Application of Microwave Treatment for the Plasticisation of Beech Wood (*Fagus sylvatica* L.) and its Densification for Flooring System Purposes

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In this study, the application of microwave treatment for wood plasticisation and its densification for flooring system purposes is presented. Microwave plasticisation was carried out using a continuous laboratory device at a frequency of 2.45 GHz, and the testing samples made from European beech (*Fagus sylvatica* L.) wood were plasticised at different power modes (2 kW, 3.5 kW, and 5 kW). Afterwards, the densification (ratio 50%) of pre-treated samples was performed. The surface temperature (T_s) and average moisture content (MC) of the samples were measured after plasticisation. The results showed the influence of the chosen mode on MC decrease and rapid T_s increase. Thus, the densification of testing samples is affected by different initial conditions that occur during the plasticisation process (MC and T_s). The Brinell hardness (H_B) of the densified samples increased by about 57% (2 kW), 103% (3.5 kW), and 83% (5 kW), compared with control samples. These results provide a better understanding of microwave plasticisation usability and its potential optimisation and application in the wood flooring industry.

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INTRODUCTION

European beech (*Fagus sylvatica* L.) is the most widespread broadleaf species in the forests of the Czech Republic, and covers approximately 7% of the total area of Bohemian and Moravian forest land (Gryc *et al.* 2008). This is an essential factor affecting the extent of the production of this raw material. Beech provides hard, strong, and resilient wood with good properties for processing, which makes it suitable for a wide range of uses (Pouchanič 2011). Beech wood is a traditional material for the wood-processing industry, although its potential has not been fully utilised (Pouchanič 2011; Dömény *et al.* 2014).

For most non-structural applications such as flooring, wood is subjected to indentation and abrasion in one form or another. This requires that wood has a certain degree of surface hardness to reduce the need for maintenance and replacement (Lamason and Gong 2007). The strength properties of wood are proportionate to its density (Kollmann and Côté 1968; Navi and Heger 2004; Kamke 2006; Fang *et al.* 2012). By mechanical densification through compression, it is possible to increase wood's strength and wear resistance. After densification, low-density wood species can be used to substitute for species with high density. Moreover, the strength and hardness of high-

density wood species could be further improved through densification. Thereby, this process can be useful in applications where wood is considered to be too soft, *e.g.*, for flooring in public environments (Blomberg 2006).

The densification of wood is a technology whereby wood is compressed in the transversal direction using heat, water, and steam to produce a product with higher density, strength, and lustre (Blomberg 2006; Lamason and Gong 2007). To improve these properties of wood, there has long been a drive to develop a process for its densification (Wingate-Hill 1983). One of the most important steps in wood densification is plasticisation. Wood exhibits its plastic behaviour when subjected to specific conditions. The cell walls of wood can be considered a matrix consisting of lignin, cellulose, and hemicellulose. Applying the right amount of heat and water in the proper combination will cause the matrix to soften significantly, changing from a glass state to a rubber state. This provides a theoretical basis for the compressibility of wood (Lamason and Gong 2007; Rautkari *et al.* 2010). The application of steam as a heating and softening medium is the most commonly used technology. However, the mechanism of heat and mass transfer during steaming means long durations of plasticisation and requires a high amount of energy. Also, the industrial processes suggested usually involve static plasticisation and compression between rigid steel plates. The densified products have not been competitive because of their low capacity and high cost (Blomberg *et al.* 2006).

Therefore, innovative wood plasticisation by microwave radiation has been studied (Norimoto and Gril 1989; Studhalter *et al.* 2009; Ozarska and Daian 2010; Gašparík and Gaff 2013). The use of microwaves for heating is well-established in society, being used in domestic and some industrial processes. However, there is potential for this technology to be introduced and applied to many other industrial heating processes (Fernández *et al.* 2011), to include the wood industry. In this sense, microwave technology is being explored as one of the methods for wood plasticisation. Plasticisation by microwave heating has several advantages over conventional methods, such as possible continuous application, reduction of plasticisation time, high energy efficiency, and volumetric heating (Seyfarth *et al.* 2003; Vongpradubchai and Rattanadecho 2009; Gašpařík and Gaff 2013).

The theoretical background of microwave heating is characterised by an internal heating process due to the direct absorption of energy by polar molecules. The key substance that absorbs microwaves is water because of its high value of dielectric permittivity (Zielonka *et al.* 1997; Oliveira and Franca 2002; Feng and Chen 2008). Wood structure also has dielectric properties due to the content of hydroxyl (-OH) and methylene (-CH$_2$OH) groups, which are mostly contained in wood polysaccharides (Torgovnikov 1993). The dielectric properties are characterised by the dielectric loss factor (ε'') and relative permittivity (ε'). Relative permittivity is the ability of the material to store energy, and the loss factor refers to the rate of energy loss in the dielectric. The ratio $\varepsilon''/\varepsilon'$, or tan δ, is called the dielectric loss tangent and represents the radiated energy that turns into heat (Torgovnikov 1993; Kabir *et al.* 1997; Brelid and Simonson 1999). Energy absorption is primarily caused by the movement of dipole molecules with the same frequency as the electromagnetic field, and the rapid change in field polarity causes the vibration and even rotation of molecules, which transforms the energy into frictional heat (Makovíny 2000; Hansson and Antti 2003).

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The goal of this work was to contribute to the knowledge of microwave heating for plasticisation in order to develop and accelerate the process of wood compression for flooring system purposes. Such an investigation could become a basis for further research of the plasticisation and mechanism of microwave (MW) pre-treatment.

EXPERIMENTAL

Materials

European beech (*Fagus sylvatica* L.) sapwood boards (300 mm × 50 mm × 10 mm, L × R × T) with an average oven-dry density (ρ_0) of 648 kg·m^{-3} were microwave-plasticised and densified. Before microwave plasticisation, the samples were soaked in distilled water for five weeks. The initial moisture content (MC) was between 91 and 100% as determined by the oven-dry method of standard EN 13183-1 (2002). In total, 40 specimens without defects (cracks, knots, *etc.*) were studied.

The testing samples were divided into four groups. Three groups were microwave-treated in different power modes (Table 1), and one group was retained as a control for hardness measurement (10 specimens).

Methods
Microwave plasticisation

The testing samples were plasticised in a continuous laboratory microwave device (Fig. 1), which operates at a frequency of 2.45 GHz and provides an adjustable power from 0.6 to 6 kW. The velocity of the conveyor can be controlled by the frequency changer (Koiš *et al.* 2014).

Fig. 1. Continuous microwave device

The first testing group was plasticised with a power of 2 kW, the second group was exposed to a power of 3.5 kW, and the third group was plasticised at 5 kW (Table 1). Plasticising modes used the same conveyor speed of 0.4 m/min.

Table 1. Microwave Treatment Specifications

Plasticisation Mode (kW)	Speed of Conveyor (m·min⁻¹)	Dimension (L × R × T) (mm³)	Average MC (%)	Number of Specimens
2.0	0.4	300 × 50 × 10	93	10
3.5	0.4	300 × 50 × 10	97	10
5.0	0.4	300 × 50 × 10	96	10

Temperature and moisture content

The surface temperature (T_s) of the testing samples was measured by a contactless infrared thermometer (IR-380, Voltcraft; Czech Republic) on the sample surfaces. The measuring was conducted at three points for each sample (front, centre, and rear) before and after microwave plasticisation. These three point values were used to calculate the arithmetic mean. The moisture content (MC) of the wood samples was determined using the oven-dry method before and after the heating process, in compliance with EN 13183-1 (2002).

Densification

The densification process was carried out using a hydraulic press (HL 400, Strozatech; Czech Republic). The device had two temperature-controlled hot plates with an area of 1200 mm x 1000 mm and a capacity of 410 tons. All groups of testing samples were densified together. The pressing parameters (Table 2), *i.e.*, temperature, time, pressure, and densification ratio, were 80 °C, 1 min, 4.5 MPa, and 50%, respectively. The testing samples were compressed in tangential direction. After the densification process, the plates were removed from the press and maintained in the closed position (0.5 MPa) for a period of five days (cooling down, stabilisation, and conditioning).

Table 2. Densification Parameters

Plasticisation Mode (kW)	Temperature of the Plates (°C)	Pressure (MPa)	Initial Thickness (mm)	Final Thickness (mm)
2.0	80	4.5	10	5
3.5	80	4.5	10	5
5.0	80	4.5	10	5

Measurement of density

The density (ρ_0) of untreated and densified beech was measured after a 24-h drying period at 103 ± 2 °C and determined in compliance with EN 323 (1993). The density value of the specimen was determined as the ratio of oven-dry mass to volume of the specimen at the oven-dry MC.

Measurement of hardness

The hardness of wood is generally measured by the ability of a steel ball to penetrate the wood's surface (Rautkari *et al.* 2010). The hardness of untreated and densified beech was measured in compliance with the EN 1534 (2000) standard for

measuring the Brinell hardness (H_B) of wood and parquet flooring. The testing samples were conditioned at 65% relative humidity (RH) and 20 °C until the equilibrium moisture content (EMC) of wood was reached. A 10-mm diameter steel ball was forced to the surface with a maximum load of 1000 N for 30 s. The diameter of the indentation was measured in two directions and the mean diameter was used to calculate the Brinell hardness using Eq. 1,

$$H_B = \frac{2F}{\pi \cdot D \cdot (D - \sqrt{D^2 - d^2})}$$

(1)

where F is the applied force, π is the ratio of a circle's circumference to its diameter, D is the diameter of the indenter, and d is the indentation diameter.

Statistical analysis

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

RESULTS AND DISCUSSION

Temperature and Moisture Content

The results of surface temperature (T_s) measurements in different power modes of microwave plasticisation are presented in Fig. 2 and Table 3. The maximum value of T_s was observed at a 5-kW power mode. The average temperature increased by 60 °C in the 5-kW mode, by 55.5 °C in the 3.5-kW mode, and by 47.7 °C in the 2-kW power mode. The average T_s data and standard deviation (σ) are given in Table 3. The results show that the power of MW radiation had a substantial effect on specimen temperature. This is because the power of MW radiation influences radiation intensity, which is converted directly to thermal energy by frictional heating.

The temperature differences in individual power modes can be caused by the uneven distribution of MC or density. Nevertheless, the variability of T_s was very small. The only statistically insignificant T_s increase, based on Tukey's HSD test, was found between the 3.5-kW and 5-kW power modes. The increase in T_s of the samples was statistically significant ($p < 0.05$) in the case of all other modes (untreated and 2 kW; untreated and 3.5 kW; untreated and 5 kW; 2 kW and 3.5 kW; and 2 kW and 5 kW).

The absorption of MW radiation is affected by the material temperature because with an increasing temperature molecule, oscillation increases and the dielectric loss tangent decreases. Moreover, individual molecules are further from each other with increasing temperature and thus their collisions, which influence the friction of molecules and the heating of the material, do not occur often. This is the reason for low differences in T_s (~5 to 12 °C) between all individual MW modes (2 kW, 3.5 kW, and 5 kW) when compared with the initial temperature (control). According to Mori *et al.* (1984), with a frequency of 2.45 GHz and an output power in the range of 0.6 to 3 kW, MW radiation will raise the internal temperature of any wood by about 90 to 110 °C after 1 to 3 min.
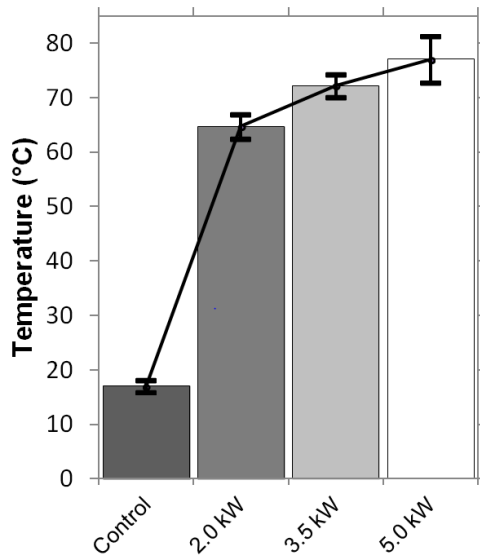
Fig. 2. Surface temperature of untreated control and treated wood with different MW plasticisation modes
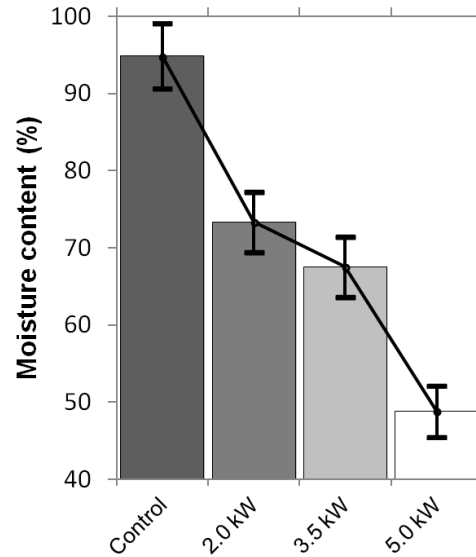

Fig. 3 Moisture content of untreated control and treated wood with different MW plasticisation modes

The measured moisture content (MC) data are presented in Fig. 3 and Table 3. Samples entering the process of MW plasticisation contained 95% of the initial MC. The results show that MW plasticisation with a 2-kW power mode reduced the MC of the testing samples by ~22%. Moisture loss of the samples treated by the 3.5-kW mode was reduced by ~27%. The highest moisture loss was found in samples treated by the 5-kW mode, where the value decreased by ~46%. The rapid loss of moisture influenced the mechanical properties of the wood due to stress caused by steam expansion.

Based on Tukey's HSD test, statistically insignificant MC was found between the 2-kW and 3.5-kW power modes. All other modes of MW plasticisation were statistically significant in terms of moisture loss. Based on the results, it can be seen that the MC of testing samples was influenced by MW power (Fig. 3). This phenomenon has also been reported by Gašparík and Gaff (2013). After MW plasticisation, the MC was still higher than the fiber saturation point, which is very important for wood softening before densification treatment.

Table 3. Results of Surface Temperature and Moisture Content of the Wood

Plasticisation Mode (kW)	Average T_s (°C)	Standard Deviation T_s (°C)	Average MC (%)	Standard Deviation MC (%)
Untreated Control	17.0 [A]	1.1	94.9 [A]	4.2
2.0	64.7 [B]	2.3	73.3 [B]	3.9
3.5	72.2 [C]	2.1	67.5 [B]	3.8
5.0	77.0 [C]	4.3	48.8 [C]	3.3

Means sharing the same letter are not significantly different (Tukey's HSD, p < 0.05)

Density and Hardness

The oven-dry density (ρ_0) values of control and densified wood are presented in Fig. 4 and Table 4. The springback effect was observed after compression. The expansion was from 5 mm to 6.11 mm (springback ~22%, in 0% MC). After the densification process, the ρ_0 of beech samples increased significantly (p < 0.05), from 648 kg·m^{-3} to about 960 kg·m^{-3} (~48%). The average ρ_0 of densified samples was similar for all individual plasticisation modes (Table 4). Therefore, the statistically significant change, based on Tukey's HSD test, was found only between the control and densified groups. Differences among the density values of specimens plasticised in modes 2 kW, 3.5 kW, and 5 kW were insignificant. The results indicate that the density of control samples exhibits enormous variability due to the differences in its composition, even within one specimen. Because of the densification treatment, the variability was eliminated from ~10% in control samples to ~2% in densified samples. This homogeneity of material is associated with the subsequent quality of the flooring.
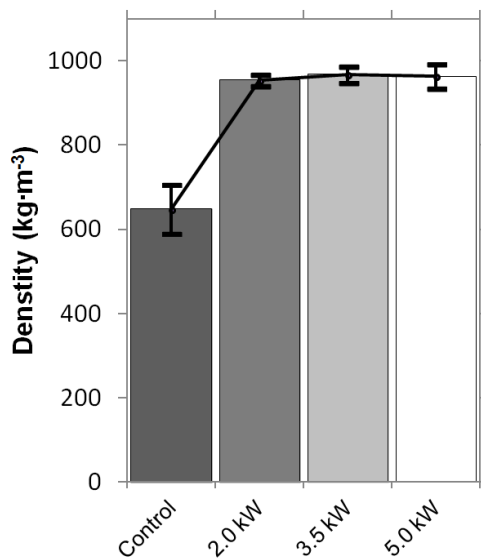

Fig. 4. Density of untreated control and densified wood with different plasticisation modes
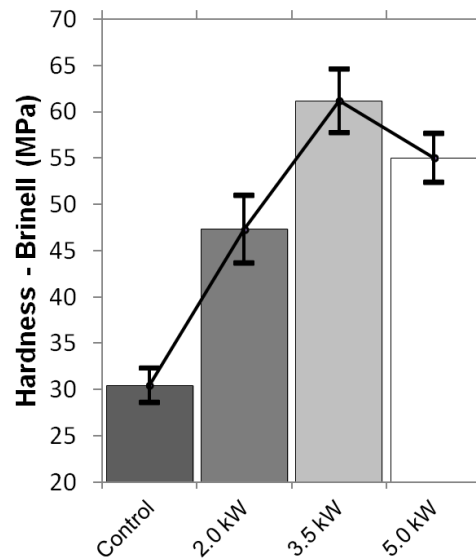

Fig. 5. Brinell hardness of untreated control and densified wood with different plasticisation modes

Table 4. Results of Density and Hardness

Plasticisation Mode (kW)	Average ρ_0 (kg·m^{-3})	Standard Deviation ρ_0 (kg·m^{-3})	Average H_B (MPa)	Standard Deviation H_B (MPa)
Untreated Control	648.0 [A]	58.6	30 [A]	1.9
2.0	954.3 [B]	14.7	47 [B]	3.7
3.5	967.7 [B]	20.3	61 [C]	3.4
5.0	963.3 [B]	29.5	55 [D]	2.7

Means sharing the same letter are not significantly different (Tukey's HSD, p < 0.05)

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The hardness values of densified wood can be increased by more than 100% (Rautkari *et al.* 2010). The results presented in Fig. 5 show the Brinell hardness (H_B) of control and densified beech wood. A significant increase ($p < 0.05$) was found for each plasticisation mode relative to the control samples. Hardness values were highly dependent upon applied MW power. The average H_B data and σ are given in Table 4. The hardness values increased by ~57% (at 2 kW), ~103% (at 3.5 kW), and ~83% (at 5 kW) when compared with the control samples.

A significant change in hardness because of densification has also been reported for different densification processes (Inoue *et al.* 1993; Navi and Heger 2004; Kamke 2006; Kutnar *et al.* 2009; Fang *et al.* 2012). Improving hardness and other mechanical properties is a primary goal of densification treatment. The greatest increase of hardness was found in the medium MW plasticisation mode (3.5 kW). The reason could be the high heat loading in the 5-kW power mode. A similar result was found in a previous study by the authors (Koiš *et al.* 2014), in which Norway spruce (*Picea abies*) was modified by MW radiation in different power modes. The results showed that the wood structure had been totally damaged when a power mode of 5 kW was used. Torgovnikov and Vinden (2009) mentioned that a high-intensity MW treatment caused a high pressure of water steam in the wood structure, which can make the cell walls delaminate. Also, Li *et al.* (2010) stated that high-intensity MW treatment created high internal steam pressure in wood cell lumens and formed a high-tensile stress in cell walls or intercellular layers. When the tensile stress in cell walls or intercellular layers was higher than its tensile strength, some cracks were generated in the cell walls and intercellular layers.

CONCLUSIONS

1. An increase in microwave power increased surface temperature significantly ($p < 0.05$).

2. Moisture content decreased significantly ($p < 0.05$) with higher modes of microwave radiation.

3. After microwave plasticisation and the densification process, the hardness of beech increased significantly ($p < 0.05$). The highest increase in Brinell hardness (103%) occurred with a plasticisation mode of 3.5 kW. Plasticisation in the highest power mode (5 kW) had a negative impact on Brinell hardness because the rapid loss of moisture content caused steam expansion and stress development.

4. Microwave heating was found to be an efficient way in terms of rapid (0.4 m/min) plasticisation as a pre-treatment for the process of wood densification. However, a good control of the process parameters is needed.

ACKNOWLEDGMENTS

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