

PERMEABILITY AND MECHANICAL BEHAVIOUR OF MICROWAVE PRE-TREATED NORWAY SPRUCE RIPEWOOD

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ABSTRACT

This is a study of the influence of microwave (MW) pre-treatment on the permeability of Norway spruce ripewood (*Picea abies* L. Karst) as it affects its mechanical properties. Specimens were treated under variable moisture content, MW intensity, and impregnation processes. According to the results, the specimens with an initial moisture content of 45–65% exhibited the highest permeability values compared to reference samples. An insignificant difference was found between MW pre-treatments at 2 and 3 kW. Statistically significant results were found after long-time (24h) vacuum-pressure impregnation (LP). The average retention value following LP was 132 kg m^{-3} , which is almost three times greater than the MW-treated groups impregnated in a short-time vacuum-pressure process. The average depth of penetration after LP was 2.0 mm and the proportion of the impregnation area following LP was 17.6%. MW pre-treatment had no effect on the impregnability or the mechanical properties of the wood; other MW regimes are open for further examination.

KEYWORDS: Norway spruce, ripewood, vacuum-pressure, permeability, microwave pre-treatment, retention, mechanical properties.

INTRODUCTION

Norway spruce (*Picea abies* L. Karst.) is one of the most important wood species in Europe for both its economic and ecological aspects, with a long tradition of cultivation and use (Caudullo et al. 2016). These days, its timber is used to manufacture wooden shingles, a traditional roof and walls covering on historical wooden buildings. Nowadays, there is great awareness about our connections with the environment and their impact on our quality of life. There is a trend to make more contact with nature, which has seen a revival of the use of traditional wooden building materials, including wooden shingles (Policinska-Serwa and Jakimowicz 2013). Historically, fir wood was used to manufacture shingles, while larch and spruce are preferred today. Shingles are exposed to continuous action and degradation by

biotic and abiotic factors. When Norway spruce is used it is vacuum-pressure impregnated due to its low natural durability. A crucial disadvantage of the wood is its low permeability, which causes many problems during impregnation. The problem occurs mainly due to the aspiration of bordered pits during drying, when the free water is removed (Fujii et al. 1997, Hacke et al. 2004, Alfredsen et al. 2007, Lehringer 2011, Durmaz et al. 2015). Several modifications to the method, such as bio-incising, mechanical incising, drilling techniques, and steaming have been applied to improve the impregnation properties of wood species that are hard to impregnate (Schwarze et al. 2006, Lehringer et al. 2009, Dashti et al. 2012, Pánek et al. 2013). In order to increase the permeability of various wood species, microwave treatment has been studied by several researchers (Trajkovic 1994, Torgovnikov and Vinden 2009, Brodie 2009, Vinden et al. 2011, Yu et al. 2011, Terziev and Daniel 2013, Dömény et al. 2014). Microwave treatment is based on the principles of electromagnetic radiation and its interaction with polar substances, such as water. The presence of water in wood has a fundamental influence on its dielectric properties (Torgonikov 1993, Paz 2010, Dömény 2017). After felling, the inner non-conductive part of Norway spruce, called ripewood, has an average moisture content of around 34–41%, while the sapwood moisture content is substantially higher. Ripewood is more or less indistinct inner wood of spruce wood, in which the sapwood has aged and presumably died with little of any deposition of the substances associated with heartwood (Haygreen and Bowyer 1982, Glass and Zelinka 2010). According to Li et al. (2009), Torgovnikov and Vinden (2009), Yu et al. (2011), Zhang et al. (2013), and Dömény et al. (2014), microwave treatment increases the permeability by raising water vapor pressure, which delaminates cell walls. Micro- or macro-cracks (depending on the radiation intensity) are formed in the wood structure, and these influence the properties of the wood – increasing its volume and changing its mechanical properties. Treu et al. (2008) increased the permeability of Scots pine heartwood and Norway spruce by MW irradiation using high intensity and 3–14 s exposure. The author stated that the longer exposure caused larger cracks in the wood structure. The effect was more pronounced for pine than for spruce wood specimens. He et al. (2014) and Ramezanpour et al. (2014) found the optimal MW parameters for improving the impregnability of fir wood. Many studies have reported that over-intensive MW treatment causes deterioration in the mechanical properties of wood (Oloyede and Groombridge 2000, Hong-Hai et al. 2005, Machado 2006, Torgovnikov and Vinden 2009). Vinden et al. (2011) irradiated railway sleepers made of pine (*Pinus radiata*) with the aim of increasing the permeability of the wood. However, after MW treatment they recorded a reduction of its bending strength. Koiš et al. (2014) modified Norway spruce (*Picea abies* L.) using MW radiation at various power levels. The results showed that the wood structure of the samples was extensively damaged by high intensities and selected conveyor speeds. Only samples treated with 3 kW MW at 0.4 m·min⁻¹ had modulus of elasticity (MOE) and modulus of rupture (MOR) values comparable to the reference specimens. Gezer et al. (2017) enhanced the impregnation properties of spruce wood by specific MW treatment. Nevertheless, the applied MW pre-treatment reduced the MOR, MOE and compressive strength parallel to the grain by 16–18% compared to reference specimens.

None of the published studies state whether the specimens tested were from sapwood or ripewood. The goal of this study was to contribute to the knowledge of MW pre-treatment

in increasing the permeability of Norway spruce ripewood in the lateral direction. The specific objective of the study was to investigate the effect of MW power and the initial MC of ripewood on improving the permeability of wood without significantly changing its mechanical properties. The results obtained by MW pre-treatment were compared to control specimens impregnated by short-time and long-time vacuum-pressure impregnation.

MATERIALS AND METHODS

Specimens were taken from one-meter-long logs of Norway spruce (*Picea abies* L. Karst.) cut from five trees (Czech Republic; diameters 25–35 cm). The sapwood boundary was marked on the logs while still fresh and the specimens were further cut only from ripewood (RW), which is more difficult to impregnate than sapwood. The dimensions of specimens for MW treatment were determined based on the technical parameters of the microwave device. Specimens from each log with dimensions $45 \times 45 \times 300$ mm (R×T×L) were divided into eight groups to maintain maximum homogeneity with the same density and early and late wood width (Tab. 1); each group contained ten specimens.

Tab. 1: Wood specimens sorted into groups according to MW regime and impregnation cycle (groups 1 and 2 were reference groups).

Group	1	2	3	4
Moisture content (%)	35 ± 2	35 ± 2	35 ± 2	35 ± 2
Microwave power (kW)	-	-	2	3
Conveyor speed (m·min ⁻¹)	-	-	0.4	0.4
Microwave application (s)	-	-	90	90
Microwave energy (kWh·m ⁻³)	-	-	41.2	61.7
Impregnation cycle	SP	LP	SP	SP

Microwave treatment Part 1

The specimens were MW treated in a continuous laboratory device that operated at a frequency of 2.45 GHz and provided adjustable power from 0.6 to 5.0 kW, under conditions stated in Tab. 1, which were chosen on the basis of preliminary tests done by Koiš et al. (2014). In the preliminary test it was found that MW power higher than 4 kW visibly damaged the structure of the tested specimens and, according to Treu and Gjolsjo (2008), MW energy of more than 50 kWh·m⁻³ at a frequency of 2.45 GHz could significantly increase the uptake of preservative after wood modification.

Specimens were MW treated with a single dose of radiation (one passage through the modification chamber). The surface temperature of the MW-treated specimens was measured by contactless infrared thermometer (IR-380, Volcraft, Czech Republic). After MW pre-treatment, cross sections of the specimens were covered with two layers of two-component epoxy resin (Epolex S1300/S7300) to prevent preservative flow in the longitudinal direction. The moisture content was measured by dielectric hygrometer (Wagner L 601-3), giving an MC of $35 \pm 2\%$.

Microwave treatment Part 2

In groups 1–4, with initial moisture content $35 \pm 2\%$, MW pre-treatment, and impregnation, better impregnability was not proven. Therefore, the initial moisture content was increased for groups 5–8. Specimens were immersed in water, and their weight was regularly measured until their moisture content reached the values given in Tab. 2. Subsequently, the same MW treatment was applied as in Part 1.

Tab. 2: Wood specimens sorted into groups according to MC and MW regime.

Group	5	6	7	8
Moisture content (%)	45–65	45–65	75–100	75–100
Microwave power (kW)	2	3	2	3
Conveyor speed ($\text{m}\cdot\text{min}^{-1}$)	0.4	0.4	0.4	0.4
Microwave application (s)	90	90	90	90
Microwave energy ($\text{kWh}\cdot\text{m}^{-3}$)	41.2	61.7	41.2	61.7
Impregnation cycle	SP	SP	SP	SP

Vacuum-pressure impregnation

The wood preservative applied was a 5% solution of Bochemit Forte Profi based on $\text{Cu}_2(\text{OH})_2\text{CO}_3$. The impregnation process was similar to Melcher and Zwiefelhofer (2013) for possible comparison of the results. It was carried out using the Bethell method in a laboratory vacuum-pressure impregnation plant (JHP-1-0072) in two steps as shown in Tab. 3.

Tab. 3: Impregnation process – under short-time (SP) and long-time (LP) vacuum pressure.

Impregnation process steps	1. Short-time vacuum pressure (SP)	2. Long-time vacuum pressure (LP)
Total duration (min)	225	1,440
Course of action (time and pressure)	90 min vacuum (10 kPa)	225 min short-time process
	120 min overpressure (900 kPa)	90 min vacuum (10 kPa)
	15 min vacuum (20 kPa)	900 min overpressure (900 kPa)
	-	225 min short-time process

After this impregnation process, the specimens were removed and weighted. Retention R ($\text{kg}\cdot\text{m}^{-3}$) values were used as an indicator of wood preservative uptake according to Eq. 1:

$$R = \frac{(m_{\text{after impregnation}} - m_{\text{before impregnation}})}{V_{\text{before impregnation}}} \quad (1)$$

Lateral penetration depth and impregnated area

The lateral penetration depth in the treated specimens was indicated by the reaction of copper with dithiooxamide (5% concentration in ethanol) in a gaseous ammonium environment. Impregnated specimens were cut at the middle of their length, the cross sections were coated with dithiooxamide solution and placed in a desiccator with 25% ammonium hydroxide for 10 min. A dark blue color indicated the impregnated area.

The impregnated area as a percentage of the total was measured using the software Image J as follows. The total cross-sectional area of the specimen surface was measured and

converted into 8-bit color depth; using the thresholding method, the impregnated area of the specimens was measured. The impregnated area was calculated thus:

$$n = \frac{S_i}{S} \times 100 (\%) \quad (2)$$

where: n is the impregnated area (%), S_i is impregnated cross-sectional area of the specimen (mm^2), and S is total cross-sectional area of the specimen (mm^2).

The measurement of penetration depth (mm) was taken using software Image J. Ten measurements were taken of each specimen and averaged.

Mechanical properties

After testing, the treated specimens were cut into samples with dimensions $20 \times 20 \times 30$ mm ($R \times T \times L$) and conditioned in a climate chamber at 20°C and 65% relative humidity. A compression test parallel to the grain was performed using a Zwick Z050/TH 3A Universal Testing Machine (Zwick Roell AG, Ulm, Germany). The Young's modulus of elasticity was obtained from the ratio of the stress (σ) to the strain (ϵ) in the linear elastic range bounded by 10% and 40% of the sample strength. The strain was determined based on the change of distance between two isolated points mechanically tracked by clip-on extensometers (Zwick Roell AG, Ulm, Germany) related to their initial distance (10 mm). The ultimate strength (σ_{\max}) was calculated from the maximum load (F_{\max}) at the point of failure related to the cross-sectional area (A) of the unloaded sample.

Statistical analysis

The measured data were processed using Statistica 12 software (StatSoft Inc., USA), evaluated using one-factor analysis of variance ANOVA, and completed with Tukey's honest significance test (HSD test).

RESULTS AND DISCUSSION

Retention

In this experimental study, the main indicator of wood permeability change due to treatment was the uptake of preservative solution in the transversal direction of Norway spruce ripewood. All groups of specimens exposed to various MW treatments exhibited a retention of preservative solution comparable to the reference group (Fig. 1).

Based on one-factor analysis of ANOVA, completed with Tukey's HSD test, variability was found to be not statistically significant. The only statistically significant difference in retention was found for the LP group. Wood specimens treated by MW power of 2 and 3 kW at $\text{MC } 35 \pm 2\%$ showed a slight retention reduction of between 18% and 20.4% compared to the reference group. Specimens with a higher MC of 75–100%, MW-treated at the same power, similarly showed about 4.1% to 13.6% lower retention than the reference group. The specimens with a MC of 45–65% had a slightly increased retention level by 9.5%.

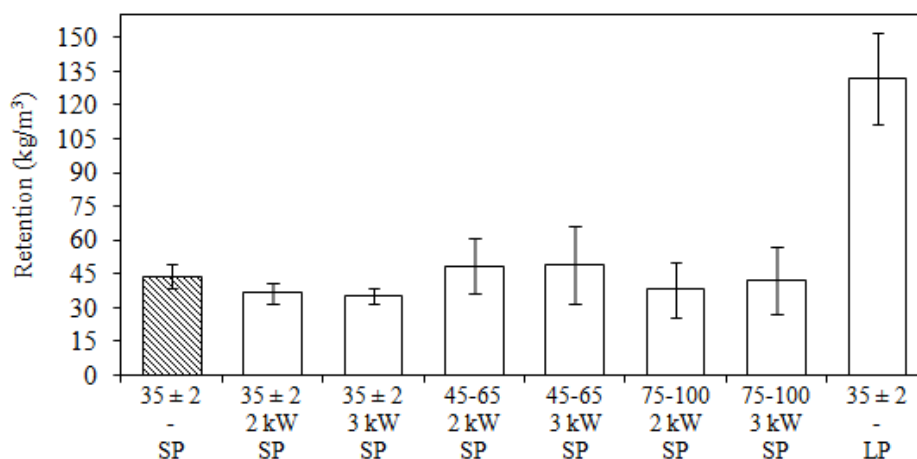


Fig. 1: Preservative solution retention of all groups treated under different conditions: moisture content, MW treatment intensity, impregnation process – short (SP) and long (LP).

The low efficacy of MW treatment was reported earlier by Treu et al. (2008) who exposed Norway spruce specimens to MW treatment repeatedly for short time periods. Even after five iterations, specimens showed no pronounced differences. He et al. (2014) found an input MC of 40–60% the optimal parameter for MW pre-treatment. The microscopic structure of wood after treatment revealed micro-cracks at the intercellular layer of the ray cells and longitudinal tracheids. Among the leading causes of a positive outcome might have been sufficient water vapor pressure in the wood structure, which was able to delaminate cell walls (Li et al. 2009, Yu et al. 2011, Zhang et al. 2013). The effect of a MC higher than 75% on MW radiation on wood may be to facilitate the absorption of microwaves and thus increase evaporation without the pressure necessary for cell-wall delamination. Treu et al. (2008) reported that the use of higher MW power and shorter exposure to MW irradiation could be more effective in producing a retention increase.

Lower initial wood MC before vacuum-pressure impregnation should be considered as it might increase retention. Many authors used an initial MC of around 12% for impregnated specimens, which caused cracks in the wood structure and e.g., altered its mechanical properties. In practice, the vacuum-pressure impregnation process uses the wood MC near the fiber saturation point (FSP) – approximately 30% (Konopka et al. 2018). Most of the authors cited did not address the difference between the sapwood and ripewood of Norway spruce during their experiments, which could be a significant reason for such high retention levels of the preservative solution after MW treatment. The density, hygroscopicity, and especially the permeability of ripewood have been reported to be quite different from those of sapwood (Kollmann and Côté 1968, Kärkkäinen, 2003).

No visible checks or cracks were observed in tested specimens with MC of $35 \pm 2\%$ after exposure to MW pre-treatment at 2 and 3 kW power, but MW power of 3 kW caused slight flow of resin to the surface. The surface temperature of specimens exposed to 2 kW was from 62 to 66°C and the 3 kW group had surface temperatures from 80 to 86°C. The temperature decreased slightly with specimens' increasing initial MC. These temperatures of treated wood specimens might have caused softening of the resins inside the wood structure and blocked the conductive paths in the lateral direction of treated wood.

Since resins are naturally present in a semi-liquid state in the wood, they can become less viscous and move – especially when the surface is heated.

The preservative retention of the ripewood was improved only by prolongation of the impregnation process to 24 hours. The average retention value of the LP group was $132 \text{ kg}\cdot\text{m}^{-3}$, which represents an almost three-fold increase in preservative solution retention over the reference group. A similar experiment was performed by Melcher and Zwiefelhofer (2013), who tried to determine the retention level and the penetration of different refractory wood species impregnated by means of two vacuum-pressure processes in which the SP lasted 2 h and LP lasted 24 h. Their findings were concordant with our experiment. The results of this work showed the dependence of treatment time on the retention of impregnating solution. According to Reinprecht (2008) the retention increase of the impregnating solution over time does not have a linear, but an exponential, dependence, which is produced by compressing the air in the treated wood when it is fully immersed (vacuum-pressure impregnation technology, dipping, immersion, etc.). Consequently, the pressure of the air in the cells of the wood increases and grows over time.

Lateral penetration depth and impregnated area

By vacuum-pressure impregnation using a water-soluble solution it's possible to impregnate spruce sapwood to a depth of 10–20 mm in the radial direction (Paserin 1970), while ripewood has a significantly lower liquid permeability and is classified in the 4th impregnability class (EN 350) as extremely difficult to impregnate with a penetration depth of several millimeters only. The results of lateral penetration depth of all groups after vacuum-pressure impregnation are observed in Fig. 2.

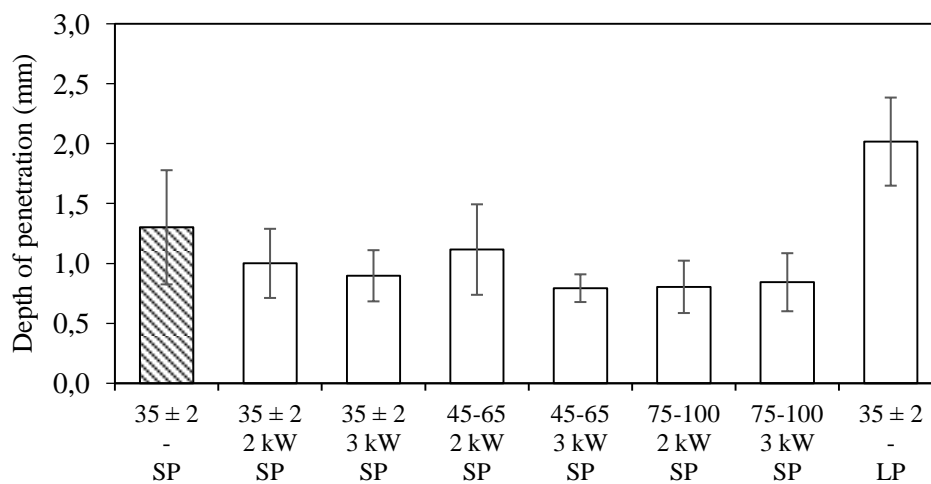


Fig. 2: Depth of penetration of all groups treated under different conditions: moisture content, MW treatment intensity, impregnation process – short (SP) and long (LP).

The only statistically significant lateral penetration depth, according to Tukey's HSD test, was found for the group of specimen's vacuum-pressure impregnated by LP cycle for 24 hours. The penetration depth of LP group was statistically significant in the case of all other groups. The average penetration depth of the LP group was 2.0 mm, which is twice as high as the other groups. All MW treated groups showed less penetration depth of

the preservative solution than the reference group. The control group impregnated with the SP cycle had an average penetration depth of 1.3 mm. The improvements in penetration depth and impregnated area by MW pre-treatment have not been demonstrated in this experiment.

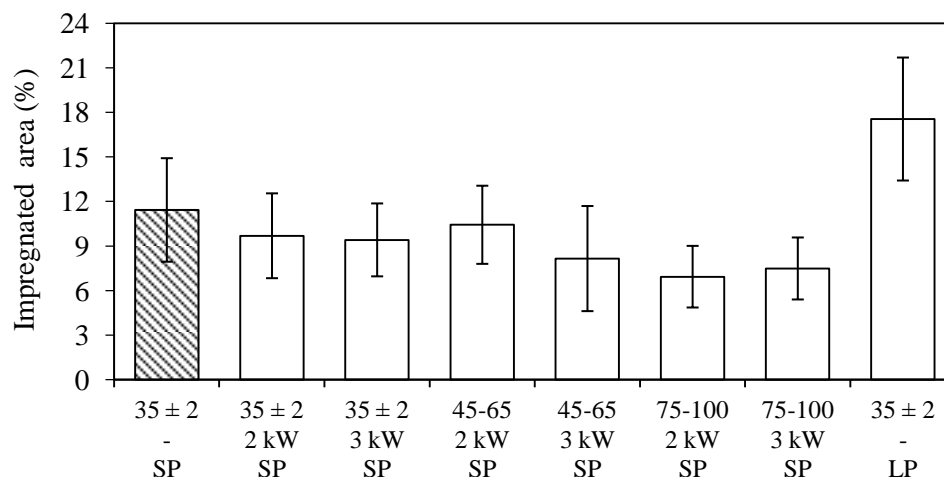


Fig. 3: Impregnated area of all groups treated under different conditions: moisture content, MW treatment intensity, impregnation process – short (SP) and long (LP).

As well as retention and penetration depth, the extent of the impregnation area was most influenced by the modification of the vacuum-pressure impregnation process. The results are observed in Fig. 3. The only statistically significant difference in impregnated area was found for the LP impregnated specimens, which differed from all other groups. The mean values of all MW-treated groups were in the range 9.7–10.0%, while specimens impregnated by LP showed twice the average value of 17.6%. From the obtained results we can say that the rate of impregnated area increase depended on the length of the impregnation process. The difference can be seen in Fig. 4.

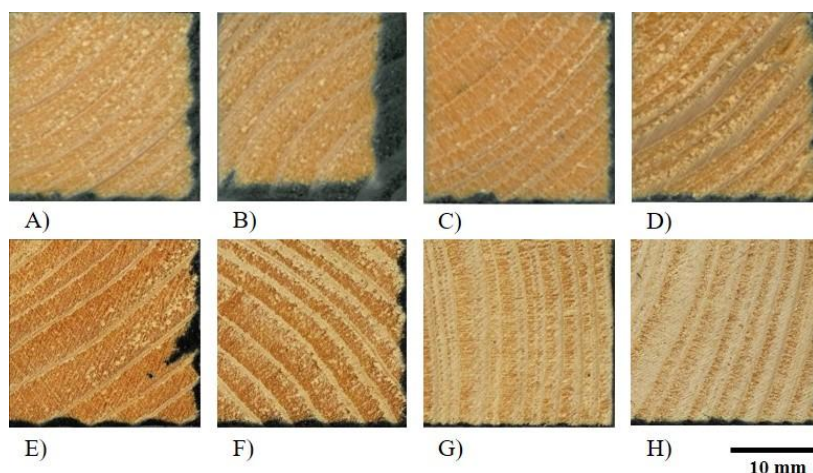


Fig. 4: Cross-sections of impregnated specimens: A) Reference (SP), B) Long process (LP), C) 2kW_MC35_SP, D) 3kW_MC35_SP, E) 2kW_MC40-60_SP, F) 3kW_MC40-60_SP, G) 2kW_MC75-100_SP, H) 3kW_MC75-100_SP.

Mechanical properties

Using a higher MW intensity or prolonged exposure to MW radiation could lead to increased permeability of Norway spruce ripewood. However, it could lead to a significant change in the mechanical properties of the wood. Mechanical testing in compression parallel to the grain was performed only for groups with an input MC of $35 \pm 2\%$, because of statistically insignificant differences in the permeability change in groups with higher initial MC. The results of compression strength (CS) and modulus of elasticity (MOE) can be observed in Tab. 4. The mean values of CS of all groups exposed to a MW intensity of 2 and 3 kW were in the range 45.7–47.6 MPa. The CS was 0.5–4.0% less than the reference group. According to statistical analysis, the influence of MW pre-treatment or impregnation modification (LP) on the mechanical properties of Norway spruce is not statistically significant. Koiš et al. (2014) reported that the wood structure of Norway spruce was destroyed after exposure to MW with an intensity of 3–5 kW and conveyor speed of $0.2 - 0.4 \text{ m} \cdot \text{min}^{-1}$. Oloyede and Groombridge (2000) examined the effect of MW pre-treatment on Caribbean pine. The specimens were exposed to MW with an intensity of 1.6 kW at frequency 2.45 GHz for two intervals. They found that the CS was reduced by 17%. Vinden et al. (2007) increased retention by MW pre-treatment of Sitka spruce wood that significantly reduced the MOR.

When a MW intensity of 2 kW at frequency 2.45 GHz was used, the Young's modulus of elasticity (MOE) increased by 7.3% compared to the reference specimens. The use of higher MW intensity (3 kW) decreased MOE by 12.4%. According to Torgovnikov and Vinden (2009), the high absorption of MW energy by the wood can create an overpressure of water vapor, which leads to the formation of cracks in the cell wall and weakening of the material strength. The authors confirmed this theory during their experiment in which they MW-treated *Pinus radiata* and caused a reduction of wood strength of 4–26 %.

Tab. 4: The compression strength (CS) and the Young's modulus of elasticity (MOE) of the MW treated and impregnated groups with MC of $35 \pm 2\%$.

Group	Reference	2 kW + SP	3 kW + SP	SP	LP
No. of specimens	50	50	50	50	50
MOE (GPa)	15.4 ^a (5.4)	16.6 ^a (5.4)	13.50 ^a (4.0)	15.5 ^a (6.7)	14.6 ^a (5.2)
CS (MPa)	47.6 ^a (7.8)	47.3 ^a (7.0)	45.7 ^a (7.5)	46.3 ^a (7.4)	45.7 ^a (6.5)

*Average standard deviation in parentheses; different letters indicate significant differences among the means according to Tukey's HSD.

CONCLUSIONS

(1) It was found that selected MW pre-treatment and different MC did not have a significant effect on the permeability of shingles made of spruce ripewood. (2) When a long-time process of impregnation was applied, in comparison with all MW-treated groups the retention level was three times higher. The results and statistical evaluation of retention, penetration depth, and level of impregnation area showed a dependency on adjusted time modification of vacuum-pressure impregnation. (3) The MW-treated and reference specimens underwent mechanical testing in which compression strength (CS) parallel to the grain and

Young's modulus of elasticity (MOE) were measured. No statistically significant differences were found. (4) MW treatment did not change the mechanical properties of the wood specimens; however, the retention increase remained unchanged. Modification of the impregnation process had a positive outcome; however, this method of production could be considered economically ineffective.

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