

Increasing the Impregnability of Oriental Spruce Wood via Microwave Pretreatment

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Microwave (MW) pretreatment was used to increase the impregnability of Oriental spruce sapwood (*Picea orientalis* (L.) Link.). Wood samples with moisture contents of 55% and 83% were subjected to different MW energy treatments (1156 MJ/m³ and 1542 MJ/m³). Additionally, the mechanical properties of the treated wood samples were tested to determine the degradation caused by exposure to the MW radiation. According to the test results, the average preservative material retention rates increased by 47.5% and 70% for the samples with initial moisture contents of 55% and 83%, respectively, compared to the reference samples. The mechanical properties of the MW pretreated wood samples decreased within the range of 1.7% to 2.9% in the case of the compression strength; changed within the range of (+)1.9% to (-)6.1% in case of the bending strength; and changed within the range of (+)0.9% to (-)6.2% in case of the modulus of elasticity (MOE). The application of MW energy at different power settings on the samples with similar moisture levels was determined to have no impact on the mechanical properties of treated wood.

Keywords: Microwave pretreatment; Impregnability; Oriental spruce; Mechanical properties

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INTRODUCTION

In the last two decades, microwave (MW) pretreatment has been the subject of extensive research. Microwave pre-treatment can be used as a means of drying, increasing permeability, increasing impregnation, and improving the physical properties of wood (Hansson and Antti 2003; Machado 2006; Torgovnikov and Vinden 2010; Beikricher *et al.* 2013; Terziev and Daniel 2013; Hermoso and Vega 2016).

The use of wood materials with low impregnability, such as Oriental spruce timber, in the industrial and construction sectors has negative ramifications. The service time of the wood material that has not been penetrated sufficiently by preservative liquids is short. Subsequently, significant maintenance and replacement costs can arise. Due to the internal morphological characteristics of Oriental spruce wood, the bordered pits are easily aspirated during drying, which creates resistance to the passage of liquids (Fig. 1). This makes the impregnation of the wood difficult (Şahin 2003). Therefore, it is beneficial to improve the impregnability of Oriental spruce wood before it is employed in applications where preservative use is mandatory.

By damaging the microstructure of the wood, the MW treatment process can increase the radii of the passages that allow liquid transition in the wood and also form micro-cracks that provide additional pathways for liquids to permeate (Mekhtiev and Torgovnikov 2004; Torgovnikov and Vinden 2009; Li *et al.* 2010). When intensive MW energy is directed to fresh sapwood, it is easily absorbed causing high vapor pressure in

tracheid lumena. Due to the high vapor pressure, the sealed off pathways that allow liquid passage are torn open, and the liquid permeability in radial and longitudinal direction increases significantly (Torgovnikov and Vinden 2009).

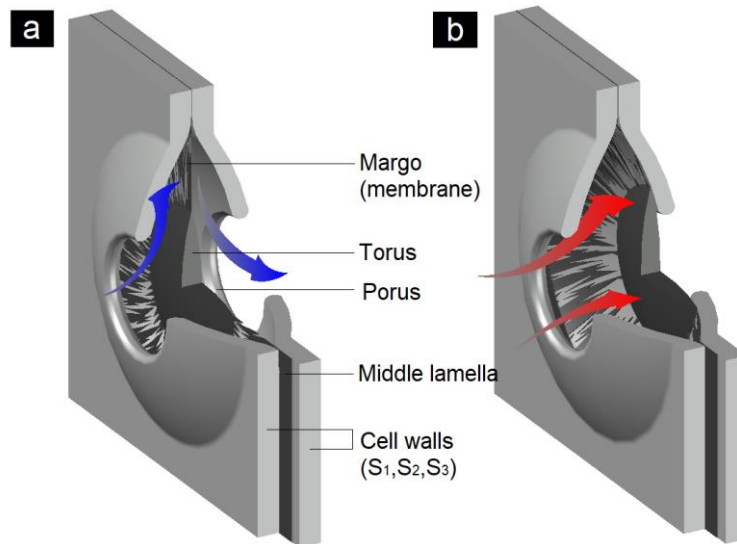


Fig. 1. Normal bordered pits allow passage of liquids between tracheids (a). In aspirated bordered pits, the torus shifts from its central position (to the right or left), covers the porus, and restricts passage of liquids (b).

A review of the literature on MW treatment demonstrates that the changes in the mechanical and physical properties are dependent on the moisture content, the MW energy intensity, and the wood species (Hong-Hai *et al.* 2005; Machado 2006; Torgovnikov and Vinden 2006; 2009; Vinden *et al.* 2011; Torgovnikov *et al.* 2015; Hermoso and Vega 2016).

Hermoso and Vega (2016) investigated the effect of MW treatment on the mechanical properties of wood by applying MW energy at different intensities on air-dried wood samples with a 12% moisture content and a moisture content greater than 30%. Both the elasticity modulus values that were within the range of 23% to 33% and the bending strength values within the range of 19% to 34% decreased. Xu *et al.* (2015) found that MW pretreatment on poplar wood resulted in an increased transverse permeability, which had a positive effect on the impregnation level. Machado (2006) exposed oak (*Quercus pyrenaica*) wood samples to MW energy at durations of 5 min and 10 min and reported strength losses of approximately 10% and approximately 20%, respectively.

Based on the results from the relevant literature, it is apparent that the MW treatment process can improve the impregnability of wood, but at the detriment of the mechanical properties. Studies on MW treatment also show that factors such as the wood type, the MW energy intensity, and the initial moisture content of wood have significant effects on the final product. Therefore, more studies should be carried out to employ various test parameters during MW treatment with different types of wood.

The goal of this study was to investigate the impact of the MW energy intensity, the MW power, and the initial moisture content on the impregnability and the mechanical properties of Oriental spruce wood. Additionally, the applied MW energy values per unit of removed moisture content were calculated to help guide future studies for preparing MW treatment procedures.

EXPERIMENTAL

Materials

Oriental spruce (*Picea orientalis* (L.) Link.) was chosen as the research material for this study. Six-hundred samples with the dimensions of 20 mm × 20 mm × 300 mm (R × T × L) were prepared from the sapwood of the fresh logs. Special attention was paid to make sure that the annual rings were parallel to the length of the samples (Fig. 2).

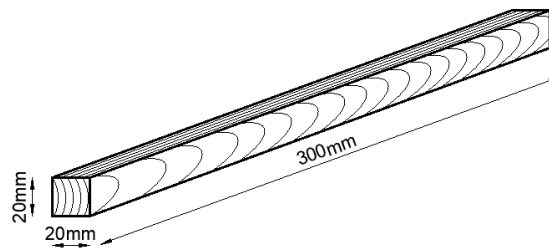


Fig. 2. Dimensions of Oriental spruce sapwood samples

Methods

Sample preparation

The freshly cut samples were weighed using an analytical balance with a range up to 200 g and a precision of 1/1000 g. The weights of the samples were recorded on their surfaces. In order to calculate the moisture content of the samples, some of the samples were picked by the random selection method and dried to a constant weight in an oven. After determining the oven-dry weight, the moisture content of the samples was calculated according to Eq. 1:

$$\text{Moisture content (\%)} = \frac{\text{Original mass} - \text{Oven dry mass}}{\text{Oven dry mass}} \times 100 \quad (1)$$

Next, the samples were divided into a reference group, a low moisture (LM) content group, and a high moisture (HM) content group. Samples in the LM group had an average moisture content of 55%, while the samples in the HM group had an average moisture content of 83%. Depending on the power level that was employed during the MW treatment, both the LM and HM groups were then sub-divided into two groups and assigned suffixes. The groups of samples that were subjected to 925 W and 1850 W MW treatment power levels were named “925” and “1850”, respectively. During the grouping based on the MW power, the utmost care was given to achieve homogeneous moisture content values within the groups.

Table 1. Sample Groups Based on the Moisture Content and the Power Level

Treatment Group	MW Power (W)	Avg. Moisture Content (%)	Std. dev.	Total Number of Samples
LM925	925	55	7.00	84
LM1850	1850	55	7.09	84
HM925	925	83	9.59	84
HM1850	1850	83	9.80	84
Reference	-	12	-	144

MW pretreatment

The MW treatment was carried out at a frequency of 2.45 GHz using a MW heating device (Model CM1929; Samsung, Seoul, South Korea) with a maximum output power of 1850 W. The interior volume of the device was 26 L (37 cm × 37 cm × 19 cm).

According to Torgovnikov and Vinden (2009), energy within the range of 250 to 1200 MJ/m³ should be applied on wood for MW modification. According to Samani *et al.* (2019), the MW energy requirement depends on the size and the initial moisture content of the specimen. It has been reported that the application of MW energy at a low intensity results in insufficient modification, and structural damage in the wood can be encountered in case of applying a high MW energy intensity (Torgovnikov and Vinden 2010). The MW energy intensity calculation can be seen in Eq. 2,

$$E = \frac{P \times T}{V} \quad (2)$$

where E is the applied energy (MJ/m³), P is the MW power (W), T is total exposure time for each treatment (hour), and V is the sample volume for each treatment (m³). The P and V variables remained constant throughout each treatment, so T was the only variable that changed to impact E .

The MW pretreatment testing was carried out with 48 samples after considering the power output capacity of the MW oven, the volume of the samples in each MW pretreatment session, and the moisture content of the samples. The preliminary test results showed that there was a 10% variation between the moisture contents of the treated samples within the same batch, even when the batch was composed of samples with very similar initial moisture contents. Based on these findings of the trial runs and considering other MW applications in the literature, the target final moisture content value was set at 18% ± 5% to achieve optimum modification. The MW pretreatment program can be seen in Table 2. The energy calculations were made based on 12 samples for each batch. Each sample had a volume of 1.440 cm³.

Table 2. MW Pretreatment Program

Treatment Group	Exposure Time (s)*	Applied Energy (MJ/m ³)	Number of Samples in the Batch	Total Number of Samples
LM925	1800	1156	12	84
LM1850	900	1156	12	84
HM925	2400	1542	12	84
HM1850	1200	1542	12	84

*The exposure time for 12 samples ($V = 1.440 \text{ cm}^3$) during each treatment.

Following the MW pretreatment program, each group was completed in seven treatment runs with 12 samples in each batch ($7 \times 12 = 84$ samples in total). In order to prevent the continuous MW energy application from damaging the wood structure during the treatment runs, four separate 30-second cooling intervals were taken. At the end of each run, the samples were cooled for 10 min and then weighed.

Upon completion of the MW pretreatment runs, the weight percent loss (WPL) values were calculated according to Eq. 3,

$$WPL (\%) = \frac{M_2 - M_1}{M_1} \times 100 \quad (3)$$

where M_1 is the initial weight of the sample prior to the MW pretreatment (g) and M_2 is the

final weight at the end of the MW pretreatment (g).

Impregnation method

In this research, the commercial wood preservative Tanalith-E 8000 (Lonza Wood Protection, Alpharetta, GA, USA) was used as the preservative material. The light green and odorless substance had a pH value of 7 and a density of 1.04 g/cm³. The product was applied as a solution with 3% concentration that was prepared by the manufacturer.

All the 20 mm × 20 mm × 300 mm samples were conditioned at a temperature of 20 °C ± 2 °C and 65% ± 5% relative humidity for approximately 2 weeks to reach the equilibrium moisture content prior to the impregnation. The weights of the conditioned samples were measured (T_1) and recorded on the surface of the samples.

The pressure method was used as the impregnation method. According to the pressure method, the samples were soaked in the solution under 4 bar pressure for 60 min. Finally, by applying vacuum on the samples, the solution remaining on the wood material surface was removed. Any leftover solution on the surface of the samples was cleaned again and the weights of the samples (T_2) were determined. After the impregnation, the preservative retention and percent of the uptake were calculated according to Eqs. 4 and 5,

$$R = \frac{(T_2 - T_1) \times C}{V} \times 100 \quad (4)$$

$$\text{Uptake } (\Delta\%) = \frac{(T_2 - T_1)}{T_1} \times 100 \quad (5)$$

where R is the amount of wood preservative that was retained in the wood specimen (kg/m³), T_2 is the sample weight after the impregnation (g), T_1 is the sample weight before the impregnation (g), C is the mass (g) of preservative in 100 g of the 3% treating solution, and V is volume of the sample (cm³).

Determination of mechanical properties

Certain mechanical properties of the modified samples, including the modulus of rupture (MOR), the modulus of elasticity (MOE), and the compression strength (CS) parallel to the grain were determined according to the TS standards 2474 (1976), 2478 (1976), and 2595 (1977), respectively. The 20 mm × 20 mm × 300 mm samples were conditioned at a temperature of 20 °C ± 2 °C and 65% ± 5% relative humidity for approximately 15 d to reach the equilibrium moisture content prior to the MOR tests.

RESULTS AND DISCUSSION

The MW Pretreatment

The WPL values ($\Delta\%$) for the MW pretreated samples are summarized in Table 3. The WPL values were within the range of 22% to 24% for the LM groups and 30% to 33% for the HM groups.

The average WPL value of the samples in the LM groups was 23% after applying 1156 MJ/m³ energy, while the average WPL value for the samples in the HM groups was 31.5% after applying 1542 MJ/m³ energy. According to these results, the energy used to obtain 1% WPL was 50.3 MJ/m³ and 49.0 MJ/m³ for the LM and the HM groups, respectively (Energy/WPL).

Table 3. WPL Values after the MW Pretreatment

Treatment Group	Applied Energy (MJ/m ³)	Weight Percent Loss ($\Delta\%$)	Std. dev.	Number of Samples
LM925	1156	22	4.01	84
LM1850	1156	24	4.59	84
HM925	1542	30	4.01	84
HM1850	1542	33	4.43	84

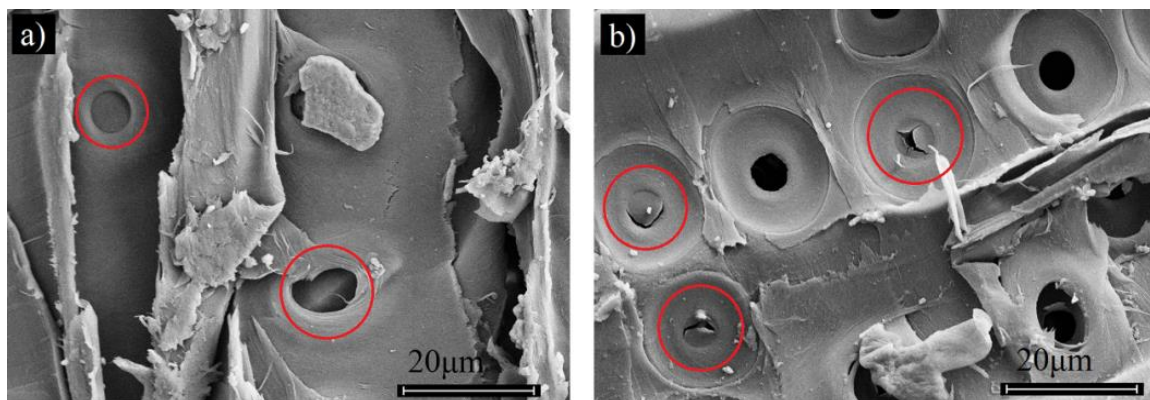
Retention

The retention results for the impregnated reference and the MW-treated samples are given in Table 4. The average retention value for the reference samples was 10.05 kg/m³. The average retention value for the MW pretreated sample groups were 14.76 kg/m³, 14.92 kg/m³, 17.30 kg/m³, and 16.90 kg/m³ for the LM925, LM1850, HM925, and HM1850 groups, respectively.

Table 4. Retention Values Observed at the end of the Impregnation Process

Treatment Group	Number of Samples	Uptake ($\Delta\%$)	Retention (kg/m ³)	Increase in Rate of Retention (%)
Reference	144	84.88	10.05	-
LM925	84	116.04	14.76	47
LM1850	84	117.11	14.92	48
HM925	84	134.11	17.30	72
HM1850	84	133.51	16.90	68

On average, the retention rates of the LM and HM groups increased by 47.5% and 70%, respectively, compared with the reference samples. These results indicate an improvement in the impregnability of the material upon MW treatment. However, higher retention rates have been reported in other relevant studies. The type of wood material and the MW energy density are important factors on these results. The opening of closed passages at high vapor pressure and formation of micro capillary cracks are important effects for explaining the increase in the retention rate after the MW treatment (Vinden *et al.* 2003; Mekhtiev and Torgovnikov 2004; Torgovnikov and Vinden 2009; 2010; Li *et al.* 2010; Torgovnikov *et al.* 2015).

**Fig. 3.** SEM images of a) control and b) MW-treated samples (3500× mag., radial section).

The scanning electron microscope (SEM) images of the control and MW-treated samples in Fig. 3 suggest that the high vapor pressure during the treatment caused partial or complete rupture of the aspirated bordered pits. The lower increase in retention rate that was encountered in this study can be assumed to be due to either solely rupture of the pit membranes in cell walls without any micro-crack formation, or both phenomena occurring only to limited extents. Jiang *et al.* (2006) reported that the moisture content of wood close to the fiber saturation point (FSP) is ideal conditions for wood permeability in MW treatment. In another study, He *et al.* (2017) reported similar results. According to the authors, when MW energy is applied to wood with high moisture content, a sufficient vapor pressure does not occur in the wood internal structure, as a significant part of the energy is absorbed by the mass of moisture. The results of these studies showed that the energy applied to wood with moisture above FSP is absorbed by the free water, reducing the effect of creating micro-macro cracks in the wood interior structure. If the FSP of the Oriental spruce wood used in this study is considered to be ~ 35%, there will be ~ 20% free water in the LM group with 55% moisture content and ~ 48% free water in the HM group with 83% moisture content. It can be concluded that a significant part of the applied energy is used in the evaporation of free water instead of the bound water in the wood cell wall, due to the high free water participation rates in the examples of MW treatment groups (LM, HM). Considering all the results and expressions, it can be said that the MW energy applied in this study did not have a significant effect on the wood structure to create micro-macro cracks, and the resulting increase in retention was due to the rupture of the aspiration of bordered pits, as determined in the SEM observations.

Mechanical Properties

Results of the CS tests

Table 5 presents the average CS value, the standard deviation, and the strength change rates. The average CS value of the reference samples was 49.68 N/mm², whereas the same values calculated for the MW-pretreated samples were between 1.7% and 2.9% lower than the reference samples. Considering the relationship between the applied MW power and the strength change rate, the 925W and 1850W groups had an average strength loss of 2.7% and 1.8%, respectively.

Table 5. Results of the CS Tests

Treatment Group	Applied Energy (MJ/m ³)	CS (N/mm ²)	Std. dev.	Change Rate of Strength (%)
Reference	-	49.68	4.46	-
LM925	1156	48.48	5.55	-2.4
LM1850	1156	48.85	5.12	-1.7
HM925	1542	48.23	5.73	-2.9
HM1850	1542	48.73	5.53	-1.9

Results of the MOR tests

Table 6 shows the average MOR values, along with the standard deviation and the percentage of strength change. The average MOR value of the reference samples were 58.05 N/mm². According to the results in Table 6, the average MOR values for the LM and HM groups were 1.5% greater and 4.8% lower than the average MOR values of the reference group, respectively.

Table 6. Results of the MOR Tests

Treatment Group	Applied Energy (MJ/m ³)	MOR (N/mm ²)	Std. dev.	Change Rate of Strength (%)
Reference	-	58.05	7.09	-
LM925	1156	58.68	9.47	+1.1
LM1850	1156	59.16	9.41	+1.9
HM925	1542	54.51	9.19	-6.1
HM1850	1542	56.05	9.20	-3.5

Results of the MOE tests

Table 7 shows the average MOE values for the sample groups, along with the standard deviation and the strength change. The average MOE value of the reference samples was 6094 N/mm². According to Table 7, the average MOE values for the LM and HM groups were 0.7% greater and 4.8% lower, respectively, compared to the average MOE value of the reference group.

Table 7. Results of the MOE tests

Treatment Group	Applied Energy (MJ/m ³)	MOE (N/mm ²)	Std. dev.	Change Rate of Strength (%)
Reference	-	6094	805	-
LM925	1156	6124	1297	+0.5
LM1850	1156	6150	1124	+0.9
HM925	1542	5893	1229	-3.3
HM1850	1542	5714	1297	-6.2

The values for the measured CS parallel to the grains decrease within the range of 1.7% to 2.9% when compared to results from other studies. Hermoso and Vega (2016) reported pressure resistance losses of 42.0% and 53.1% in two different sample groups of *Eucalyptus* wood that were subjected to high intensity MW treatment. Machado (2006) observed approximately 10% and 20% reductions in pressure resistance when MW energy at two different intensities were applied on oak wood samples.

The reduction in the MOR and the bending MOE values, within the range of 3.5% to 6.2% were very low compared to the reported losses in other studies. However, these losses were only observed for the HM groups that were exposed to the MW energy for longer periods of time. Improvements within the range of 0.5% to 1.9% were observed for the LM groups after the MW treatment. Hermoso and Vega (2016) reported bending resistance losses of 23.3% and 52.6% for the LM and HM groups, respectively.

When the overall losses in the strength of the material were interpreted, the calculated losses in this study can be considered as insignificantly small. In similar studies, increases in retention rates along with degradations in mechanical properties were commonly attributed to microcracks that formed during the MW modification (Torgovnikov and Vinden 2006; Torgovnikov *et al.* 2015; Terziev *et al.* 2020; Weng *et al.* 2020). Negligible decreases in mechanical properties resulting in this study support the view that micro-macro cracks in the wood structure were of minor importance. According to Terziev *et al.* (2020), intense MW modification transform wood into a highly porous material with plenty cavities, dramatically changes the physical and mechanical properties

of wood. According to Weng *et al.* (2020), the high intensity MW process causes macroscopic cracks that may adversely affect the appearance quality and mechanical properties of the wood. Although the high retention increase occurs as a result of the damages in the wood structure, the preservative may not cause a high level of protective effect similarly, since the preservative cannot stay adhered to wood over long periods.

CONCLUSIONS

1. Power levels of 925 W and 1850 W were employed to determine the effect of the microwave (MW) power factor on the impregnability of oriental spruce wood. The MW pretreatment applications at 1850 W resulted in a greater weight percent loss (WPL) than the applications at 925 W. Additionally, greater losses in the pressure resistance and the bending resistance were observed for the samples that were subjected to the 925 W treatment. Based on these results, it can be suggested that it is more beneficial to apply higher power for a shorter duration in MW pretreatments, to preserve the mechanical properties and shorten the treatment time.
2. The literature has reported on the role that MW energy treatment on wood has in creating high vapor pressure to rupture the previously sealed off passages, which are important for liquid transition, to increase the retention. In accordance with the literature, the average retention increased by 70% for the HM groups that were subjected to MW treatment. If the MW pretreatment is carried out to increase the impregnability, better results may be achieved with the use of fresh wood with a high moisture content.
3. The insignificant losses in the mechanical properties upon the MW treatments may be an indication for estimating the optimum parameters for MW treatment programs. As a result of employing 1156 MJ/m³ MW energy for the group with the 55% moisture content and 1542 MJ/m³ MW energy for the group with the 83% moisture content, high extents of improvement in retention were achieved with a minimal effect on the mechanical properties. In the future studies, if a higher retention increase is desired, an increase in the energy density can be made by if lower mechanical properties are acceptable.
4. Providing that a very high increase is desired in the method of increasing impregnability by microwave treatment, rupturing must occur in the elements of wood structure besides opening the liquid passage paths. The condition for this may be to apply appropriate MW energy by choosing a value close to the fiber saturation point of the initial moisture content of the wood, or to apply very intense MW energy to wood with high moisture content. However, it is necessary to accept the significant decrease in mechanical properties as a result of this process.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Karabuk University Scientific Research Project Coordination Office, Grant No. KBÜ-BAP-14/2-DS-043.

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Article submitted: December 25, 2020; Peer review completed: January 31, 2021;
Revised version received and accepted: February 11, 2021; Published: February 12, 2021.
DOI: 10.15376/biores.16.2.2513-2523