Composites

One-sided surface charring of beech wood

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ABSTRACT

One-sided surface charring of beech wood (*Fagus sylvatica* L.) was analyzed. Specimens were charred on one surface using contact heating system with a hot plate, at a temperature of 220 °C and atmospheric pressure for 15 or 40 min. Surface charring was applied on the radial or tangential surface. Spectrophotometric chemical analysis was carried out to determine the total amount of soluble carbohydrates and phenolic compounds beneath the surface in order to evaluate the depth (within 1, 2, 3 and 4 mm) of the degradation affected by the charring. Specimens of dimensions $14 \times 14 \times 14$ mm were used to determine the moisture behavior of charred specimens. The equilibrium moisture content (EMC) of specimens was calculated at a temperature of 20 °C and 65% relative humidity. Water absorption (WA) was recorded during the water immersion for 240 h at various intervals. Finally, the three-point bending tests were undertaken and modulus of rupture (MOR) and modulus of elasticity were calculated. Degradation of the main wood compounds due to chemical changes occurred during the surface charring is closely associated with a mass loss (3.5–5.5%) and further related to the severity of process and orientation of specimens to heat flow. Application of surface charring resulted in the significant reduction in EMC ($\sim 20\%$) as a result of a decrease in hydroxyl groups, an increase in cellulose crystallinity as well as further crosslinking of lignin. Furthermore, the WA of one-sided surface-charred wood considerably decreased (15%) when compared to that of the control specimens. A significant increase in soluble carbohydrates as well as phenolic compounds was found, and it can be stated that one-sided charring affected wood properties to depth 2–3 mm from the surface. Significant differences (14–24%) of bending strength MOR results are most likely due to improved sorption behavior of wood. The results show that beech wood charring improved moisture-related characteristics and consequently led to better mechanical behavior, but more studies are needed to exploit the potential of surface charring method for future use.

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Introduction

Due to environmental challenges that society is facing, material science has been increasingly focusing on lignocellulose-based biopolymer materials such as wood. There is a scientific effort to enhance wood or its derivatives to obtain materials that maintain their advantages over time and at the same time reduce their drawbacks such as natural variability, dimensional instability or biological degradability [20, 30]. Innovative wood modification methods allow reduction or even elimination of natural disadvantages of wood in order to obtain better performance over the service life of wood-based materials. Chemical, thermal and mechanical treatments are commonly applied to wood resulting in permanent or temporary changes in wood properties [10, 17, 23]. Thermal modification at elevated temperature has been long considered as the most commercially successful and efficient method to decrease the equilibrium moisture content (EMC) of wood and consequently improve its dimensional stability and biological durability [7, 39, 40, 43]. The main drawback of thermal modification is low physical strength that prevents its use in load-bearing structures [3, 19, 35].

It is known that thermal degradation of wood is a set of overlapping reactions. At low temperatures between 20 and 150 °C, the wood dries, beginning with the loss of free water and finishing with bound water. At 180–250 °C, the temperature ranges commonly used for traditional thermal modification, wood undergoes important chemical transformations and results in decomposition of the non-crystalline carbohydrates, while lignin and cellulose degradation occurs rather above 280 °C [12, 14, 42]. Heating to 300 °C results in depolymerization of cellulose, production of volatile compounds, formation of oxidation products (carbonyl and carboxyl functionalities) and finally charring [5, 11].

As an alternative to traditional thermal modification, wood could be modified only from the exposed surface(s), sparing time and costs and preserving the structural properties of wood [13, 21, 22]. One-sided surface charring has been traditionally utilized predominantly in Japan [1]. In this technique, the wood is burned with naked flame or a heated iron pad, and the result is said to last for decades and be practically maintenance free. So far, one-sided surface charring of beech wood at moderate temperature has not been scientifically investigated and knowledge about its performance is rather limited [22].

Beech wood is the most spread broadleaf species in forests of the Czech Republic. Its wood is a traditional material for wood-processing industry; however, the potential of use is not fully utilized. In order to investigate the effect on one-sided charring on properties of beech, several characteristics were studied. The results form a part of a project aiming to develop a new technique for beech wood modification with added value and improved performance.

Materials and methods

European beech (*Fagus sylvatica* L.) wood obtained from Czech forest enterprise was studied. Specimens were cut from a single-quarter sawn board with an average oven-dry density 688 kg m^3. Afterward, the specimens were sorted into six groups with 30 specimens in each group representing the orientation of charred surface and time used (Table 1). The orientation of specimens regarding heat flow is further illustrated in Fig. 1.

One-sided surface charring

Specimens were charred on one side using the contact system with hot plate, at a moderate temperature of 220 °C and atmospheric pressure for 15 or 40 min on the radial and tangential surface of specimens. Specimens were conditioned at a temperature of 20 °C and 65% RH before the surface charring.

The degree of thermal degradation of wood compounds was determined by mass loss (M_L), based on oven-dry (103 ± 2 °C for 24 h) mass before and after the heating process. The M_L was calculated according to Eq. (1).

$$\text{Mass loss}(\%) = \left(\text{dry mass}_{[\text{reference}]} - \text{dry mass}_{[\text{charred}]}\right)/$$
$$\text{dry mass}_{[\text{reference}]}. \tag{1}$$

Determination of total carbohydrates and phenolic compound surface layers

Furthermore, chemical analysis of the charred surface was carried out within various depths in order to propose a methodology for the evaluation of degraded distance (depth) affected by temperature and

Table 1 Coding of specimens referring to the section and time of heating

Group	Reference		One-sided charring			
Charred surface	Radial	Tangential	Radial	Tangential	Radial	Tangential
Charred time	0 min		15 min		40 min	
Coding	Ref_{RAD}	Ref_{TAN}	$Heat_{RAD1}$	$Heat_{TAN1}$	$Heat_{RAD2}$	$Heat_{TAN2}$
No. of specimens	30	30	30	30	30	30

Figure 1 Annual ring orientation in specimens related to heating direction.
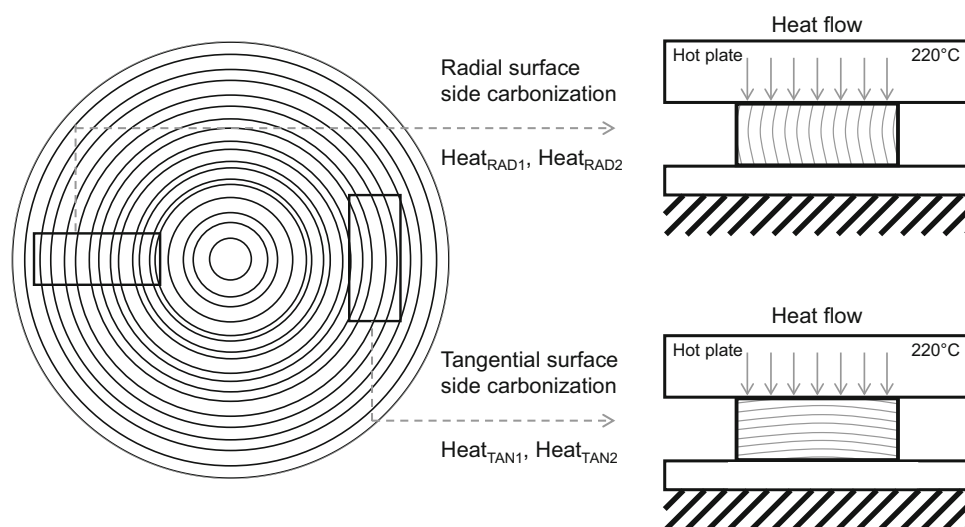
length of the heating process. Four thin layers of wood (4×1 mm) were prepared from the charred surface using microtome (Core-Microtome WSL) and grounded using laboratory mill (Retch MM 400) to obtain a homogeneous mixture. Afterward, 0.1 g of dry homogenous mass from the individual layers of specimens was placed into the vial. 5 mL of aqueous methanol (Penta, Czech Republic), 50/50, v/v, was added. Samples were extracted according to the following protocol: 15 min of shaking on an orbital shaker (Miulab, China), 100 rpm, followed 15 min sonication in an ultrasound bath (GT Sonic, China). Extraction continued for another 15-min shaking at 100 rpm. Sample filtration followed. All stages were performed at room temperature ($\pm$ 20 °C). Thus, the prepared extract was used for subsequent spectrophotometric analysis of total carbohydrates and total phenolic compounds.

Spectrophotometric determination of total carbohydrates—phenol–sulfuric acid method

Carbohydrates dehydrated by reaction with concentrated sulfuric acid produce furfural derivatives that react with phenol and give red-colored detectable compounds. A slightly modified method for determination of total carbohydrates was used [2]. To a 0.5 mL aliquot of extract from wood mass in a screw-on tube (prepared according to the protocol mentioned above) were added 1.5 mL of demineralized water and 1 mL of freshly prepared 5% aqueous phenol (Penta, Czech Republic) solution. The mixture was vortexed (Miulab, China). Immediately, 5 mL of concentrated sulfuric acid (Penta, Czech Republic) was added. The sample was mixed manually and left for 10 min to stand. After that, the sample was again thoroughly mixed on a vortex and allowed to heat for 20 min in a thermostated water bath (Benchmark, USA) at 30 °C. The 30-min standing of the sample at room temperature followed for stabilization of color development. Thus, the prepared sample was measured on a spectrophotometer (Metash, China) at 490 nm against demineralized water. A calibration curve for quantification was constructed using glucose (Sigma-Aldrich, Czech Republic) standard solutions. The concentration of total carbohydrates was expressed as the glucose equivalent (GLUE) per 1 g of dry wood mass. For each series of samples, one blank determination was measured by the same procedure.

Spectrophotometric determination of total phenolic compounds—Folin–Ciocalteu method

The Folin–Ciocalteu method is based on the reduction of phosphotungstate and phosphomolybdate complexes by phenolic to blue reaction products. A modified method for the determination of total phenolic compounds was used [31]. 0.2 mL aliquot of the extract from wood mass (prepared according to the protocol mentioned above) was mixed with 4.8 mL of demineralized water in a tube, and 0.2 mL of Folin–Ciocalteu reagent (Sigma-Aldrich, Czech Republic) was added. The mixture was vortexed. After 3 min, 1 mL 20% Na_2CO_3 solution was added and sample was vortexed again. After keeping the sample at room temperature for 20 min and last shaking, absorbance at 700 nm was measured against demineralized water. A calibration curve for quantification was constructed using gallic acid (Sigma-Aldrich, Czech Republic) standard solutions. The concentration of total phenolic compounds was expressed as the gallic acid equivalent (GAE) per 1 g of dry wood mass. For each series of samples, one blank determination was measured by the same procedure.

Moisture behavior

Specimens of dimensions $14 \times 14 \times 14$ mm were cut from the charred and reference samples to determine equilibrium moisture content (EMC) and water absorption (WA). Specimens were conditioned at 65% relative humidity (RH) and 20 °C using climate chamber (Memmert GmbH), and EMC was calculated according to Eq. (2). Further, the oven-dry (103 ± 2 °C for 24 h) specimens were entirely immersed under water for 240 h and water absorption (mass of specimens) was recorded in the selected intervals. Afterward, moisture content (MC) of water soaked specimens was calculated according to Eq. (2).

$$\text{Moisture content}\,(\%) = (\text{wet mass} - \text{dry mass})/ \text{dry mass}.$$

(2)

Mechanical properties testing

The static three-point bending ($14 \times 14 \times 210$ mm) was carried out using universal testing machine Zwick Z050/TH 3A (Zwick Roell AG, Ulm, Germany). The experimental procedure was controlled by software test Xpert v11.02. The static three-point bending was tested according to BS 373 [4]. The force during the bending test was applied on the charred surface until the failure by the 50 kN load cell through the single load head at the midspan. The radius of supports and loading heads was 15 mm. The support span of 196 mm was determined as $14 \times$ sample height oriented in the load direction, i.e., 14×14 mm, whereas the free sample ends were equal to half height = 7 mm. The failure recognized by a significant drop in the force along with the visible sign of the failure occurred between the 30 s and 60 s. The modulus of rupture (MOR) was calculated from the maximum force. The calculation of modulus of elasticity (MOE) was based on forces recorded at 10% and 40% of the maximum loading force and corresponding deflections of bended specimens as measured by extensometers.

Statistical analysis

The data were processed in Statistica 10 software (StatSoft Inc., USA) and evaluated using one-factor analysis of variance (ANOVA), completed with Tukey's honest significance test (HSD test).

Results and discussion

Mass loss (M_L) of wood is one of the most important parameters to consider as an indicator of degree of thermal degradation when applying elevated temperature [6, 16, 23]. The M_L basically depends on the wood species, heating system, temperature and time, with temperature having the most dramatic effect. As expected, higher M_L was found as the duration of one-sided surface charring was extended (Fig. 2a). Slightly higher values of M_L were found for specimens heated on the tangential surface. This might be explained by the presence of more early/latewood in surface as well as higher thermal conductivity in the radial direction compared to the tangential direction ($\lambda_R > \lambda_T$). It is believed that these differences can be attributed to a significant amount and orientation of wood rays in hardwoods [28, 33, 34]. According to the literature, at least 4% mass loss should be achieved to reasonably change the moisture behavior
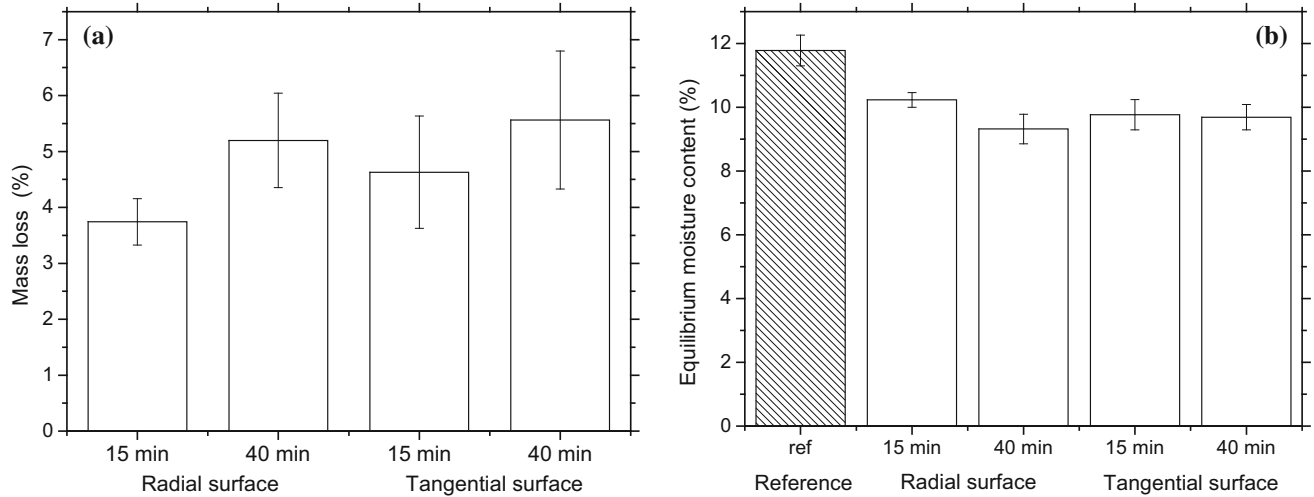
Figure 2 Mass loss of one-sided surface-charred specimens (**a**), equilibrium moisture content of specimens conditioned at 65% RH and 20 °C (**b**).

and biological durability of treated wood [7, 29, 40, 42].

An average EMC of control beech wood was 11.8% (0.4%) after conditioning (Fig. 2b). A statistically significant decrease (up to 20%) was observed for all tested groups. Even though specimens tended to better performance after long-term surface heating (40 min), there was no statistically significant difference between heated groups. The main reason for the decrease in the EMC is that less water is absorbed by the cell walls after the wood charring as a result of chemical change with a decrease in hydroxyl groups. Furthermore, reduced accessibility of free hydroxyl groups (–OH) to water molecules may be also due to the increase in cellulose crystallinity as well as further cross-linking of lignin as result of polycondensation reactions might also contribute to decrease in EMC [8, 18, 38].

Average water absorption curves of the control and charred specimens during 240 h of water immersion are shown in Fig. 3. The water absorption of one-sided surface-charred wood decreased compared to that of the control specimens. Maximum water absorption of beech wood was reported to be 100–118% [28]. After 240 h of water immersion, specimens reached the maximum MC of 117% (5.2%), while one-sided surface charring generally decreased the water absorption of specimens. Water absorption decreased to 106% (3.4%) for the radial surface-heated specimens and to 99% (3.9%) for tangential surface-charred specimens heated for 40 min. These findings correspond to values of M_L as a result of
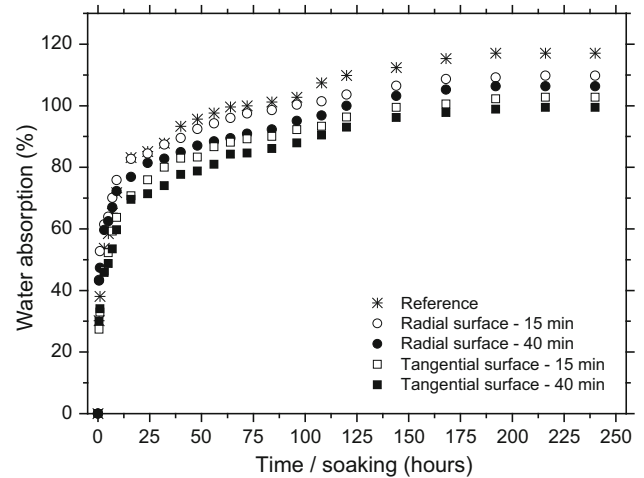

Figure 3 Results of water absorption during 240 h of water immersion.

degradation of wood components. During water absorption, liquid penetrates into wood by capillary force. The water absorption behavior of wood is closely related to its properties because the swelling and linear expansion are associated with the amount of water absorbed [8, 32]. Previous studies showed that elevated temperature leads to degradation of wood compounds, reducing the relative content of polysaccharides and the number of hygroscopic groups, such as hydroxyl groups and carbonyl groups [9, 24].

The reduction in the hydroxyl groups results from the depolymerization and hydrolyzation of hemicelluloses and structural change of lignin due to the

cross-linking at high temperature and therefore blocking of the access of water to wood cell walls. Thus, the water absorption of wood was reduced and its dimensional stability was increased [7]. In addition to the effects of changes in chemical composition, a large number of pits might be closed when the heating temperature (220 °C) was applied [36]. Thus, the water absorption paths of free water were blocked and the transport of free water was impeded. According to results, it is evident that all tested groups absorbed more water (up to 80% of MC) and more rapidly at the early stage of water immersion (up to 25 h) when compared to the second stage. No differences between tested groups were found at the early stage of water immersion as mainly untreated part of specimens affects water absorption. This phenomenon is generally attributed to the natural capillaries present in the wood, which quickly attains

equilibrium with hydration medium by capillary inhibition. At the beginning of the water absorption process, capillaries and cavities near the surface are filled up very fast. Hence, it can be assumed that the water concentration of the surface is raised to saturation almost instantaneously. The moisture movement is restricted to inside material only. Water moves freely in the large cavities, but in the small ones, the presence of trapped air bubbles influences the water movement inside the material.

Quantitative data from analytical technique providing results of total soluble carbohydrates and phenolic compounds are presented in Figs. 4 and 5. According to this procedure, the effective depth of charred surface can be evaluated. One-sided surface charring modifies the structure of wood cell wall polymers conferring new properties of the desire material. Thermal stability of the different polymers

Figure 4 Total amount of soluble carbohydrates (mg/g) within analyzed specimens at various depths from the charred surface.
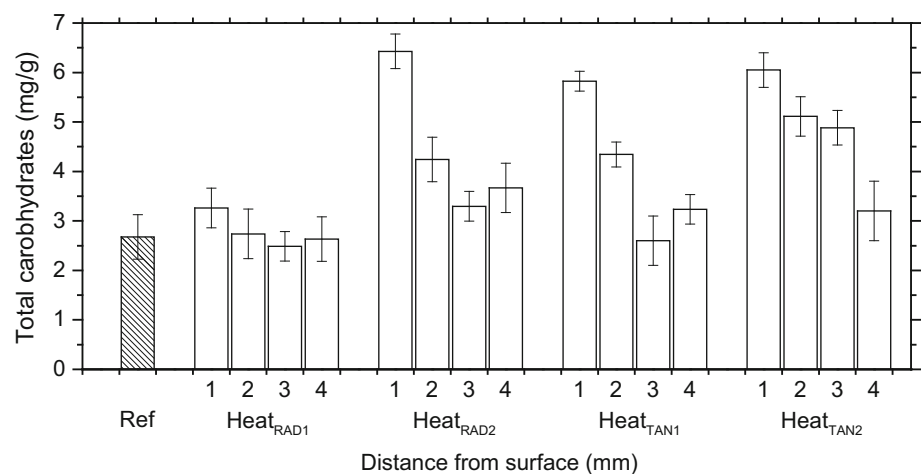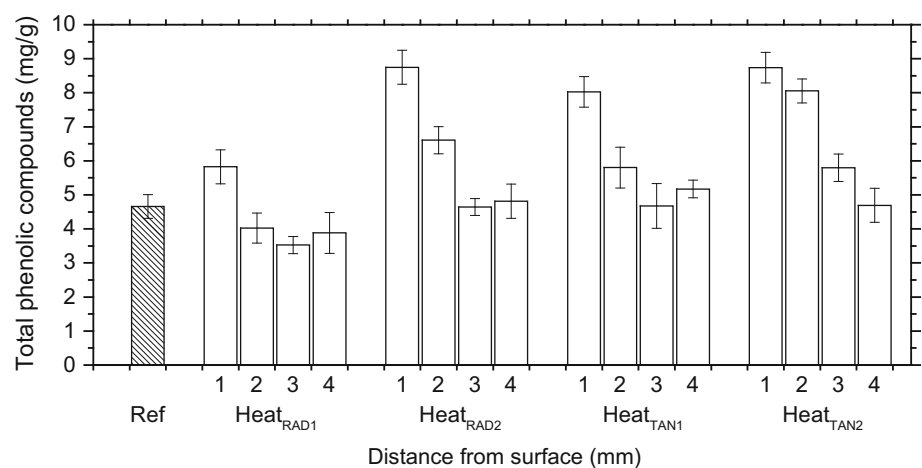

Figure 5 Total amount of soluble phenolic compounds (mg/g) within analyzed specimens at various depths from the charred surface.

constitutive of wood differs according to the chemical structure. Hemicelluloses present a lower degree of polymerization and higher reactivity due to the amorphous structure, which are degraded first, followed by lignin and cellulose [15]. The main volatile compounds are formic acid, acetic acid, furfural resulting from hemicelluloses degradation. Acetic acid formed during degradation of acetylated xylans catalyzes depolymerization reactions reducing the degree of polymerization of hemicelluloses [23]. Cellulose, which presents a high degree of polymerization and crystallinity, is generally considered as relatively resistant under heat treatment conditions used. The decomposition of lignin is observed for temperatures starting from 220 °C with the appearance of phenolic substances such as vanillin. The main results in Fig. 4 showed that the one-sided surface charring induced a change in the soluble carbohydrates content of wood specimens. The most remarkable change was the quantitative increase in soluble sugars (including cellulose and glucose structural units of the hemicelluloses polymer), as a consequence of thermal degradation of wood compounds.

An average total amount of carbohydrates of reference specimens was measured to be 2.7 mg/g of dry wood mass. Due to the thermal degradation, the sum of soluble carbohydrates increased significantly within surface layers and decreased with increasing distance from the heated surface. Specimens heated on the tangential surface shows the most significant increase through analyzed layers of wood, which are also corresponding to measured mass loss. According to analyzed data, it can be assumed that one-sided charring affected material properties to minimum of 2–3 mm from the surface.

Very minor changes through the analyzed layers were measured on specimens heated at the radial surface for 15 min. However, this might be further verified by increasing the number of measurements. Niemz et al. [25] also stated that during the degradation of hemicelluloses soluble carbohydrate compounds are produced in wood. Furthermore, it is believed that the structure of wood is altered and with increasing soluble sugar concentration, the bending (MOR) as also modulus of elasticity (MOE) decreases.

A reduction in the hemicelluloses content during heating is commonly measured with an increase in the total phenol concentration [25]. This statement was also confirmed for one-sided surface charring, as presented in Fig. 5. During the thermal degradation of hemicelluloses, the lignin–carbohydrate connections are also cleaved. The cleavage leads to the easier depolymerization of this non-carbohydrate-bonded lignin fraction, yielding simple phenolic compounds. The transformation of lignin and the increase in the concentration of the low molar mass phenolic compounds contribute to the reduced water absorption and consequently reduced swelling and shrinkage characteristics [7, 16, 26, 37].

The mechanical properties of reference and one-sided surface-charred specimens are shown in Fig. 6. The radial and tangential surface charring of wood affected the bending strength (MOR) of tested specimens, which is significantly higher when compared
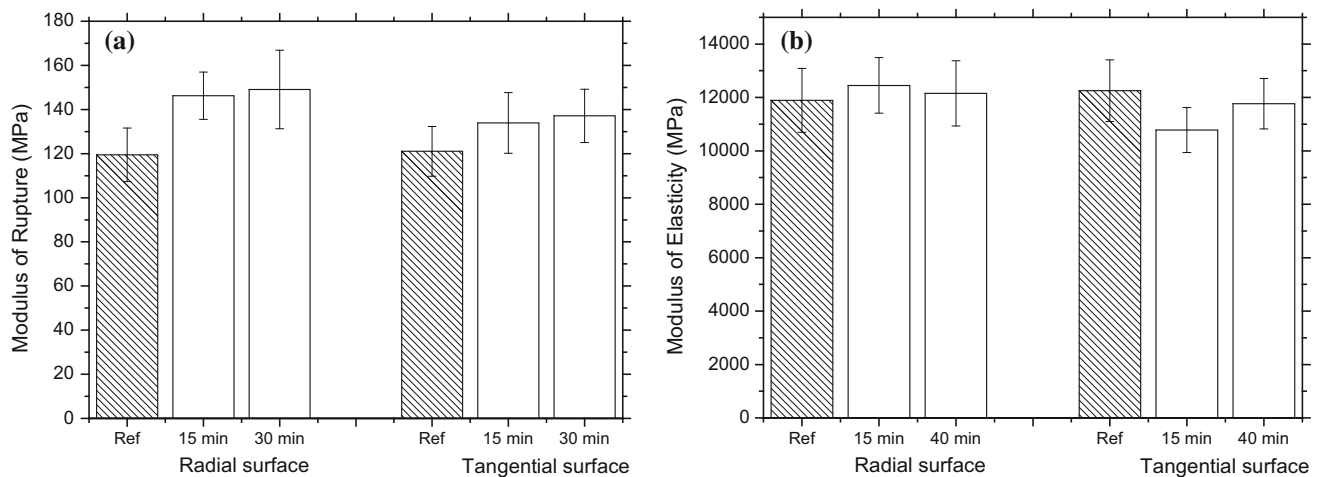
Figure 6 Modulus of rupture in three-point bending (**a**) and modulus of elasticity (**b**).

to reference (14–24%). This might be most likely explained by several factors.

Firstly, the natural wood characteristics (density variation, annual ring width and its orientation) variability may have a minor effect on results even though the specimens were made from the same boards. Secondly, surface charring of wood led to decreased EMC ($\sim$ 20%) and therefore specimens with different moisture content were measured. According to the literature, with 1% MC decrease (within range of bound water) approx. 3–4% of wood strength is increased. Thirdly, charred surface might be compressed as a consequence of surface softening (plasticizing) by elevated temperature and pressure due to heated plate weight. This would considerably increase strength on the compression side during a flexural test. Furthermore, no statistically significant differences were found for modulus of elasticity (MOE). Niemz et al. [25] also stated that thermal treatment generally gave no important impact on the MOE. An average bending strength (MOR) and modulus of elasticity (MOE) of reference beech wood specimens were measured to be 119.5 MPa (12.1 MPa) and 12 000 MPa (1200 MPa), respectively, and this is consistent with other authors [27, 28, 41].

Conclusions

The one-sided surface charring of beech wood (*Fagus sylvatica* L.) at a temperature of 220 °C was studied. Application of surface heating led to a significant reduction in equilibrium moisture content (EMC) as a result of chemical changes with a decrease in hydroxyl groups, increase in cellulose crystallinity as well as further cross-linking of lignin as result of polycondensation reactions. The reduction is closely associated with the mass loss as the main indicator of degree of surface charring. Mass loss is related to the severity of process (influence of duration) as well as specimen's annual rings orientation (influence of thermal conductivity). Furthermore, the kinetics of water absorption of one-sided surface-charred wood considerably decreased compared to that of the reference specimens. Chemical analysis provided the total amount of soluble carbohydrates and phenolic compounds within various depths from the heated surface. A significant increase in soluble carbohydrates as well as phenolic compounds, as a result of thermal degradation of wood compounds, can be

further used as a tool for the indication of depth affected by elevated temperature. Additionally, the radial and tangential surface heating affected the mechanical behavior of tested specimens. Significantly higher bending strength (MOR) is attributed to improved sorption behavior of wood, eventually to compressed surface of charred wood as a consequence of surface softening by elevated temperature and pressure due to heated plate weight. Nevertheless, concluding from the results of this study, beech wood exhibits favorable results in terms of moisture-related characteristics as well as mechanical behavior when one-sided surface charring at moderate temperature (220 °C) and moderate time (15 or 40 min) is applied. Therefore, this might be a suitable technique for one-sided exposed wood surfaces (wood cladding, wooden shingles) where wood–water interaction has major importance, but also for those elements where the stiffness of the material is important (decking, wooden beams, etc.). Furthermore, more detailed studies of surface charring process should be undertaken and unanswered behavior of one/all sided charred surface described (e.g., different wood swelling behaviors through specimen cross section, biological durability, fire resistance, etc.).

Acknowledgements

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