 2009; Esteves *et al.* 2013). Changes in the structure of lignin, such as an initial depolymerisation by splitting of aliphatic side chain (*e.g.*, C-O bonds of C3 side chain and especially β-(O-4) ether linkages) and further condensation reactions (Gerardin 2015 and literature cited herein) are revealed in FTIR spectra by a higher intensity of the aromatic skeletal vibrations at 1600 and 1505 cm^{-1}, which also shifts to 1510 - 1512 cm^{-1}, and the higher intensity of the absorption at 1330 cm^{-1}, as well as the shift of 1245 to 1220 cm^{-1} absorption band (C-O bonds) (Windeisen *et al.* 2007; Esteves *et al.* 2013). Aldehydes including formaldehyde, acetaldehyde, furfural, and hydroxymethyl-furfural are also involved in reticulation reactions of lignin with formation of methylene bridges. Extractives (*e.g.*, terpenes and polyphenols naturally occurring in wood) evaporate in the first stages and then new extractives (including polycyclic phenols with antioxidant activity, according to Abajji *et al.* 2009) are formed above 160 °C by thermal degradation of wood components and migrate to the surface contributing to its low wettability (Gerardin 2015 and literature cited herein).

During the last decades, several heat treatment processes reached industrial application in supplying products to the European market. The main differences between them lie in the nature of the inert atmosphere and the curing conditions used during the heat treatment. The heat treatment conditions involve mass loss during the process and changes to the product properties. It is generally acknowledged that thermal decomposition reactions of the wood polymers is accentuated by the presence of oxygen, while also thermal degradation is more rapid in systems were moisture is present due to the catalytic effect of the acids (acetic acid) resulting by hydrolysis. However, more research has been focused on the effect of treatment intensity (temperature, time) (*e.g.*, Chaouch *et al.* 2010; Baysal *et al.* 2014; Olarescu *et al.* 2014; Olarescu and Campean 2014; Cheng *et al.* 2016) than on the influence of the atmosphere during the process (*e.g.*, Wetzig *et al.* 2011; Chen *et al.* 2012; Bal 2015), though some differences in the chemical processes occurring in different environments and, therefore, differences in the properties of the treated material should be expected.

OBJECTIVES

The main aim of this research was to investigate comparatively the color and chemical changes revealed by FTIR as result of two different heat-treatments applied to beech wood: heat-treatment in superheated steam in an industrial-scale kiln based on the ThermoWood process and heat treatment in a laboratory-scale electric oven, in air at atmospheric pressure. These treatments were applied on pre-steamed beech samples, and therefore color and chemical changes due to this initial treatment were also analyzed by employing the same methods.

The present research is part of a more extensive project emerged from the idea to use an existing manufacturing line of solid wood panels from pre-steamed beech wood strips and adapt it so as to produce on the same line solid wood panels from heat-treated beech wood strips.

EXPERIMENTAL

Wood Material and Thermal Treatments

To manufacture solid wood panels, 400 × 50 × 28 mm beech wood strips were steamed as in Table 1 and dried to a moisture content of 7 ± 2% according to the schedule presented in Table 2. These light steamed samples were coded M_S, while the original untreated beech wood samples were coded M in this research.

Table 1. Light Steaming Schedule for 28-mm Beech Strips (3 successive phases)

Temperature (°C)	Duration (h)	
40	3	12h
60	5	
80	4	

Note: The process in continuous, starting with the top row and proceeding downwards.

Table 2. Drying Schedule for 28-mm Beech Strips

Phase	Wood Moisture Content (MC) (%)	Temperature (°C)	Drying Gradient
Actual drying	Initial MC … 40%	40	2.1
	40% … 30%	45	2.3
	30% … 25%	50	2.4
	25% … 16%	55	2.8
	16% … 10%	60	3.0
	10% … 7%	65	3.2
Conditioning	7 ± 2%	60 °C, 10 h	
Cooling	7 ± 2%	30 °C, 2 h	

Half of the steamed samples were heat-treated in superheated steam in an industrial-scale kiln based on the ThermoWood method (Fig. 1, black line) and were coded HT_1. This process involves three phases (ThermoWood Handbook 2003):

Phase 1 - The chamber temperature is raised rapidly up to 100 °C. Then, the temperature is increased steadily to 130 °C, during which time the high-temperature drying takes place and the moisture content in the wood decreases to nearly zero;

Phase 2 – Once high-temperature drying has taken place, the temperature inside the chamber is increased to between 185 °C and 200 °C, depending on the type of wood (in case of beech it is 200 °C). When the target level has been reached, the dry-bulb temperature remains constant for 2-3 hours (2.5 hours in case of 25 mm thick beech). Throughout this phase, the wet-bulb temperature stays constant at 99 °C;

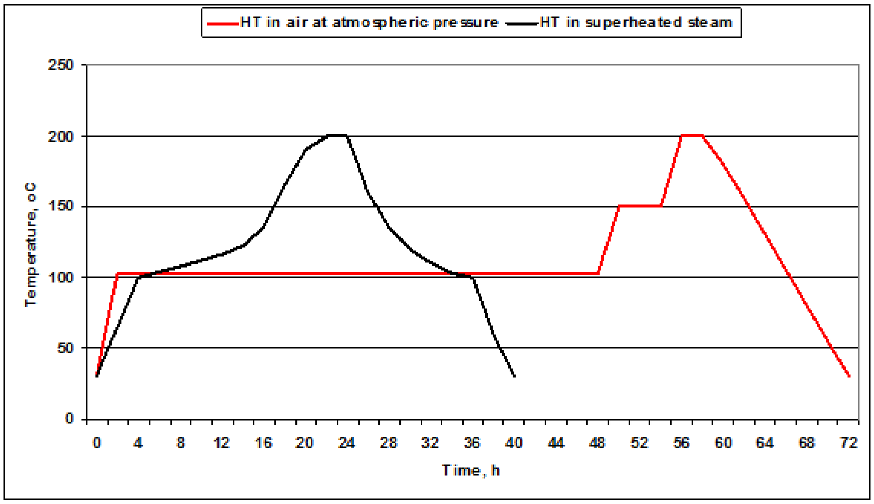


Fig. 1. Comparative treating diagrams for the two heat treatment techniques: superheated steam in an industrial-scale kiln (black) and air at atmospheric pressure in a laboratory oven (red)

Phase 3 – Cooling and moisture conditioning represents the final stage and it consists of lowering the temperature. When the temperature has reached 80 to 90 °C, re-moisturizing takes place by using a water spray system to bring the wood moisture content to a usable 4% to 7%. The maximum pressure used within this process was 253 Pa.

The other samples were heat-treated in a laboratory-scale electric oven, in air at atmospheric pressure (Fig. 1, red diagram) and were coded HT_2. Although the media were different, the temperature (200 °C) and duration (2.5 h) of the heat treatment were the same in the two cases. However, there were differences in the total process duration, in the heating dynamics up to the final heating temperature of 200 °C, and in the cooling time (Fig. 1; Table 3).

Table 3. Heat-Treatment Schedule for 26-mm Beech Strips for the Two Experimental Methods

Phase	Parameters (Temperature / Time)	
	ThermoWood process (heat treatment in superheated steam)	Heat treatment in air at atmospheric pressure
Pre-heating / Oven-drying	100 °C / 3 h	103 °C / 48 h
Heating	100 °C…150 °C / 16 h 150 °C…200 °C / 5 h	103 °C...150 °C / 6 h 150 °C…200 °C / 0.5 h
Actual heat treatment	**200 °C / 2.5 h**	**200 °C / 2.5 h**
Cooling	200 °C…30 °C / 13.5 h	200 °C...30 °C / 15 h
Total process duration	**40 h**	**72 h**

The mass loss due to the heat treatment was calculated based on the weighting of each sample before and after the heat treatment according to Eq. 1,

$$ML = \frac{M_i - M_{HT}}{M_i} \cdot 100 \qquad [\%] \qquad (1)$$

where M_i represents the mass of the sample before the heat treatment (g) and M_{HT} is the mass of the heat-treated sample (g).The four faces of all wooden strips (control, steamed, and further heat treated) were planed to remove external layers before investigation.

Color Measurements

The color of the samples was assessed in the CIE Lab system by an AvaSpec-USB2 spectrometer (10° standard observer, under standard D65 illuminant, Avantes Apeldoorn, Netherlands), equipped with an integrating AVA sphere with a diameter of 80 mm, interconnected by optical fibers. Data were processes using Ava Soft version 7.7 software. The color coordinates lightness *L** (varying from 0 for black to 100 for white), redness *a** (varying from negative values for green to positive values for red on the green-red axis), and yellowness *b** (varying from negative values for blue to positive values for yellow on the blue-yellow axis) were measured for control (M), slightly steamed (M_S), and heat treated samples (HT_1, HT_2). For each variant, three strips were measured in 3 different randomly chosen areas, so that the results represented the average of nine measurements of small areas with a diameter of about 10 mm.

Three slices of about 5 mm were cut transversally from each strip to determine if color was uniform inside of the 28 thick strips. As the color was uniform and also the FTIR spectra registered on different areas of these transversal sections were similar, the color measured on the surface was considered representative for the bulk material.

The color changes associated with light steaming and the two different heat treatments were evaluated by calculating the color difference Δ*E* (Eq. 2),

$$\Delta E = [\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}]^{1/2} \qquad (2)$$

where ΔL^*, Δa^*, and Δb^* are the differences between the respective coordinates of control samples and those for the thermally treated ones.

As previously noted (Allegretti *et al.* 2008; Klement and Marko 2009), the smallest color changes visible by human eye are associated with Δ*E* values higher than 0.2 and lower than 2.0, while Δ*E* values between 2 and 6 account for clearly visible differences. Δ*E* values higher than 6 and smaller than 12 are high color differences. A color difference higher than 12 is considered as emerging from two different colours.

FTIR Investigation

FTIR spectra were measured on samples heat-treated by the two different processes (H_T1 and H_T2), untreated wood (M), and slightly steamed wood (M_S) to evaluate and compare the changes in the chemical composition of beech wood, first the ones caused by steaming, then, comparatively, the ones caused by the two heat-treatments. A Bruker Alpha spectrometer (Bruker Optik GMBH, Ettlingen, Germany) with an ATR unit was employed to record spectra in the range of 4000 to 400 cm^{-1}, at a resolution of 4 cm^{-1} and 24 scans. For each type of sample, spectra were recorded on three different slices of about 5 mm thick, cut from the strips.

Spectra were recorded on three randomly chosen areas of each strip to cover both outer layers and the middle of the sample. Each individual spectrum was then baseline corrected and smoothed. Average spectra were first computed for each slice (3 spectra) and then as a general average for the sample type (average of averages on 3 slices), so that finally an average of 9 spectra were considered for each case.

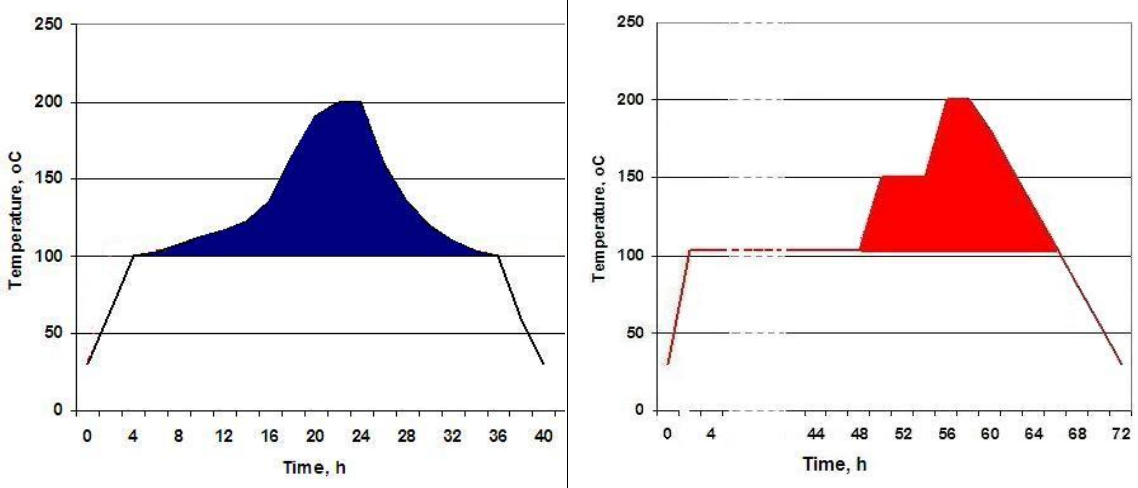
The resulting average spectra of control and thermally treated samples were compared to observe differences brought about by the different thermal treatments in this research. Assignment of absorption bands was done based on the literature (Tolvaj and Faix 1995; Colom *et al.* 2003; Pandey 2005; Pandey and Vuorinen 2008; Rosu *et al.* 2010; Chang *et al.* 2010a, b; Emandi *et al.* 2011; Chen *et al.* 2012, 2014). Moreover, the areas of some absorption bands were integrated, and some ratios of some relevant absorption peaks were computed to assist interpretation of results. All spectra processing and calculation including integration was done using OPUS software (Bruker Optik GMBH, Ettlingen, Germany).

Integration of areas was based on the “tangent skim method”, which measures the area between the curve and the linear baseline drawn across the bottom of the peaks (http://terpconnect.umd.edu/~toh/spectrum/Integration.html), the borders of integration resulting from the position of this tangent line (Table 5), slightly adjusted if necessary for each type of sample.

RESULTS AND DISCUSSION

Mass Loss

The average mass loss of the 10 samples heat-treated by the ThermoWood method was 13.18 ± 1.36%, while that of the 10 samples heat-treated in air was 4.10 ± 0.24%, which counts for a mass loss 3.2 times higher for ThermoWood method in superheated steam compared to heat treatment in presence of air at atmospheric pressure.



a. Heat treatment in superheated steam: relative area = 1415 °C·h

b. Heat treatment in air at atmospheric pressure: relative area = 625 °C·h

Fig. 2. Relative area calculation according to method proposed by Candelier *et al.* (2015)

The importance of the total time of exposure at temperatures above 100 °C (heating + actual heat treatment) has been emphasized by Candelier *et al.* (2015), who found that the temperature kinetics during heat treatment may be used to quantify the treatment effectiveness. The relative area situated below the temperature curve up to 100 °C on the temperature/time diagram is a reliable indicator of the heat treatment intensity. Higher relative area indicates greater mass loss and, thus, more severe degradation. The result obtained within the present research regarding the lower mass loss when treating wood at atmospheric pressure can be well explained by applying this instrument of quantification. The relative area below the temperature curve in case of the heat treatment in superheated steam was 1415 °C·h, which was 2.3 times higher than for the heat treatment at atmospheric pressure, where the relative area measures 625 °C·h. (Fig. 2).

Color Changes

Color change is an important effect of heat treatments with positive impact on the consumers and market. Visual assessment of color changes revealed that slight steaming of beech wood samples caused a slight color change visible as turning from white - yellow to a pale reddish shade, whilst further heat treatments of these slightly steamed samples determined a major color change to dark brown. The samples treated by the two different procedures appeared quite similar on average, though small differences among samples of the same group (HT_1 or HT_2) were visible. Color variability of wood is well known, and this seemed to remain the case also for heat-treated wood.

The color measurements on the investigated samples are presented in Table 4. These results were in good accordance with visual perception, though more relevant as free of subjective evaluation, and at the same time representing an average of more measurements (min 3/sample) and samples (3 replicates).

Table 4. Color Measurements and Calculated Differences

Color Coordinate	Type of Sample				Modification of Color Coordinates (calculated differences)		
	Control M	Steamed M_S	Heat treated HT_1	Heat treated HT_2	Steaming M_S – M	Heat treatments	
						HT_1 - M_S	HT_2 - M_S
*L**	72.34 *(1.09)*	64.76 *(0.78)*	43.47 *(1.71)*	44.65 *(2.07)*	-7.58	-21.29	-22.50
*a**	6.92 *(0.26)*	11.99 *(0.41)*	7.90 *(0.74)*	7.78 *(0.48)*	5.07	-4.09	-4.21
*b**	15.13 *(0.38)*	20.81 *(0.80)*	12.63 *(1.34)*	13.39 *(2.34)*	5.68	-8.18	-7.42
Color difference Δ*E*					10.75	23.17	21.84

Note: Average values are shown (standard deviation in brackets)

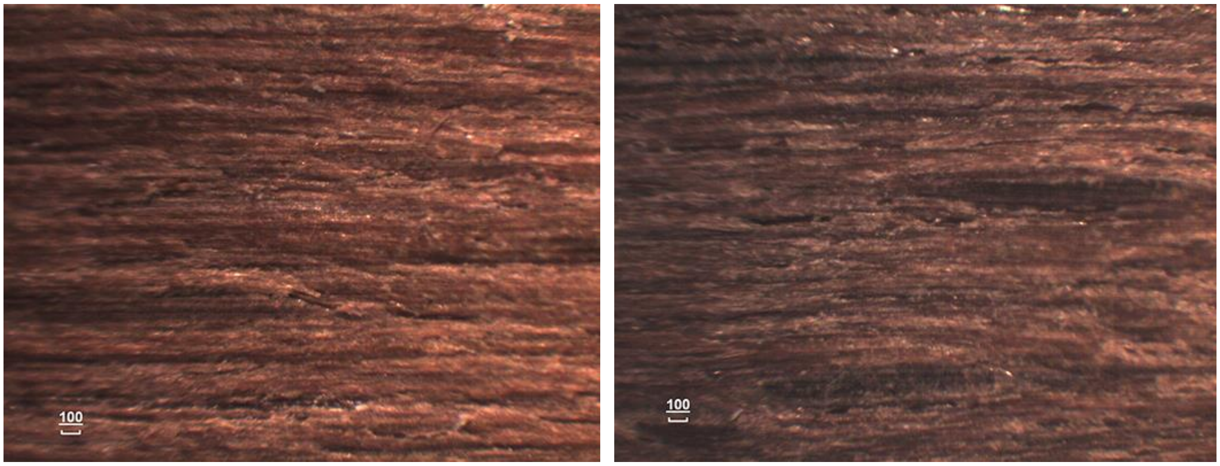
Color data show that steaming results in decreased lightness (Δ*L* = -7.58) and a chromatic shift in the color space to red (Δ*a** = +5.07) and yellow (Δ*b** = +5.68). The overall color change by light steaming was expressed by an Δ*E* value of 10.75, which accounts for a high color difference compared with control beech.

Further heat treatments of lightly steamed beech resulted in noticeable higher color changes influenced by the actual treatment applied. For heat treatment 1 (ThermoWood process in superheated steam), there was a clear decrease in lightness (Δ*L* = -21.29) accompanied by a decrease in redness (Δ*a** = -4.09) and a more important decrease of yellowness (Δ*b** = -8.18), with a total color difference Δ*E* of 23.17. Compared with the control un-steamed beech, redness of heat treated beech HT_1 increased, while yellowness decreased. For heat treatment 2, the decrease of lightness (Δ*L* = -22.50) and redness (Δ*a** = -4.21) were only slightly different (with 1 unit or less) compared with HT_1. Overall, heat treatment of steamed beech by the second procedure produced a Δ*E* of 21.84, close to that computed for the first procedure (with only 1.33 units lower). These color difference values (above 12 units) accounted for different colours.

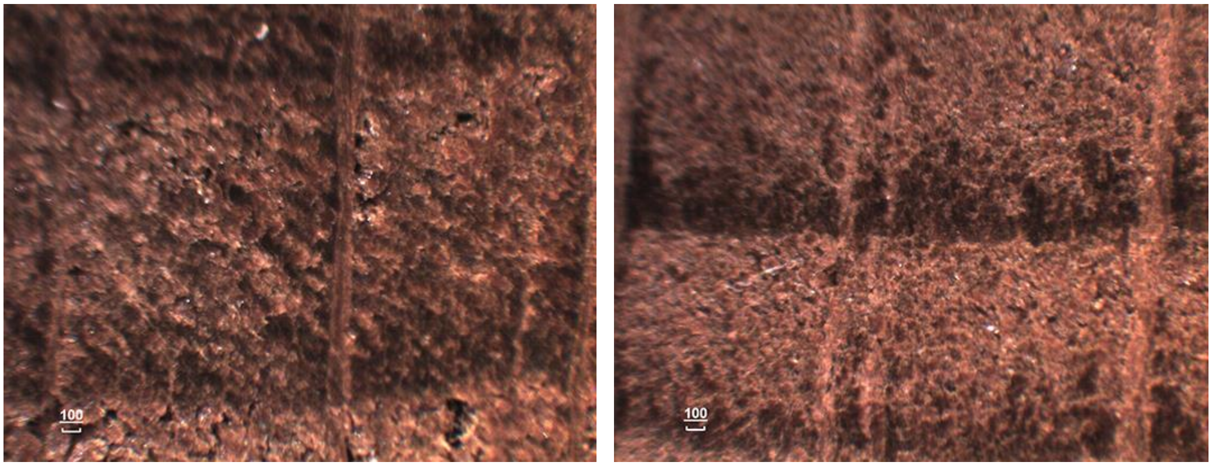
Color evolution during heat treatment is used to classify treated wood, as there are significant correlations of Δ*E* values with treatment intensity, the mass loss, and the thermal process used (Candelier *et al.* 2015). However, a correlation related to evolution of heat-treated wood color, which is valid for a certain treatment method cannot be used to compare the color changes resulting from different methods. Moreover, chemical changes in the presence of oxygen, especially polyoses degradation and oxidation, are accelerated (Gerardin 2015), and specific chromophores such as quinones are formed (Chen *et al.* 2012). This may explain why in the present research quite similar color differences resulted from the two heat treatments in different environments, though the mass losses were noticeably different. Heat treatment in presence of air (HT_2) with mass loss of about 4% resulted in a color change (Δ*E* = 21.84), which was only slightly lower than that (Δ*E* =

23.17) corresponding to a mass loss of about 18% in a heat treatment in over heated steam. However, due to the variability of color as wood property, measurements on a higher number of replicates are necessary for a clear conclusion.

The presence of air in the second procedure likely causes accentuated degradation of hemicelluloses and amorphous cellulose (Sandquist 2004; Hill 2006) and dehydration reactions, which form carbonaceous material within the wood structure (Gerardin 2015 citing Nguila *et al.* 2007). This may have contributed to the accentuated darkening of these samples at lower mass loss values. Microscopic investigation of the heat-treated samples seemed to confirm this hypothesis (Fig. 3). Thus, more dark inclusions with carbonized aspect and reflective properties were present on the surface of beech samples heat treated in the presence of air (HT_2) than in steam atmosphere (HT_1). Steam acts as a blanket protecting wood surfaces from contact with oxygen (Hill 2006). Moreover, a clearly higher contrast between latewood and early wood was visible (at both macro and micro levels) on the transversal section of heat-treated beech HT_2 compared with H_T1.



HT1 – surface (30X) **HT2 – surface (30X)**



HT1 – transverse section (30X) **HT2 – transverse section (30X)**

Fig. 3. Comparison of heat-treated samples HT_1 and HT_2 viewed under a stereomicroscope

FTIR Investigation

The spectra of the investigated samples are depicted in Figs. 4 and 5. Only spectra of control and light steamed beech wood are presented on the whole range of wave-numbers 4000 to 400 cm^{-1} in Fig. 4a. The other spectra are only presented in the region 1800 to 800 cm^{-1}, where most of the specific vibrations occurred and some changes were easily observed. The spectra showed strong bands of H bonded O-H stretching absorption at around 3330 to 3340 cm^{-1} and C-H asymmetric and symmetric stretching at around 2880 to 2930 cm^{-1}.

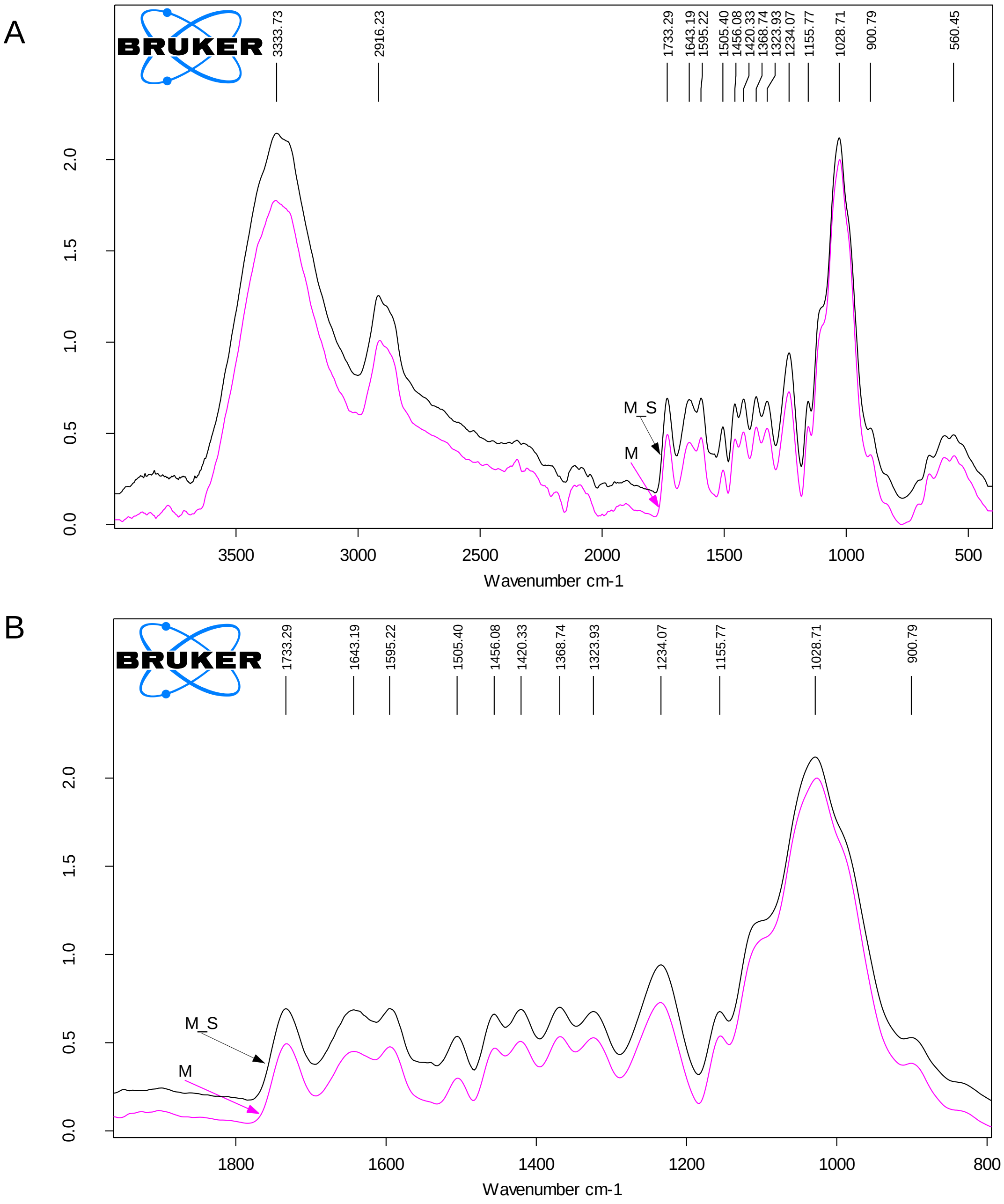


Fig. 4. Comparative FTIR spectra of control and light steamed beech wood. (A) The whole registration range (4000 to 400 cm^{-1}); (B) the fingerprint region 1800 to 800 cm^{-1}

Other peaks in the fingerprint region (1800 to 800 cm^{-1}) included 1730 cm^{-1} (unconjugated C=O groups, mostly present as acetyl groups in hemicelluloses), 1643 cm^{-1} (conjugated C=O and C=C, aromatic ketones, *e.g.*, in α position of the side chain of lignin units), 1595 cm^{-1} (aromatic skeletal vibration of syringyl lignin plus C=O stretch), 1505 to

1506 cm^{-1} (aromatic skeletal vibration of lignin), 1455 cm^{-1} (aromatic CH deformation and asymmetric bending of CH_3 in lignin), 1422 cm^{-1} (C–H deformation in lignin and carbohydrates; CH_2 bending in cellulose), 1367 to 1370 cm^{-1} (CH deformation in carbohydrates: cellulose, hemicelluloses), 1324 cm^{-1} (syringyl ring breathing with CO stretch and C_1–O vibration in syringyl derivatives of lignin and C–H vibration in cellulose), 1234 cm^{-1} (C–O stretch in lignin and xylan, syringyl ring), 1155 cm^{-1} (C–O–C asymmetric bridge stretching vibration in cellulose and hemicelluloses), 1110 cm^{-1} (aromatic skeletal and C-O stretch in lignin and polysaccharides), 1028 to 1030 cm^{-1} (C-O stretching of primary alcohols in cellulose and hemicelluloses), and 900 cm^{-1} (C_1-H deformation of glucose ring in cellulose) (Tolvaj and Faix 1995; Colom *et al.* 2003; Pandey 2005, Pandey and Vuorinen 2008; Chang *et al.* 2010a,b; Rosu *et al.* 2010; Emandi *et al.* 2011; Chen *et al.* 2012, 2014).

The integrated areas of some of these absorption bands further employed in semi-quantitative evaluation of chemical changes are presented in Table 5, while the resulting calculated ratios are summarized in Table 6.

Table 5. Integrated Areas of Selected Absorption Bands in the FTIR Spectra (Average Values)

Absorption Peaks [cm^{-1}]	Borders of integrated areas [cm^{-1}]	Integrated Areas (*A*)			
		M	**M_S**	**HT_1**	**HT_2**
3340	3648 - 3006	454.79	505.21	402.09	436.57
2920	2981 - 2758	47.18	49.79	39.16	42.24
1730	1775 - 1693	14.05	15.57	15.34	15.79
1643	1689 - 1622	4.97	6.84	0.47	1.10
1595	1613 - 1566	3.91	3.19	5.17	4.86
1506	1532 - 1484	3.15	4.26	4.66	4.50
1421	1442 - 1398	2.52	2.92	2.25	2.59
1370	1391 - 1349	2.76	3.19	2.37	2.92
1234	1289 - 1187	26.30	28.82	24.31	25.05
900	912 - 878	1.10	0.97	1.48	1.23

Table 6. FTIR Ratios of Relevant Absorption Bands for the Different Beech Wood Samples

FTIR Ratio	**Assignment**	**M**	**M_S**	**HT-1**	**HT_2**
A1730 /A1370	Non conjugated carbonyl / Carbohydrates	5.08	4.88	6.46	5.40
A1506 /A1370	Lignin / Carbohydrates	1.14	1.33	1.96	1.54
A1730 /A1643	Non conjugated carbonyl / conjugated carbonyl	2.82	2.27	32.34	14.38
A3340 /A1506	Hydroxyl / Lignin	144,30	118,71	86,26	96,99

Light steaming did not cause readily detectable chemical changes in the FTIR spectra (Fig. 4). This result was expected, as only limited hydrolysis of hemicelluloses and dissolution/decomposition/oxidation of extractives (usually not detectable by FTIR) could actually occur in the mild steaming conditions performed in this research. Hydrolysis of hemicelluloses, especially xylan (pentosan), present in higher proportions in hardwoods (beech), starts with deacetylation of esterified hydroxyl groups; therefore, a slight decrease in non-conjugated carbonyl groups (present mostly in hemicelluloses as acetyl) and slight increase in free hydroxyl groups was expected. This was reflected in the slight decrease of the ratios A1730/A1370 (non-conjugated carbonyl/ carbohydrates) from 5.08 (M) to 4.88 (M_S) and A1730/A1643 (non-conjugated carbonyls/conjugated carbonyls) from 2.82 to 2.27.

Furthermore, the FTIR ratios A1506/A1370 (increasing from 1.14 to 1.33) and A3340/A1506 (decreasing from 144.30 for control M to 118.71 for M_S) suggested that light steaming led to a surface richer in lignin and less hydrophilic. This effect might be explained by decreased hemicellulose content following removal of water-soluble decomposing fragments from the wood surface. Moreover, lignin-carbohydrates linkages may be broken during steaming (Kosikova *et al.* 1999). The hydrolytic cleavage of β-O4-aryl-ether linkages in lignin leading to water-soluble lignin fragments and formation of free phenolic hydroxyl groups may occur (Hill 2006), though more likely at higher temperatures than those employed in this research. The presence of sugars, phenols, and lignin-derived soluble monomers in the water effluents from beech steaming reported by Ledig *et al.* (2004) supports these results.

Heat treatment of the light-steamed beech resulted in clearly visible changes in the FTIR spectra (Fig. 5). In the case of heat treatment in overheated steam atmosphere (HT_1), the most obvious changes consist in a significant decrease of the absorption band for conjugated carbonyls at 1643 cm^{-1} and a slight increase of the absorption bands for lignin at 1506 and 1595 cm^{-1}.

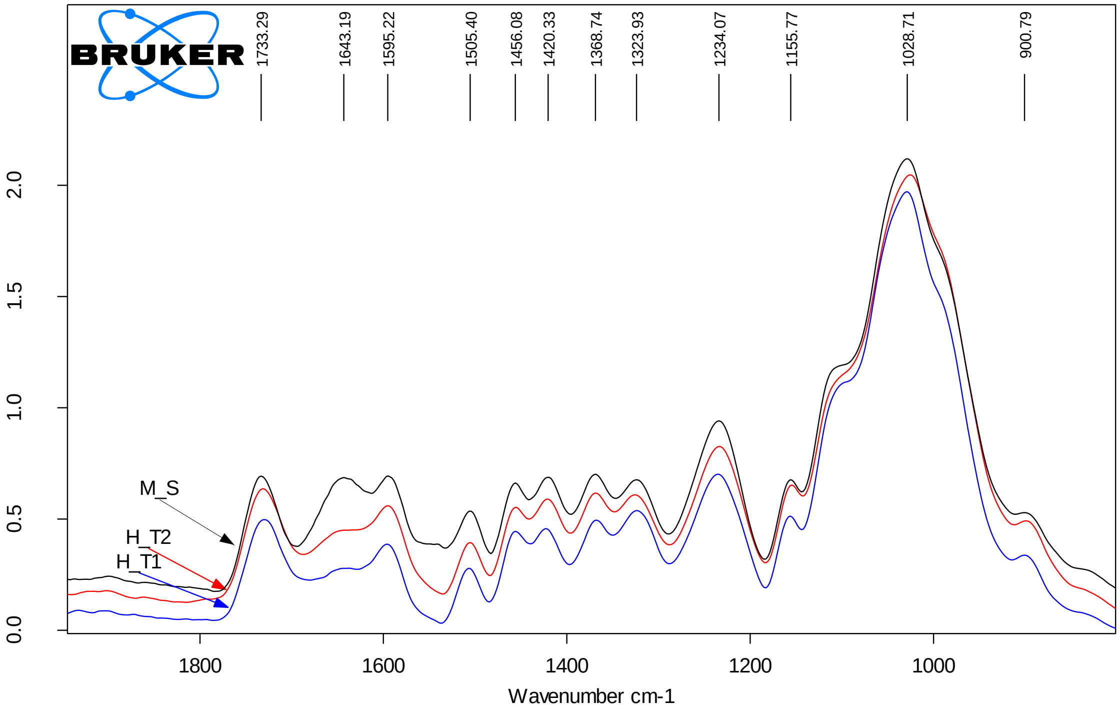


Fig. 5. Comparative spectra of light steamed (M_S) and heat treated beech in the two variants HT_1, HT-2 (different environments –atmosphere) in the fingerprint region 1800 to 800 cm^{-1}

The increase of lignin absorption bands is explained by the degradation of polysaccharides (hemicelluloses) leaving a surface richer in lignin (Hill 2006), while some successive processes in the structure of lignin are associated with the decrease of conjugated carbonyl groups present mostly in the structure of lignin aliphatic chain at the Cα. Initial cleavages in the lignin network, occurring at the Cα and β-O4-(aryl-ether linkage) positions, are followed by further condensation of lignin units through the Cα position of the side chain (Hill 2006; Windeisen *et al.* 2007; Esteves *et al.* 2013) to a more cross-linked structure, which can also include furfural units resulted from sugars by dehydration.

Similar changes in the FTIR spectra were recorded in the case of beech heat-treated in the laboratory oven in presence of air (HT-2), though the intensity of these changes was different, as resulting from the superimposed spectra in Fig. 5 and the data in Tables 5 and 6. Notably, the intensity of the non-conjugated carbonyls absorption band at 1730 cm^{-1} remained unchanged for both HT_1 and HT_2 compared with the steamed beech. However, this was slightly shifted from 1733 cm^{-1} to lower wave numbers (1730 cm^{-1} for HT_1 and 1731 cm^{-1} for HT_2) as it actually represents different types of carbonyl groups than the acetyl moieties in hemicelluloses, which are the first to be hydrolyzed during the heat treatment of wood due to their lability (Fengel and Wegener 1984; Tdjeersma and Militz 2005; Hill 2006). The non-conjugated carbonyl groups in the heat-treated beech should be associated with more free carboxylic acid groups and new ester groups formed during later stages of heat treatment in the structure of both lignin (Tdjeersma and Militz 2005) and carbohydrates (González-Peña *et al.* 2009; Esteves *et al.* 2013) by esterification, carboxylation, and oxidative processes. Depolymerisation of amorphous cellulose and oxidative processes of cellulosic hydroxyls leading to carbonyl and carboxyl groups can also occur in the case of heating in air atmosphere (Hill 2006).

The calculated FTIR ratios presented in Table 6 allowed an easier identification of chemical changes associated with the thermal processes investigated in this research, including the subtle ones associated to the light steaming, alongside a comparison between the HT_1 and HT_2 heat treatments. The values of these ratios suggest that heat treatments caused decrease of carbohydrate content and oxidative effects (increase of A1730/A1370), leading to a relative increase of lignin content (increase A1506/A1370), and a reduced relative ratio between hydrophilic/hydrophobic components (decrease of A3340/A1506). A clear increase in the ratio between the areas of absorption bands of non-conjugated (1730 cm^{-1}) and conjugated (1643 cm^{-1}) carbonyl groups, from 2.27 for steamed beech to 32.34 for HT_1 and 14.38 for HT_2 was registered, in accordance to previously reported research (González-Peña *et al.* 2009; Esteves *et al.* 2013).

Moreover, a comparison of all the calculated FTIR ratios indicates that these changes were more advanced for HT_1 than HT_2. This is in accordance with the higher mass loss value determined for HT_1 (13.18%) compared with HT_2 (4.10%). Mass loss is closely related to the thermal degradation of wood during a heat treatment and appears to be the main indicator of the treatment intensity (Allegretti *et al.* 2012), which is strongly influenced by the heating medium and treatment intensity (couple temperature-duration).

Analyzing the relative values of relevant FTIR ratios (area of a certain peak for heat treated wood) in relation to the mass loss registered in the two treatments, a correlation could be observed (graphs not shown). The obtained results support the reliability of FTIR as an investigation tool for monitoring heat treatment processes. However, tests with more heat treatment conditions need to be performed in order to validate such an instrument and this will be the subject of further research. A slightly different approach, employing FTIR and SD-IR (second derivative – infrared) correlation spectroscopy, has been recently reported by Li *et al.* (2015), showing a strong relation between treatment intensity and chemical changes of wood main components, as revealed by FTIR.

CONCLUSIONS

1. Light steaming caused expected color changes (pale reddish shade), but the changes in the FTIR spectra were minor and difficult to be observe directly. These were detectable only by calculating ratios of relevant absorption peaks. They suggest only a slight hydrolysis of hemicelluloses, which was expected considering the low steaming temperature (max. 80 °C).
2. The mass losses recorded after the two heat treatment techniques were situated at a ratio of 3.2 (with values of 13.18% for superheated steam treatment and 4.10% for heating in air, respectively), although the actual heat treatment temperature and duration were the same (200 °C/ 2.5 h). This might be correlated with the different heating kinetics (time to raise temperature from 100 to 200 °C) and the relative areas in °C × h units delimited in the treating diagrams at temperatures above 100 °C, as proposed by Candelier *et al.* (2015). The heating time was 3.2 times higher in the case of the heat treatment in superheated steam (21 h) compared with the heat-treatment in air at atmospheric pressure (6.5 h), while the calculated relative areas for the two methods were 1425 °C×h and 625 °C×h, respectively, which corresponds to a ratio of 2.3/1.
3. Both heat treatments resulted in noticeable and comparable color changes (ΔE = 23.17 and 21.84, respectively). The presence of air in the second process might have caused accentuated darkening of these samples at lower mass loss values. Microscopic investigation indicated the presence of carbonaceous inclusions on the samples heat treated in the presence of oxygen.
4. Both heat treatments caused clearly visible changes in the FTIR spectra, though they were more advanced for the heat treatment resulting in the higher mass loss (HT in superheated steam). The most obvious changes consisted in decrease of the absorption band for conjugated carbonyls at 1643 cm^{-1} and a slight increase of the absorption bands for lignin at 1506 and 1595 cm^{-1}.
5. Chemical changes were better reflected by the ratios of the relevant absorption bands. The values of these ratios suggest a decrease of the carbohydrates content and oxidative effects (increase of A1730/A1370), leading to a relative increase of lignin content (increase A1506/A1370), with a more condensed structure (increase of A1730/A1643), and, respectively, reduced relative ratio between hydrophilic/hydrophobic components (decrease of A3340/A1506).

ACKNOWLEDGMENTS

The authors acknowledge the structural founds project PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009) for providing the infrastructure used in this work.

REFERENCES CITED

Allegretti, O., Cividini, R., and Travan, L. (2008). "Drying process for white beech," *Pro Ligno* 4(1), 11-19.

Allegretti, O., Brunetti, M., Cuccui, I., Ferrari, S., Nocetti, M., and Terziev, N. (2012). "Thermo-vacuum modification of spruce (*Picea abies* Karst.) and fir (*Abies alba* Mill.) wood," *BioResources* 7(3), 3656-3669.

Altinok, M., Ozalp, M., and Korkut, S. (2010). "The effect of heat treatment on some mechanical properties of laminated beech (*Fagus orientalis*) wood," *Wood Research* 55(3), 131-142.

Bal, B. C. (2015). "Physical properties of beech wood thermally modified in hot oil and in hot air at various temperatures," *Maderas: Ciencia y Technologia* 17(4), 789-798. DOI: 10.4067/S0718-221x2015005000068

Baysal, E., Kart, S., Toker, H., and Degirmentepe, S. (2014). "Some physical characteristics of thermally modified oriental beech wood," *Maderas: Ciencia y Technologia* 16(3), 291-298 DOI: 10.4067/S0718-221x2014005000022

Candelier, K., Hannouz, S., Elaieb, M. T., Collet, R., Dumarçay, S., Pétrissans, A., Gérardin, P., and Pétrissans, M. (2015). "Utilization of temperature kinetic as a method to predict treatment intensity and corresponding treated wood quality: Durability and mechanical properties of thermally modified wood," *Maderas: Ciencia y Technologia* 17, 253-262. DOI: 10.4067/S0718-221x2015005000024

Candelier, K., Thevenon, M. F., Petrissans, A., Dumarcay, S., Gerardin, P., and Petrissans, M. (2016). "Control of wood thermal treatment and its effects on decay resistance: a review," *Annals of Forest Science*. DOI: 10.1007/s13595-016-0541-x

Chang, T. C., Chang, H. T., Wu, C. L., and Chang, S. T. (2010a). "Influences of extractives on the photodegradation of wood," *Polymer Degradation and Stability* 95, 516-521. DOI:10.1016/j.polymdegradstab.2009.12.024.

Chang, T. C., Chang, H. T., Wu, C. L., Lin, H. Y., and Chang, S. T. (2010b). "Stabilizing effect of extractives on the photo-oxidation of *Acacia confusa* wood," *Polymer Degradation and Stability* 95, 1518-1522. DOI: 10.1016/j.polymdegradstab.2010.06.012

Chaouch, M., Petrissans, M., Petrissans, A., and Gerardin, P. (2010). "Prediction of decay resistance of different softwood and hardwood species after heat treatment based on analysis of wood elemental composition," in: *Proceedings of the Fifth European Conference on Wood Modification*, Riga, Latvia, 135-142.

Cheng, S., Huang, A., Wang, S., and Zhang, Q. (2016). "Effect of different heat treatment temperatures on the chemical composition and structure of Chinese fir wood," *BioResources* 11(2), 4006-4016. DOI: 10.15376/biores.11.2.4006-4016

Chen, Y., Gao, J., Fan, Y., Tshabalala, M. A., and Stark, N. M. (2012). "Heat-induced chemical and color changes of extractive-free black locust (*Robinia pseudoaccacia*) wood," *BioResources* 7(2), 2236-2248. DOI: 10.15376/biores.7.2.2236-2248

Chen, Y., Tshabalala, M. A., Gao, J., Stark, N. M., and Fan, Y. (2014). "Color and surface chemistry changes of extracted wood flour after heating at 120 °C," *Wood Science and Technology* 48, 137-150. DOI: 10.1007/s00226-013-0582-3

Colom, X., Carrillo, F., Nogués, F., and Garriga, P. (2003). "Structural analysis of photodegraded wood by means of FTIR spectroscopy," *Polymer Degradation and Stability* 80, 543-549. DOI: 10.1007/s13595-016-0541-x

Dzurenda, L. (2013). "Modification of wood colour of *Fagus sylvatica* l. to a brown-pink shade caused by thermal treatment," *Wood Research* 58(3), 475-482.

Emandi, A., Vasiliu, C. I., Budrugeac, P., and Stamatin, I. (2011). "Quantitative investigation of wood composition by integrated FT-IR and thermogravimetric methods," *Cellulose Chemistry and Technology* 45, 579-584.

Esteves, B., Velez Marques, A., Domingos, I., and Pereira, H. (2013). "Chemical changes of heat treated pine and eucalyptus wood monitored by FTIR," *Maderas: Ciencia y Technologia* 15(2), 245-258. DOI: 10.4067/S0718-221X2013005000020

Fabiyi, J. S., and Ogunleye, B. M. (2015). "Mid-infrared spectroscopy and dynamic mechanical analysis of heat treated obeche (*Triplochiton scleroxylon*) wood," *Maderas: Ciencia y Technologia* 17(1), 5-16. DOI: 10.4067/s0718-221x2015005000001.

Fengel, D., and Wegener, G. (1984). *Wood: Chemistry, Ultra-Structure, Reactions*, Verlag Walter de Gruyter & Co., Berlin, Germany.

Ferrari, S., Cuccui, I., and Allegretti, O. (2013). "Thermo-vacuum modification of some European softwood and hardwood species treated at different conditions," *BioResources* 8(1), 1930-2126. DOI: 10.15376/biores.8.1.1100-1109

Finnish Thermowood Association (2003). *ThermoWood Handbook*, Finnish Thermowood Association, Helsinki, Finland.

Gerardin, P. (2015), "New alternatives for wood preservation based on thermal and chemical modification of wood: A review," *Annals of Forest Science.* DOI 10.1007/s13595-015-0531-4

González-Peña, M. M., Curling, S. F., and Hale, M. D. C. (2009). "On the effect of heat on the chemical composition and dimensions of thermally-modified wood," *Polymer Degradation and Stability* 94, 2184-2193. DOI: 10.1016/j.polymdegradstab.2009.09.003.

González-Peña, M., and Hale, M. (2009) "Colour in thermally modified wood of beech, Norway spruce and Scots pine, Part 2: Property predictions from colour changes," *Holzforschung* 63, 394-401. DOI: 10.1515/HF.2009.077

Hill, C. (2006). *Wood Modification: Chemical, Thermal and Other Processes*, Wiley & Sons, Hoboken, NJ, USA.

Kaymakci, I., and Akyildiz, M. H. (2011). "Dimensional stability of heat treated Scots pine and oriental beech," *Pro Ligno* 7(4), 32-38

Klement, I., and Marko, P. (2009), "Color changes of beech wood (*Fagus sylvatica* l.) during high temperature drying process," *Wood Research* 54(3), 45-54.

Kollmann, F. (1962). *Furniere, Lagenhoelzer und Tischlerplatten*, Springer Verlag, Berlin, Germany.

Kosikova, B., Hricovõni, M., and Cosentino, C. (1999). "Interaction of lignin and polysaccharides in beech wood (*Fagus sylvatica*) during drying processes," *Wood Science and Technology* 33, 373-380.

Ledig, S. F., Slavik, I., Broege, M., and Mollenkopf, N. (2004). "Characterization of effluent from direct and indirect steaming of beech timber," *Holz als Roh- und Werkstoff* (2004) 62, 346-348. DOI: 10.1007/s00107-004-0507-9

Li, M.-Y., Cheng, S.-C., Li, D., Wang, S.-N., Huang, A.-M., and Sun, S.-Q. (2015). "Structural characterization of steam-heat treated *Tectona grandis* wood analyzed by FT-IR and 2D-IR correlation spectroscopy," *Chinese Chemical Letters* 26, 221-225. DOI: 10.1016/j.cclet.2014.11.024

Lohmann, U. (1998) *Holz Handbuch*, DRW-Verlag, Leinfelden, Germany.

Marinescu, I. (1980). *Drying and Thermal Treatment of Wood* (Volume II), Technical Publishing House, Bucharest, Romania (in Romanian).

Militz, H. (2002). "Heat treatment technologies in Europe: Scientific background and technological state-of-art," in: *Conference on Enhancing the Durability of Lumber and Engineered Wood Products*, Orlando, FL, USA.

Mitani, A., and Barboutis, I. (2012). "Assessment of color changes and hygroscopic properties of beech (*Fagus sylvatica*) wood after thermal modification," in*: 23rd International Scientific Conference Wood is Good – With Knowledge and Technology to a Competitive Forestry and Wood Technology Sector*, pp. 111-118.

Niemz, P., Hofmann, T., and Retfalvi, T. (2010). "Investigation of chemical changes in the structure of thermally modified wood," *Maderas: Ciencia y Technologia* 12(2), 69-78. DOI: 10.4067/SO718-221X2010000200002

Olarescu, C. M., and Campean, M. (2014). "Color change – mass loss correlations for heat treated wood," in: *CBU International Conference of Innovation, Technology Transfer and Education*, Prague, Czech Republic, pp. 345-352.

Olarescu, C. M., Campean, M., and Olarescu, A. (2014). "Dimensional stabilization of wood originating from small-diameter trees through heat treatment," *BioResources* 9(3), 4844-4861. DOI: 10.15376/biores.9.3.4844-4861

Pandey, K. K. (2005). "Study of the effect of photo-irradiation on the surface chemistry of wood," *Polymer Degradation and Stability* 90(1), 9-20. DOI: 10.1016/j.polymdegradstab.2005.02.009

Pandey, K. K., and Vuorinen, T. (2008). "Comparative study of photodegradation of wood by a UV laser and a xenon light source," *Polymer Degradation and Stability* 93, 2138-2146. DOI: 10.1016/j.polymdegradstab.2008.08.013

Rosu, D., Teaca, C. A., Bodirlau, R., and Rosu, L. (2010). "FTIR and colour change of the modified wood as a result of artificial light irradiation," *Journal of Photochemistry and Photobiology B: Biology* 99(3), 144-149.

Sundqvist, B. (2004). *Colour Changes and Acid Formation in Wood during Heating*, Ph. D. Dissertation, Lulea University of Technology, Skelleftea, Sweden.

Tdjeersma, B. F., and Militz, H. (2005). "Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood," *Holz als Roh- und Werkstoff* (2005) 63, 102-111. DOI 10.1007/s00107-004-0532-8

Todaro, L., Zuccaro, L., Marra, M., Basso, B., and Scopa, A. (2012). "Steaming effects on selected wood properties of Turkey oak by spectral analysis," *Wood Science and Technology* 46(1), 89-100. DOI: 10.1007/s00226-010-0377-8

Todorovic, N., Popovic, Z., Miic, G., and Popadic, R. (2012). "Estimation of heat treated beechwood properties by color change," *BioResources* 7(1), 799-815. DOI: 10.15376/biores.7.1.0799-0815

Tolvaj, L., and Faix, O. (1995). "Artificial ageing of wood monitored by DRIFT spectroscopy and CIE *L*a*b** colour measurements," *Holzforschung* 49(5), 395-404. DOI: 10.1515/hfsg.1995.49.5.397

Tolvaj, L., and Molnar, S. (2006). "Colour homogenisation of hardwood species by steaming," *Acta Silvatica and Lignaia Hungarica* 2, 105-112.

Tolvaj, L., Papp, G., Varga, D., and Lang, E. (2012). "Effect of steaming on the colour change of softwoods," *BioResources* 7(3), 2799-2808. DOI: 10.15376/biores.7.3.2799-2808

Tolvaj, L., Molnar, Z., and Nemeth, R. (2013). "Photodegradation of wood at elevated temperature: Infrared spectroscopic study," *Journal of Photochemistry and Photobiology B: Biology* 121, 32-36. DOI: 10.1016/j.jphotobiol.2013.02.007

Tolvaj, L., Nemeth, R., Pasztory, Z., Bejo, L., and Takats, P. (2014). "Colour stability of thermally modified wood during short-term photodegradation," *BioResources* 9(4), 6644-6651. DOI: 10.15376/biores.9.4.6644-6651

Trübswetter, T. (2006). *Holztrocknung. Verfahren zur Trocknung von Schnittholz – Planung von Trocknungsanlagen*, Carl Hanser Verlag GmbH & Co. KG, Munich, Germany.

Varga, D., and van der Zee, M. E. (2008). "Influence of steaming on selected wood properties of four hardwood species," *Holz als Roh- und Werkstoff* 66(1), 11-18. DOI: 10.1007/s00107-007-0205-5

Wagenführ, R. (2008). *Holzatlas*, Carl-Hanser Verlag GmbH & Co. KG, Munich, Germany.

Wetzig, M., Heldstab, C., Tauscher, T., and Niemz, P. (2011). "Ermittlung augewählter mechanischen Kennwerte thermisch modifizierter Buche," *Bauphysik* 33, (6), 366-374. DOI: 10.1002/bapi.201190045

Windeisen, E., Strobel, C., and Wegener, G. (2007). "Chemical changes during the production of thermo-treated beech wood," *Wood Science and Technology* 41(6), 523-536. DOI: 10.1007/s00226-007-0146-5

Yildiz, S., Tomak, E. D., Yildiz, U. C., and Ustaomer, D. (2013). "Effect of artificial weathering on the properties of heat treated wood," *Polymer Degradation and Stability* 98(8), 1419-1427. DOI: 10.1016/j.polymdegradstab.2013.05.004

Article submitted: May 26, 2016; Peer review completed: July 22, 2016; Revised version received and accepted: August 3, 2016; Published: August 11, 2016.
DOI: 10.15376/biores.11.4.8325-8343