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Faculty of Forestry and Wood Technology

Department of Wood Science

Microwave Modification of Wood

Doctoral Thesis

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Abstract

Dömény, J. (2016) Microwave modification of wood, Doctoral thesis, Mendel University in Brno, 116p.

The microwaves as a rapid and highly effective heating was well-established in society and was recognised as potentially useful method for many industrial applications. On the contrary, wood modification using microwave (MW) energy is not very common. Therefore, the main motivation of this thesis was to enhance the knowledge about the utilization of MW treatment in wood technology.

The aims of this thesis can be divided into two main tasks. In first task the author has been intended to develope, analyse and optimise various MW modification techniques. The second task investigates the relationship between MW modification techniques and selected physical and mechanical properties of treated wood. The basic information and theoretical background as well as the application of MW heating were given in the introduction and literature review.

The experimental part consisted of several investigations dealing with MW wood modification. These investigations were focused on the improving of the permeability and impregnability, pre-drying, plasticization, and acceleration of chemical reactions. Differences between particular modification treatments can be found in the power levels and duration of treatment. Within the work, the different wood species *i.e.* European beech (*Fagus sylvatica* L.), hybrid poplar (*Populus hybrids*) and Norway spruce (*Picea abies* L.) have been studied.

The effect of MW radiation on the fluid permeability and compression strength parallel to the grain was investigated in the first experiment on the beech false heartwood. The treatment was performed in discontinues MW device in different modification modes. The coefficient of specific permeability was calculated using Darcy's law and results showed the increase of specific permeability by 159%. On the contrary, the compression strength parallel to the grain decreased by up to 15%. Following experiment evaluated the influence of MW modification on impregnability of wood. The evaluation of impregnability was calculated by weight percentage gain (WPG). The samples exposed for shorter time intervals showed the improvement of impregnability (WPG 33.84%). On the contrary, samples treated for longer time results lower substance uptake.

Investigation dealing with wood pre-drying was performed using a continual laboratory MW device, which was developed for the wood modification purposes. The effect of MW radiation on moisture loss, surface temperature, and mechanical properties of Norway spruce (*Picea abies*) has been studied. The results indicated that by using low power mode and fast conveyor speed the mechanical properties of the material will be maintained. On the contrary high power mode and slow conveyor speed will have a negative impact on the changes in the wood structure and the mechanical properties when high initial moisture content (MC) of wood is used.

Investigation of MW plasticization was focused on wood densification and following application of material for flooring system purposes. The plasticization treatment was performed in different power modes. The results showed the influence of the chosen power mode on moisture decrease and rapid temperature increase, which had significant effect on plasticization and following densification process. This investigation was followed by experiment which evaluated the efficacy of MW plasticization by microstructural analysis using a scanning electron microscope (SEM) and by density profiles measurement. Results showed uniformity of density profiles through samples thickness which confirmed even plasticization in sample volume. Microscopic structure observation revealed that the MW plasticization was not accompanied with any fractures and that deformations present in the densified wood were due to viscoelastic buckling of cell walls without cracks propagation.

The investigation dealing with MW acceleration of chemical reaction has been carried out in the process of wood acetylation. Beech (*Fagus sylvatica* L.) and poplar (*Populus hybrids*) wood were impregnated using acetic anhydride, and chemical reactions were initiated by MW and conventional heating. The results showed that no significant differences were found between conventional (60 min) and MW heating (15 min). From this point of view, the MW heating can reduce the reaction time and make the process more effective.

This investigation should provide a better insight into details of the MW wood modification and could became a basis for future research.

Key words: microwave; modification; high-frequency energy; permeability; impregnability; drying; plasticization; densification; acetylation; dimensional stability; temperature; density; compression strength; bending strength; SEM

Abstrakt

Dömény, J. (2016) Mikrovlnná modifikace dřeva, Disertační práce, Mendelova univerzita v Brně, 116 s.

Mikrovlnná technologie je dnes dobře známá, používá se v domácnostech pro ohřev potravin i v různých průmyslových odvětvích, ve kterých se v posledních letech její použití rychle rozvíjí. Nicméně ve dřevařském průmyslu není tato technologie příliš využívána, proto bylo hlavními cíli předkládané disertační práce rozšířit základní poznatky o mikrovlnném ohřevu a uvést možné způsoby mikrovlnné modifikace dřeva. V první řadě šlo o vývoj, analýzu a optimalizaci různých modifikačních technik. V druhé řadě se jednalo o zhodnocení mikrovlnné modifikace ve vztahu k vybraným fyzikálním a mechanickým vlastnostem dřeva. Literární část práce obsahuje ucelený přehled teorie mikrovln a výzkumy autorů zabývajících se mikrovlnným ohřevem v dřevařství.

Experimentální část práce se skládá z několika experimentů využívajících mikrovlnné záření k modifikaci dřeva. Tyto experimenty byly zaměřeny na změnu propustnosti, impregnaci, předsušení, plastifikaci a urychlení chemických reakcí. Rozdíly mezi těmito úpravami můžeme nalézt v použitých výkonech a v době ozařování. Pro experimentální část bylo vybráno dřevo buku (*Fagus sylvatica* L.), topolu (*Populus hybrids*) a smrku (*Picea abies* L.).

Náplní prvního experimentu bylo posoudit vliv mikrovlnného záření na axiální propustnost dřeva pro kapaliny a mechanickou pevnost dřeva. Zkoušeným materiálem byl buk lesní obsahující špatně propustné nepravé jádro. Mikrovlnná úprava byla provedena v diskontinuálním mikrovlnném zařízení ve dvou variantách s rozdílnou dobou ozařování. Koeficient specifické propustnosti byl vypočítán na základě Darcyho zákona. Výsledky vykazovaly zvýšení propustnosti až o 159 %, avšak současně snížení meze pevnosti dřeva o 15 %. Následující experiment posuzoval vliv mikrovlnné úpravy na impregnovatelnost dřeva. Vyhodnocení experimentu bylo provedeno pomocí výpočtu hmotnostního přírůstku impregnované látky. Zkušební vzorky vystavené ozařování v kratších intervalech vykazovaly zvýšení impregnovatelnosti dřeva (WPG 33,84 %), naopak vzorky vystavené ozařování v delších intervalech mikrovlnné úpravy vykazovaly nižší příjem impregnační látky.

Experiment zabývající se mikrovlnným předsušením byl proveden na zkušebních vzorcích dřeva smrku ztepilého (*Picea abies* L.). K ohřevu byla použita kontinuální mikrovlnná linka, která byla vyrobená pro úpravu vlastností dřeva. Studie sledovala závislost výkonu mikrovlnného záření na výslednou vlhkost dřeva, teplotu povrchu a mechanickou pevnost. Z výsledků je patrné, že mechanické vlastnosti materiálu jsou zachovány při použití nižších výkonů ozařování a větších rychlostí posuvu materiálu modifikační komorou.

Experiment mikrovlnné plastifikace byl zaměřen na lisování dřeva pro výrobu podlahových dílů. Plastifikace byla provedena na kontinuální mikrovlnné lince při různých výkonech záření. Výsledky studie prokázaly vliv výkonu záření na teplotu a vlhkost materiálu, což mělo významný vliv na plastifikaci dřeva a jeho následné zhuštění. Na tento experiment navázala studie pro vyhodnocení efektivnosti mikrovlnné plastifikace z pohledu mikroskopické analýzy a hustotních profilů zhuštěného materiálu. Výsledky hustotních profilů prokázaly, že materiál byl zhuštěn rovnoměrně v celém průřezu, z čehož plyne, že materiál byl plastifikován v celém objemu. Mikroskopická analýza neprokázala vznik trhlin vlivem mikrovlnné plastifikace ani vlivem zhušťování dřeva.

Experiment, zaměřený na urychlení chemických reakcí byl ověřen v procesu acetylace dřeva. Po impregnaci dřeva buku (*Fagus sylvatica* L.) a topolu (*Populus hybrids*) anhydridem kyseliny octové, byly vzorky zahřívány pomocí mikrovln i klasickým konvenčním ohřevem. Z výsledků je patrné, že nedošlo k statisticky výrazným rozdílům mezi mikrovlnnou (15 min) a konvenční (60 min) acetylací dřeva. Z tohoto pohledu může mikrovlnný ohřev urychlit chemické reakce mezi dřevem a látkou.

Tato práce by měla rozšířit základní znalosti z oblasti mikrovlnného ohřevu a poskytnout komplexní přehled o možných způsobech modifikací dřeva vedoucí ke zlepšení jeho užitných vlastností.

Klíčová slova: mikrovlny; modifikace; vysokofrekvenční energie; propustnost; impregnovatelnost; sušení; plastifikace; zhušťování; acetylace; rozměrová stálost; teplota; hustota; pevnost v tlaku; pevnost v ohybu; mikroskopická analýza

Preface

The present study was carried out at the Department of Wood Science at Faculty of Forestry and Wood Technology, Mendel University in Brno (Czech Republic).

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Brno, January 2016

Jakub Dömény

List of Papers

This doctoral thesis is a summary of the following papers, which are referred in the text by Roman numerals I–VI.

I. Dömény, J., Koiš, V., Dejmal, A. (2014) Microwave radiation effect on axial fluid permeability in false heartwood of beech (*Fagus sylvatica* L.). BioResources 9(1): 372–380. DOI: 10.15376/biores.9.1.372-380.

II. Dömény, J., Koiš, V., Pařil, P. (2013) Impregnability of European beech false heart wood after microwave treatment. Pro Ligno 9(4): 190–194. ISSN: 1841-4737.

III. Koiš, V., Dömény, J., Tippner, J. (2014) Microwave device for continuous modification of wood. BioResources 9(2): 3025–3037. DOI: 10.15376/biores. 9.2.3025-3037.

IV. Dömény, J., Koiš, V., Zapletal, M. (2014) Application of microwave treatment for the plasticization of beech wood (*Fagus sylvatica* L.) and its densification for flooring system purposes. BioResources (9)4: 7519–7528. DOI: 10.15376/biores.9.4.7519-7528.

V. Dömény, J., Čermák, P., Koiš, V., Tippner, J., Rousek, R. Density profile and microstructural analysis of densified beech wood (*Fagus sylvatica* L.) plasticized by microwave treatment. Prepared manuscript.

VI. Dömény, J., Čermák, P., Pařil, P., Fodor, F. P., Dejmal, A., Rademacher, P. (2015) Application of microwave heating for acetylation of beech (*Fagus sylvatica* L.) and poplar (*Populus hybrids*) wood. BioResources 10(4): 8181–8193. DOI: 10.15376/biores.10.4.8181-8193.

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List of Abbreviations

ANOVA	analysis of variance
ASE	anti-swelling efficiency
EMC	equilibrium moisture content
H_B	Brinell hardness
HSD	Tukey's honest significance test
ISM	industrial, scientific and medical frequencies
MC	moisture content
MOE	modulus of elasticity
MOR	modulus of rupture
MW	microwave
R	retention
RH	relative humidity
S	swelling
SD	standard deviation
SEM	scanning electron microscope
VOC	volatile organic compound
WPG	weight percentage gain

Symbols

f	frequency (Hz)
ω	angular frequency ($rad \cdot s^{-1}$)
λ_w	wavelength (m)
c	speed of propagation ($m \cdot s^{-1}$)
B	magnetic flux density ($N \cdot m^{-1} \cdot A^{-1}$)
D	electric flux density ($C \cdot m^{-2}$)
E	electric field strength ($V \cdot m^{-1}$)
E_0	maximum electric field ($V \cdot m^{-1}$)
H	magnetic field strength ($A \cdot m^{-1}$)
J	current density ($A \cdot m^{-2}$)
ε	relative complex permittivity
ε^*	complex permittivity

ε_0	permittivity of vacuum ($\varepsilon_0 = 8.85 \cdot 10^{-12}$)
ε'	relative permittivity
ε''	relative dielectric loss factor
μ	relative complex permeability ($H \cdot m^{-1}$)
μ^*	complex permeability ($H \cdot m^{-1}$)
μ_0	magnetic permeability of vacuum ($\mu_0 = 4 \cdot \pi \cdot 10^{-7}$)
$\tan \delta$	dielectric loss tangent
F	farad
σ	conductivity ($S \cdot m^{-1}$)
e	Euler's number ($e = 2.718$)
j	imaginary number ($j = \sqrt{-1}$)
γ	complex distribution factor
α	attenuation factor – real part of the complex wave
β	phase factor – imaginary part of the complex wave
D_p	penetration depth (m)
t	time (s)
P	polarization ($C \cdot m^{-2}$)
T_s	surface temperature (°C)
Q	generated heat ($W \cdot m^{-3}$)
ρ	density ($kg \cdot m^{-3}$)
C	specific heat capacity ($J \cdot kg^{-1} \cdot K^{-1}$)
λ	thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)
η	dynamic viscosity ($Pa \cdot s$)
V	volume (m^3)
S	area of the flow (m^2)
$\Delta\rho_p$	difference in pressures (Pa)
K	coefficient of specific permeability ($m^3 \cdot m^{-1}$)
m	weight (g)

1 INTRODUCTION

1.1 Background

Wood as an important renewable resource, has a broad range of material properties for various applications, *e.g.,* relatively high strength and stiffness, low specific weight, natural appearance with interesting texture, good insulation properties, and easy machinability (Kollmann 1951; Stamm 1964; Wagenführ 2000; Skodras *et al.* 2004). The huge diversity in wood species guarantees that there is always a species with the required properties for a specific purpose (Boonstra 2008). Unfortunately rising demand for consistent, high quality material increases prices and availability of some wood species, especially tropical hardwood species. Therefore, in last decades raised interest in different wood modification techniques as use of the adequate reserve of less durable wood species. To that end, technologies for improving wood properties, *e.g.*, bio-durability, dimensional stability, colour, wettability, etc. are indispensable (Hill and Jones 1996; Militz 2002; Rowell 2005; Hill 2006; Čermák *et al.* 2015). Hill (2006) explains that wood can be modified chemically, biologically or physically, with purpose of fundamentally changing the certain properties. These treatments are well known nowadays and some of them are widely used in wood industry. The most important thing in industries, apart from producing the high quality products for the market, is to increase productivity and to reduce production cost. In general, several wood modification techniques are related to heating of the wood, *e.g.,* heat treatment, chemical modification (acetylation and butylation), densification, etc.*,* which is most energy intensive and costly process in wood products industry. Commonly used conventional heating in modification processes based on convective heat transfer from hot air or different media to wood surface, followed by subsequent conductive heat transfer from surface to internal parts of wood might limit the range of feasible applications. This requires a considerable amount of energy and long lasting heating in order to reach the required temperature in all cross-section of the material. Such heating levels usually results in low production rate and high cost products. Therefore, the application of MW energy has been recognised as potentially useful method for reduce the heating time and make modification processes more effective. The main advantage of this method is the rapid heat of the material throughout the whole cross-section due to dielectric properties of wood and water (Torgovnikov 1993;

Larsson *et al.* 1999). Another advantage is the ability to use MW heating in continual process, which may be included in the flow technology production. The principle behind MW heating is based on the polar characteristic of molecules and their ability to absorb and transform MW radiation into heat (Metaxas and Meredith 1983; Torgovnikov 1993; Hansson and Antti 2003). Permanent dipoles of molecules begin to move with the same frequency as the electromagnetic field. Therefore, rapid changes in the field polarity cause vibration and rotation of molecules, which transforms the MW energy into frictional heat (Makovíny 2000; Hansson and Antti 2003).

Since the 1970s, several investigations have been carried out in the field of MW wood modification, which was basically focused on acceleration of chemical reactions, permeability changes, drying, reducing the growth tension, wood plasticization and elimination of volatile organic compound (VOC). Unfortunately, published studies related to modification techniques using MW radiation are still limited. Therefore, this work was performed and it should provide a better insight into details of the MW wood modification.

1.2 Aims of the study

The main aim of the presented doctoral thesis was to enhance the knowledge about the utilization about MW treatment in wood technology. The derived aims were to (a) develop, analyse and optimize various MW modification techniques, and (b) investigate the relationship between MW modification and selected physical and mechanical properties of wood. The results of the thesis and papers should provide a better understanding of MW modification, which probably contributes to a more controlled use of MW treated wood in service conditions. The thesis pursues the following objectives:

Part a: Microwave modification techniques

I. Find out the influence of MW radiation on axial wood permeability and increase of impregnability in transverse directions.
II. Examine the effect of MW radiation on continual wood pre-drying.
III. Contribute to the knowledge of MW heating for plasticization in order to develop and accelerate the process of wood compression.
IV. Accelerate the chemical reactions between wood and chemicals using MW heating.

Part b: Material properties of microwave modified wood

V. Evaluate the change in microscopic structure using a scanning electron microscope (SEM).
VI. Evaluate the physical properties of MW modified wood (equilibrium moisture content, swelling, density, surface temperature).
VII. Evaluate the mechanical properties of MW modified wood (compression strength, bending strength, hardness).

1.3 Outline of the study

This doctoral thesis summarizes MW modification techniques together with articles published in scientific journals and conference proceedings that were authored during the years 2013 to 2016. The work done during this time in various fields of MW modification processes has traditional outlines and consists the theoretical background, materials and methods and results together with a discussion and the conclusions drawn from this work. Introduction chapter presents general background of the field of work as well as the main motivation and aims of the thesis. The literature chapter describes electromagnetic theory, MW heating, wood structure and MW effect on physical and mechanical properties. Several studies related to the application of MW treatments in wood technology have been published in last decades and therefore the next part summarized the review about this modification techniques. The following part listed scientific papers published in peer review journals as output of this thesis and finally there is a summary of the conclusions made.

2 LITERATURE REVIEW

2.1 Electromagnetic heating

The existence of electromagnetic heating was known from 19th century and by the late 1920s industrial technologies were already available to warm up materials with frequencies of around 3 kHz (Bolourian 2010; Vikberg *et al.* 2014). The magnetrons for MW heating began to be used in the 1940s and this technology allowed frequencies higher than 3 GHz to be generated (Redhead 2001; Hansson 2007). Field of wood industry started to use magnetron in the early 1960s (Egner and Jagfeld 1964). According to the literature, dielectric heating was first applied in the production of plywood and in curing of synthetic resins (Vikberg *et al.* 2014).

Depending on frequency, dielectric heating can be divided into two technologies, radio frequency and MW. Radio frequencies below 100 MHz are generated with open-wire circuits and applied between metallic electrodes. MWs are emitted from vacuum tubes direct into the materials trough metallic tubes called waveguides and can be generated at 85–94% electrical efficiency (Resch 2006; Saitou 2006; Vikberg *et al.* 2014). The operational frequency spectrum of MWs ranges from 300 MHz to 300 GHz with corresponding wavelengths ranging from 1 m to 1 mm. The most commonly used frequencies for MW heating are 2.45 GHz and 915 MHz (Starck *et al.* 2005). The power penetration depth decreased with using the higher frequency. For example, maximum absorption for water is located near 10 GHz, but only surface heating can be reached. The actual frequencies used for industrial high frequency heating are regulated to avoid interference (Resch 2006). Basically, small number of ISM (industrial, scientific and medical) frequencies are internationally reserved for use in process heating, medical diathermy equipment, MW oven, etc. (these frequencies are 5.8 GHz, 2.45 GHz, 40.68 MHz, 27.12 MHz, 13.56 MHz, etc.). The ISM bands can be used without a license in most countries (ITU-R 2012), however these regulations can deviate depending on the country specific rules (Imenokhoyev *et al.* 2013).

The common understanding of dielectric heating is based upon the motion of ions and permanent (or induced) dipoles as they attempt to follow the applied electric field component (Metaxas and Meredith 1983; Torgovnikov 1993; Hansson and Antti 2003). These fields create a molecular friction, which results in heating throughout the mass of the material (Jumiang *et al.* 2002). This differs from conventional methods in which heat is transferred between objects or medium through the mechanisms of conduction and convection. Because the material itself generates the heat, heating is very rapid and selective (Sutton 1989). This technology promises several advantages over conventional methods, such as reduction of processing time, saving of energy and costs by high energy efficiency, possible continuous application and volumetric heating (Seyfarth *et al.* 2003; Vongpradubchai and Rattanadecho 2009; Gašparík and Gaff 2013; Imenokhoyev *et al.* 2013).

Industrial and commercial MW heating is used for a number of applications such as cooking, curing, evaporation, drying, steriling, wireless signals transmission, medical treatment, vulcanizing, and many other applications. However, use of MW energy is not so common in wood industry.

2.1.1 Electromagnetic wave

The electromagnetic waves include two components: electric and magnetic field. These two fields oscillate vertically relative to each other and they are perpendicular to the direction of propagation. The influence of the magnetic field on wood is negligible, because in dielectric materials magnetic permeability is comparable to that of free space, therefore is not taken into consideration for practical purposes. On the other hand the influence of the electric field on wood is very high. Thus, the high frequency electromagnetic waves are able to polarize charges in wood material (Torgovnikov 1993).

Monochromatic electromagnetic wave (Figure 1) is sinusoidal wave determined with one frequency (f) and one wavelength (λ_w). The wavelength is related to frequency through the speed of propagation (c):

$$c = \lambda_w \cdot f \quad (\mathrm{m \cdot s^{-1}}) \tag{1}$$

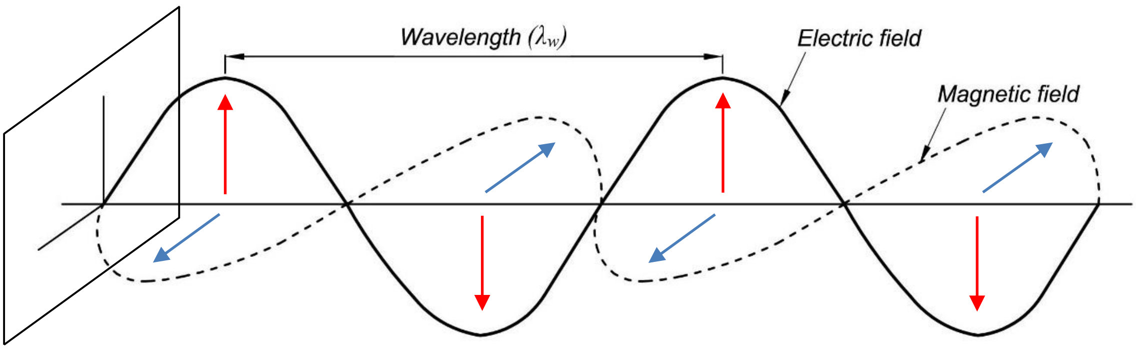

Figure 1 Monochromatic electromagnetic wave showing the perpendicularly-oriented waves of electric and magnetic fields, and the characteristic wavelength

The speed of propagation in vacuum is $3 \cdot 10^8$ m·s^{-1}, but when the wave enters a dielectric medium, the speed and wavelength are reduced. Electromagnetic behaviour in dielectric media is governed by Maxwell's equations. James Maxwell worked out a theory of electromagnetism in 1864, supported by twenty equations describing the behaviour of electric and magnetic fields (Fleisch 2008). These equations were later combined and simplified into four equations, known as Maxwell's equations. Three constitutive equations (2), (3), and (4) describing the relationship between electromagnetic fields and materials can be derived from Maxwell's equations (Chen *et al.* 2004):

$$D = \varepsilon \cdot E \qquad (\mathrm{C \cdot m^{-2}}) \tag{2}$$

$$B = \mu \cdot H \qquad (\mathrm{N \cdot m^{-1} \cdot A^{-1}}) \tag{3}$$

$$J = \sigma \cdot E \qquad (\mathrm{A \cdot m^{-2}}) \tag{4}$$

where: D (C·m^{-2}) is electric flux density, B (N·m^{-1}·A^{-1}) is magnetic flux density, E (V·m^{-1}) is electric field strength, H (A·m^{-1}) is magnetic field strength, J (A·m^{-2}) is current density, ε is relative complex permittivity of material, μ (H·m^{-1}) is the relative complex permeability of the material and σ (S·m^{-1}) is conductivity of the material (Torgovnikov 1993). Permittivity describes the interaction of a material with an electric field and shows how the electric charge distribution in the material is changed by the application of an electric field (Chen *et al.* 2004). Permeability describes the interaction of a material with a magnetic field. As stated above, the permeability of dielectrics is comparable to that of vacuum and is therefore not considered in dielectric measurements. Conductivity is a measure of how easily electrons can travel through the material under the influence of an external electric field (Paz 2010).

In effect, Maxwell's equations are a set of relations linking the values of a number of quantities that describe electric (D, E) and magnetic (B, H) fields. All are vector quantities and are functions not only of the three spatial coordinates x, y and z but also of time t (Syms and Cozens 1992). With respect to Maxwell's equations, the electric field from a wave moving in the z direction at time t can be described by the equation (Hippel 1954):

$$E = E_0 \cdot e^{j\omega \cdot t - \gamma \cdot z} \qquad (\mathrm{V \cdot m^{-1}}) \tag{5}$$

where: E_0 ($V \cdot m^{-1}$) is maximum value of electric field, e is Euler's number (e = 2.718), j is imaginary number $\left(j = \sqrt{-1}\right)$, t (s) is time, γ is complex distribution factor, z (m) is distance of the dielectric from the surface and ω ($rad \cdot s^{-1}$) is the angular frequency of the wave, which can be defined by the equation:

$$\omega = 2 \cdot \pi \cdot f \quad (rad \cdot s^{-1}) \tag{6}$$

The electric field is transverse to the z direction and move through space or material with a complex distribution factor (Metaxas and Binner 1991):

$$\gamma = j\omega\sqrt{\varepsilon^* \cdot \varepsilon_0 \cdot \mu^* \cdot \mu_0} = \alpha + j\beta \tag{7}$$

where: ε^* is the complex permittivity, which indicates the ability to absorb and store energy, ε_0 is the permittivity of vacuum ($\varepsilon_0 = 8.85 \cdot 10^{-12}$), μ^* is the complex permeability, which describes the interaction of a material with a magnetic field, and μ_0 is the permeability of vacuum ($\mu_0 = 4 \cdot \pi \cdot 10^{-7}$). The real part of γ can be defined as an attenuation factor α and the imaginary part as a phase factor of the wave β. The complex dielectric constant ε^* and relative complex permittivity ε can be defined by the equations (Hippel 1954):

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \tag{8}$$

$$\varepsilon = \varepsilon_0 \cdot \varepsilon^* \tag{9}$$

where: ε' is the relative permittivity (real part) and ε'' is the relative dielectric loss factor (imaginary component). Relative permittivity shows how much slower an electromagnetic wave penetrates trough the wood in relation with vacuum, respectively represents the energy that is stored in the polarized dipoles (Erchiqui 2013). Relative dielectric loss factor includes all losses which can occur during penetration of electromagnetic wave trough wood, respectively determines the energy that is transformed into heat or elastic forces during the polarization (Vikberg *et al.* 2014).

The ratio between the real and imaginary components of the dielectric constant can be expressed by dielectric loss tangent (tan δ). It is the indicator of material properties which reflects the effect of applied electric field vectors and direct current conductivity.

It stands for the high frequency energy dissipated in material and subsequently transformed into thermal energy (Torgovnikov 1993):

$$tan\,\delta = \frac{\varepsilon''}{\varepsilon'} \tag{10}$$

Combining equations 7 and 9 the real part can be divorced from imaginary part of complex propagation factor γ and equations for attenuation factor α and phase shift factor β can be achieved (Hippel 1954):

$$\alpha = \omega\sqrt{\varepsilon_0 \cdot \mu_0}\left(\frac{\varepsilon'}{2}\left(\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right)\right)^{\frac{1}{2}} \tag{11}$$

$$\beta = \omega\sqrt{\varepsilon_0 \cdot \mu_0}\left(\frac{\varepsilon'}{2}\left(\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}+1\right)\right)^{\frac{1}{2}} \tag{12}$$

2.1.2 Wave attenuation

The interaction of an electromagnetic field with a material may cause several responses, and MWs can be reflected, absorbed or transmitted. Reflective materials tend to be bulk metals with many free electrons (Saitou 2006). Transparent materials tend to have low conductivities associated with members of the glass and ceramics family (Imenokhoyev *et al.* 2013). Absorbing materials consist of all those which exhibit dielectric character (Torgovnikov 1993).

When an electromagnetic field is penetrating a dielectric material, *i.e.* wood, the energy is gradually absorbed by the material due to the polarity. The electromagnetic field strength at the surface is therefore decreasing exponentially during the penetration. The attenuation inside the material can be explained by the exponential equation (Torgovnikov 1993):

$$E(z) = E_0 \cdot e^{-\alpha \cdot z} \qquad (\mathrm{V \cdot m^{-1}}) \tag{13}$$

where: α is attenuation factor and z is distance of the dielectric from the surface. The influence of attenuation and phase shift on the wave is shown in Figure 2.

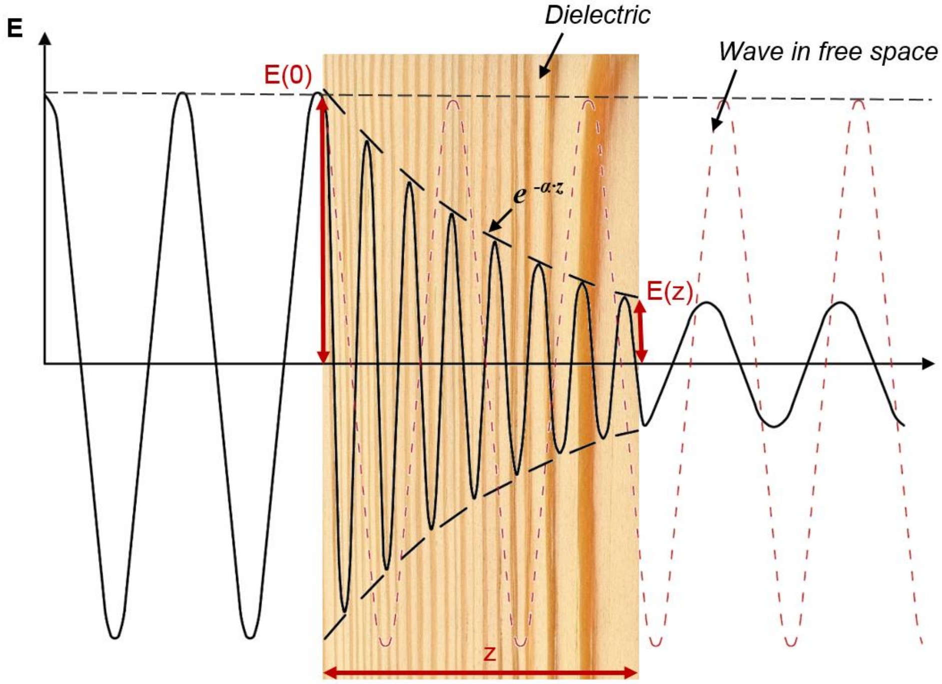

Figure 2 Wave attenuation: electromagnetic wave transmitted into wood

The penetration depth (D_p) depends on dielectric properties of wood, density, MC, temperature, grain orientation, etc. The D_p is defined as the thickness of the material when the transmitted power is reduced to $1/e$ of its original value. That mean when material would absorb approximately 63% of the incident electromagnetic power. The penetration depth is given by equation (Metaxas and Binner 1991; Koubaa *et al.* 2008):

$$D_p = \frac{1}{2 \cdot \alpha} \qquad \text{(m)} \qquad (14)$$

Material with large thickness and high loss factor may cause that heating occurs only in surface layers. To prevent this phenomenon, proper electromagnetic heating must be chosen so that enough time is provided for a subsequent heat flow between surface and core layers. When thickness of the material is less than the D_p, only part of supplied energy will be absorbed (Metaxas and Binner 1991; Vongpradubchai and Rattanadecho 2009; Kormin *et al.* 2013).

2.1.3 Polarization

The electromagnetic waves are able to polarized charges in dielectric materials. Polarization is categorized into 4 mechanisms, *i.e.* electronic, ionic, dipole and interfacial (Skaar 1988). Electronic and ionic polarization are not connected with energy losses. These polarizations play the main role when optical or infrared spectroscopy is used, therefore they are not taken into consideration for heating purposes. Dipole polarization consists in displacement and rotation of molecules with an asymmetric charge distribution in direction of an external field and creating molecular friction (Metaxas and Meredith 1983). The interfacial polarization play role in the dielectric properties of the wood, because it occurs in heterogeneous dielectric materials. Wood consists of components in solid, liquid and gaseous state with different potentials causes a charge in these components and free electrons and ions, starts moving through the volume (Skaar 1988; Sugimoto and Norimoto 2004).

Polarization (P) is a quantity which numerically characterizes the substance polarization effect under the influence of the external electric field through its relative dielectric constant (Torgovnikov 1993):

$$P = (\varepsilon' - 1) \cdot \varepsilon_0 \cdot E \qquad (\mathrm{C \cdot m^{-2}}) \tag{15}$$

The total polarization of the substance is generally expressed as the sum of electronic, ionic, dipole and interfacial polarizations.

2.1.4 Thermal distribution during the microwave heating

According to Maxwell's equations approaches, the MW heat source is computed from the knowledge of the electric field strength (Curet and Rouaud 2011). The heat Q ($\mathrm{W \cdot m^{-3}}$) generated per unit volume in a material can be calculated using equation (Torgovnikov 1993):

$$Q = \omega \cdot \varepsilon_0 \cdot \varepsilon'' \cdot E^2 \qquad (\mathrm{W \cdot m^{-3}}) \tag{16}$$

When wood absorbs MW energy, its volume will be instantly heated. However heating will be not uniform through the volume due to the nature of MW and material properties, *i.e.* density and MC. Heat conduction will serve to level out the uneven temperature distribution (Hansson 2007). Heat transfer within the wood is based on the generalized heat equation, which depends on its thermo-physical properties. The tree-dimensional heat equation may be used to describe heating of material, as follows (Lundgren *et al.* 2006):

$$\rho \cdot C \frac{\partial T}{\partial t} = \nabla \cdot (\lambda \nabla T) + Q \tag{17}$$

where: ρ (kg·m^{-3}) is density, C (J·kg^{-1}·K^{-1}) is specific heat capacity, T (K) is temperature, t (s) is time, λ (W·m^{-1}·K^{-1}) is thermal conductivity and Q (W·m^{-3}) is generated heat.

2.2 Wood and microwave heating

Wood is a natural composite material and chemical complex of cellulose, hemicelluloses, lignin, and extractives (Hon and Shiraishi 2001). Cellulose and hemicelluloses are the polysaccharides which constitute about 70% of the wood substance and determine the dielectric properties of the cell wall (Torgovnikov 1993). Lignin is a mixture of irregularly ramified copolymers based on phenyl-propane units. All of these three main components are polar polymers characteristic by process of dipole polarization. Cellulose polarization is associated with a displacement and rotation of hydroxyl groups (-OH), methylene groups (-CH_2OH) and glucose residues (Metaxas and Meredith 1983). Hemicelluloses polarization is close to cellulose, mainly glucomannan which is one of the main hemicellulose in softwood. Their polarization is associated mainly with methylene groups. Xylan hemicellulose does not contain methylene groups, and therefore its dielectric properties are not very significant. Lignin has lower dielectric properties in comparison with main wood polysaccharides despite the presence of hydroxyl and methylene groups (Torgovnikov 1993).

The importance of dipole polarization in dry wood is very small compared with its effect in water (Paz 2010). The presence of moisture in wood has biggest influence on dielectric properties due to strong dipole moment of water molecules (William 1975). Water in wood is primarily held by chemical bonds. These are hydrogen bonds formed by the hydroxyl groups in the wood cell walls (Paz 2010). As MC increases, water occupies the cell cavities and pores, held only by mechanical forces (Berry and Roderick 2005).

Monomolecular MC (~0–6%) possess the most stable bonds with the hydroxyl groups, therefore molecules exhibit limited mobility under the external electromagnetic field (Torgovnikov 1993). With increasing MC the higher dielectric parameters could be reached, not only due to larger amounts of polar molecules, but also because of their greater mobility.

The presence of all of mentioned dipole molecules have effect on dielectric loss factor and this making wood a good candidate for processing with high frequency energy. However, wood as an anisotropic material has different dielectric properties in longitudinal, radial and tangential directions, which have to be taken into account mainly for dielectric measurements (Koubaa *et al.* 2008). On the contrary, complex dielectric constants in the radial and tangential directions do not differ much so they can be considered as equal (Torgovnikov 1993). Dielectric losses are normally higher along the grain than across the grain (Samson 1984).

2.2.1 Anatomical structure changes

Some published studies report that MW radiation may cause the delamination of wood structure. According to Merenda and Holan (2008), Torgovnikov and Vinden (2009), Li *et al.* (2009), Yu *et al*. (2011) and Vinden *et al*. (2011) applying of MW radiation to the wood caused a high pressure of water steam in the wood structure, which can make the cell walls delaminated. Terziev (2002) investigated the microscopic changes in microwave-dried Scots pine (*Pinus sylvestris* L.). The author stated that after the modification significant changes occurred in the structure of the wood. Zhang *et al.* (2013) examined the effects of MW radiation on the microscopic structure changes in the wood of European larch (*Larix decidua*). They evaluated the wood with an electron microscope and revealed changes in cell wall pits. Originally closed double pits of tracheids were ruptured consequent to the MW irradiation, which opened the conductive paths and shortened the drying time. Hong-Hai *et al.* (2005) also dealt with the microscopic changes in the European larch (*Larix decidua*) in relation to various outputs and exposure times. The results showed that with high outputs the membranes in the cell wall pits of parenchymatic cells cracked, which increased the radial permeability of wood for gases and liquids. Jiang *et al.* (2006) reached the same results as Hong-Hai *et al.* (2005). Their microscopic analysis discovered micro-cracks in the area of parenchymatic cells. Nasswettrová and Nikl (2011) explored the changes in the structure of microwave-dried wood of beech (*Fagus sylvatica* L.) and Scots pine (*Pinus sylvestris* L.). Following their results, they reported no deformations of the anatomical elements of wood. XiMing *et al.* (2002) tested various times of treatment using the wood of ash (*Fraxinus mandshurica*) and poplar (*Populus canadensis*). Their experimental results were based on the slides of the electron microscope. It turned out that after MW treatment the cell wall pits ruptured and the tyloses in vessels were realigned. In some cases, the tyloses were even washed away from the wood structure. Vongpradubchai and Rattanadecho (2009) compared the structure of wood dried by MW and conventional heating. The authors stated that the microscopic structure was very similar in both drying methods and did not contain any visible defects. However, the authors reported a better structure in the microwave-dried samples due to lower values of shrinking. Torgovnikov and Vinden (2009) dealt with the MW modification of wood and evaluated the changes of anatomical elements. The results indicated that parenchymatic cells are the first to get destroyed. Depending on the output used, subsequently micro-cracks occur in tracheids, libriform fibres and vessels.

2.2.2 Thermal and moisture behaviour during microwave heating

The temperature of wood during MW heating depends on several factors, *i.e.* power of MW radiation, time of treatment, dimensions of material, MC, frequency and permeability of wood (Norimoto and Gril 1989; Leiker *et al.* 2004; Studhalter 2005; Brodie 2007b; Studhalter *et al.* 2009). According to Mori *et al.* (1984), using the power in the range of 0.6 to 2.4 kW, at a frequency 2.45 GHz and time of treatment 1–3 minutes, the surface temperature will reach 90–110 °C and internal layers of wood will reach 100–130 °C. The temperature cannot exceed these values as long as free water remains (Mori *et al.* 1984; Norimoto and Gril 1989). The higher temperature levels were obtained only in the wood species with low permeability, where exceptionally low vapour diffusion rate occurred (Norimoto and Gril 1989). Vongpradubchai and Rattanadecho (2011) reported, that MC has significant effect on the temperature development, because MW energy is absorbed mainly by water in the wood. Studhalter *et al.* (2009) stated, that the larger temperature gradient occurred in the green timber, where was found the lowest surface temperature and the highest internal temperature. Zielonka and Dolowy (1998), Antti *et al.* (1999), Zielonka and Gierlik (1999) and Brodie (2007b) demonstrated that when thick material is MW heated, the highest temperature is located at a distance from the surface. The temperature increases in the direction from the surface to this critical point and vice versa, it decreases linearly from this point away (Brodie 2007b). To prevent such a phenomenon and obtain the temperature in whole cross-section, the MW treatment must be well chosen so that enough time is provided for the subsequent conductive heat transfer between the boundary and the core (Vongpradubchai and Rattanadecho 2011). It was also found that timber with lower MC can be heated more uniformly (Studhalter *et al.* 2009).

2.2.3 Mechanical properties

Studies of many authors indicate that high values of MW output result in the reduction of wood mechanical properties. For example, Oloyede and Groombridge (2000) examined the effect of MW drying of Caribbean pine (*Pinus caribaea*) on the tensile strength parallel to the grain. The authors concluded that the strength decreased by up to 60% when the highest possible output (1.6 kW) was used. Antti *et al.* (2001) challenged the results explaining them by an inappropriate selection of the drying mode. They stated that the process was hard and not possible to control. Hansson and Antti (2003) conducted a study dealing with comparison of the MW, conventional and high-temperature drying of spruce (*Picea abies* L.) focusing on the changes of mechanical properties. They measured the modulus of rupture (MOR) and modulus of elasticity (MOE) in static bending. Their results did not manifest any statistically significant differences between the methods of drying. Terziev and Daniel (2002) conducted a similar experiment using the Scots pine (*Pinus sylvestris* L.). The authors stated that the most chemical and structural changes of the material occurred in the case of the MW drying. Hong-Hai *et al.* (2005) measured the bending strength of microwave-modified European larch (*Larix decidua*) and complemented their study with a microscopic analysis. They recorded the decrease in the strength, occurrence of micro-cracks and rupturing of the cell wall pits. The authors reported that the micro-cracks especially emerged between parenchymatic cells, therefore the reduction of the bending strength was not very marked. Machado (2006) examined the influence of the MW heating on the compressive strength of oak (*Quercus pyrenaica* Wild.). The results of his experiment indicated the decrease in mechanical properties by 10% to 20%, depending on the mode applied. Leiker *et al.* (2005) dried the wood of beech (*Fagus sylvatica* L.) with a combination of MW heating and vacuum. The dried material was subsequently subjected to the bending strength test and the results were compared with conventional drying and MW drying in the atmospheric pressure. No statistically significant differences were found among these three groups as regards the wood strength. The author mentioned that the results were affected by the variability of the measured data. Vinden *et al.* (2011) irradiated railway sleepers from pine (*Pinus radiata*) with the aim to increase the impregnability of the material. However, after this MW treatment they recorded decreased MOR. According to the Australian standard AS 3818.2 (2004), the values of the strength were still suitable for use for railway sleepers. According to Torgovnikov and Vinden (2009), the high absorption of MW energy in the wood can cause an overpressure of the water vapour, which leads to the formation of cracks between cells and weakening of the material strength. The authors proved this theory by an experiment in which they irradiated the wood of pine (*Pinus radiata*). This treatment led to a reduction of strength by 4–26%. Lawrence (2004) also recorded decreased MOE and MOR in microwave-irradiated pine (*Pinus radiata*). The reduction of mechanical properties was reportedly caused by the modification of tracheids. The author stated that the reduction of mechanical properties was influenced in the process rather by the movement of the material than the output of radiation. Lawrence (2003) reported the same dependence of the feed speed on the decrease in hardness of Eucalyptus (*Eucalyptus obliqua*). He recorded up to 54% decrease in the hardness of the material in comparison with the reference samples.

2.3 Application of microwave treatments in wood technology

MW application n for wood industry has been studied by several scientists in last decades. This technology can be used, for example for wood drying due to which the drying time shortens significantly (Zielonka and Gierlik 1999; Dedic and Zlatanovic 2001; Hansson and Antti 2003; Hunt *et al.* 2005) or it can be used in various combinations, such as MW-vacuum drying, MW-sublimation drying, MW-conventional drying, etc. (Mollekopf and Wagenführ 2002; Seyfarth *et al.* 2003; Leiker and Adamska 2004). In addition to the shortening of the drying time, MW modification increases the permeability and facilitates the uptake of protective substances by the wood structure (Simon *et al.* 2007; Torgovnikov and Vinden 2008; Torgovnikov and Vinden 2009; Vinden *et al.* 2011). According to Vinden *et al.* (2011), it helps reduce the growth tension in wood. BinHua *et al.* (2012) summarized the possible applications of the MW technology for the wood industry, like wood drying, curing of adhesives, defect detection of wood, MC measuring and VOC emission reduction. This treatment can be used also for plasticization of wood (Studhalter *et al.* 2009) and for acceleration of chemical reactions, *e.g.* MW acetylation (Larsson and Simonson 1999; Treu *et al.* 2007; Yu *et al.* 2009).

2.3.1 Drying

The first attempts with wood drying using MW radiation were made on thin materials as thin lumber, pencil slats and veneers. These experiments faced problems with the field inhomogeneity and the impossibility to measure parameters during the process. To overcome the problem with inhomogeneity, continuous MW devices and applicators with more sources were designed (Egner and Jagfeld 1964; Barnes 1976; Antti 1999; Hansson and Antti 2003; Seyfarth *et al.* 2003; Leiker *et al.* 2004; Vongpradubchai and Rattanadecho 2009; Prasad 2012). Egner and Jagfeld (1964) used a continuous MW tunnel with a frequency of 2.45 GHz and a maximum output of 6 kW to dry spruce and beech boards. The author found that the MW drying does not affect the wood hygroscopicity. Barnes (1976) developed a prototype for wood drying at a frequency of 915 MHz and an output of 25 kW, by which he dried large pieces of Douglas fir (*Pseudotsuga menziesii*) and hemlock (*Tsuga spp.*). The depth of penetration was higher at this frequency than in the case of 2.45 GHz. The results showed that he was able to dry the material within 5–10 hours (depending on the species) with minimum deterioration of mechanical properties. In 1999, Antti and Perré (1999) published their work dealing with the MW drying of wood. They provided the temperature and MC distribution in the material during the drying process. The measured data showed an opposite MC gradient inside the material in comparison with conventional wood drying and a higher temperature near the surface of the material. Klement (1999) published a study on the distribution of moisture in MW drying of various tree species. However, these data did not show a reduced MC in the central parts of the samples. Zielonka *et al.* (1997) measured the temperature distribution in the material during the drying process and reached similar results as Antti and Perré (1999). In the process of MW drying, it is also possible to use low heating temperatures. For example, Leiker and Adamska (2004), Seyfarth *et al.* (2003), Cividini *et al.* (2003) and Resch (2006) suggested using MW drying in combination with the vacuum, which substantially shortens the drying time. The drying temperature can be low (30–40 °C), thus avoiding thermal degradation of wood and changes in the colour of the material. Besides the combination of MW heating and the vacuum, there are also several others, such as the MW heating in combination with solar drying (Brodie 2007a; Brodie 2008), conventional drying (Lee 2003; Wang 2005), infrared heating (Hanghi and Ghanadzadeh 2006) and sublimation drying (Wang and Shi 2008). Brodie (2008) stated that the combination of solar and MW drying allows for a 17% reduction of the time needed for drying of eucalyptus (*Eucalyptus* sp.) 30 mm thick sawn timber and 33% reduction of the drying time of poplar (*Populus alba* L.) timber of the same thickness. The results showed that in this type of drying, there was a 9% decrease in the wood density due to the formation of internal micro-cracks in the structure. Brodie (2008) explained the quick drying by a higher permeability and faster outflow of moisture from the wood. Lee (2003) tested the combined MW and conventional heating for the drying of the Korean pine (*Pinus koraiensis*). He managed to reduce the drying time from 60 h to 90 min for sawn timber 25 mm thick. Timber 50 mm thick was dried for 190 min instead of the original 400 h. Dedic and Zlatanovic (2001) dried red oak (*Quercus rubra*), which is sensitive to cracks during MW heating. It turned out that in combination with hot air the final quality of the material was higher than in the case of common drying methods. Wang (2005) focused on the drying of the tree species for which there is the risk of internal tensions during the drying process. The emergence of this tension is caused by the high moisture gradient, which is commonly avoided by a long time of drying. However, Wang (2005) used the MW radiation to balance the gradient, which resulted in a considerable reduction of the wood tension.

According to Resch (1968), Barnes (1976) and Vikberg *et al.* (2014), the MW heating at frequency 2.45 GHz is probably not practical for drying thick wooden product due to its small penetration depth in the wet material.

2.3.2 Plasticization

Desh and Dinwoodie (1996) stated that plasticization is used for a temporary change in mechanical properties of the material which facilitates bending or densification of wood. According to Norimoto and Gril (1989), it is possible to use MW heating for plasticization. Hansson (2007) reported that during MW heating the plasticization heat is produced directly in the structure of the material. However, the temperature distribution in the cross-section of the material depends on the moisture profile (Brodie 2007b).

According to Ozarska and Daian (2010), MW bend consists of three phases: 1) MW heating to soften the wood (plasticization), 2) Bending into the desired shape and stabilization of the workpiece, and 3) Accelerated drying using the MW technology. Studhalter (2005) and Studhalter *et al.* (2009) researched MW plasticization with the purpose of bending the wood. In their experiments they identified the distribution and transmission of the temperature throughout the cross-section of material using optical fibers. As a result, the authors provided the MC and temperature profiles of three groups of samples with different input MC (20%, 35%, 80–120%) and found out the MC had significant effect on temperature development. Kuiyan and Jian (2009) conducted the MW plasticization of elm (*Ulmus*), which was subsequently longitudinally compressed and bent. In this way, they achieved the optimal bending factor (minimum radii). Juniper (2008) dealt with numerical simulations. He studied the mechanical loading of wood during MW plasticization and bending. He compared MW models of plasticization and steaming of wood and he attempted to find the optimum parameters for bending and determine MOR in bending. The use of MW heating for drying and stabilisation of bent parts was studied by Harris *et al.* (2007). The aim of their research was to find out whether it is possible to reduce the time required to stabilize the parts using MW heating, without a reduction of mechanical properties. They compared the MW and the conventional heating. The results showed that MW heating is able to dry and stabilize the parts with an initial wood MC of 45 to 50%. However, to maintain the mechanical properties it is necessary to use a mild drying mode (up to several hours). Gašparík and Gaff (2013) reported that the advantages of the MW plasticization are the possible high-capacity operation (continuous process), easy temperature control, simple operation of the device, and simple technology for which no other media (*e.g.* steam) are required. Compared to commonly used processes (steaming, boiling), the MW process of plasticization is much faster and also has lower energy demands (Gašparík and Gaff 2013).

2.3.3 Modification of permeability

Studies of the MW modification revealed a significant increase in wood permeability for liquids and gases (Trajkovic 1994; Torgovnikov and Vinden 2004; Merenda and Holan 2008; Torgovnikov and Vinden 2008; Torgovnikov and Vinden 2009; Vinden *et al.* 2011 and Yu *et al.* 2011). According to Vinden *et al.* (2011) and Yu *et al.* (2011), the principle of MW increase in permeability is based on the origination of water vapour pressure, which is able to delaminate the cell walls. Then micro and macro cracks are formed in the wood structure (depending on the intensity of radiation) and affect other properties of wood, *e.g.,* the volume increases and mechanical properties are reduced (Torgovnikov and Vinden 2009).

An experimental setting of the radiation intensity was discussed by Torgovnikov and Vinden (2008). The result of their work was the division of modifications into three levels: low, medium, and high. The low level offers 1.1 to 1.5 times increased wood permeability. The medium level increases the permeability up to a thousand times, and the high level modifies the wood structure into a porous material (Torgovnikov and Vinden 2008). According to Yu *et al.* (2011) and Torgovnikov and Vinden (2004), the cracks are formed mostly within pith rays, because they have thinner cell walls than the other wood elements. This fact can be detected in the results of Torgovnikov and Vinden (2006), who obtained increased permeability values in the radial direction in a modified material. Dashti *et al.* 2012 also found that the MW radiation at frequency of 2.45 GHz can increase the wood radial permeability. On the contrary, Trajkovic (1994) in his experiment demonstrated an increased permeability in the longitudinal direction. The differences in transverse permeability were negligible. MW modification in relation to an increase in impregnability was studied by Chovanec *et al.* (1994), Kang (1998), Vinden *et al.* (2000), Liu *et al.* (2005), Jiang *et al.* (2006), Zhang and Cai (2006), Treu *et al.* (2008), Torgovnikov and Vinden (2008, 2009, 2010), Yu *et al.* (2011) and Beikircher *et al.* (2012). Treu *et al.* (2008) irradiated samples of spruce (*Picea abies* L.) and Scots pine (*Pinus sylvestris* L.) in a special continuous device with an output of 6 kW and a frequency of 2.45 GHz. The aim of the experiment was to reduce the material density and raise the wood impregnability. The modification was controlled by changes of the speed of the conveyor belt. The results showed an increased impregnability of all groups of samples. As the author pointed out, the low speed of the conveyor causes the creation of large cracks in the wood, therefore he recommended short exposure times of treatment and high output of irradiation. Liu *et al.* (2005) examined the effect of MW radiation on the impregnability increase of European larch (*Larix decidua*) with distilled water. The author found statistically significant differences in the water uptake in the treated samples. A similar experiment was carried out by Jiang *et al.* (2006), who also irradiated the wood of the larch (*Larix decidua*). He achieved at the same results as Liu *et al.* (2005), and added a microscopic analysis. The analysis explained the increase in the permeability by the rupture of the cell wall pits and the formation of micro-cracks in the area of parenchyma cells. Kang (1998) used MW heating to dry six different tree species (*Robinia sp., Alnus sp., Fraxinus sp., Pinus resinosa, Pinus radiata, Tsuga heterophylla*). He used the dried samples to measure the wood permeability and determined its relation to the radiation intensity. After the statistical evaluation, he found that there is a linear relationship between the treatment intensity and permeability within a species. Beikircher *et al.* (2012) modified the permeability of Norway spruce (*Picea abies*) by a mild mode of MW radiation. The results showed that the wood treatment affected the increase in protective agent uptake. Torgovnikov and Vinden (2010) pointed to the commercial use of MW modification of wood and indicated its possible application to increase permeability of timber, logs, railway sleepers, bridge structures, buttstocks, garden timber, chips for pulping and many others.

2.3.4 VOC emissions

Volatile Organic Compounds (VOCs) are substances which, under normal pressure and temperature conditions (20 °C; 101.3 kPa), are easily released into the air. Most of these substances have negative effect on health and the environment (Act No. 86/2002 Coll.). For example, they are released from heat-treated wood (Kamdem *et al.* 2000), coating and impregnation substances, glues, etc. (Kim 2009). Wang *et al.* (2005) examined the VOC emissions in the manufacture of particleboards. The results of their work showed that with a proper drying mode setting and selection of an appropriate input conditions, MW drying releases a smaller amount of VOC than conventional drying. These results were achieved in a mild mode, in which no high temperatures of the material were reached and thus the amount of VOC was low. However, the authors did not deal with the VOC emissions from the final product. On the contrary, Saito *et al.* (2004) used a stronger mode of MW radiation for the maximum VOC (formaldehyde) emission from melamine resin glued plywood. The emission in the environment of the MW device rose significantly. After removing and conditioning of the sample, VOC emission was many times lower than in the case of untreated samples. MW treatment was also applied to new wood-based composites as accelerated fixation of copper-ethanolamine treated wood and as the reducing of formaldehyde emissions (Simon *et al.* 2007).

2.3.5 Acceleration of chemical reactions

Chemical treatment methods effectively protect wood against the effects of the external environment, *e.g.*, light, weathering and wood-destroying insects and fungi (Hill and Jones 1996; Li *et al.* 2000; Chang and Chang 2001; Militz 2002; Rowell 2005; Hill 2006); they also improve dimensional stability in response to changes in the relative humidity (Tarkow *et al.* 1950; Chang and Chang 2002; Homan and Jorissen 2004; Popescu *et al.* 2013). Some chemical modifications require heating for the chemical reaction of the substances with wood (acetylation, DMDHEU, etc.). Commonly, conventional heating is used for this purpose but it can have a negative effect on the quality of the final product as well as the economy of operation (Grelier *et al.* 1997). Research in this area indicated that MW heating designed for chemical reaction of substances with wood is much faster, more economical and well suited for continuous production processes (Grelier *et al.* 1997; Larsson *et al.* 1999; Larsson and Simonson 1999; Sethy *et al.* 2012).

Microwave heating in the process of acetylation

Rowell *et al.* (1987), Nilsson *et al.* (1988), Militz (1991) and Larsson *et al.* (1999) dealt with the modification of wood by acetylation (acetic anhydride). In the process of acetylation, hydroxyl groups (-OH) in the wood cell walls are replaced with acetyl groups which significantly improves the dimensional stability of the wood in relation to the relative humidity (Militz 1991; Rowell 2013). The process of acetylation finishes with a chemical reaction that is activated by heating or a catalyst. Sethy *et al.* (2012) compared the acetylation time and degree of a chemical reaction activated by a catalyst (potassium acetate) and a chemical reaction activated by MW heating. The results of this study showed that the MW reaction was much faster and contained a higher degree of acetylation. Larsson *et al.* (1999) in their work compared the acetylation degree after heating in an oil bath and MW heating. The results showed the same degree of acetylation, for both applied treatments, but in other time modes. MW heating was many times faster. Larsson *et al.* (1999) stated that the MW energy activates the acetylation reaction only by the influence of heating, and it is not a direct reaction of radiation with the substance. Katović *et al.* (2004) compared the activation of the reaction of citric acid and a catalyst SHP (sodium hypophosphite) using MW and conventional heating. The results of the experiment indicated that in both types of heating a proper reaction of the substances occurred. The polarizability of the acetic anhydride (Ac_2O) in the MW field has been extensively studied (Baghurst and Mingos 1992; Larsson *et al.* 1999). Baghurst and Mingos (1992) stated that during MW heating, the temperature rise of the Ac_2O was about two times higher than water.

Microwave heating in the process of butyrylation

Chang and Chang (2003) used MW heating for wood butyrylation. Butyrylation is a modification aimed to increase the dimensional stability and light resistance of wood. Chemical changes of wood were analysed using magnetic resonance and diffuse reflectance. The results manifested that the degree of butyrylization is higher in the central part of the sample than in the surface layer when MW radiation is used. The authors of the study proved that wood has better resistance to photodegradation and better dimensional stability after butyrylation.

Microwave heating in the process of furfurylation

Treu *et al.* (2007) impregnated wood of Scots pine (*Pinus sylvestris* L.) by furfuryl alcohol, which they subsequently irradiated with MWs. The authors in this experiment detected the appropriate process parameters (output, time and initial wood MC) for the ideal degree of an agent fixation. The evaluation consisted in the mass uptake of the agent and the extent of leaching of the agent from wood. The author stated that the use of MW heating slightly increased the fixation of the agent, but on the other hand, the power consumption was higher.

Microwave heating in the process of DMDHEU

Timber treatment by dihydroxy dimethylol ethylene urea (DMDHEU) improves dimensional stability and protects the wood against biological agents. Katović *et al.* (2004) conducted an experiment using fir (*Abies alba* Mill.) and beech (*Fagus silvatica* L.) in which they compared the DMDHEU reaction activated by MW and conventional heating. The results indicated that conventional heating is more efficient for the chemical reaction. The indicator selected was the dimensional stability of soaked samples.

Microwave heating in the process of polycarboxylic acids (PCAs)

PCAs modification improves dimensional stability of wood (Katović et al. 2004). Vukusic *et al.* (2006) modified fir (*Abies alba* Mill.) and beech (*Fagus silvatica* L.) chemically using polycarboxylic acids, specifically the citric acid and 1,2,3,4-butanetetra-carboxylic acid. They used sodium sulfate as a catalyst and activated the reaction by MW heating. The authors stated that the effect of the modification was very intense thanks to this type of heating. The results showed that the chemical treatment improved the dimensional stability as well as the tensile strength of the wood.

Curing of adhesives and coatings

BinHua *et al.* (2012) compared the reaction time of adhesives in the case of the MW heating and the conventional heating. The results of MW heating exhibited extremely fast curing of adhesives. Grelier *et al.* (1997) used the MW heating to cure coating substance with ultraviolet (UV) radiation resistance. The author described that in this method no catalysts had to be used. The authors stated that MW heating is sufficient for the process of curing.

3 MATERIAL AND METHODS

3.1 Material and testing samples

The most common European hardwood and softwood species were used in this study. The European beech *(Fagus sylvatica* L.) (Papers IV, V and VI), European beech containing false heartwood (Papers I and II) and hybrid poplar (*Populus hybrids*) (Paper VI) were chosen from hardwood species and the Norway spruce (*Picea abies* L.) (Paper III) was chosen from softwood species.

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. 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. This wood is used for flooring, furniture, handles, veneer, woodenware, containers, pallets, etc., when treated with preservative agents, beech is suitable also for railway sleepers.

Hybrid poplar (*Populus hybrids*) is a fast-growing species, which can be used in number of applications as production of cheap plywood, boxes, pellets, etc. Because of its white colour and the length of the wood fiber, poplar is used in the production of paper. This wood has a diffuse-ring porous structure with little difference between earlywood and latewood. The wood of the poplar is soft, lightweight, and has a moderately high shrinkage.

Norway spruce (*Picea abies* L.) is the most economically valuable conifer in Europe. This species is known for its resonance, lightweight and springiness. Spruce wood is used for many purposes such as musical instruments, wooden aircraft, paddles, construction work as structural wood, etc.

These wood species were selected for different MW modification techniques and detailed information about the material used in each study and samples preparations are presented in Papers I–VI.

3.2 Modification methods

The experimental investigation of MW modification have been carried out using discontinues or continues MW device. MW modification for improving the permeability and impregnability of the wood (Papers I and II) was carried out using discontinues device and modification was performed by using two treatment modes. Afterwards, the permeability for liquids in the axial direction (Paper I) and substance uptake in transverse directions (Paper II) were measured. Following experiments dealing with wood pre-drying, plasticization, and acceleration of chemical reactions (Papers III–VI) were performed in new MW continuous laboratory device. Differences between particular modification treatments can be found in the power levels and duration of treatment (conveyor speed). Detailed information about the treatment is presented in Papers I–VI.

3.3 Microwave devices

First investigations of MW treatment were performed in discontinues MW device (Figure 3, Table 1), which operates at a frequency of 2.45 GHz with fix power 0.9 kW.

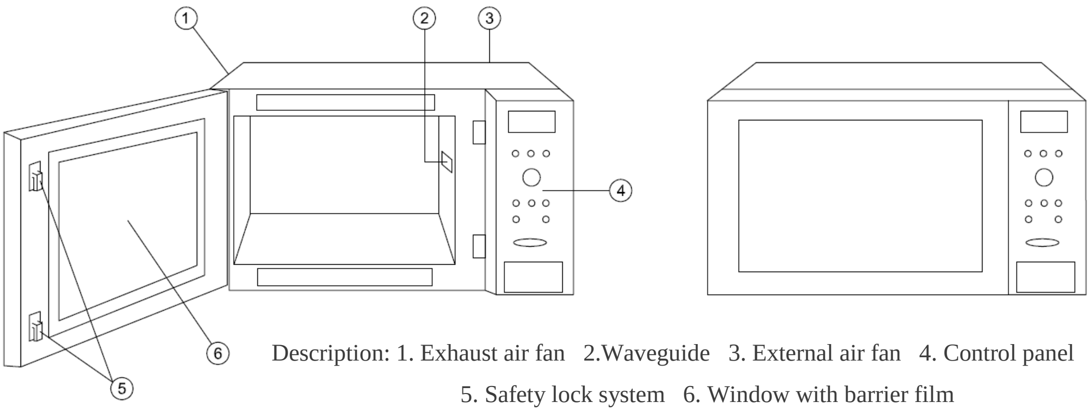

Figure 3 Discontinues laboratory MW device

The internal dimensions of chamber are 515×415×290 mm^3 (width×depth×height). This discontinues MW device helped to found out that MW technology is a potentially useful method to improve the physical and technological properties of wood.

Based on activities in this investigation, research team from Department of Wood science in co-operation with Romill company (CZ) developed a unique continuous laboratory MW device (Figure 4, Table 2), consist of the bearing construction, conveyor, modification chamber, waveguide, and generator depicted in scheme in Figure 5.

Magnetron in devices generator operates at a frequency of 2.45 GHz and disposes adjustable power from 0.6 to 5 kW. The conveyor controlled by the frequency changer can reached the speed of material in range 0.1–1 $m{\cdot}min^{-1}$. To achieve the maximum possible absorption of the radiation by the material, the waveguide is equipped with two electric motors that modify the geometry of the electromagnetic wave entering the chamber.

Figure 4 Continues MW device – basic view of conveyor, generator, modification chamber and waveguide

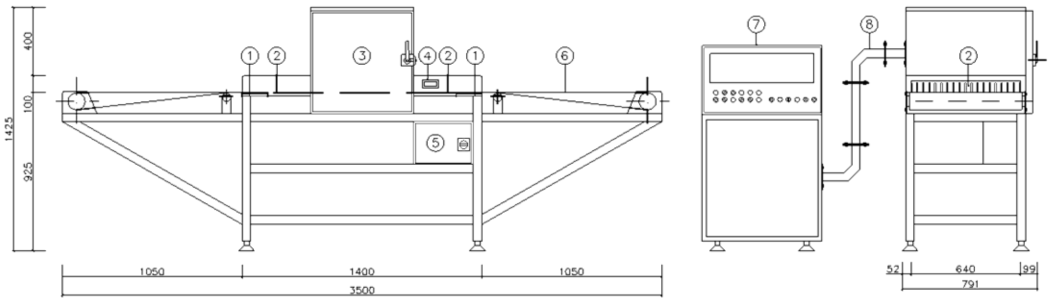

Figure 5 Scheme of continues MW device

Table 1 Discontinues MW device – technical specification

Technical specification	
Power supply	230 V
Frequency	2.45 GHz
Output power	0.9 kW
Cooling of magnetron	air cooling

Table 2 Continuous MW device – technical specification

Technical specification	
Power supply	400V + N + PE, 32A
Frequency	2.45 GHz
Output power	0.6–5 kW
Cooling of electronics	air cooling
Cooling of magnetron	water cooling from an external source (minimal flow rate: 5 litters per minute)

3.4 Testing methods

The MC of the wood samples (Papers I–VI) was determined using the oven-dry method in compliance with EN 13183-1 (2002). The surface temperature (T_s) of the testing samples (Papers III–VI) was measured by a contactless infrared thermometer (Voltcraft IR-380, accuracy ±1.0 °C). The density (ρ) (Papers IV–VI) was determined in compliance with EN 323 (1993) as the ratio of mass to volume of the samples at the oven-dry MC. Permeability (Paper I) was measured experimentally with distilled water using a measuring device designed for testing the axial permeability of fluids through porous materials. The coefficient of specific permeability was calculated using a formula based on Darcy's law. Uptake of substance (Paper II) was calculated using the weight percentage gain (WPG) (Equation 19). The swelling (*S*) (Paper VI) was calculated from dry and equilibrium states of samples under different RH levels using the Equation 27. The anti-swelling efficiency (ASE) represented differences between the swelling of the treated and untreated wood. The ASE was calculated according to the Equation 28 in Paper VI. The microstructure of MW densified and control samples was examined in co-operation with Thünen Institute of Wood Research (Hamburg-Bergedorf, Germany) using scanning electron microscopy (FEI Quanta 250, FEG). Mechanical properties (bending strength and strength in compression parallel to the grain and Brinell hardness) were measured using a universal testing machine Zwick Z050 (Papers I, III, and IV).

PAPER I.

Microwave Radiation Effect on Axial Fluid Permeability in False Heartwood of Beech (*Fagus sylvatica* L.)

Dömény, J., Koiš, V., Dejmal, A. (2014)

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Microwave Radiation Effect on Axial Fluid Permeability in False Heartwood of Beech (*Fagus sylvatica* L.)

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ABSTRACT

This study evaluated the effect of MW radiation on the fluid permeability and compression strength parallel to the grain of beech false heartwood. European beech (*Fagus sylvatica* L.) was selected, and samples of false heartwood with dimensions of 30×20×20 mm^3 were used. The MW treatment was carried out in a laboratory device at a frequency of 2.45 GHz. The testing samples were divided into three groups (untreated, treated at 20s intervals, and treated at 30s intervals). The permeability was measured in the axial direction using distilled water. The coefficient of specific permeability was calculated using Darcy's law. The results showed that the coefficient of specific permeability increased by up to 159% in comparison with untreated samples. The compression strength parallel to the grain decreased by up to 15%.

Key words: European beech; false heartwood; high-frequency energy; microwave treatment; permeability; compression strength

INTRODUCTION

European beech (*Fagus sylvatica* L.) is a species that is heavily used by industry. It provides hard, strong, and resilient wood with good properties for processing, which makes it suitable for a wide range of uses (Pouchanič 2011). *Fagus sylvatica* L. wood density ranges between 620 and 720 kg/m^3 on average, depending on altitude and the specific site (Gryc *et al*. 2008a). The density of the false heartwood is usually higher. Many properties of *Fagus sylvatica* surpass the properties of other common European species. Beech wood also has several disadvantages, which limit its possible use as a material. One of the most significant is that it forms false heartwood, which is seen in practice as a flaw, reducing the quality of material entering the production process and thus also reducing its utilization (Pouchanič 2011). The formation of false heartwood in beech is determined by two main factors: the presence of ripe wood (the central part of the trunk has a lower MC than that of sap wood), and air entering the wood structure. Both of these factors must be fulfilled for false hardwood to form. A wound in the stem or a branch is the primary cause of air entering the tree stem. Oxygen contained in the air causes oxidation and the production of brown, polyphenolic compounds (Koch *et al*. 2001). At the same time, tyloses grow from parenchyma cells through pits between parenchyma cells and vessels and block them. Such tissue allows very little fluid to permeate beyond the wound, and many investigators thus see the false heartwood of beech as a protective tissue that prevents the origination and development of fungal hyphae (Račko and Čunderlík 2010).

MW radiation is used to modify wood to achieve an improvement in its natural drying (Brodie 2004a), plasticization (Makovíny and Zemiar 2004; Yu *et al*. 2011), and wood synthesis with other natural materials, *e.g.*, resins (Torgovnikov and Vinden 2004). Structural changes through wood modification can lead to changes in physical wood properties such as permeability, density, strength, flexibility, heat conductivity, shrinkage and swelling (Torgovnikov and Vinden 2009).

The principle of MW heating is based on the polar molecules (H_2O, -OH, $-CH_2OH$, -CHOH, -COOH) contained in the wood (Hansson and Antti 2003). If the wood is exposed to an electromagnetic field of MWs, molecules that are dipolar in character will begin to move with the same frequency as the electromagnetic field, and the rapid change in field polarity causes vibration and even rotation of molecules, which transforms the energy of MWs into frictional heat (Makovíny 2000; Hansson and Antti 2003; Nasswettrová and Nikl 2011). Water is the best absorber of MW radiation with the frequency 2.45 GHz (Nasswettrová and Nikl 2011). By applying MW radiation to the wood with certain MC, the water in the cell cavity absorbs the energy and boils off quickly, forming high steam pressure; the pressure causes cell microstructure change, resulting in the properties change including the permeability. Moreover, Merenda and Holan (2008), Yu *et al*. (2011) and Vinden *et al*. (2011) state that when steam is created, the cell walls become delaminated and wood permeability thus increases. According to Torgovnikov and Vinden (2004), cell walls of ray cells are the easiest to destroy by water steam pressure during MW heating in which micro-cracks are created in the wood, leading to an increased volume of the wood and a decrease in density. However, the mechanical properties deteriorate. Oloyede and Groombridge (2000) studied the effect of drying Caribbean pine (*Pinus caribaea*) timber using MWs and concluded that MW drying could reduce timber's tensile strength by 60%. The authors hypothesized that peculiar mechanisms involved in MW heating caused this harmful effect on the strength and fracture toughness. Torgovnikov and Vinden (2000 and 2001) treated the wood of *Eucalyptus* by MW radiation. Steam expansion, caused by MWs of high intensity, modified selected hardwoods by increasing wood permeability. The MOE was decreased by 12 to 17% and the volume increased by 13.4%. Torgovnikov and Vinden (2009) performed a MW modification of the wood of Paulownia (*Paulownia fortunei*) at an output of 30kW and a frequency of 0.922 GHz. They concluded that the MW modification caused its high heat loading, which led to a decrease in MOE in the tangential direction from 3400 MPa to 1600 MPa and a decrease in MOR in the tangential direction from 39.3 MPa to 16 MPa. Terziev and Daniel (2013) treated samples of Norway spruce (*Picea abies*) for improved wood permeability. Results showed that the water uptake of the treated samples was doubled compared to the untreated controls, but the MOE decreased with 10% after the treatment.

Beech wood has a relatively low resistance to biodegradation factors, which is usually compensated for by chemical treatments (impregnation by natural oils, waxes, etc.). The determining factor deciding the uptake of the chemical treatment is wood permeability. The measure of permeability is not only wood porosity but also the interconnection of free spaces in the capillary system. Concerning wood, this means the connection of cell lumina by perforated screens-cell wall pits or cross-fields of rays (Požgaj *et al*. 1997). The actual permeability depends on many factors: from the wood element distribution and the size of pits in the cell walls to the anatomical direction, density, moisture, etc.

The goal of this work was to find out if MW radiation will increase the axial permeability of beech false heartwood. Should this hypothesis be confirmed, impregnation and durability of false heartwood in beech would increase. Healthy false heartwood would not have to be excluded and after impregnation; rather it would become an adequate material for the construction and furniture industries.

EXPERIMENTAL

Materials

The testing samples were selected with dimensions of 30×20×20 mm^3 (length×width×height) from a section of European beech (*Fagus sylvatica* L.) containing false heartwood. The size of the testing samples was used with respect for measuring the axial permeability and strength in compression parallel to the grain. The section was chosen without fungi or insect damage. The samples did not contain defects, such as cracks, knots, pits, fiber deflection, or reaction wood. Before MW treatment, the samples were soaked in distilled water for 3 weeks. Before beginning the modification process, the material contained 85% MC on average (standard deviation, SD 8.2%). The testing samples were divided into three groups. Each group (untreated, first treated group, second treated group) contained 70 samples.

Microwave Treatment

The samples were treated in a small laboratory MW device with chamber dimensions 515×415×290 mm^3 (width×depth×height). The device works with a frequency of 2.45 GHz and an output of 900 W. Testing samples were rotated during the treatment process, and MWs came from a linear direction. The treatment process consisted of two intervals with a 30s pause in between for material relaxation (balancing tension and decreasing wood temperature). The experiment included two treatment groups. The first group of samples was exposed to MWs at 20s intervals (20s of treatment, 30s of relaxation, and 20s of treatment); the second group of samples was treated at 30s intervals (30s of treatment, 30s of relaxation, and 30s of treatment). To ensure homogeneity, the samples were treated separately. After MW treatment, the testing samples were soaked to 85% MC (SD 12.4%) and then the permeability was measured.

Measuring of Permeability

Permeability was measured experimentally with distilled water using a measuring device designed for testing the axial permeability of fluids through porous materials (Figure 1). The test was conducted with water and also a sample temperature of 20 °C and a fluid pressure of 0.5 MPa; the diameters of the openings for distilled water flow were 10 mm. The MC of samples in the permeability test was above the saturation point, and the following coefficient of specific permeability was calculated using a formula based on Darcy's law:

$$K = \frac{V \cdot L \cdot \eta}{t \cdot S \cdot \Delta\rho_p} \tag{18}$$

where: K ($m^3 \cdot m^{-1}$) is the coefficient of specific permeability, η (Pa·s) is the dynamic viscosity of water by Čmelík *et al.* (2001), V (m^3) is the volume of fluid flowed through, S (m^2) is the area of the flow, t (s) is the time of flow duration, $\Delta\rho_p$ (Pa) is the difference in pressures at the ends of samples, and L (m) is the length of a sample.

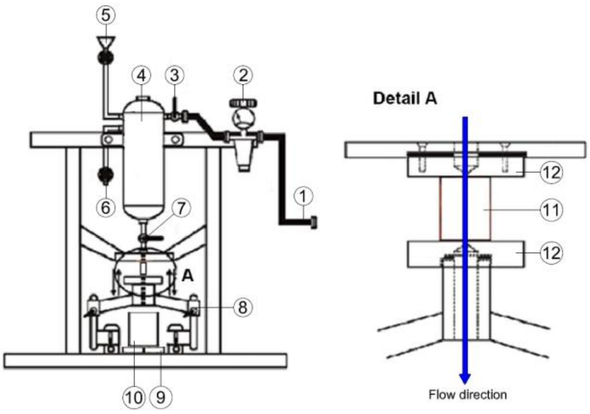

Figure I. 1 Permeability measuring device – scheme and detail A with clamping of the testing sample (Nasswettrová 2011)

Label key: 1) Supply of pressured air, 2) Pressure regulator with manometer, 3) Ball valve (air), 4) Pressure tank, 5) Fill valve, 6) Safety valve, 7) Ball valve (distillate water), 8) Eccentric support for clamping the sample, 9) Scale, 10) Beaker, 11) Testing sample, 12) Washers with a hole (diameter 10 mm)

Measurement of Mechanical Properties

After MW treatment and permeability tests, the samples were kiln dried to 0% MC following the European standard EN 13183-1 (2002). Subsequently, the wood strength in compression parallel to the grain was measured using a universal testing device.

RESULTS AND DISCUSSION

Permeability

The samples treated by MWs showed higher permeability than the untreated samples. Figure 2 depicts the calculated coefficients of specific permeability. The mean permeability of untreated beech false heartwood was $4.72 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$ (SD $8.8 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$). The samples exposed at 20s intervals showed an increase in the mean permeability value of 28% (to $6.04 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$, SD $6.81 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$) compared to the untreated samples. The samples exposed at 30s intervals with a mean coefficient of $12.2 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$ (SD $19.1 \cdot 10^{-13}$ $m^3 \cdot m^{-1}$) exhibited a higher increase in permeability, representing a 159% increase in mean permeability. This phenomenon explains the findings of Torgovnikov and Vinden (2009): the longer the wood is exposed in MW radiation, the more the structure of wood micro-cracks are developed, which increases the permeability.

Although the standard deviations showed high variability, statistically significant differences could be found between untreated samples and treated samples at 30s intervals, as well as between both treated groups (Tukey's test). The differences can be explained by the different treatment time and thus a different mode of heat stress.

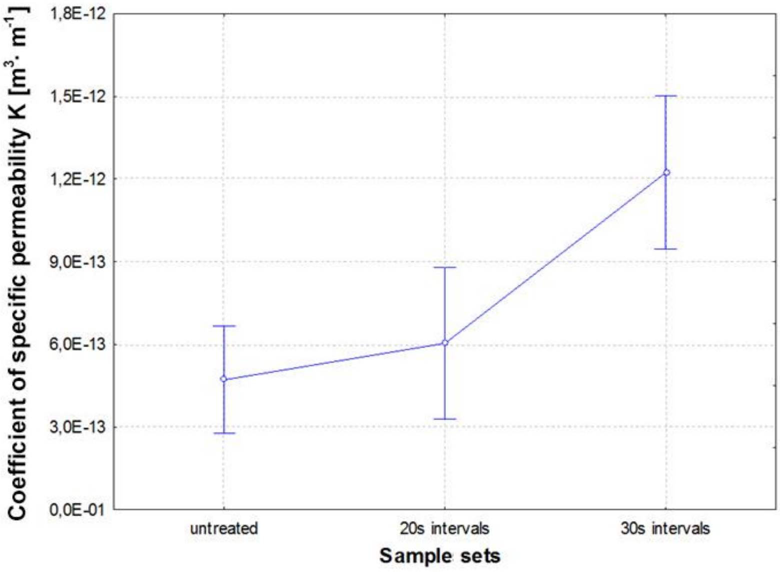

Figure I. 2 Coefficients of specific permeability of the different sample sets

The observed data cannot be compared with literature because no scientific papers were found dealing with the issue of permeability of beech false heartwood. However, the presented data can be compared with studies that discuss the permeability of *Fagus sylvatica* L. wood without false heartwood. The conclusion seems to be feasible if common values of permeability can be reached by MW. Several authors have observed the permeability of beech wood, as listed in Table 1. It is apparent that permeability is highly variable. High variability in the beech wood may be related to differences in the number of vessel per mm^2 (80 to 130) and their different radius (15 to 50 m·10^{-6}) (Požgaj *et al.* 1997). Different permeability of sapwood and heartwood may be attributed to differences in chemical composition and anatomical structure. Microscopic studies by Gholamiyan and Tarmian (2010) revealed that the presence of tyloses in the vessels as a result of false heart wood formation is the main factor for the decreased axial permeability.

Table I. 1 Coefficients of specific permeability of beech according to various authors

Author	Permeability [m^3·m^{-1}]
Hudec (2002)	2.91·10^{-11}
Kurjatko *et al.* (2002)	1.39·10^{-11}
Čunderlík and Hudec (2002)	1.3·10^{-11}
Babiak and Kúdela (1993)	4.9·10^{-12}
Požgaj *et al.* (1997)	8.51·10^{-12}
Kúdela (1999)	10·10^{-12}
Hudec and Danihelová (1992)	7.56·10^{-12}

Similarities can be found between the permeability of samples treated at 30s intervals and the results of permeability in normal beech without the false heart wood presented by Babiak and Kúdela (1993).

Mechanical Properties

Figure 3 shows the mean values of strength in compression parallel to the grain in the false heartwood of beech. The mean value of strength of the untreated samples was 102.95 MPa (SD 9.83 MPa). The samples treated at 20s intervals reached a mean value of 94.41 MPa (SD 8.97 MPa), and those treated at 30s intervals had a mean value of 89.22 MPa (SD 10.48 MPa). The wood strength decreased by 9% in the first group; there was a 15% reduction of wood strength in the second group, compared with the untreated samples. Samples treated in 30s intervals achieved much better permeability with an insignificant decrease in strength compared with samples treated in 20s intervals.

Statistically significant differences were found between untreated samples and both treated groups (Tukey's test). Measurements of mechanical properties showed that the treatment did not cause a large decrease in strength. The values suggested that the treated material would be acceptable for further use in practice. Moreover, there was better impregnation capacity, which could substantially improve the mechanical properties in some cases.

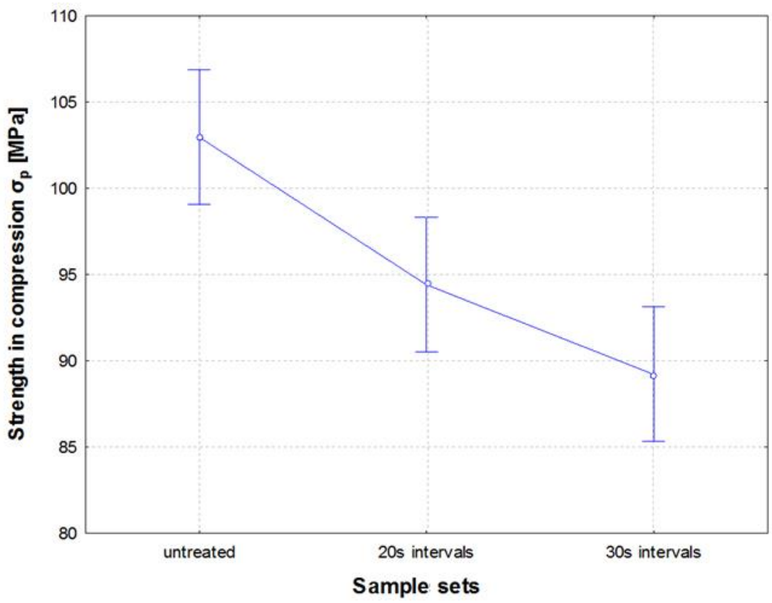

Figure I. 3 Compression strength parallel to the grain

Figure 4 illustrates the correlation between permeability and strength in compression parallel to the grain. The correlation coefficient was very low (r = -0.53). It seems that with increasing permeability due to MW treatment, the strength in compression parallel to the grain decreased. Micro-cracks, which tend to increase the wood permeability, occurred during the MW treatment. With longer treatment, there were increases in the number and size of the micro-cracks. Therefore the mechanical properties such as strength in compression were decreased. This influence on micro-cracks was demonstrated by Torgovnikov and Vinden (2009).

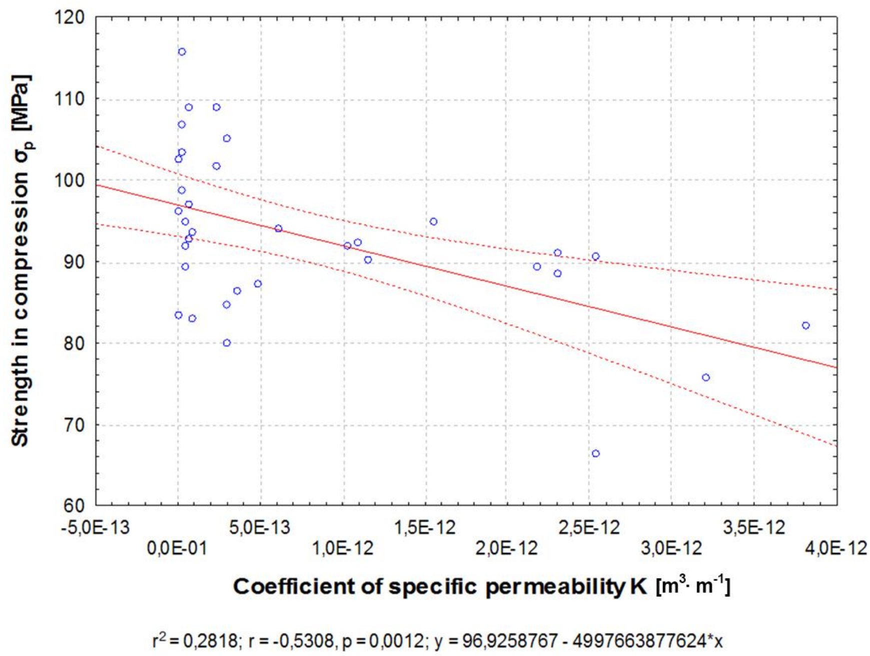

Figure I. 4 Compression strength in dependence of wood specific permeability

CONCLUSIONS

1. The aim of this paper was to determine if MW radiation influences the axial permeability by distilled water and the compression strength parallel to the grain of beech false heartwood. MW radiation increased the mean specific permeability at group 20s intervals of treatment by 28% (statistically insignificant) and at group 30s intervals of treatment by 159% (statistically significant) compared to the untreated samples. The difference between the two treated groups (20s intervals of treatment and 30s interval of treatment) was statistically significant.

2. The mean compression strength parallel to the grain decreased at group 20s intervals treated up to 9% (statistically significant) and at group 30s intervals up to 15% (statistically significant) compared to the untreated samples. There were no statistically significant differences between the two treated groups (20s intervals of treatment and 30s interval of treatment).

PAPER II.

Impregnability of European Beech False Heart Wood after Microwave Treatment

Dömény, J., Koiš, V., Pařil, P. (2013)

Pro Ligno 9(4): 190–194.

Impregnability of European Beech False Heart Wood after Microwave Treatment

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ABSTRACT

The purpose of this study was to experimentally evaluate the MW radiation effect on weight percentage gain (WPG). The species European beech (*Fagus sylvatica* L.) was selected and testing samples from false heartwood with dimensions of 20×20×30 mm^3 were used. The MW treatment was carried out on laboratory device at a frequency of 2.45 GHz. Afterwards the oil impregnation in transverse directions was performed. Results were compared with the reference samples (untreated). The samples exposed at 20s intervals (20s treatment, 30s relaxation and 20s treatment) shows improvement of impregnability (WPG 33.84%), which is with agreement of authors hypothesis. The WPG at 30s intervals of exposure (30s treatment, 30s relaxation and 30s treatment) decreased to 26.59%. Based on results, the future work dealing with time influence of exposure in MW treatment is needed.

Key words: European beech; *Fagus sylvatica* L.; false heartwood; impregnability; microwave modification

INTRODUCTION

Beech is the most spread broadleaf species in forests of the Czech Republic. Its wood is a traditional material for wood-processing industry, although potential of use is not fully utilized. One of the most significant disadvantage is that it forms false heartwood, which is in the practise seen as a flaw reducing the quality of material entering the production process and thus also its utilization (Pouchanič 2011).

The formation of false heartwood in the beech is determined by two main factors: presence of ripe wood and air entering the wood structure. An absence of one of them prevents false heartwood from forming. A wound of the stem or a branch is a primary cause of air entering the tree stem. Oxygen contained in the air causes oxidation of soluble hydrocarbons and starch in living or partially dead parenchyma cells, while brown coloured polyphenolic compounds arise and enter the neighbouring tissues colouring them (Koch *et al.* 2001). At the same time, tyloses grow from parenchyma cells through pits between parenchyma cells and vessels and block them (Račko and Čunderlík 2010). The beech false heart wood is characterized by low durability and impregnability, which are important wood properties for exterior use. Species with low durability are usually improved by impregnation (Liu *et al.* 2005). However, the anatomical structure of few species is due presence of several compounds in the cell walls characterized by low permeability (Torgovnikov and Vinden 2009). Vinden *et al.* (2000) mentioned that pine heart wood is almost unimpregnable. Similar results were obtained by Walker *et al.* (1993), which states that the heartwood cells are darker coloured because they contain extractable substances. These substances penetrate through the cell wall and lumen which leads to lower permeability of heart wood than sapwood. MW treatment leads to increase of permeability of heart wood and improve impregnability of substances into the wood structure (Vinden *et al.* 2000). Bao and Lu (1992) published study, where testing samples with average MC 24.5% were treated by using 5.7 kW and 9 kW of MW energy. The experiments were done in two stages (20 or 30s intervals). Between stages was 25–30s lag for relaxation of material. Afterwards, the samples were impregnated. The higher WPG was observed in the samples treated by using 5 kW in first stage and 9 kW in second stage. It is believed that by using lower power during first stage occurs gradual plasticization of resin. In the next stage (with higher power) is the resin by steam and water forced out of the wood.

Aim of this paper was to find out the influence of MW treatment on impregnability of false beech heartwood. The increase of permeability as well as impregnability by MW radiation should be confirmed.

MATERIAL AND METHOD

Preparation of samples

The testing samples with dimensions 20×20×30 mm^3 (R×T×L) selected from a section of beech (*Fagus sylvatica* L.) containing false heartwood were used. The section was without any indication of fungi or insect damage. The samples did not contain defects, such as cracks, knots, pith, fibre deflection, or reaction wood. Before MW treatment the samples were soaked in distilled water for 2 weeks. The material entering the process of modification contained 90% of MC on average. Each group (untreated, first treated group, second treated group) contained 50 samples.

Microwave treatment

The samples were treated in a laboratory MW device at a frequency of 2.45 GHz and output of 900 W. The treatment process consisted of two intervals with a 30s pause in between for material relaxation (balancing of tension and decreasing of wood temperature). The experiment included two groups of treatment. The first group of samples was exposed to MWs at 20s intervals (*20s treatment, 30s relaxation and 20s treatment*), the second group of samples was treated at 30s intervals (*30s treatment, 30s relaxation and 30s treatment*). To ensure homogeneity, the samples were treated separately.

Coating of cross sections

The samples were after MW treatment dried to 0% MC in an oven set at 103 ±2 °C for 24 hours as mentioned in EN 13183-1 (2002). To prevention that oil will be impregnated just from radial and tangential surfaces were cross section coated by urea-formaldehyde glue (purpose of the experiment was to find impregnability of MW treated wood just from transverse directions). Kiln at 70°C was used to curing of glue.

Impregnation

Impregnation was carried out on laboratory vacuum-pressure impregnation device JHP1-0072 (Figure 1). Initial MC of testing samples was 0%. Impregnation was performed by using impregnating oil Naturol (combination of hemp, soybean and linseed oil), which is suitable for wood protected against weather conditions. Impregnation was done in vacuum, by using pressure 10 kPa for 120 min.

Figure II. 1 Vacuum-pressure impregnation equipment: JHP 1-0072

Uptake of substance

Uptake of substance (oil) was calculated using the WPG according to the equation:

$$WPG = \frac{m_m - m_n}{m_n} \cdot 100 \ [\%] \tag{19}$$

where: WPG (%) is the weight percent gain, m_m (g) is weight of impregnated sample and m_n (g) is the weight of unimpregnated sample.

RESULTS AND DISCUSSION

Changes in impregnability of MW treated beech false heartwood were observed. Table 1 shows the calculated WPG for all testing groups. The WPG of samples exposed at 20s intervals show an increase of impregnability in comparison with reference group. The average WPG was 29.37% for the reference group and 33.84% for treated group. The lower penetration uptake in comparison with references occurred in testing samples which were treated at 30s intervals. The average WPG of this treated group was 26.59%. Increase of WPG for samples treated at 20s intervals can be explained by generating steam in structure of wood. Internal steam pressure had effect on the pit membranes in cell walls, tyloses in vessels, and the weak ray cells, which leads to rupture wood pathways. This had a positive effect on the wood permeability and impregnability. It is believed that during MW treatment the heartwood extractives (terpenes, phenolic compounds etc.) are plasticized in addition to lignin plasticization. Those extractives could be by using longer MW exposure forced from cell walls to lumens and intercellular spaces. Due that occurs lower permeability as well as impregnability.

Table II. 1 Results of weight percent gain (WPG)

Sample groups	Average [%]
Reference	29.37
20s intervals	33.84
30s intervals	26.59

Authors of paper expected increase of impregnability of treated wood; this hypothesis was in short treated group confirmed. Decrease of wood impregnability by using longer exposure (as can be seen in 30/30s group) should be studied in next steps of research. The microscopic images could be useful to prove or disprove authors theory about the leaching of heartwood extractives to lumens and intercellular spaces as a reason to decrease of WPG.

New microwave device

This study fallows the research of MW treatment as a potentially useful method to improve the physical, construction and technological properties of wood. Based on recent activities in this topic, was on Department of Wood Science established a group dealing with the MW modification. In co-operation with Romill company (CZ) was developed unique continuous MW device (Figure 2) with power range from 0.6 to 5 kW. In these days the testing experiments are in progress.

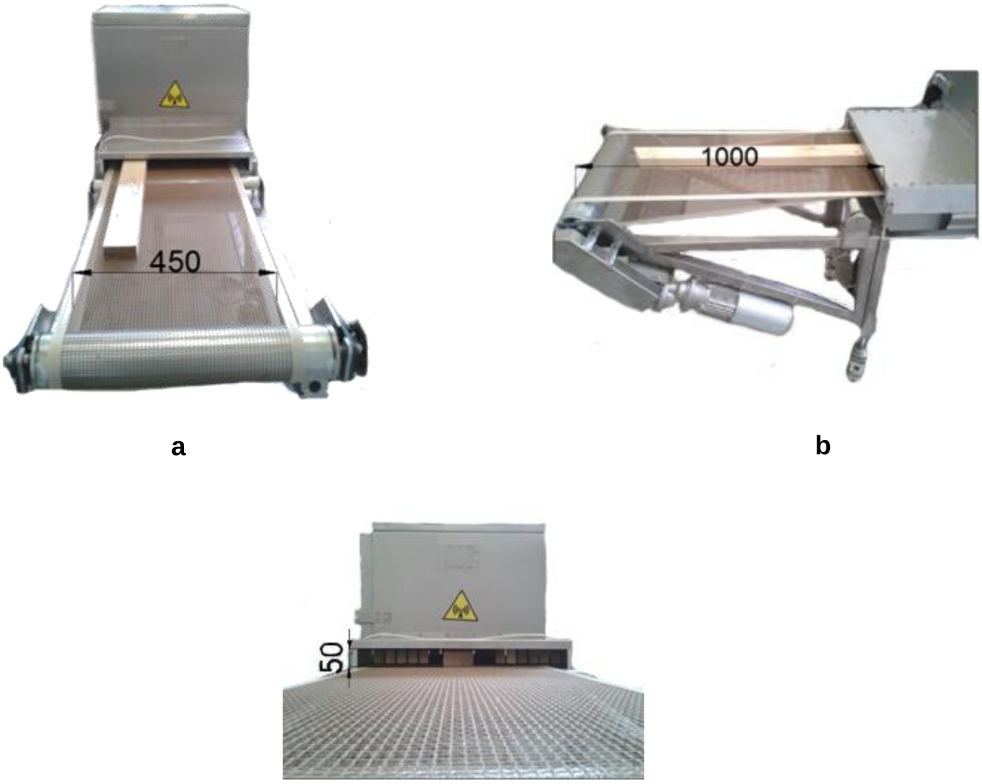

Figure II. 2 New MW device: **a**) front view **b**) side view **c**) front detail

CONCLUSIONS

The purpose of this study was to evaluate the influence of MW treatment on impregnability of beech false heart wood. The MW treatment was carried out on laboratory device at a frequency of 2.45 GHz and output of 900 W. The samples were divided to two groups and MW treated at different time intervals of exposure (20s and 30s). Afterwards all samples were impregnated and the WPG test was used to assess of impregnability. Reference group shows the average WPG 29.37%. The samples exposed at 20s intervals show the average WPG 33.84% which is with agreement of authors hypothesis, based on literature review (higher WPG after MW treatment). The WPG of the samples exposed at 30s intervals decreased on average 26.59%. Based on these results the further experimental work needs to be done to study the effects of time exposure on impregnability of wood. Results provide a better understanding of MW treatment, which probably contributes to a more controlled use of MW treated wood in service conditions.

PAPER III.

Microwave Device for Continuous Modification of Wood

Koiš, V., Dömény, J., Tippner, J. (2014)

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Microwave Device for Continuous Modification of Wood

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ABSTRACT

The aims of this study were to introduce a new laboratory MW device developed for the modification of wood properties and to examine the effect of MW radiation on moisture loss, surface temperature, and mechanical properties (the static modulus of elasticity – MOE, and the modulus of rupture – MOR) of Norway spruce (*Picea abies*). The device was developed for a continuous modification process. The MW generator works at a frequency of 2.45 GHz, and the adjusted output ranges from 0.6 to 5 kW. The experiment was based on four different modes of MW modification, each of them with a varied generator output and conveyor speed. Regarding mechanical properties, the results showed that a feasible output for the MW modification of the samples was up to 3 kW, with a conveyor speed of around 0.4 m/min. The greatest moisture loss, approximately 40%, was found in the group treated at 5 kW and 0.2 m/min. The highest surface temperature, 87 °C, was measured in the group treated at 5 kW and 0.4 m/min after the second passage through the modification chamber.

Key words: device; microwave; drying; high-frequency energy; modification; wood

INTRODUCTION

MW modification of wood has been used in experiments for drying (Hansson and Antti 2003), pre-drying (Awoyemi 2004; Beikircher *et al.* 2012; Harris *et al.* 2008), better fixation of impregnation agents in the wood structure (Yu *et al.* 2009), better permeability and impregnability (Torgovnikov and Vinden 2009), and to achieve plasticization (Studhalter *et al.* 2009).

A laboratory MW device has been introduced (Brodie 2004b, 2007a; Dömény *et al.* 2014b; Hansson and Antti 2003, 2006; Hansson *et al.* 2005; Harris *et al.* 2011; Sethy *et al.* 2012; Sugiyanto *et al.* 2010; Zhao *et al.* 1998). A device for industrial applications has also been presented (Torgovnikov and Vinden 2004;

Torgovnikov and Vinden 2009; Vinden *et al.* 2011). The drawback indicated by Manickavasagan *et al.* (2006), Sebera *et al.* (2012), and Nasswettrová *et al*. (2013) is the non-homogeneity of the MW field. This negative feature causes an uneven cross-sectional distribution of material drying. The non-homogeneity of the MW field can be eliminated through a continuous modification (Torgovnikov and Vinden 2009), adding a special homogenizing element into the application chamber (Nasswettrová *et al*. 2013), or increasing the number of waveguides in the application chamber (Sebera *et al.* 2012).

Oloyede and Groombridge (2000) concluded that the tension inside wood decreases by up to 60% when dried by MW energy as compared to conventional drying. Hansson and Antti (2006) investigated the effect of the drying methods (MW and convectional drying) and temperature (60 to 110 °C) on wood hardness. Provided the drying process is controlled, they concluded that variables such as anatomical direction, anatomical structure, and wood density have more effect on hardness than the drying method or the temperature. Antti (1995) presented the results of MW dried samples of the pine and the spruce at 8% moisture content (MC). The process was controlled based on the internal temperature (not exceeding 140 °C), internal vapour pressure, and moisture vapourisation velocity. The author concluded that the moisture loss at moisture contents over the fibre saturation point was 0.20 to 0.45% *per* min, and that below the fibre saturation point was 0.10 to 0.20% *per* min. The study showed that spruce samples were dried 1.6 times faster than pine samples. No conditioning of the wood was necessary since the wood was free of stresses. Brodie (2008) stated that the dielectric properties of most materials depend on temperature, frequency, and MC. According to Torgovnikov (1993), wood has dielectric properties due to the water it contains and the content of hydroxyl (-OH) and methylene (-CH_2OH) groups. These are mostly contained in the cellulose amorphous part. The dielectric properties of wood vary in the three basic directions, *i.e.* radial, tangential, and longitudinal (R, T, L), and are characterised by two main parameters: the dielectric loss tangent (tan δ) and the relative permittivity (ε'). The dielectric loss tangent represents the radiated energy that turns into heat and is absorbed by the material under the influence of the electric field (Torgovnikov 1993). The relative permittivity is the ratio between the capacity (C) of a capacitor filled with the dielectric and the capacity (C_0) of the same capacitor filled with a vacuum. According to Brodie (2008), the electromagnetic wave transmitted through a dielectric material will manifest a dampening and delay in signal when compared to the same wave passing through a vacuum.

Device Parameters

The research team at the Department of Wood Science, Mendel University in Brno, developed a device with continuous operation to further understand the effects of wood MW modification. The team cooperated with the Romill Company, which has extensive experience regarding research and development due to the high number of MW applications for various industrial fields in Central Europe they have provided.

The device consists of the bearing construction, conveyor, modification chamber, waveguide and generator. The dimensions are 3400×500×1425 mm^3 (length×width×height). The conveyor velocity is controlled by the frequency changer. The dimensions of the modification chamber are 600×600×600 mm^3. The maximum possible dimensions of the treated material are 3000×450×50 mm^3. The center of the device is the MW generator, containing a magnetron with an output adjustable from 0.6 to 5 kW. To achieve the maximum possible absorption of the output radiation by the material, the waveguide is equipped with two electric motors that modify the geometry of the electromagnetic wave entering the chamber. The device is equipped with two inserts from attenuation ceramics to prevent leaks of the unabsorbed output from the chamber.

The entire construction of the device and the modification chamber were developed as a unique prototype. Therefore, one of the aims of the study is to examine the effect of MW radiation on moisture loss, surface temperature, and mechanical properties of the wood of Norway spruce (*Picea abies*) using this device.

EXPERIMENTAL

Sample Preparation

Samples with dimensions of 40×110×500 mm^3 (radial×tangential×longitudinal) were made from Norway spruce (*Picea abies*) wood. The materials used were free of defects such as cracks, pith, fibre deflection, reaction wood, fungi, and insects. Before MW drying, the samples were soaked in distilled water for five weeks. The material entering the modification process had on average 77% MC (Table 1).

Microwave Modification

The samples tested were divided into four groups, based on the mode of MW modification. The groups differed by the different radiation outputs and the speed of movement through the modification chamber, *i.e.* conveyor speed. The first group was treated with an output of 3 kW at a conveyor speed of 0.4 m/min in two cycles (2 passages through the modification chamber). The second group was exposed to an output of 5 kW with the same speed in two cycles. The third and fourth groups were only treated by one cycle, at outputs of 3 kW and 5 kW, respectively, and a conveyor speed of 0.2 m/min (Figure 2). The laboratory was conditioned at 20 °C and 40% RH.

Moisture Content

The samples were weighed before entering the device and after coming out at each cycle. Then, the samples were dried to 0% MC in a conventional laboratory drying chamber. The MC was calculated by the oven-dry method in compliance with EN 13183-1 (2002).

Temperature

The temperature of the samples was measured using a contactless infrared thermometer (IR-380, Voltcraft) at three points (front, centre, and rear) before and after the modification (Figure 1). These values were used to calculate the arithmetic mean.

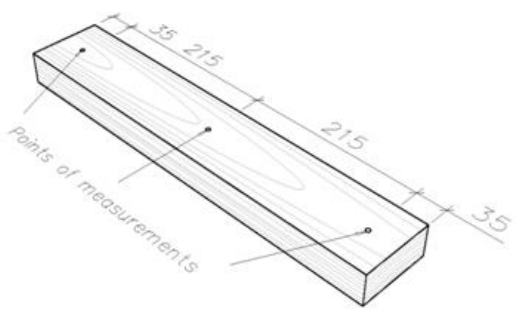

Figure III. 1 Temperature measurement points

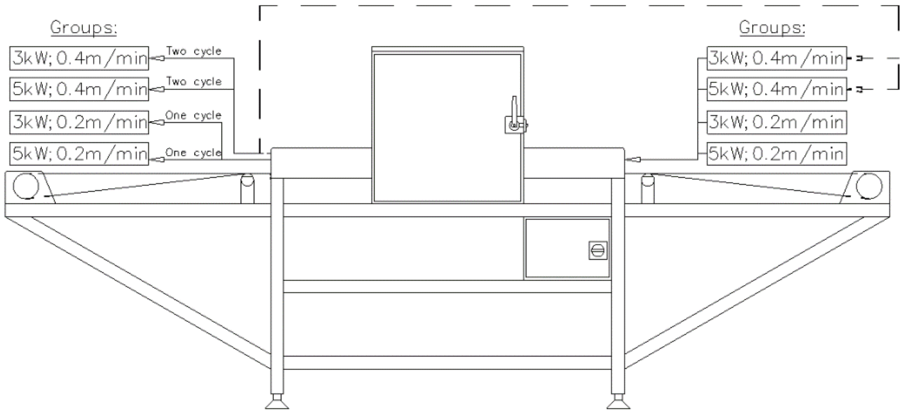

Figure III. 2 Scheme of tested procedures (groups and cycles)

Mechanical Properties

After the MW modification and the measuring of temperature and MC, the samples were cut (Figure 3) into bending samples with dimensions of 20×20×300 mm^3 (R×T×L). The modulus of elasticity (MOE) and the modulus of rupture (MOR) in static bending perpendicular to the grain in the radial direction (Figure 4) were measured in at 12% MC.

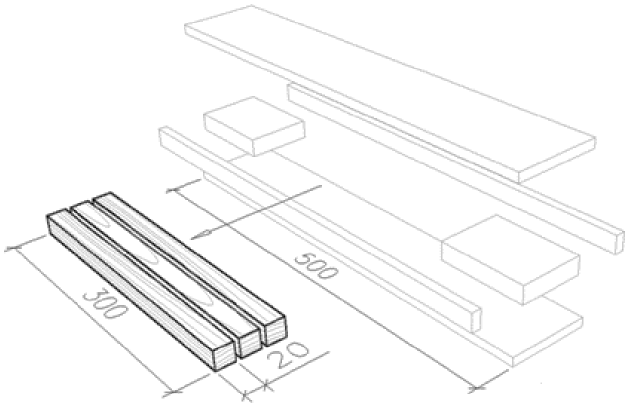

Figure III. 3 Cutting of a sample for the bending measurement

The span of supports was 240 mm, the radius of supports and the forcing head was 15 mm (Figure 4). The value of MOR was calculated from the maximum loading force, as given in equation:

$$MOR = \frac{3 \cdot F_{\max} \cdot l}{2 \cdot b \cdot h^2} \tag{20}$$

where: F_{max} (N) is the maximum loading force, l (mm) is the span of supports, b mm) is the width of a cross-section of the sample, and h (mm) is the thickness of the sample (height of a cross-section).

The calculation of MOE was based on forces measured at 10% and 40% of the maximum loading force (force of destruction) and the corresponding deflections of a bended beam measured by an extensometer. The MOE was calculated from equation:

$$MOE = \frac{(F_{40\%} - F_{10\%}) \cdot l^3}{4 \cdot b \cdot h^3 \cdot (u_{40\%} - u_{10\%})} \qquad (21)$$

where l (mm) is the span of supports, $F_{40\%}$ and $F_{10\%}$ (N) are forces at 40% and 10% level of the maximum force F_{max}, respectively, b (mm) is the width of the sample cross-section, h (mm) is the thickness of the sample (height of a cross-section), and $u_{40\%}$ and $u_{10\%}$ are deflections at forces $F_{40\%}$ and $F_{10\%}$, respectively.

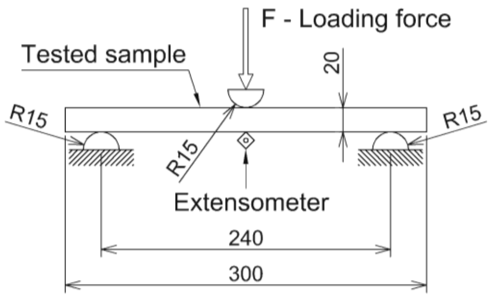

Figure III. 4 Measuring modulus of elasticity and modulus of rupture

Processing of Results

The results were processed in STATISTICA. They were evaluated using one-factor analysis of variance (ANOVA), complemented with Tukey's HSD test.

RESULTS AND DISCUSSION

Moisture Content

Table 1 shows the statistical analysis of the MC data for all MW modification modes.

Table III. 1 Moisture content

Moisture content						
Cycle	Number of samples	Average (%)	Median (%)	Minimum (%)	Maximum (%)	Variability (%)
Untreated (3 kW; 0.4 m/min)	6	77	77	74	82	4
1st 3 kW; 0.4 m/min	6	73	73	71	77	3
2nd 3 kW; 0.4 m/min	6	64	64	58	69	6
Untreated (5 kW; 0.4 m/min)	6	79	78	69	90	9
1st 5 kW; 0.4 m/min	6	68	68	59	78	9
2nd 5 kW; 0.4 m/min	6	45	46	38	53	12
Untreated (3 kW; 0.2 m/min)	5	76	75	72	82	5
1st 3 kW; 0.2 m/min	5	57	56	51	62	8
Untreated (5 kW; 0.2 m/min)	5	77	80	69	82	7
1st 5 kW; 0.2 m/min	5	37	36	33	43	10

Figure 5 shows the moisture loss of the samples (wood). The MW modification mode with an output of 3 kW and a conveyor speed of 0.4 m/min reduced the wood MC by 4% after the first passage through the modification chamber, which is 10 times more than that published by Antti (1995). She concluded that the loss of moisture over the fibre saturation point was 0.20 to 0.45% *per* min. After the second passage, the wood MC was up to 13% lower than the initial MC. The doubled moisture loss after the second passage was caused by the increased material temperature (Table 2 and Figure 7). The MW modification mode with an output of 5 kW and a conveyor speed of 0.4 m/min caused an 11% MC decrease at the first passage through the chamber and a 34% MC decrease at the second passage, when compared with the initial wood MC. The MW modification modes with a conveyor speed of 0.2 m/min consisted of only one passage through the modification chamber. The output of 3 kW resulted in a 19% MC decrease; the output of 5 kW decreased the MC by up to 40%. Figure 6 displays the dependence of MC on the speed of the conveyor and MW power, and Equation 22 describes this relationship:

$$MC = 76.9899-(4.0197\cdot POW)-(5.2876\cdot CS)+(12.6141\cdot POW^{2})+(7.9003\cdot POW\cdot CS)-(0.8603\cdot CS^{2}) \quad (22)$$

where: *MC* is moisture content, *POW* is MW power, and *CS* is conveyor speed.

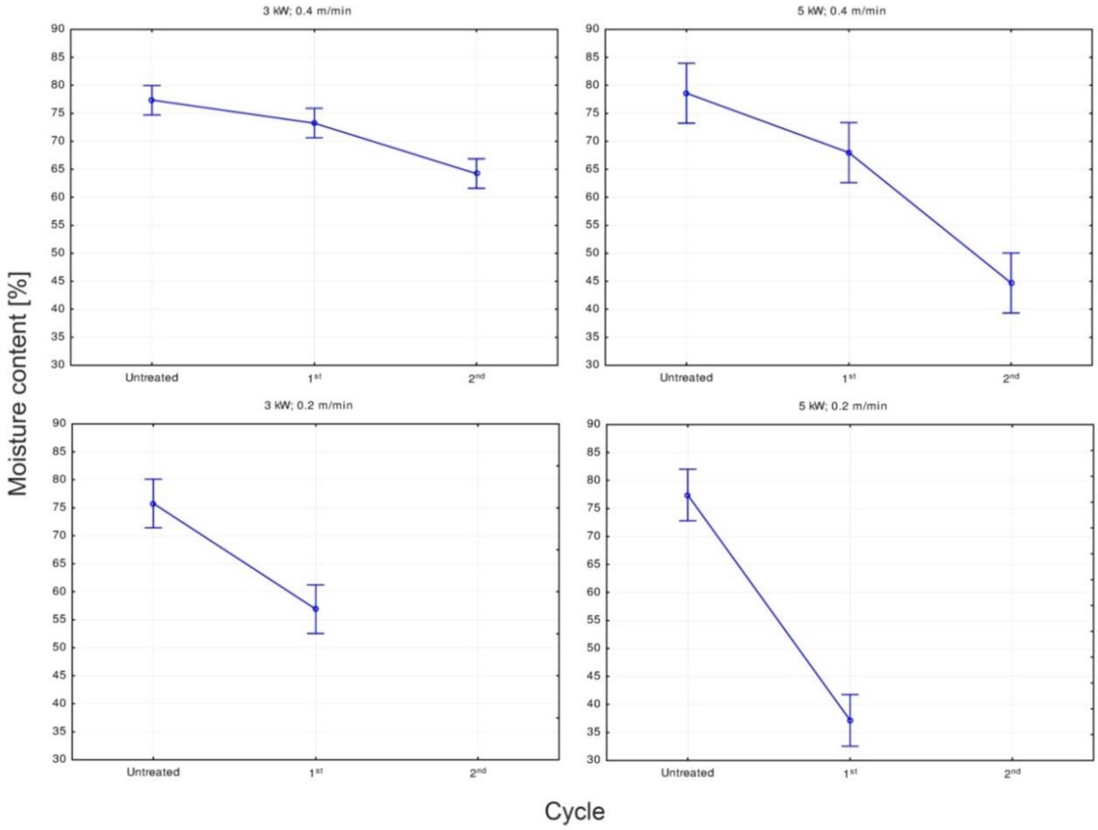

Figure III. 5 Moisture content with different modification cycles

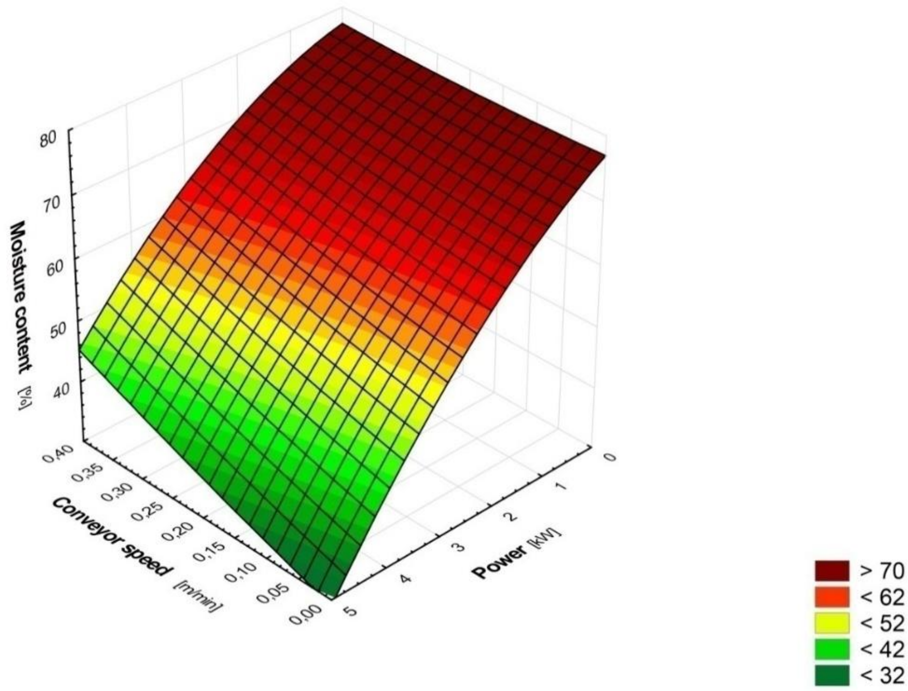

Figure III. 6 Influence of MW power and conveyor speed on moisture content

The only statistically insignificant MC reduction, according to Tukey's HSD test, was found for the first passage through the modification chamber at an output of 3 kW and a speed of 0.4 m/min. The decrease in the MC of the samples was statistically significant in the case of all the other modes (2^{nd} 3 kW, 0.4 m/min; 5 kW, 0.4 m/min; 3 kW, 0.2 m/min; 5 kW, 0.2 m/min).

Surface Temperature

The surface temperature of all the wood samples entering the MW modification was 16 °C. Table 2 presents the surface temperatures of the samples after they came out of the modification chamber. Figure 8 displays the dependence of surface temperature on the conveyor speed and MW power, and Equation 23 describes this relationship,

$$TEMP = 16.052+(22.4409 \cdot POW)+(20.7302 \cdot CS)-(1.7989 \cdot POW^2)-(7.1643 \cdot POW \cdot CS)- (65.529 \cdot CS^2) \quad (23)$$

where: *TEMP* is surface temperature, *POW* is MW power, and *CS* is conveyor speed.

The temperature of the wood surface after the first passage through the chamber at an output of 3 kW and a speed of 0.4 m/min increased by 36 °C when compared with the initial temperature. The second passage increased the temperature by another 23 °C, which was an increase of 59 °C when compared to the initial temperature. The MW modification with an output of 3 kW and speed of 0.2 m/min increased the temperature by 53 °C. The MW modification with an output of 5 kW and a speed of 0.2 m/min increased the temperature by 59 °C when compared with the initial temperature. In the case of a speed of 0.4 m/min and an output of 5 kW, the temperature increased by 54 °C after the first passage and another 9 °C after the second passage of the samples through the modification chamber, a difference of 63 °C from the initial temperature. The smaller increase in temperature after the second passage is caused by the lower content of water in the wood (Table 1, Figure 5) and thus also a lower absorption of MW radiation. The absorption of MW radiation decreases when the temperature of the material increases because water molecules oscillate by themselves due to their high enthalpy. Moreover, individual molecules are further from each other with an increased temperature and thus their collisions, influencing the friction of molecules and heating up of the material, do not occur often. Based on Tukey's HSD test, all modes and cycles of the MW modification were statistically significant from the perspective of the temperature increase.

Table III. 2 Sample surface temperatures

Surface Temperatures						
Cycle	Number of samples	Average (°C)	Median (°C)	Minimum (°C)	Maximum (°C)	Variability (%)
1st 3 kW; 0.4 m/min	6	52	50	50	56	5
2nd 3 kW; 0.4 m/min	6	75	77	69	80	7
1st 5 kW; 0.4 m/min	6	70	70	65	75	5
2nd 5 kW; 0.4 m/min	6	79	77	75	87	7
1st 3 kW; 0.2 m/min	5	69	70	65	71	4
1st 5 kW; 0.2 m/min	5	75	75	70	80	6

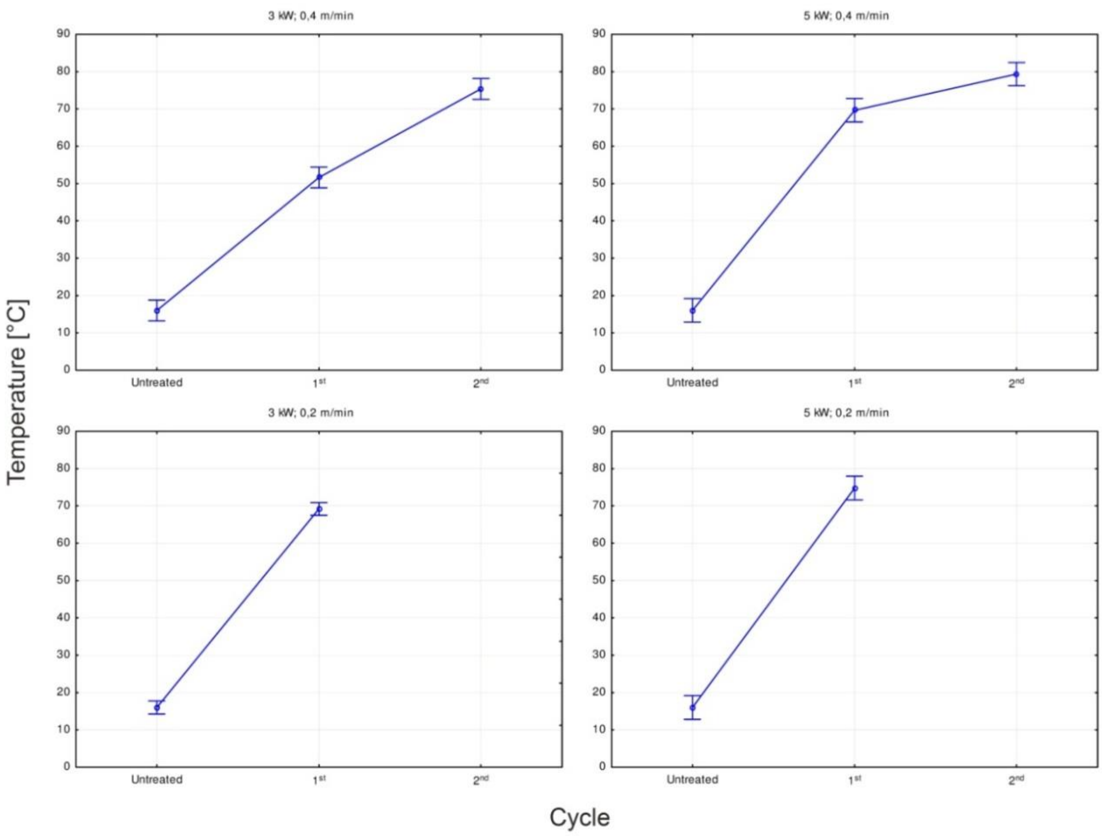

Figure III. 7 Temperature with different modification cycles

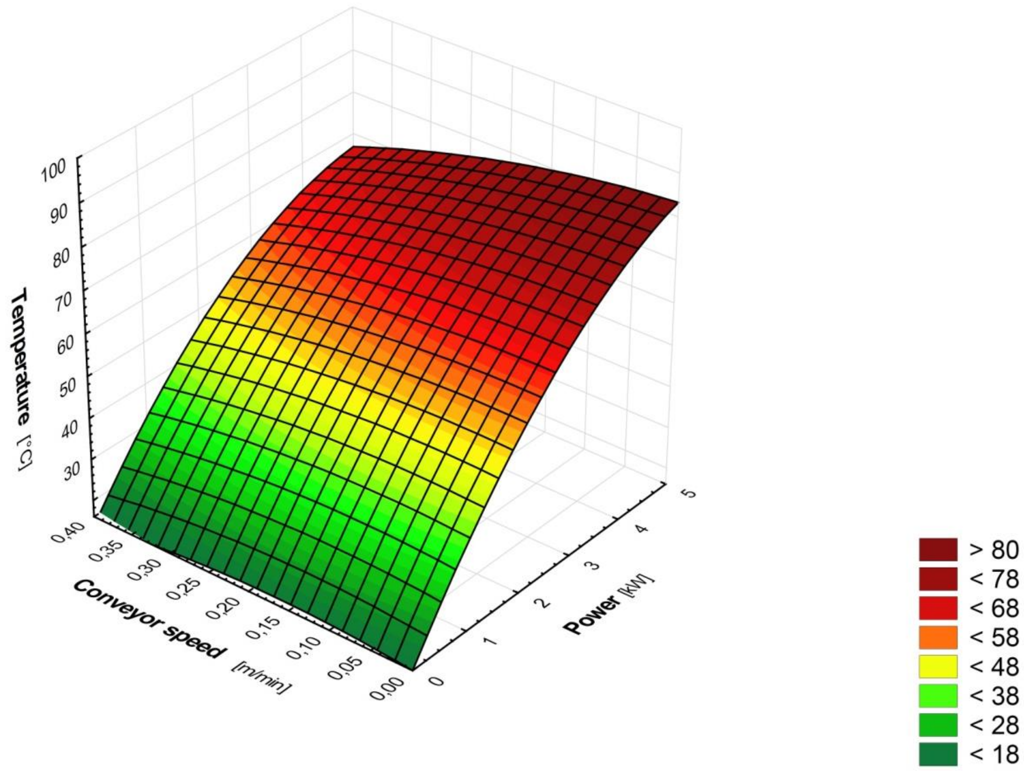

Figure III. 8 Influence of MW power and conveyor speed on the sample surface temperature

Mechanical Properties

The mechanical properties of the MW treated wood were assessed with static bending perpendicular to the grain. The mechanical properties of the samples exposed to (5 kW, 0.4 m/min), (3 kW, 0.2 m/min), and (5 kW, 0.2 m/min) could not be measured. Their structures were totally damaged, indicating an improper setting of MW modification parameters. Table 3 shows that the MW modification parameters (generator output and conveyor speed) were set correctly only in the case of the group treated at 3 kW and 0.4 m/min. The values of MOE (8289 N/mm^2) and MOR (74 N/mm^2) in this group are comparable to the reference group and data published in the literature (Požgaj *et al.* 1997).

Hansson and Antti (2006) stated that variables such as MC of the initial material, anatomical structure, and density affect the hardness of wood more than the selected mode of drying (MW × convectional). According to our results, the parameters that significantly affected the mechanical properties of MW treated wood are the MW output and the conveyor speed used. The variables mentioned by Hansson and Antti (2006) are difficult to control. On the contrary, parameters such as the output power and the conveyor speed can be adjusted precisely. However, if they are set wrong, it has fatal consequences for the treated material (Table 3). Torgovnikov and Vinden (2009) performed a MW modification of Paulownia (*Paulownia fortunei*) wood at an output of 30 kW and frequency of 0.922 GHz. They concluded that the MW modification caused high heat loading. Moreover, water vapour developed a high pressure in its structure and delaminated cell wall tracheids and pith rays. This led to a decrease in the tangential direction MOE from 3400 to 1600 N/mm^2 and a decrease in the tangential direction MOR from 39.3 to 16 N/mm^2. Torgovnikov and Vinden (2000; 2001) treated eucalyptus wood with MW power. The MOE decreased by 12 to 17% and the volume increased by 13.4%. Liu *et al.* (2005) treated the wood of larch (*Larix olgensis*) with MW power and then measured the permeability, MOE, and MOR of the samples in bending and compared the acquired values with data from reference samples. They stated that the correct selection of MW modification parameters improved the permeability of the treated samples, without any considerable decrease in the MOE and the MOR. The permeability increased due to the delamination of the pit membranes in the radial parenchyma and the occurrence of small cracks in cell walls. Zhou *et al.* (2007) exposed the larch wood to intensive MW heating and then examined the changes in bending properties and toughness. The results showed that a correct setting of the MW modification parameters decreased the MOE and impact toughness by only about 10%.

Table III. 3 Sample bending test results (MOE and MOR)

Bending	**Reference**	**3 kW, 0.4 m/min**	**5 kW, 0.4 m/min**	**3 kW, 0.2 m/min**	**5 kW, 0.2 m/min**	**Literature (Požgaj *et al.* 1997)**
MOE [N/mm^2]	*8463*	*8289*	*unmeasurable*	*unmeasurable*	*unmeasurable*	*8210*
MOR [N/mm^2]	*75*	*74*	*unmeasurable*	*unmeasurable*	*unmeasurable*	*73*

CONCLUSIONS

1. Because of the unique construction of the MW device, the results of our experiments differ from the results of similar studies (Brodie 2004b, 2007a; Hansson and Antti 2003, 2006; Hansson *et al.* 2005).

2. The average surface temperature was 75 °C, and the average moisture loss was 13% after two cycles of modification in the group treated at 3 kW and 0.4 m/min. The static bending perpendicular to the grain manifested values of MOE and MOR of 8289 N/mm^2 and 74 N/mm^2, respectively. The average surface temperature of the group treated at 5 kW and 0.4 m/min was 79 °C, and its average moisture loss was 34%. The MOE and MOR could not be measured. The average surface temperature of the group treated at 3 kW and 0.2 m/min was 69 °C, and its average moisture loss was 19%. The MOE and MOR could not be measured. The average surface temperature of the group treated at 5 kW and 0.2 m/min was 75 °C, and its average moisture loss was 40%. The MOE and the MOR could not be measured.

3. The acquired data show that the primary parameters affecting the properties of MW treated spruce (*Picea abies*) wood are the generator output and conveyor speed. A high power output (5 kW) and a slow conveyor speed (0.2 m/min) had a negative impact on the changes in the wood structure and the mechanical properties of the wood. Wood treated in this way can be used for pressure impregnation by synthetic resins with a following material densification or for pulping during the production of fibreboards, cellulose, and paper (Torgovnikov and Vinden 2009). Another possible application could be the drying of firewood and wood chips.

4. The results of this study indicate that it is necessary to conduct further experiments to optimize the process of wood MW modification. Based on the data obtained during the first experiment, a lower output and a faster movement through the modification chamber will be used for the modification to maintain the mechanical properties of the material. The authors are planning to conduct experiments in which the internal temperature of the material would also be measured (optical sensors), microscopic analysis of the wood structure will be performed, and the changes in the density of the modified material will be examined.

PAPER IV.

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

Dömény, J., Koiš, V., Zapletal, M. (2014)

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

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ABSTRACT

In this study, the application of MW treatment for wood plasticization and its densification for flooring system purposes is presented. MW plasticization 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 plasticized 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 MC of the samples were measured after plasticization. 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 plasticization 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 MW plasticization usability and its potential optimisation and application in the wood flooring industry.

Key words: microwave treatment; high-frequency energy; plasticization; density

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.* 2008b). 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.* 2014a).

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 plasticization. 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 plasticization and requires a high amount of energy. Also, the industrial processes suggested usually involve static plasticization 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 plasticization by MW radiation has been studied (Norimoto and Gril 1989; Studhalter *et al.* 2009; Ozarska and Daian 2010; Gašparík and Gaff 2013). The use of MWs 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, MW technology is being explored as one of the methods for wood plasticization. Plasticization by MW heating has several advantages over conventional methods, such as possible continuous application, reduction of plasticization time, high energy efficiency, and volumetric heating (Seyfarth *et al.* 2003; Vongpradubchai and Rattanadecho 2009; Gašparík and Gaff 2013).

The theoretical background of MW heating is characterised by an internal heating process due to the direct absorption of energy by polar molecules. The key substance that absorbs MWs 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_2OH) 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; Larsson 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).

The goal of this work was to contribute to the knowledge of MW heating for plasticization 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 plasticization and mechanism of MW pre-treatment.

EXPERIMENTAL

Material

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

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

Methods

Microwave plasticization

The testing samples were plasticized in a continuous laboratory MW device (Figure 1), which operates at a frequency of 2.45 GHz and provides an adjustable power from 0.6 to 5 kW. The speed of the conveyor can be controlled by the frequency changer (Koiš *et al.* 2014).

Figure IV. 1 Continuous MW device

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

Table IV. 1 MW treatment specifications

Plasticization mode (kW)	Speed of conveyor (m·min^{-1})	Dimension (L×R×T) (mm^3)	Average MC (%)	Number of samples
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 MW plasticization. These three point values were used to calculate the arithmetic mean. The 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 × 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 IV. 2 Densification parameters

Plasticization 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 sample was determined as the ratio of oven-dry mass to volume of the sample 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% 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 equation:

$$H_B = \frac{2F}{\pi \cdot D \cdot \left(D - \sqrt{D^2 - d^2}\right)} \tag{24}$$

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 MW plasticization are presented in Figure 2a 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 (SD) are given in Table 3. The results show that the power of MW radiation had a substantial effect on sample 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.

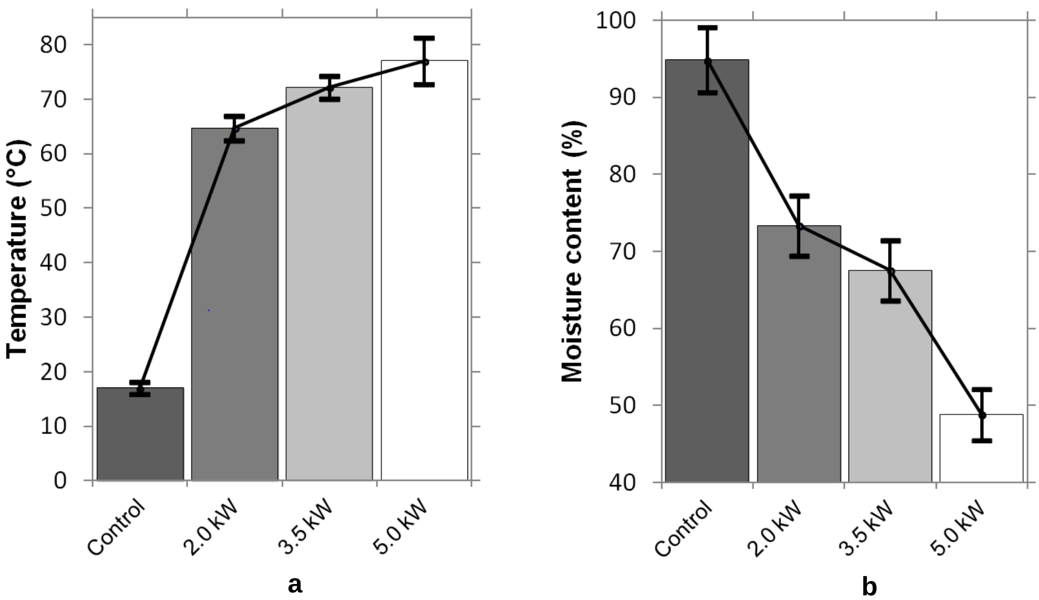

Figure IV. 2 Surface temperature (**a**) and moisture content (**b**) of untreated control and treated wood with different MW plasticization modes

The measured MC data are presented in Figure 2b and Table 3. Samples entering the process of MW plasticization contained 95% of the initial MC. The results show that MW plasticization 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 plasticization 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 (Figure 2b). This phenomenon has also been reported by Gašparík and Gaff (2013). After MW plasticization, the MC was still higher than the fiber saturation point, which is very important for wood softening before densification treatment.

Table IV. 3 Results of surface temperature and moisture content of the wood

Plasticization 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 Figure 3a 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 plasticization 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 samples plasticized 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 sample. 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.

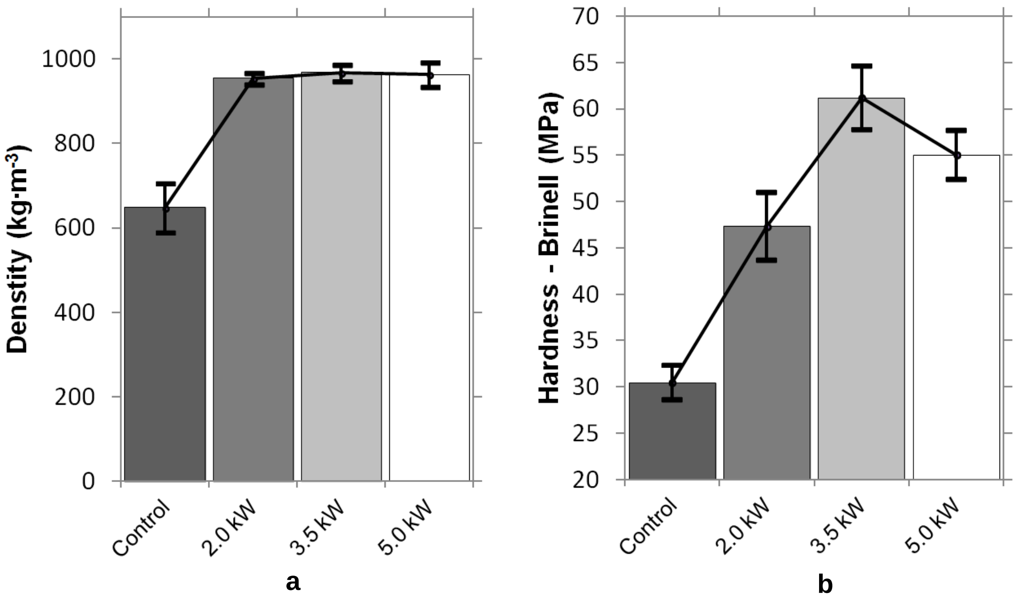

Figure IV. 3 Density (**a**) and Brinell hardness (**b**) of untreated control and treated wood with different MW plasticization modes

Table IV. 4 Results of density and hardness

Plasticization 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, $p < 0.05$)

The hardness values of densified wood can be increased by more than 100% (Rautkari *et al.* 2010). The results presented in Figure 3b show the Brinell hardness (H_B) of control and densified beech wood. A significant increase ($p < 0.05$) was found for each plasticization mode relative to the control samples. Hardness values were highly dependent upon applied MW power. The average H_B data and SD 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 plasticization 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 MW power increased surface temperature significantly ($p < 0.05$).
2. MC decreased significantly ($p < 0.05$) with higher modes of MW radiation.
3. After MW plasticization and the densification process, the hardness of beech increased significantly ($p < 0.05$). The highest increase in Brinell hardness (103%) occurred with a plasticization mode of 3.5 kW. Plasticization in the highest power mode (5 kW) had a negative impact on Brinell hardness because the rapid loss of MC caused steam expansion and stress development.
4. MW heating was found to be an efficient way in terms of rapid (0.4 m/min) plasticization as a pre-treatment for the process of wood densification. However, a good control of the process parameters is needed.

PAPER V.

Density Profile and Microstructural Analysis of Densified Beech Wood (*Fagus sylvatica* L.) Plasticized by Microwave Treatment

Dömény, J., Čermák, Koiš, V., Tippner, J., Rousek, R.

Prepared Manuscript

Density Profile and Microstructural Analysis of Densified Beech Wood (*Fagus sylvatica* L.) Plasticized by Microwave Treatment

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ABSTRACT

This study was carried out in order to determine the efficacy of MW plasticization for wood densification purposes. The plasticization process was carried out using a continuous laboratory device at a frequency of 2.45 GHz. The testing samples made from European beech (*Fagus sylvatica* L.) wood were MW treated with an output of 3.5 kW at a conveyor speed of 0.4 m/min. Afterwards, MW plasticized samples were densified with ration 50%. The microscopic structure changes after densification process were detected using a scanning electron microscope (SEM) and density profiles were measure during the X-ray densitometer. Results show uniformity of density profiles through samples thickness which confirmed even plasticization in sample volume. Microscopic structure observation revealed that the MW plasticization was not accompanied with any fractures and that deformations present in the densified wood were due to viscoelastic buckling of cell walls without cracks propagation.

Key words: beech wood; microwave; plasticization; densification; density profile; density; SEM

INTRODUCTION

Wood, as renewable resource, plays an important role in our daily life as well as industry area. European beech (*Fagus sylvatica* L.) as the most widespread broadleaf species in the forests of the Czech Republic is a traditional material for wood-processing industry (Dömény *et al.* 2014b). Rising demand for consistent, high quality material in last decades has raised interest in different wood modification techniques which considerably improve certain material properties, *i.e.* dimensional stability, bio-durability, thermal properties, colour shade, etc. (Hill and Jones 1996; Militz 2002; Rowell 2005; Hill 2006; Rautkari 2012; Čermák *et al.* 2015).

Density is commonly considered as one of the most important material characteristic, because density strongly correlated with wood strength (Wangaard 1950; Fang *et al.* 2012). Therefore by mechanical densification through compression, it is possible to increase wood's strength (Kollmann and Côté 1968; Kamke 2006). Several densification processes have been developed for that purpose (Kollmann *et al.* 1975; Inoue *et al.* 1993; Higashihara *et al.* 2000; Kamke 2006; Boonstra and Blomberg 2007; Fukuta *et al.* 2008; Gabrielli and Kamke 2008).

One of the most important steps in wood densification is softening of wood structure, *i.e.* plasticization. It is hydrothermal treatment resulting in considerable changes of the compressive behaviour of wood, which can be explained by its viscoelastic nature (Morsing 2000). Applying the certain amount of heat and water vapour in the proper combination will cause softening of the wooden matrix consisting of cellulose, hemicellulose, and lignin (Rautkari *et al.* 2010). This softening behaviour is characterised by phase transition of matrix from a glass to a rubber state (Lamason and Gong 2007). Wood plasticization can be done by using various procedures. The application of steam as a heating and softening medium is the most traditionally used method for wood softening. Unfortunately, due to mechanism of heat and mass transfer during steaming of wood, time consumption and energy requirements, are important issues of this method.

In order to reduce the plasticizing time an innovative way of plasticization using MW heating has been recently studied (Norimoto and Gril 1989; Seyfarth *et al.* 2003; Vongpradubchai and Rattanadecho 2009; Gašparík and Gaff 2013; Dömény *et al.* 2014b). Plasticization by MW heating brings several advantages over conventional water vapour method, such as possibility to lower energy consumption, rapid heating of wood over sample volume, application for continues purposes (Vongpradubchai and Rattanadecho 2009; Gašparík and Gaff 2013). The theoretical principle of MW heating is based on the polar characteristic of molecules and their ability to absorb and transform MW radiation into heat energy (Metaxas and Meredith 1983; Torgovnikov 1993; Hansson and Antti 2003). Permanent dipoles of molecules rapidly absorb the electromagnetic radiation which is converted to kinetic energy and into frictional heat (Makovíny 2000; Hansson and Antti 2003). On the contrary the structural changes during MW treatment can appear and this may lead to changes in wood strength (Oloyed and Groombridge 2000; Hansson and Antti 2003; Hong-Hai *et al.* 2005; Leiker *et al.* 2005;

Machado 2006). By applying MW radiation to the wood with certain MC, the water in the cell cavity absorbs the energy and vaporized (Merenda and Holan 2008; Torgovnikov and Vinden 2009). The internal high steam pressure can delaminate the cell walls and create narrow voids in wood structure (Torgovnikov and Vinden 2004; Li *et al.* 2009; Yu *et al.* 2011; Vinden *et al.* 2011).

Present study aims to (1) evaluate the effect of MW (MW) plasticization and the following densification on density profile and (2) to visually evaluate the microscopic structure changes measured by scanning electron microscopy (SEM). This work should provide better insing into details of wood plasticization using MW heating and its potential use in industry scale.

MATERIAL AND METHODS

Tangential and radial samples (50×40×8 mm^3; length×wide×thickness) made of European beech (*Fagus sylvatica* L.) sapwood were MW plasticized and densified afterwards. Prior MW plasticization, the samples were soaked in distilled water for two weeks. An average initial MC was 79% as determined by the oven-dry method of standard EN 13183-1 (2002). In total, 32 samples (16 radial and 16 tangential) without defects (cracks, knots, etc.) were studied. 8 samples from both groups were MW plasticized and densified in sample thickness direction, rest of the samples was kept as a controlled.

The process of plasticization have been carried out using continuous laboratory MW device, which operates at a frequency of 2.45 GHz and provides an adjustable power from 0.6 to 5 kW (Dömény *et al.* 2014b; Koiš *et al.* 2014). The testing samples were plasticized with an output of 3.5 kW at a conveyor speed of 0.4 m/min. Process parameters were found to be as the most effective in terms of rapid plasticization for subsequent wood densification (Dömény *et al.* 2014b).

The surface temperature (T_S) of the testing samples was measured by a contactless infrared thermometer (IR-380, Voltcraft) on the sample surfaces. The 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).

Once the MW plasticization is done, the samples were immediately densified to the final thickness of 4 mm (ratio 50%), using a hydraulic press (HL 400, Strozatech). Temperature-controlled plates were heated to 80 °C and pressing time was set to 1 minute.

Testing samples from the same testing group were densified in one batch. After the densification, the press was released and the plates were maintained in the closed position for a period of five days (cooling down, stabilization, and conditioning).

Afterwards the densified as well as control samples were oven dried to 0% MC, according to EN 13183-1 (2002) and density profiles were measured for all samples. Density profiles were measured at interval 0.1 mm trough the sample thickness (4 mm; densification direction) using an X-ray densitometer (X-RAY, Dense-Lab).

The microstructure of MW densified and control samples were examined using field emission scanning electron microscopy (FE-SEM - FEI Quanta 250, FEG). Samples were trimmed to a final size of about 4×4×4 mm³. Subsequently were mounted onto aluminium stubs with conductive carbon paste and were sputter-coated with gold. Aim of this analysis was to found out if MW pre-treatment (plasticization) can cause the structural changes in the wood.

RESULTS AND DISCUSSION

Temperature and moisture content during the plasticization

An average surface temperature (T_s) of all MW plasticized samples increased significantly ($p < 0.05$) from initial temperature (20 °C) to 72.2 °C (standard deviation 2.2 °C). The T_s differences given by standard deviation, can be caused by the uneven distribution of MC or density in samples at the beginning of process (treatment). No significant differences between surface temperature of the radial and tangential samples were found.

The radial and tangential samples entering the process of MW plasticization contained an average 80.4% (3.4%) and 76.9% (4.6%) of MC (with standard deviation), respectively. MC of samples decreased by ~38% (2.8) for radial and by ~33% (2.1%) for tangential samples during MW pre-treatment.

After MW plasticization, the temperature and MC state was sufficient for phase transition of wooden matrix from a glass state to a rubber state. Wood softening occurs at lower temperatures when MC increases, because glass transition is highly dependent on MC of wood (Hillis and Rozsa 1978; Uhmeir *et al.* 1998; Rautkari *et al.* 2011).

Density and density profile

After the densification process, the original density (with standard deviation) of 770.8 kg·m^{-3} (31.4) and 676.6 kg·m^{-3} (23.6) increased significantly ($p < 0.05$) to 1194 kg·m^{-3} (46.8) for radial samples and to 950.7 kg·m^{-3} (25.4) for tangential samples. The minor springback effect was observed after the densification process. The expansion was from original thickness, *i.e.* 4 mm to 4.08 mm and 4.52 mm, for radial and tangential samples, respectively (springback ~2% and ~13%, at 0% MC).

The density profiles, represented by example of the radial and tangential sample, are depicted in Figure 1 and 2. The control as well as densified samples exhibited almost uniform density distribution through its thickness. Such behaviour was expected for control samples because beech wood as a diffuse porous hardwood species has negligible difference in density over earlywood and latewood sections (Bouriaud *et al.* 2004). On the other hand, the density profile of densified wood is depended on the pressing parameters (press, time) as well as on softening (plasticization) procedure (moisture and temperature distribution in samples). Uniform distribution of density over sample thickness confirmed that MW plasticization was effective as the heating and softening pre-treatment, *i.e.* the samples were plasticized evenly in the entire wood volume and therefore also evenly densified.

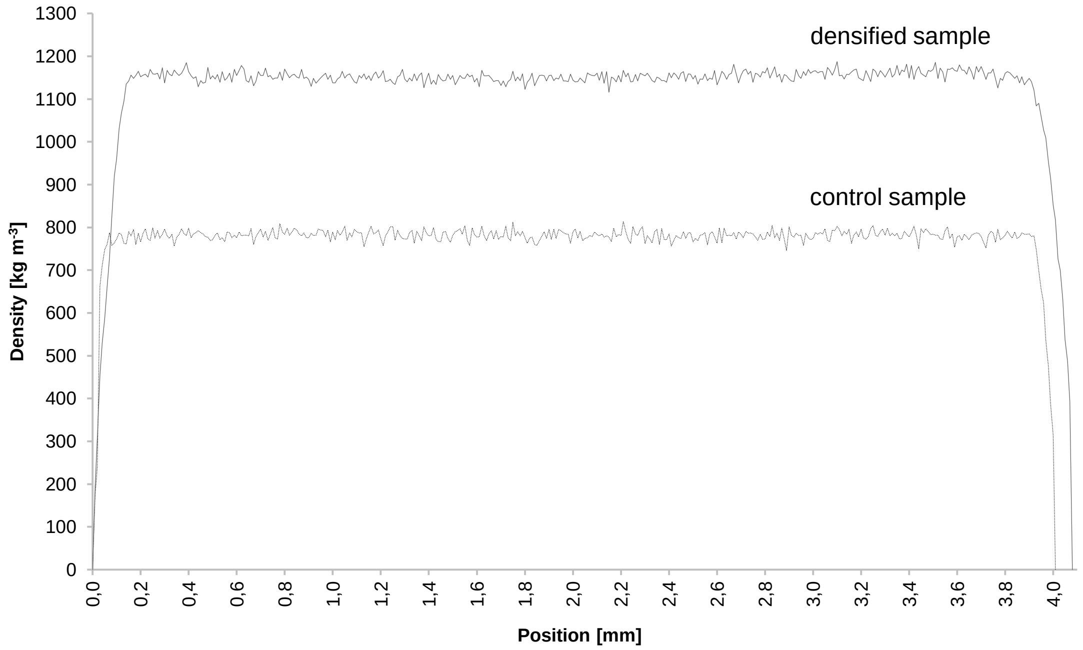


Figure V. 1 Density profiles of the control and densified radial sample

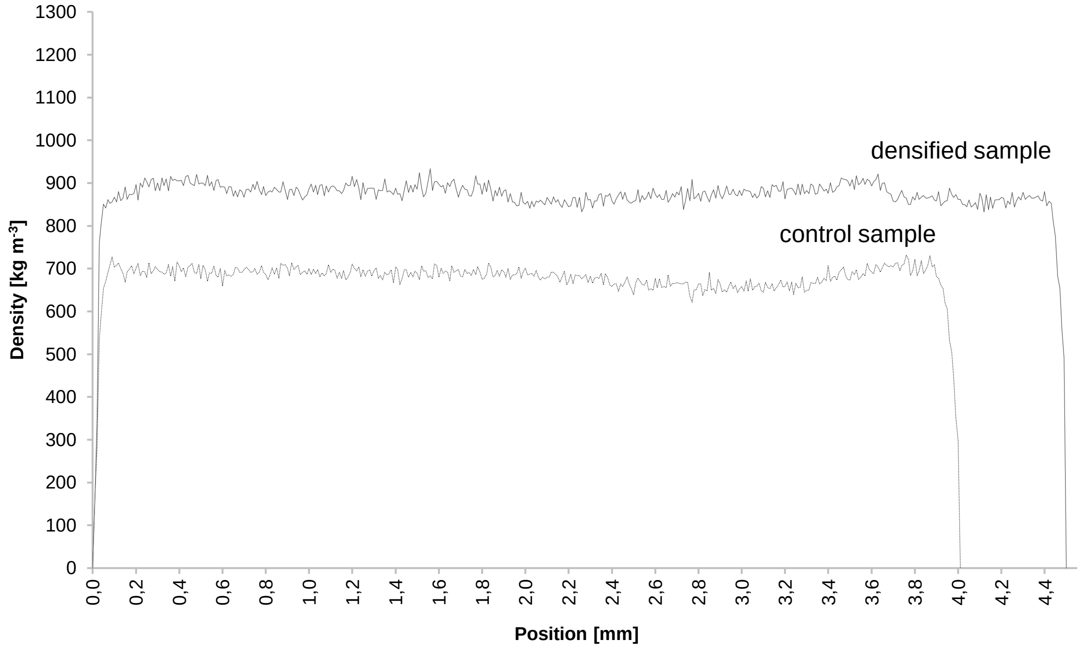


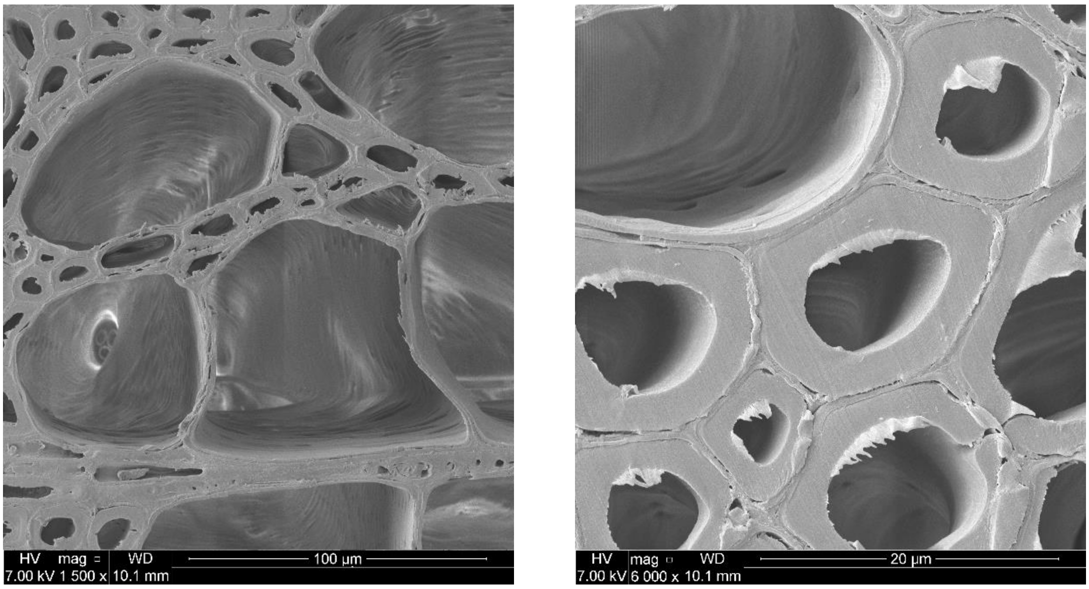
Figure V. 2 Density profiles of the control and densified tangential sample

The density of radial samples is in agreements with data reported by Skyba *et al.* (2009) who reached an average density 1140 kg·m^{-3} for beech densified with ratio of 45%.

Even though, the effect of wood densification is obvious, the density variation can be found between the radial and tangential samples. More significant increase in density was found for radial samples due to pith rays orientation perpendicular to the loading direction. In the tangential samples the pith rays probably reinforced compression due to their orientation in to the loading direction, which had effect on the greater springback effect. Deformations perpendicular to the loading direction could occur and probably be also assumed as the reason for lower density in the tangential samples. Furthermore, pith rays orientation in radial samples could then prevent this deformations perpendicular to the loading direction.

Microstructure analysis

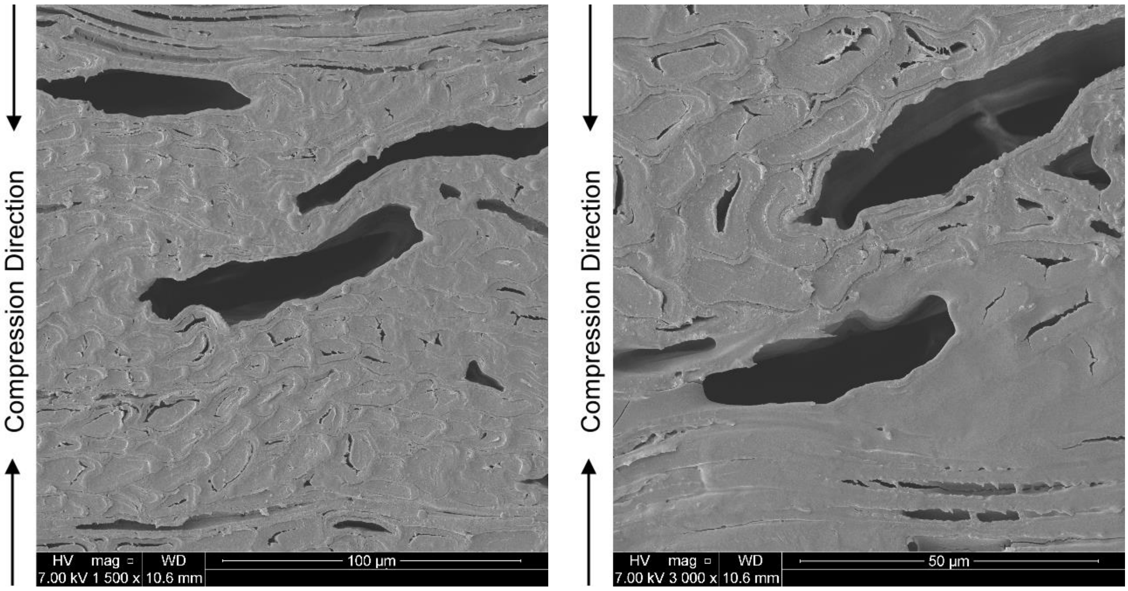
Microstructure analysis was carried out to examine the efficiency of MW plasticization, potential micro cracks propagation and mechanisms of cells deformation during densification process. The electron microscopy was focused on the cross section of the wood samples, where most of the anatomical elements is shown in its transverse directions.



a **b**

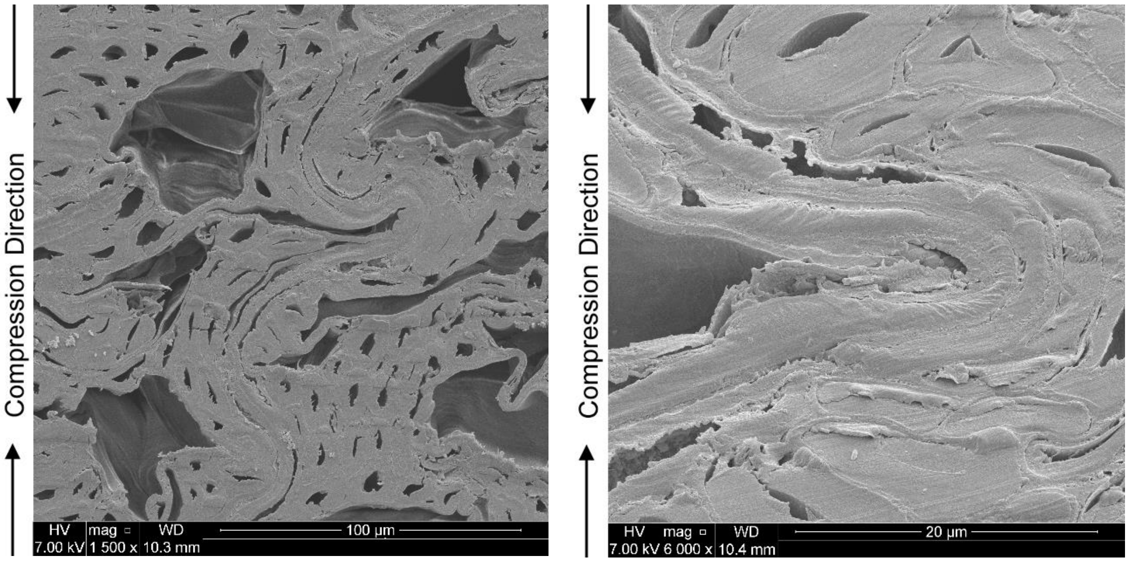
Figure V. 3 SEM image of control sample with magnification 100 µm (**a**) and 20 µm (**b**)

SEM analysis shows the microstructure of control (Figure 3) and densified samples (Figure 4 and 5) at various magnifications. It can be seen that after densification the morphology of cells was changed. Cells were deformed in the direction of densification and volume of cell lumen was significantly reduced. The densification of radial samples (Figure 4) resulted in cell wall collapse and therefore the lumens of cells nearly disappeared. Since the structural variation between earlywood and latewood in beech wood can be consider as insignificant, the morphology after densification process cannot be different. The cell wall damages (micro cracks) due to MW radiation, *i.e.* insufficient plasticization was not observed, even though cells were deformed.



a b

Figure V. 4 SEM image of radial sample with magnification 100 µm (**a**) and 50 µm (**b**)



a b

Figure V. 5 SEM image of tangential sample with magnification 100 µm (**a**) and 20 µm (**b**)

The densification of tangential samples (Figure 5) resulted in the deformation of the ray cells which significantly affected the results of densification process, *i.e.* springback effect. This was also previously confirmed by Haygreen and Bowyer (1996), who stated that wide ray cells of hardwoods can reinforce compression in the tangential samples.

From analysis it is evident that cells from both transverse densification processes were deformed without fractures and dislocations of the cell walls. This confirmed that the selected mode (based on previous pre-tests) of MW plasticization was sufficient as the pre-treatment for wood densification purposes.

A few published studies reported that MW radiation might cause the delamination of wood structure. Torgovnikov and Vinden (2009) stated that applying of MW radiation to the wood caused a high pressure of water vapour in the wood structure, which can make the cell walls delaminated. According to Merenda and Holan (2008), Li *et al.* (2009), Yu *et al*. (2011) and Vinden *et al*. (2011) the fractures and voids can occur in the wood structure when vapour is generated. However, authors of published papers used wood species with rather low permeability, where high pressure vapour is closed in the wood structure. High permeability of the beech wood is considered as a reason that the cell wall damages were not observed in the present study.

CONCLUSIONS

1. The results show that the density significantly increased after the densification of wood and samples exhibit uniformity of density profile, *i.e.* samples were densified evenly in the entire cross-section.
2. Microscopic analysis shows that MW plasticization as well as subsequent densification was performed without any fractures of the cell walls.
3. Different densities have been gained for densification in the radial and tangential samples due to pith rays orientation to loading direction.
4. MW heating was found to be sufficient way for wood plasticization of the beech wood. However, when other wood species are applied the MW plasticization process should be optimized properly.

PAPER VI.

Application of Microwave Heating for Acetylation of Beech (*Fagus sylvatica* L.) and Poplar (*Populus hybrids*) Wood

Dömény, J., Čermák, P., Pařil, P., Fodor, F. P., Dejmal, A., Rademacher, P. (2015)

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Application of Microwave Heating for Acetylation of Beech (*Fagus sylvatica* L.) and Poplar (*Populus hybrids*) Wood

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ABSTRACT

MW and conventional acetylation of wood was carried out to determine its efficacy on the material properties. Beech (*Fagus sylvatica* L.) and poplar (*Populus hybrids*) samples with dimensions 14 mm × 14 mm × 14 mm were impregnated using acetic anhydride, and chemical reactions were initiated by MW and conventional heating. The MW acetylation process was carried out using laboratory device at a frequency of 2.45 GHz in several testing modes to reduce time of the reaction. The uptake of substance, EMC, wood swelling, and dimensional stability were determined in order to evaluate the efficacy and degree of acetylation. Both MW and conventional heating positively affected the selected material properties. The results showed that no significant differences were found between MW and conventional heating; therefore, MW heating can be used as a valid replacement in the acetylation process. MW power of 2 kW and 0.1 m·min^{-1} conveyor speed were the optimum conditions for MW acetylation. These process parameters resulted in 39.4% ASE $_T$ and 35.2% ASE $_R$ for beech and 38.0% ASE $_T$ and 16.3% ASE $_R$ for poplar samples. This work provides insight into the details of wood acetylation using MW heating.

Key words: acetic anhydride; chemical reactions; dimensional stability; wood impregnation; microwave treatment; wood modification

INTRODUCTION

Wood, as an important renewable resource, has a broad range of material properties for various applications, *e.g.*, relatively high strength and stiffness, low specific weight, natural appearance with interesting texture, insulation properties, and machinability

(Kollmann 1951; Stamm 1964; Wagenführ 2000; Skodras *et al.* 2004). However, natural wood also has undesirable properties that might limit the range of feasible applications. Wood is a natural heterogeneous composite and is considered to be dimensionally unstable when exposed to wet conditions (Kumar 1957; Rowell 1983; Skaar 1988; Hunter 1995). Hydroxyl (-OH) groups of hemicellulose and cellulose chains are mainly responsible for the highly hygroscopic behaviour of wood (Stamm 1964; Rowell 1983). The dimensional instability of wood under different moisture/humidity conditions is considered a major drawback of wood performance (Stamm 1964; Hill and Jones 1996; Homan and Jorissen 2004; Popescu *et al.* 2013).

Wood modification techniques can be applied in order to improve certain wood properties, *e.g.*, bio-durability, dimensional stability, colour, wettability, etc. (Hill and Jones 1996; Militz 2002; Rowell 2005; Hill 2006; Čermák *et al.* 2015). Chemical modification can, for instance, be used as an efficient way to transform hydrophilic OH groups into larger hydrophobic groups (Kollmann 1951; Rowell 1983; Skaar 1988; Bodîrlău *et al.* 2009). Little to no water can penetrate the permanently swollen cell wall of wood as a result of chemical treatment (Homan and Jorissen 2004).

Acetylation of wood is one of the most commonly used chemical treatments to improve the dimensional stability and biological durability of wood (Tarkow *et al.* 1950; Larsson and Simonson 1994; Popescu *et al.* 2013). Moreover, this treatment retains to wood its original colour and improves acoustical, dielectric, and strength properties (Tarkow *et al.* 1950; Dreher *et al*. 1964, Homan *et al.* 2000).

Acetylation effectively changes free hydroxyls within the wood into acetyl groups (Rowell 1983; Hill and Jones 1996; Rowell 2013). This is done by reacting wood with acetic anhydride (Ac_2O) (Militz 1991; Sander *et al.* 2003). The standard acetylation process includes impregnation of oven-dried wood with Ac_2O, followed by conventional heating to initiate the chemical reactions with wood polymers (Bongers and Beckers 2003). Acetic acid is then released as a by-product of the reactions (Homan and Jorissen 2004). Application of the Ac_2O without a catalyst or cosolvent is the preferred method for wood acetylation (Rowell 2013). Time consumption is an important issue for the proposed acetylation method (Yang *et al.* 2014).

In order to reduce the reaction time and make the process more effective, an innovative wood acetylation process that uses MW energy has been recently studied

(Larsson *et al.* 1999; Larsson and Simonson 1999; Larsson 2002; Li *et al.* 2009; Diop *et al.* 2011; Yang *et al.* 2014). The application of MW energy rapidly heats the material throughout the whole cross-section using the dielectric properties of wood (Torgovnikov 1993; Larsson *et al.* 1999) instead of commonly used convection and conduction heat flux (Koskiniemi *et al.* 2013). The principle behind MW heating is based on the polar characteristic of molecules and their ability to absorb and transform MW radiation into heat (Metaxas and Meredith 1983; Torgovnikov 1993; Hansson and Antti 2003). Permanent dipoles of molecules begin to move with the same frequency as the electromagnetic field. Therefore, rapid changes in the field polarity cause vibration and rotation of molecules, which transforms the MW energy into frictional heat (Makovíny 2000; Hansson and Antti 2003; Dömény *et al.* 2014a). The polarizability of the Ac_2O in the MW field has been extensively studied (Baghurst and Mingos 1992; Larsson *et al.* 1999). Baghurst and Mingos (1992) stated that during MW heating, the temperature rise of the Ac_2O was about two times higher than water.

Unfortunately, published studies related to the acetylation process conducted using MW heating are still limited. Therefore, the present study aims to (1) analyse the acetylation process using MW heating, (2) evaluate the efficacy of MW heating on the chemical reactions during the process and its similarities with conventional methods, and (3) evaluate material properties (uptake of substances, equilibrium moisture content, wood swelling, and anti-swelling efficiency). This work should provide a better insight into details of the wood acetylation.

EXPERIMENTAL

Materials

Beech (*Fagus sylvatica* L.) and poplar (*Populus hybrids*) sapwood were studied. Samples with dimensions 14 mm × 14 mm × 14 mm were oven dried to 0% MC, according to EN 13183-1 (2002). The average oven-dry density (ρ_0) of testing samples was 694 kg·m^{-3} for beech and 316 kg·m^{-3} for poplar. Afterward, samples were sorted into groups of 10 for each species and acetylation treatment (Table 1).

Table VI. 1 List of treatments and process parameters

Material	No. of samples	Treatment	Mode	Speed of conveyor	Time
Beech/Poplar	10/10	Control	-	-	-
Beech/Poplar	10/10	MW I	1.5 kW	0.1 m·min^{-1}	15 min
Beech/Poplar	10/10	MW II	2.0 kW	0.1 m·min^{-1}	15 min
Beech/Poplar	10/10	MW III	2.0 kW	0.025 m·min^{-1}	60 min
Beech/Poplar	10/10	Conventional heat	100 °C	-	60 min

Methods

Acetylation process

Prior to treatment, samples were pressure impregnated with Ac_2O (Sigma-Aldrich, analytical grade ≥ 99%) in a laboratory plant JHP1-0072 (Figure 1).

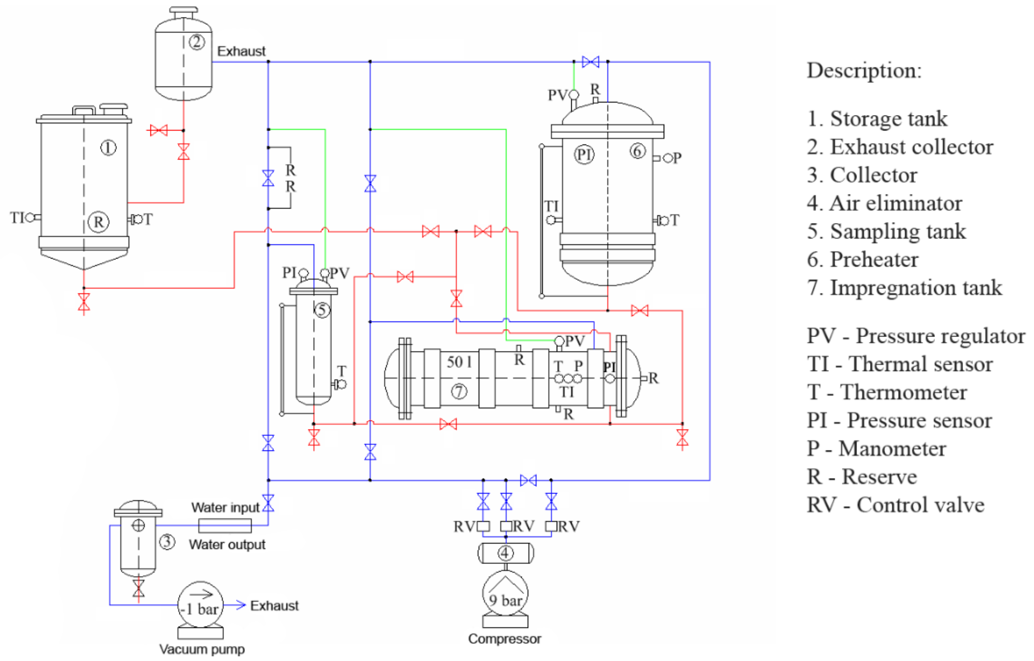


Figure VI. 1 Scheme of vacuum-pressure impregnation equipment (Dejmal *et al.* 2012)

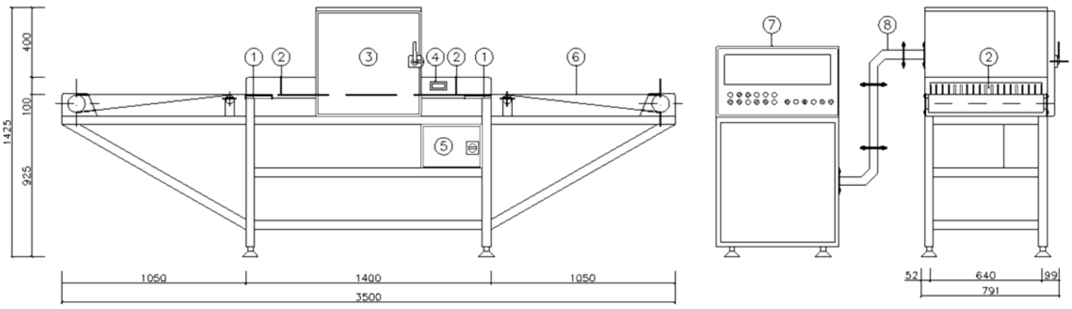
Wood species were impregnated separately using same process parameters, *i.e.* 0.8 MPa pressure for 120 min at 20 °C. Weight percentage gain (WPG) and retention (R) values were used as an indicator of substance uptake according to the following equations:

$$WPG\ (\%) = (m_2 - m_1)/\ m_1 \quad (25)$$

$$R\ (kg \cdot m^{-3}) = (m_2 - m_1)/V \quad (26)$$

where: m_1 is the sample weight before impregnation, m_2 is the sample weight after impregnation, and V is volume of the sample.

Afterward, MW and conventional heating were used to induce chemical reactions taking place during the acetylation process (Table 1). A continuous laboratory MW device (Figure 2) that operates at a frequency of 2.45 GHz with adjustable power from 0.6 to 5 kW was used for the MW heating. Conventional heating was carried out in a standard laboratory drying oven (Sanyo MOV 112).



Description: 1. Duced ceramics 2. Flexible copper plate 3. Modification chamber 4. Speed control panel 5. Power button 6. Conveyor 7. Microwave generator with control panel 8. Waveguide

Figure VI. 2 Scheme of continuous MW device

Once chemical reactions took place, the testing samples were placed into the impregnation plant in vacuum at 10 kPa for 60 min to eliminate the residual acetic anhydride and acetic acid from wood structure.

Uptake of the substance was determined in two steps: after the impregnation of testing samples and after the chemical reaction when residuals were eliminated.

Surface temperature

The surface temperature of the testing samples was measured by a contactless infrared thermometer (Voltcraft IR-380, accuracy ±1.0 °C) during a short interruption of the chemical reactions (turning off the power and opening the modification chamber for a few seconds) for both methods of heating. Measurement was done in the middle of the reaction time.

Equilibrium moisture content and Dimensional stability

The samples were conditioned in a climate chamber (Sanyo MTH 2400) at different relative humidities (30%, 65%, and 99%) at 20 °C until the EMC was reached for certain conditions. Afterward, the MC was determined using the oven-dry method according to EN 13183-1 (2002).

Dimensional stability in radial and tangential directions was determined by estimating wood swelling ($S_{T,R}$) and anti-swelling efficiency ($ASE_{T,R}$). The swelling was calculated in dry and equilibrium states at the RH under study. ASE represents difference between the swelling of the treated and untreated wood. $S_{T,R}$ and $ASE_{T,R}$ were calculated according to the following equations:

$$S_{T,R}\ (\%) = 100\ (D_{T,R,2} - D_{T,R,1})/\ D_{T,R,1} \qquad (27)$$

$$ASE_{T,R}\ (\%) = 100\ (S_{T,R,u} - S_{T,R,t})/S_{T,R,u} \qquad (28)$$

where $D_{T,R,1}$ (mm) is the radial or tangential dimension of the oven-dried sample, $D_{T,R,2}$ (mm) is the radial or tangential dimension of the conditioned sample, and $S_{T,R,u}$ (%) and $S_{T,R,t}$ (%) are wood swelling of the untreated and treated sample, respectively.

Statistical analysis

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

RESULTS AND DISCUSSION

Uptake of Substance

Average WPG and retention values are shown in Table 2. The amount of Ac_2O impregnated within the wood structure was identical for all testing groups (MW I, MW II, MW III, and conventional heating) after the impregnation process within the same wood species group. The results showed significant differences between the WPG of beech (66%) and poplar (211%), even though the impregnation process parameters were the same. The major differences can be explained by different densities of wood species, which is reflected in calculating of WPG (Eq. 25). In fact it was caused also by the structural and chemical composition of the different wood species. For practical applications, it is rather important to know the amount of the impregnated substance expressed by weight in the wood volume. Therefore, retention was used as a second indicator of the substance uptake.

Based on the Tukey's HSD test, statistically insignificant WPG and retention were found between conventional heating, MW I, and MW III treatments for beech and conventional heating, MW II, and MW III treatments for poplar. All other acetylation treatments were statistically significant in terms of substance uptake (Table 2). After the chemical reaction, the WPG decreased by ~57% for beech and ~202% for poplar. This shows that a relatively high uptake of substance was reached but a small amount of the Ac_2O reacted within and remained in the wood structure. This can also be seen from the retention results. After the chemical reaction, the retention decreased from 656.2 $kg \cdot m^{-3}$ to ~30 $kg \cdot m^{-3}$ for poplar and from 452.7 $kg \cdot m^{-3}$ to ~65 $kg \cdot m^{-3}$ for beech. Values of beech retention (after the reaction) were two times higher than poplar. However, this does not mean that the efficacy of acetylation was also higher because of the higher retention. The efficacy depends on the degree of substitution and the amount of free hydroxyl groups, which is associated with wood density. Therefore, WPG is an appropriate indicator of the substance uptake and the acetylation process efficacy. Some authors have evaluated the effectiveness of acetylation by acetyl content using HPLC analysis (Larsson and Simonson 1999) or by degree of substitution determined by a back-titration method (Li *et al.* 2009). However, WPG is a well-known indicator used in most studies dealing with wood acetylation. In the present study, WPG values of the acetylated beech and poplar samples were similar for all treatment modes, on average ~9%. Eranna and Pandey (2012) published data of the substance uptake for rubberwood (*Hevea brasiliensis*) conventionally treated at a temperature of 120 °C, after 15 min, 30 min, 60 min, and 120 min and reported that the WPG reached ~7%, ~8%, ~9%, and ~12%, respectively. Yang *et al.* (2014) compared the different types of acetylation reactions: liquid, MW, and vapour phase after 60 min of reaction time. Sugi wood (*Cryptomeria japonica*) acetylated using a MW reaction exhibited the highest WPG (19.4%), followed by the liquid phase reaction (19.1%), and vapour phase reaction (18.0%). Li *et al.* (2009) acetylated cellulose by MW heating with iodine as a catalyst. Results of the WPG after 15 min of the treatment at temperatures of 80 °C, 100 °C, and 130 °C were 13%, 16%, and 25%, respectively (Li *et al.* 2009). Pries *et al.* (2013) conventionally acetylated beech for a fungal decay test by Ac_2O at 120 °C for 120 min and observed 10.2% WPG. Results of the substance uptake (WPG, R) were difficult to compare with previously published data because of different wood species and process parameters used.

Table VI. 2 Results of substance uptake (weight percentage gain and retention)

Uptake of substance	Treatment	WPG (%)		Retention (kg·m^{-3})	
		Beech	Poplar	Beech	Poplar
After impregnation	-	66.0 (3.8) ^{A}	211.3 (12.0) ^{A}	452.7 (23.2) ^{A}	656.2 (17.2) ^{A}
After chemical reaction	MW I	9.3 (1.1) ^{B}	7.7 (1.8) ^{B}	65.4 (6.4) ^{B}	24.2 (5.4) ^{B}
	MW II	10.2 (0.5) ^{C}	9.6 (0.8) ^{C}	70.0 (3.3) ^{C}	30.3 (2.3) ^{C}
	MW III	8.8 (0.8) ^{B}	9.3 (1.4) ^{C}	61.4 (5.5) ^{B}	30.1 (6.1) ^{C}
	Conventional Heat	9.5 (0.8) ^{B}	9.6 (1.3) ^{C}	64.7 (4.9) ^{B}	30.1 (4.1) ^{C}

Means sharing same letter in column are not significantly different (Tukey's HSD, $p < 0.05$)
Numbers in parentheses indicate standard deviation

Temperature

A maximum surface temperature was observed when MW treatment at 2 kW with 0.025 m·min^{-1} conveyor speed (mode MW III) was used, as well as conventional heating. The surface temperature increased from 20 °C to 103 °C and 100 °C, respectively. Moreover, when milder MW heating modes were used, the surface temperature increased from 20 °C to 83 °C (MW II) and 69 °C (MW I). The results showed that the MW power and conveyor speed had a substantial effect on the sample temperature, influenced by various radiation intensities converted directly to thermal energy by frictional heating.

Equilibrium Moisture Content

By converting the hydroxyl groups of cell wall polymers into hydrophobic acetyl groups, the hygroscopicity of the wood was reduced. Values of the EMC at different relative humidities are presented in Figure 3. Significant differences were found between the control, MW, and conventionally heated samples at all RH levels (30%, 65%, and 99%). However, insignificant differences were found between various modes of MW treatment. In the case of poplar, there were insignificant differences between MW II and MW III treatments. Similar results were recorded for beech in 99% RH. In that respect, 60 min (MW II) and 15 min (MW III) treatments had identical results for EMC.

Yang *et al.* (2014) reported that the acetylation efficacy increased with reaction time. Such a statement was not confirmed in the present study. From the results, it can be concluded that the MW treatment provided the same degree of acetylation independent of the duration of the reaction in the range of 15 to 60 min. However, it should be considered that only two time modes (conveyor speeds) were used in the experiment and deeper investigation is needed to confirm this statement. Larsson *et al.* (1999) studied the MW acetylation of pine and stated that the microwave-heated wood gives a higher degree of acetylation during the initial phase of the reaction compared to the conventional method. With a prolonged reaction time, the degree of acetylation was about the same, because only thermal effects are included when MW heating is applied to wood acetylation (Larsson *et al.* 1999).

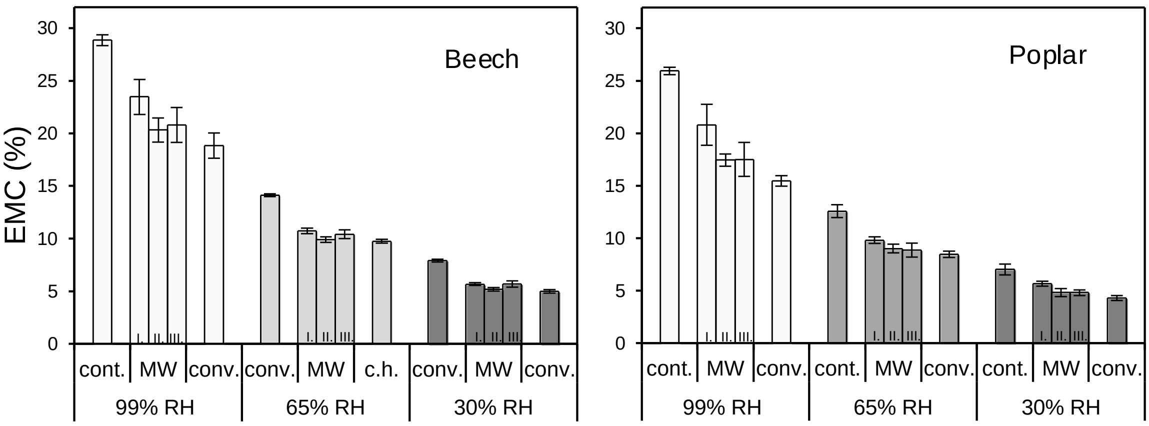


Figure VI. 3 EMC of beech and poplar under different RH; Roman numerals indicate MW modes (MW I–III)

Using conventional heating for 60 min, the EMC of beech samples in 99% RH decreased by 35% when compared to the control. By using 2 kW MW heating for 15 and 60 min (MW II and MW III), the EMC decreased by ~29%. Similar results were found for poplar samples. The EMC of conventionally heated poplar decreased by ~40% and by about 32% for MW heating samples (MW II, MW III). The MW I mode provided a lower degree of acetylation even though a longer period (60 min) of heating was used. This was probably caused by a low increase of temperature during the acetylation process. EMC values at 65% and 30% RH showed a similar trend as 99% RH (Figure 3). The degree of acetylation increased with an increase in the MW power; therefore, it can be stated that MW power has a significant effect on wood acetylation.

Dimensional Stability

The radial and tangential swelling of wood (S_R and S_T) at different RH levels are presented in Figure 4. Results at 99% RH were used for the ASE, indicating the effectiveness of treatments on the dimensional stability (Figure 5).

The control samples had a higher radial and tangential swelling than acetylated samples. Since acetyl groups occupy space within the cell wall, the wood is not able to absorb water molecules and therefore wood swelling is reduced (Tarkow *et al.* 1950; Rowell 1983). When conventional and MW acetylation are compared, only very minor differences in the wood swelling can be found. Moreover, statistically insignificant differences were found between 60 min (conventional, MW III) and 15 min treatments. Heating by MW can accelerate chemical reactions in the acetylation process, whereby the same dimensional stability is reached. Therefore, the reaction time of acetylation can be reduced and the process made more effective.

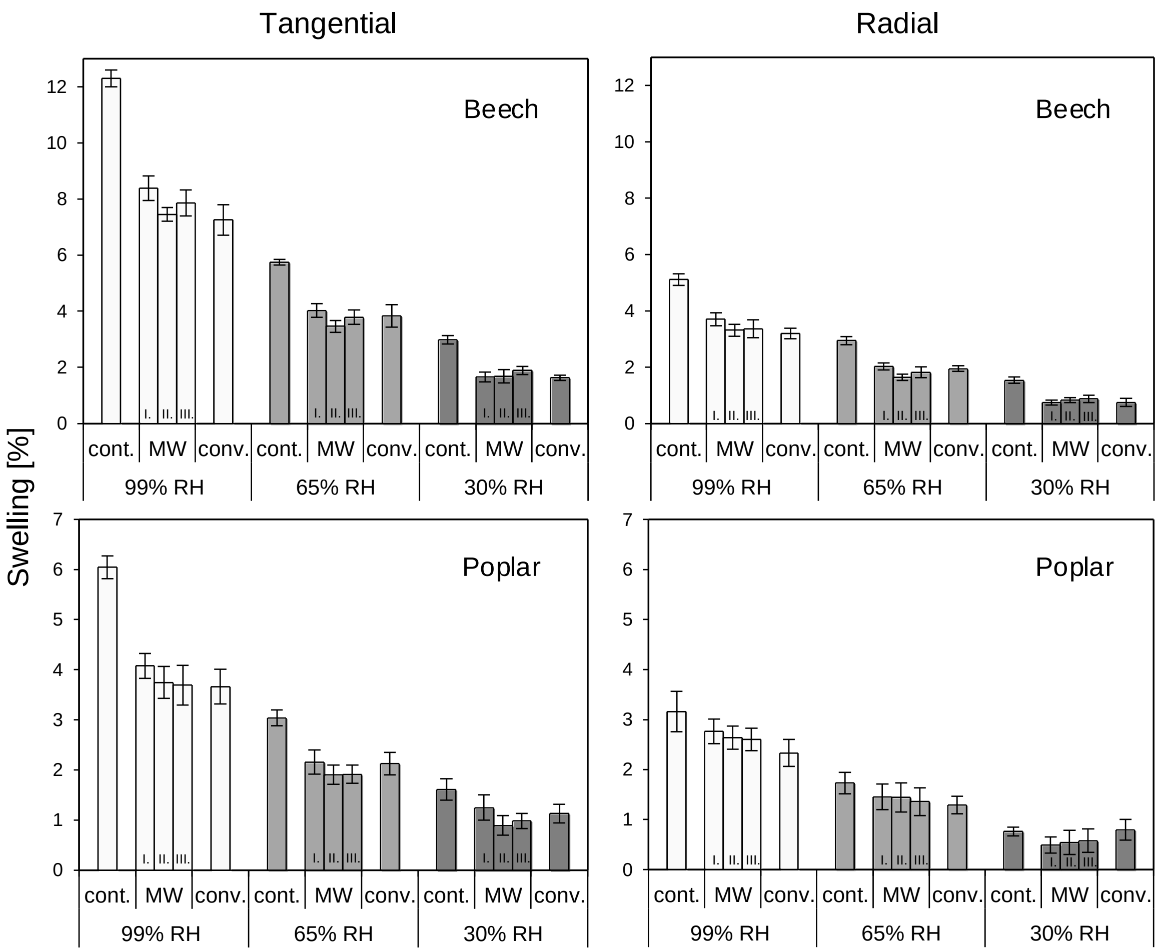


Figure VI. 4 Swelling of beech and poplar in the tangential and radial directions

Anti-swelling efficiency is the most commonly used method to evaluate the dimensional stability of the modified wood (Santos 2000; Čermák *et al.* 2015). The dimensional stability was considerably improved for all applied acetylation treatment modes. The ASE of the acetylated beech in modes MW I, MW II, MW III, and conventional heating were 31.8%, 39.4%, 36.1%, and 41.0% in the tangential and 27.7%, 35.2%, 34.3%, and 37.6% in the radial direction, respectively. Similar values of ASE_T and lower values of ASE_R were found for poplar samples, *i.e.* 35.8%, 38.0%, 38.9%, and 39.4% in the tangential and 12.4%, 16.3%, 17.0%, and 26.1% in the radial direction, respectively.

Unfortunately, it was difficult to compare the ASE data of the acetylated wood, because no study dealing with the same wood species was found. Nevertheless, the data are comparable with previous studies published by Hill and Jones (1996) and Rowell *et al.* (2008). Hill and Jones (1996) stated that after the acetylation of Corsican pine (*Pinus nigra*), ASE reached 35% at 10% WPG. Similar results were found by Rowell *et al.* (2008), who reported 34% ASE (at 10% WPG) for southern pine (*Pinus taeda*).

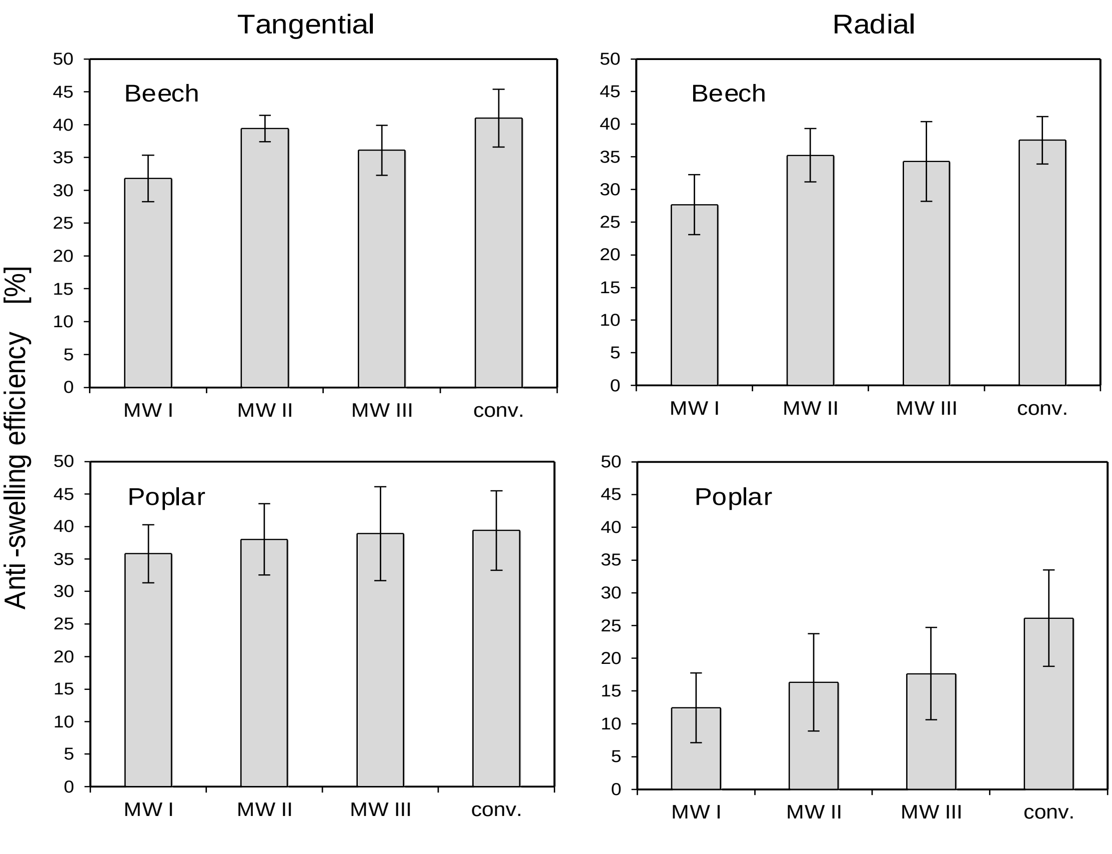


Figure VI. 5 ASE of beech and poplar in tangential and radial directions in 99% relative humidity

It is well known that wood is an anisotropic material with different dimensional changes in different anatomical directions (Kollmann 1951; Boutelje 1962; Stamm 1964; Skaar 1988). According to Niemz *et al.* (1993), swelling in the tangential and radial directions can be expressed by a 2:1 (S_T:S_R) ratio. The tangential and radial swelling ratios ($S_{T,99\%}$:$S_{R,\,99\%}$) for control, MW, and conventional heating were 2.4:1, 2.3:1, and 2.3:1 for beech and 1.9:1, 1.4:1, and 1.6:1 for poplar. From the results, it can be concluded that acetylated beech samples provided negligible swelling ratio improvement. However, poplar samples showed much more significant swelling ratio improvement (S_T:S_R). This improvement is attributed to the lower ASE of poplar in the radial direction (Figure 5) compared to the tangential direction. The acetylated wood (species-dependent) can be therefore considered more homogenous, but anisotropy of swelling remains.

CONCLUSIONS

1. The acetylation of samples using different treatment modes (MW I, MW II, MW III, and conventional heating) provided similar WPG values after chemical reactions (~9%). Retention, expressed by weight in the wood volume, has been suggested as a better indicator of substance uptake. Beech wood had approximately two times higher substance retention compared to poplar, due to its structural and chemical composition.

2. The MW and conventional acetylation positively affected EMC, wood swelling, and consequently the dimensional stability. The improvement in the investigated properties was nearly identical in both types of treatment. Therefore, the efficacy of the acetylation process carried out using MW or conventional heating is comparable.

3. The rate of acetylation increased with an increase of MW power. However, the treatment time (15 and 60 min) did not affect the degree of acetylation. These results were confirmed for all RH levels (30%, 65%, and 99%).

4. The optimum mode of MW acetylation was found at the MW power of 2 kW using 0.1 m·min^{-1} conveyor speed (15 min). This mode resulted in 39.4% ASE_T and 35.2% ASE_R for beech and 38.0% ASE_T and 16.3% ASE_R for poplar samples.

5. MW heating was found to be an efficient rapid acetylation process (15 min, 0.1 $m \cdot min^{-1}$). However, more detailed investigation of the time-dependency of MW heating should be done in the future.

4 CONCLUSIONS

The presented doctoral thesis summarizes the research in field of MW treatment techniques application and presents the experimental results focused on selected wood properties when MW radiation is applied. Results of scientific papers published in peer review journals can be concluded as follows:

- MW radiation can highly influence the permeability of the wood in all anatomical directions of wood. Even though, the axial permeability has been significantly increased for both treatment modes (Paper 1), the transverse permeability is highly influence of used treatment modes (Paper 2). The impregnability (oil uptake) increased when milder (20s interval) mode was used, and decreased with more severe mode (30s interval). Moreover, mechanical properties might be decreased, mainly due to the mechanism of the treatment, *i.e.* presence of micro-cracks in wood structure.

- The material properties and characteristics, *i.e.* mechanical properties (bending strength), moisture content and surface temperature of the pre-dried wood (Paper 3) are highly influenced by process parameters, *i.e.* MW power and time of exposure (conveyer speed), when MW treatment is used. Results indicate that only low MW power (3 kW) and time of exposure (conveyor speed – 0.4 $m \cdot min^{-1}$) can maintain the strength properties of MW treated material on reasonable level, while longer exposure and higher MW power lead to cell-wall delamination and complex failure in the wood structure.

- MW heating was found to be an efficient way in terms of rapid plasticization as a pre-treatment for the wood densification process (Paper 4 and Paper 5). Results showed the influence of the selected power mode on MC decrease and rapid surface temperature increase. Therefore, the power mode had significant effect on plasticization and following densification process. Microscopic structure analysis (Paper 5) revealed that the MW plasticization as well as subsequent densification was, for used MW mode, performed without any fractures of the cell walls.

- Heating by MW can accelerate the chemical reactions in the acetylation process (Paper 6). The results showed that the degree of acetylation increased with a higher MW power. Subsequently, the EMC and dimensional stability were positively affected as result of the chemical transformations. Furthermore, no significant difference was found when conventional (100°C, 60 min) and MW heating (15 min) were compared. Therefore it’s believed that MW heating can reduce the reaction time and make the process of acetylation more effective.

Results of this doctoral thesis show that MW modification has many potentials and can be consider as an appropriate technology for wood industry. Details which led to better understanding of this technology were explained in published scientific papers presented in this study. Considering high interest of international scientists and institutions in field of wood modification increasing demands for material treated/modified by MW could be also expected in future on local production. Therefore, future investigation dealing with application of MW radiation and its effect on permanent or temporary changes in material properties should be done closely (*i.e.* effect of MW exposure on VOC emissions; application of MW heating for curing of glues and coatings; numerical models of MW heating in modification processes; time-dependency of MW radiation on impregnability and microscopic changes in parenchyma cells after MW treatment).

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