

MICROSCOPIC IDENTIFICATION OF CHANGES IN BEECH (*FAGUS SYLVATICA* L.) AND PINE (*PINUS SYLVESTRIS* L.) CELL STRUCTURE AFTER DRYING USING HIGH-FREQUENCY ENERGY OF THE MICROWAVE BAND

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Abstract

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High-frequency energy transfer represents a progressive technology with an increasing range of industrial application. One of the main advantages of microwave technology is the volumetric principle of energy transfer. Based on this fact, the gradients of moisture content and temperature are identical and when the wood is dried it helps transport moisture from porous material and it also helps and transport free water whit lumen of cells. From a practical viewpoint, microwave heating increases the quality of the dried material and reduces the necessary processing. The quality of a dry material is an essential input parameter for other technological procedures and it depends on the deformations created in its cell structure. Therefore, the monitoring of changes brought about during the drying process is necessary. The aim of this study was to identify the changes in the microscopic structure of the wood of beech (*Fagus Sylvatica* L.) and pine (*Pinus Sylvestris* L.) dried using the high-frequency energy of the microwave band. The microscopic structure of a material modified by microwaves was photographed by means of a low-vacuum microscope and then visually compared with the native structure. The results show that the structure of beech and pine wood during the time of the proposed drying regime does not differ considerably from the native structure. This outcome is documented in a digital form and it confirms the harmless character of microwave heating towards wood structure in the conditions of optimum drying parameters.

wood, drying, *Fagus sylvatica*, *Pinus sylvestris*, high-frequency energy, electromagnetic field, microwave heating

Wood drying is one of the most significant industrial processes within the processing industry (Siau, 1984). It directly affects mechanical and physical properties of wood through the loss of moisture and the creation of internal pressure causing tension (Plumb *et al.*, 1985). Generally, drying can be described as an almost concurrent movement of heat and moisture at the local thermodynamic balance at each point of the body, with the simultaneous consideration of the local changes in coefficients determining the movement of mass and energy in the

body (Crank, 1966). The actual heat propagation in wood and materials based on wood is one of transfer phenomena which are of high significance from the point of view of manufacturing technologies and usage (Babiak, 1976). Especially dielectric heating, which is used in wood drying as well, is seen as very interesting for the fields of science and technology. Its principle is based on the transformation of the alternating electromagnetic field energy into heat energy in materials with non-uniform distribution of electric charges in molecules (Kauman, 2006).

Within dielectric heating, both of wood and wood materials, a narrow band of the frequency spectrum is preferred, i.e. the microwave band.

Microwave heating represents a progressive technology with an increasing range of industrial application. Its main advantages are the volumetric principle of energy transfer, the shortening of the time necessary for processing, savings of energy, the reduction of the load on the environment and safe operation. For wood drying, mainly the volumetric heating, which is determined by the identical gradient of moisture and temperature, is important. This concurrent movement of mass and energy helps transport moisture from a porous material and it also helps the from cell cavities and intercellular spaces of wood and transport free water whit lumen of cells. The equipment for industrial applications in Europe is constructed using the worldwide used frequency of 2450 MHz. The usage of this frequency is based on two reasons predominantly. The first reason is physical and it is related to the absorption of microwave energy in polar molecules, i.e. molecules with an asymmetric distribution of electric charges, and its change into kinetic energy with simultaneous creation of heat. In the microwave process, both the intermolecular friction when surpassing intermolecular attractive forces and the hysteresis between the affecting field and the induced electric response due to inertia dependent on the electric charge, mass and shape of molecules are applied (Torgovnikov, 2006). The most frequent representative of a polar molecule is a water molecule, which can appear in all three states of matter in wood. The second reason is technical as the above mentioned frequency solves the issue of the possible interference with telecommunication frequencies.

The microwave energy affecting the anisotropic wood structure causes evaporation and the movement of water molecules in the gaseous state over a complex inhomogeneous structure, and the consequence of this movement can be the deformation of wood cell elements. The origination of deformations is determined by many factors based on the interaction of the microwave radiation and the wood structure, which can, at the same time, be considered the basic parameters of microwave drying of wood. The dielectric behaviour of wood in the alternating electric field is directed by factors, such as frequency, temperature, moisture, density, electric field intensity and wood fibre direction (Torgovnikov, 1993). Fundamental works of degrees value moisture for microwave drying describe Makovíny (1995). From the perspective of the wood anatomical structure, especially the radius of conducting ways seems to be significant for microwave heating. The real process of drying is always a combination of diffusion and permeability. The intended increase in permeability in all directions of wood on the basis of wood structure change is determined by the intensity and the time of the exposure to the microwave radiation and the content of moisture. An intensive exposure and a longer time of exposure of water

molecules in wood structure to microwave radiation lead to the increase in vapour pressure in wood cells and among them and the increase in permeability.

The exceeding of the tensile strength perpendicular to the grain of thin walls of parenchymatic cells results in the dislocation of walls and other wood elements. The border between the intended change in permeability and the use of microwave energy for drying depends on the setting of optimal parameters and the resulting quality of the dry material. The quality of the dry material is a basic input parameter for further technological processes and it is related to the deformations created in the cell structure of the dry wood. Therefore, the monitoring of changes occurring during the process of drying is necessary. When appropriate parameters for drying are kept, the microwave heating contributes to a higher quality of the dried material and it reduces the processing. The aim of the presented paper is to document the microscopic structure of selected wood species treated by microwave heating for a time specified by the proposed drying regime.

MATERIAL AND METHODS

The wood of beech (*Fagus sylvatica* L.) and the wood of pine (*Pinus sylvestris* L.) were selected as the representatives of hardwood and softwood respectively for the microscopic analysis of the changes in wood structure at microscopic and submicroscopic levels caused by high-frequency electromagnetic energy of the microwave band.

Testing specimens for the analysis of wood structure changes were cut from a log from the radial timber and their dimensions were 20 × 20 × 150 mm guaranteeing for a special orthotropic character of the specimens. The specimens did not include any defects which would affect the current of physical fields (moisture and temperature) in a significant way. This means mainly defects like cracks which would damage the examined specimens during the microwave heating and thus also the results of the analysis. Further, knots were considered unacceptable as their structure would cause inhomogeneous heating in their vicinity and this would also negatively affect the final evaluation. Also the decay and the presence of wood-damaging fungi in a more developed stage of growth, pith or reaction wood, or coloured heartwood could not be considered standard properties for the explored identification of changes based on the microwave energy. The chosen angle of fibre deviation from the vertical axis of specimens was 10° at maximum. Two sets of specimens were used; the first set served for reference, for the examination of the initial moisture and for the comparison of the microscopic structure with microwave treated material; the second set served for the actual microscopic identification. The sampling of beech and pine was conducted before drying, while the initial moisture of specimens was around the saturation point which better corresponded to the real conditions during micro-

wave drying. The testing specimens from each species were extracted in compliance with valid norms (ČSN 49 0103, ČSN 49 0123).

The specimens prepared in this way were exposed to electromagnetic radiation of the microwave band by means of the Sencor SMW 7223 microwave oven. The energy source was a CW magnetron with 850 W output which worked at frequency of 2450 MHz. For the purpose of the microscopic identification of changes in wood after the application of microwave heating, 10 specimens of each wood species were tested. The drying of specimens was conducted in the conditions interpreted as two types of drying.

The conditions and parameters of the first type of drying corresponded to microwave drying and the drying was maintained until all specimens achieved a constant weight loss by evaporation. The time of microwave energy exposure in this case was 60 s, followed by 300 s of relaxation and the entire process was applied periodically with the total time of drying 120 min. The specimens in the space of the cavity resonator with material movement were heated up to the temperature of 35–65 °C. The places with a differing intensity of high-frequency electromagnetic field, which are unchangeable in time and cause local increase in temperature and thus an overall inhomogeneity of the field, were eliminated by means of a turning plate inside the Sencor SMW 7223 oven. During the relaxation time, the specimens remained inside the oven so that the accumulated heat and moisture in the heated material could get balanced.

The specimens within the second type of drying were exposed for 60 s and relaxed for 120 s until there were visual signs of scorching. Both types of drying were conducted continually. At the beginning of drying a half of the specimens were placed in the microwave oven at the same time and then taken out in 6-minute intervals and analysed at each interval. In each interval the same number of specimens were taken and their weight loss was determined. During the relaxation stage, the specimens were again left in the switched-off oven for the balancing of accumulated heat and moisture. After the last cycle of drying, the specimens were conditioned in an air-conditioned room to balanced moisture corresponding to the relative air moisture of $60 \pm 5\%$ and air temperature of 20 ± 5 °C.

For the following microscopic analysis itself the specimens with an appropriate (“the best readable”) structure were chosen. To identify the changes in the microscopic structure based on the microwave heating a low-vacuum microscope (MIRA 3 LMU High Resolution Schottky FEG-SEM, producer TESCAN, a.s.) belonging to the Masaryk University in Brno was used within the microscopic centre of the department of anatomy of the Faculty of Medicine. The low-vacuum microscope works at the pressure of 50–100 kPa. When this is used, the specimens do not have to be modified by wet processes which would affect the possible anomalies created by the microwave energy. For the purpose of the presented paper, the specimens were not metal coated and scanning was conducted at the pressure of 50 kPa. For the monitoring by means of the low-vacuum microscope it was necessary to prepare special specimens with specific dimensions out of the microwave treated specimens. Because of the character of this paper, the cells of axially placed anatomical elements of individual wood species were cut in their cross sections, which demanded the removal of the front side of the specimens in the order of micrometers (10–20 µm) and their mounting to the port (a specialized mounting device $d = 10$ mm) which enables fitting the specimens in the microscope chamber. To obtain a readable front the microtome was used as it allowed us to change the geometry of the blade. The microtome was used horizontally while the specimen remained stationary. Because of their small contact area some specimens were treated by a razor with an appropriate secondary pressure. The entire process of the scanning of the microscopic structure was computer-controlled and the output was a graphical image in a digital form (DETECTOR = BSE DET).

RESULTS AND DISCUSSION

The DETECTOR = BSE DET graphical image of the microscopic structure of the beech and pine wood treated by microwaves, using the types and parameters of drying presented in Table I, was analysed for the detail evaluation of changes in the wood structure. The microscopic identification was based on the observation of the modified microscopic structure at the microscopic cross-section. The method uses the identification of the fibrous

I: Drying parameters and the size of specimens

Drying parameters	Type 1	Type 2
Ambient operating temperature	60 °C	60 °C
High-frequency output	850 W	850 W
Frequency	2,450 MHz	2,450 MHz
Regime	CW (continuous wave)	CW (continuous wave)
Output density	32 mW/cm ³	32 mW/cm ³
	60 s exposure, 300 s relaxation	60 s with 120 s relaxation until signs of local scorching!
Specimen dimensions	20 × 20 × 150 mm	20 × 20 × 150 mm

cells of the microwave-treated wood which quantitatively differed from the specific signs of the fibrous cells of native (reference) wood.

The microscopic images of beech (*Fagus Sylvatica* L.) and pine (*Pinus sylvestris* L.) treated by the first type of microwave drying are displayed from the cross-section and with the detail of the cell structure, or the fragments of cell walls. In the images there are apparent burrs caused by the modification by the razor; these cannot be considered dislocations due to the microwave drying of wood. They are secondary deformations of the surface caused by the created pressure and the cutting speed necessary for the preparation of the surface for scanning.

Figures 1–5 show that the resulting structure after the microwave heating treatment does not qualitatively differ from the native structure of the selected wood species. Further, the results show that the chosen type of drying did not change the cellular structure of the selected wood species significantly, even in the places where deformations could be expected. The specific fragments of the cellular structure, most notably bordered pits, remained without any apparent signs of damage.

The graphical images of the microscopic structure of the specimens modified by the second type of drying.

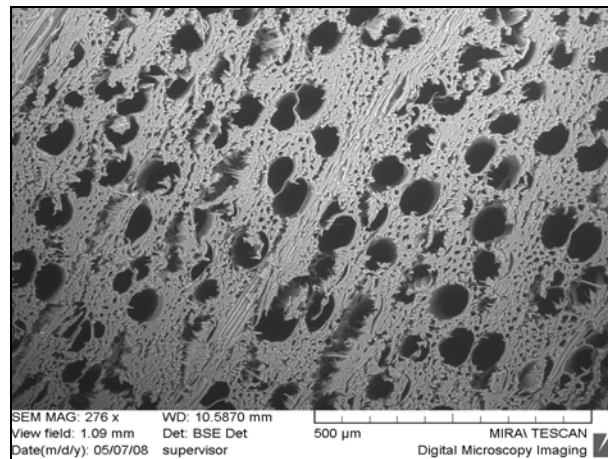
Figures 6–9, which represent the results of the second type of drying with the creation of local scorches, show that the resulting structure does not contain any apparent deformations. The longer exposition to the high-frequency heating caused the overheating of the structure and the plasticisation of lignin. This, in combination with the secondary pressure applied because of the preparation of the surfaces for scanning, caused a minor condensation of the structure, especially in beech (Fig. 6 and Fig. 7).

Microwaves are generated in the source, 'magnetron', and then they are led through the waveguide into the inside of the microwave oven. Once there, the microwaves are reflected from the metal coat and thus they create a place- and time-variable space field. When the testing specimens are inserted, the field gets deformed in dependence on their dielectric properties and volume. Wood does not have a polar character in a significant way, therefore, it hardly gets heated in an electromagnetic field. Its structure is only heated up by the secondary heat from the heated water molecules which are in the mass of wood the best absorbents of the microwave radiation with consequent increase in temperature depending on the content of moisture. The higher amount of water molecules the wood contains, the higher is the value of the loss factor and the temperature gradient in the section increases. Based on the volumetric principle of energy transfer, the gradients of moisture content and temperature are identical, which helps transport moisture from porous material and it also helps and transport free water whit lumen of cells. When the frequency of the applied high-frequency electromagnetic

field is 2 450 MHz, its polarity changes 2.45 billion times a second. A water molecule which has a polar character with its own dipole moment is forced to adapt to the changes immediately, it means to change its orientation. The result is vibration or even rotation in the direction of the field, with the result of forced movement causing the friction of molecules, the change of the state of matter into vapour and the creation of secondary heat.

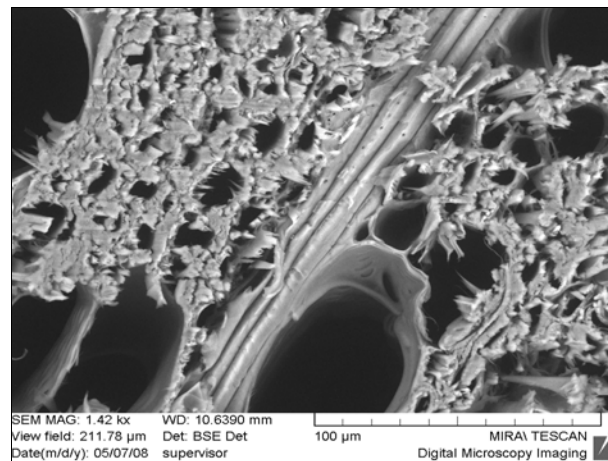
Based on the results we can conclude that the selected types of drying did not have significant impact on the changes in the cell structure of the chosen wood species. The model of the first type represented the drying parameters corresponding to the optimal regime. The actual movement of the moisture field over the complex inhomogeneous and anisotropic structure of wood was accompanied by a phase change, evaporation on the surface of specimens. The moisture gradient during microwave heating of wood by drying depended on the permeability of wood and its moisture conductivity. The analysis was conducted in the cross-section as most diagnostic signs are located there and it is possible to monitor the changes as a transformation of a solid mass. The harmless character of microwave heating was especially apparent for beech, which has the tendency to develop cracks and collapse when dried. In the ambient conditions hydrogen bonds were freed and thus water in wood was freed from the hydroxyl groups. The arising vapour pressure escaped from the wood structure, the assumption being that the vapour pressure will develop tension in the cells and between them and this will cause the deformation of the wood structure. The critical points, where deformations were expected, were the pits of cell elements. However, the results show that no changes due to the microwave energy such as wood cell dislocations or deformations occur. This fact is apparent from the presented microscopic images Fig. 1–5. It can be assumed that the tension created in the wood cells did not exceed the strength limits and did not cause the change in the structure. The explanation mainly lies in the power density and the time of the exposure to microwave radiation. An important parameter is also the initial moisture of the specimens which was at the saturation point. If the loss factor gained high values, the moisture content was high and the time of exposure longer, we would most probably identify changes in the wood structure due to the deformation of cell structures. If we applied these parameters, we would direct the research into the intended change in permeability, which is the subject matter of the future research of the authors. Nevertheless, for the purpose of complexity, it would be appropriate to make an analysis from radial and tangential directions, and aim the attention at parenchymatic cells of pith rays especially.

The model of the second type of drying represented the parameters of the environment of wood microwave heating in which local scorches are developed. There was a longer exposure of specimens



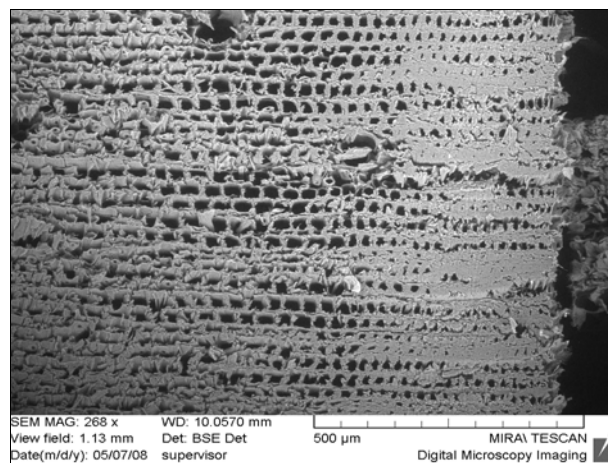
(Nasswettrová, Nikl, 2010)

1: A microscopic image of a beech (*Fagus Sylvatica* L.) specimen cross-section with a visible diffuse-porous structure and an extended strong pith ray



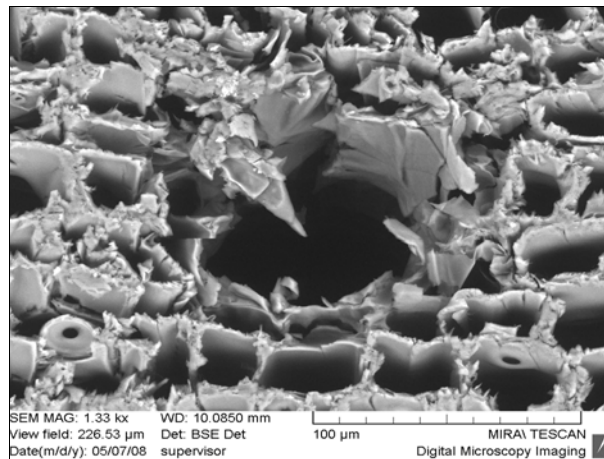
(Nasswettrová, Nikl, 2010)

2: A microscopic image of a beech (*Fagus Sylvatica* L.) cell structure, showing a detail of the layers of the pith ray with simple pits and the inner surfaces of the early-wood vessel with bordered pits on the surface in a line



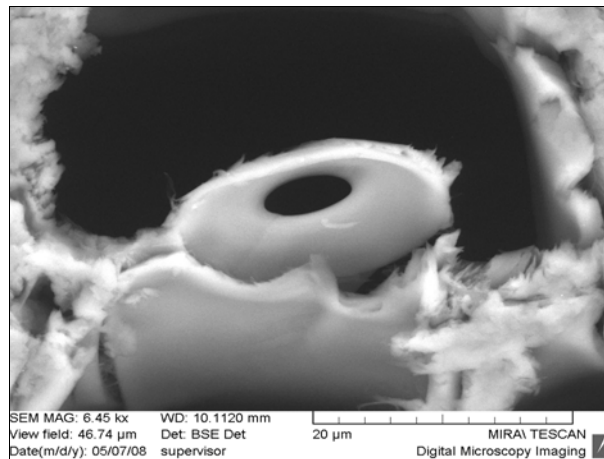
(Nasswettrová, Nikl, 2010)

3: A microscopic image of a pine (*Pinus sylvestris* L.) specimen cross-section, showing the sharp transition between early and late wood and also the prismatic shapes of tracheid cross-sections



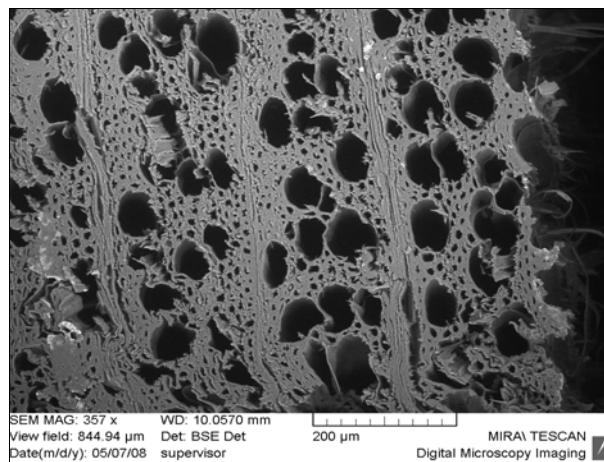
(Nasswettrová, Nikl, 2010)

4: A microscopic image of a pine (*Pinus sylvestris* L.) specimen, showing the detail of a vertical resin canal in late wood without epithelial cells and bordered pits on radial walls of late-wood tracheids



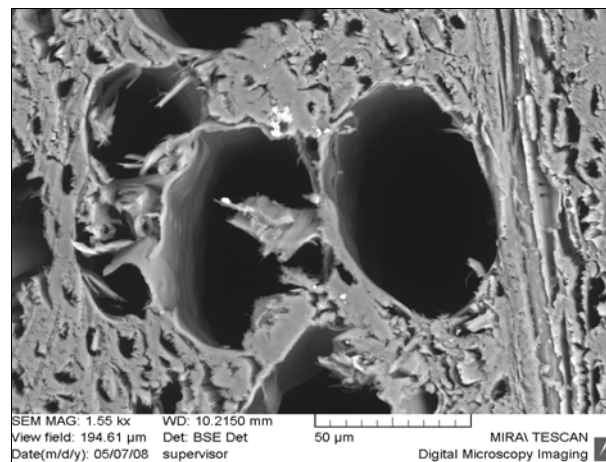
(Nasswettrová, Nikl, 2010)

5: A microscopic image of a pine (*Pinus sylvestris* L.) specimen, showing the detail of the inner surface of a bordered pit with an aperture, without visible dislocation. The bordered pit has been torn from the tracheid surface by secondary pressure applied during the sample preparation.



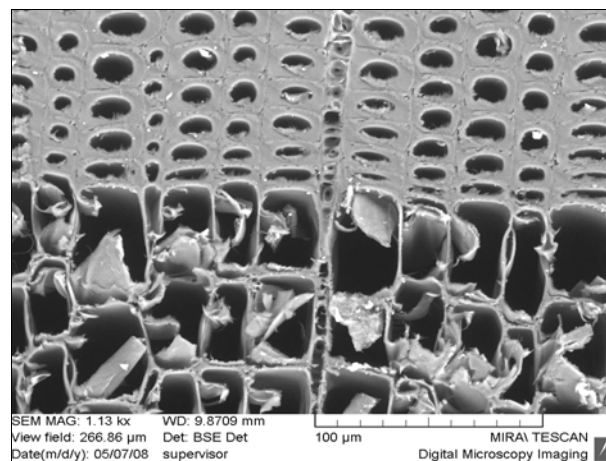
(Nasswettrová, Nikl, 2010)

6: A microscopic image of a beech (*Fagus Sylvatica* L.) specimen cross-section with visible strong pith rays and a diffuse-porous structure



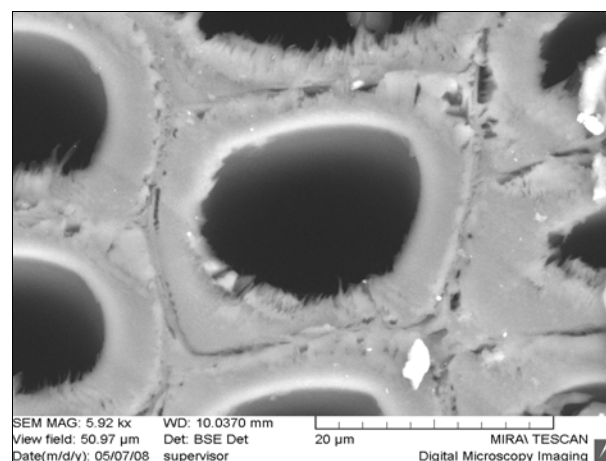
(Nasswettrová, Nikl, 2010)

7: A microscopic image of a beech (*Fagus Sylvatica* L.) specimen, showing the detail of a cross-section of a bundle of vessels



(Nasswettrová, Nikl, 2010)

8: A microscopic image of a pine (*Pinus sylvestris* L.) specimen cross-section, showing the sharp transition between early and late wood and also the cross-section of tracheids. In the middle, a pith ray visible as a one-layer broader band.



(Nasswettrová, Nikl, 2010)

9: A microscopic image of a pine (*Pinus sylvestris* L.) specimen, showing the detail of late-wood tracheids with thicker cell walls. The traces of cell wall structure disruption are not apparent.

in the environment determined by the intensity of the microwave energy. The surface of specimens was prepared and the surface with the visible structure change caused by scorching was analysed. The structure of the cell walls of the specimens of beech (*Fagus sylvatica* L.) and pine (*Pinus sylvestris* L.) did not show any damage caused by the high-frequency microwave energy at the level of microscopic structure, Fig. 6–9. The microwave heating caused the loss of bound water from the wood mass, which decreased the value of the loss factor which expresses the amount of energy transformed into heat. After a longer exposure to the microwave radiation, the plasti-

cisation of lignin and local visual overheating of the structure occurred, which can be attributed to the uneven balance between the small diameter of conducting ways and the relatively large volume of the created vapour escaping from the wood structure. However, the cell elements of the selected wood species from the microscopic point of view remained without apparent anomalies. This conclusion can be explained by the proposed cycles of microwave heating where the moisture gradient, which was created during the decrease of bound water in the cross-section of the dried profile, induced a drying tension without exceeding the wood strength limits.

SUMMARY

The aim of the study was to identify the changes in the wood structure when treated by microwave heating at the level of cell elements. For the microscopic analysis of the changes wood of beech (*Fagus sylvatica* L.) and pine (*Pinus sylvestris* L.) were chosen. Beech wood has a diffuse-porous structure and when dried there is an increased tendency for the appearance of cracks, which enabled us to monitor whether microwave heating is harmless for it. The high-frequency energy of the microwave band was applied to the native structure of wood in two types of drying. The first type represented the optimum parameters of microwave drying; in the second type the specimens were exposed to microwave energy until visible local scorches appeared. The results lead us to the conclusion that deformations of the cell structure in both wood species modified by high-frequency electromagnetic energy of the microwave band for the established drying time were not observed. This outcome is visible in all digital photographs of separate microscopic sections taken by a low-vacuum microscope. The critical points in the native structure of wood, as are especially bordered pits of pine (*Pinus sylvestris* L.) (Fig. 4) remained without any visible damage after microwave rating (Fig. 5). To sum up, we can see that the proposed regime of microwave heating has no significant impact on the changes in the microscopic structure of the examined wood species, therefore, it can be considered harmless to the inhomogeneous structure of wood.

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