Measuring the Modulus of Elasticity of Thermally Treated Spruce Wood using the Ultrasound and Resonance Methods

Tomáš Holeček,[a] Miroslav Gašparík,[a,]* Rastislav Lagaňa,[b] Vlastimil Borůvka,[a] and Eliška Oberhofnerová [c]

The effect of thermal treatment temperatures on the dynamic and static modulus of elasticity (MOE) of Norway spruce wood (*Picea abies* (L.) H. Karst.) was evaluated. The dynamic MOE was measured using ultrasound and resonance methods in the longitudinal and transverse directions. The static MOE was determined by the three-point bending test. The dynamic MOE values determined by the ultrasound method were higher than the static MOE values in each case. As the temperature of the thermal treatment increased, the difference between the dynamic and static MOE values decreased. The MOE increased with increasing temperature, and it was more pronounced on a tangential surface. Increasing the sensor distance had a positive effect on the correlation between the static and dynamic MOE, and the effect from the increased temperature decreased. Measurement by the resonance method showed twice as high MOE values in the transverse direction than in the longitudinal direction. The thermal treatment caused a significant decrease in the MOE only in the transverse direction, and the differences were insignificant in the longitudinal direction. The dynamic MOE values measured by the resonance method were higher than the static MOE values but slightly lower than the values measured by the ultrasound method.

Keywords: Modulus of elasticity; Thermal treatment; Spruce wood; Ultrasound method; Resonance method

*Contact information: a: Department of Wood Processing, Faculty of Forestry and Wood Sciences, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521, Czech Republic; b: Department of Wood Science, Faculty of Wood Sciences and Technology, Technical University in Zvolen, T. G. Masaryka 2117/24, Zvolen, 960 53, Slovakia; c: Department of Wood Products and Wood Constructions, Faculty of Forestry and Wood Sciences, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521, Czech Republic; *Corresponding author: gathiss@gmail.com*

INTRODUCTION

Although wood is a versatile natural material with desirable properties, there are some manufacturing processes that change the structure of wood and thus, its characteristic properties. Some of these changes are targeted (plasticization, densification, pressing, impregnation, *etc*.), which improve certain properties of the wood and its resistance (Borůvka *et al*. 2015). However, the side effects of processes such as drying, surface embossing, and others may result in unwanted changes (cracks, staining, surface roughness, *etc*.) (Gaff and Gáborík 2014).

A typical example of such processes is thermal treatment, which changes the physical and mechanical properties of the wood through high temperatures with oxidation or an inert atmosphere (Ates *et al*. 2009; Rousset *et al*. 2009). Thermal treatment

temperatures are usually in the range of 150 °C to 280 °C (Martinka *et al*. 2014), and significant changes occur when the temperature exceeds 200 °C. Thermal treatment improves wood durability, resistance to biological factors and weathering, hygroscopicity, and dimensional stability (Allegretti *et al*. 2008; Murata *et al*. 2013; Navickas *et al*. 2015), but it significantly reduces its mechanical properties (Weiland and Guyonnet 2003; Borrega and Kärenlampi 2008; Niemz *et al*. 2010; Kačík *et al*. 2012; Kubš *et al*. 2016). Depending on the temperature and thermal treatment conditions, the mechanical properties are reduced by 10% to 30%.

The modulus of elasticity (MOE) is an essential characteristic that describes the wood stiffness. A high MOE means that the wood has a high resistance to deformation (Liang and Fu 2007). The MOE is one of the most basic bending properties, and it is also influenced by the ambient temperature, just like other mechanical properties of wood (Ayrilmis *et al*. 2010). This fact is important in terms of the use of wood elements for construction purposes in an environment with varying temperature conditions. Although the static MOE is the most commonly used characteristic, the dynamic MOE is also an important characteristic. While static stress is based on an increase in the load force over a specific period of time, dynamic stress takes place immediately (Bal and Bektaş 2012). Some studies have found significant linear correlation between the dynamic and static MOE of wood (Liu *et al*. 2006; Sales *et al*. 2011; Baillères *et al*. 2012).

The ultrasound and resonance methods are suitable non-destructive methods that determine the modulus of elasticity (MOE) and modulus of rupture (MOR) of wood. A large number of studies have used these methods and compared them with the static method of determining MOE (Vafai *et al*. 1976; Zhang *et al*. 2009; Bučar and Bučar 2011; Liu *et al*. 2014). Both methods are based on measuring the shock wave transition time through wood and then calculating the sound wave velocity. Increased temperature and moisture content reduce the sound wave velocity in wood depending on the effect that these variables have on the MOE and density (Halabe *et al*. 1997; Karsulovic *et al*. 2000; Kretschmann 2010). The sound wave velocity of wood is closely linked to the density and MOE of wood. The wood species and grain direction also affect the sound wave velocity of wood (Mishiro 1996; de Oliveira and Sales 2006). The highest sound wave velocity is measured in the longitudinal direction, which is due to the longitudinal arrangement of most wood elements. According to Halabe *et al.* (1993), the sound wave velocity in the tangential and radial directions is only 30% and 45% of the value achieved in the longitudinal direction, respectively.

This work assessed the effect of thermal treatment on the MOE of spruce wood. The thermal treatment was carried out at various treatment temperatures (165 °C, 180 °C, and 210 °C). The dynamic and static MOE values were determined using the non-destructive ultrasound and resonance methods, as well as a bending test. The ultrasound and resonance methods were used to measure the MOE in the longitudinal and transverse direction.

EXPERIMENTAL

Materials

Eighty-five-year-old Norway spruce (*Picea abies* (L.) H. Karst.), used in this study, was harvested from the central region of the Czech Republic, near Kostelec nad Černými lesy, east of Prague. The mature wood zones selected for samples were located

in the middle distance between the pith and bark at a height from 1.5 m to 5 m from the stump. Suitable parts were cut into 3.5-m-long planks. These planks were cut into 650-mm-long pieces. Samples that were defectless (without knots, cracks, or reaction wood, and annual growth rings slope < 5°) and with dimensions of 20 × 20 × 650 mm were used for the experiments (Fig. 1). The samples were conditioned for 2 months in a HCP 108 humidity chamber (Memmert, Schwabach, Germany) (humidity (ϕ) = 65 ± 3% and temperature (t) = 20 ± 2 °C) to achieve a 12% equilibrium moisture content (EMC).

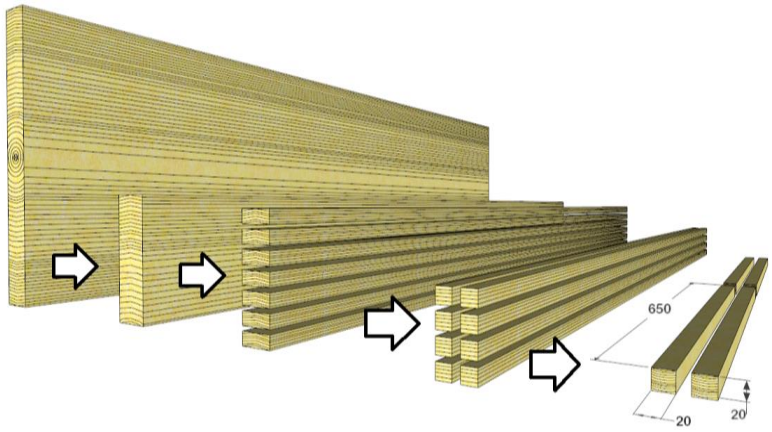

Fig. 1. Preparation of testing samples

After conditioning, the samples were divided into two groups. The first group contained samples intended for the thermal treatment and the second group was untreated wood, which were used as reference samples. The whole investigation involved 112 samples.

Procedure

Thermal treatment

The wood samples were placed into the S400/03 thermal chamber (LAC Ltd., Rajhrad, Czech Republic). The thermal chamber specifications are listed in Table 1. The thermal treatment was based on the ThermoWood® process principle developed by VTT, Finland, and carried out in an atmosphere with a protective medium (in this case, a water curtain) to prevent overheating and burning. The whole thermal treatment process is divided into three basic parts (Fig. 2).

Table 1. Parameters of the Thermal Chamber

Technical Parameters	
Moisture content of wood	12%
Filling capacity of TW furnace	0.38 m^3
Power consumption	6 kWh
Maximum temperature reached	165 °C, 180 °C, and 210 °C

The first stage was drying, which was started at an initial temperature (ambient temperature) of 20 °C. The first step heated the wood to 40 °C at a set rate of 20 °C/h. When this temperature was reached, the temperature increase rate was reduced to 12

°C/h, until the desired temperature of 130 °C was reached. This stage was the same for each type of treatment.

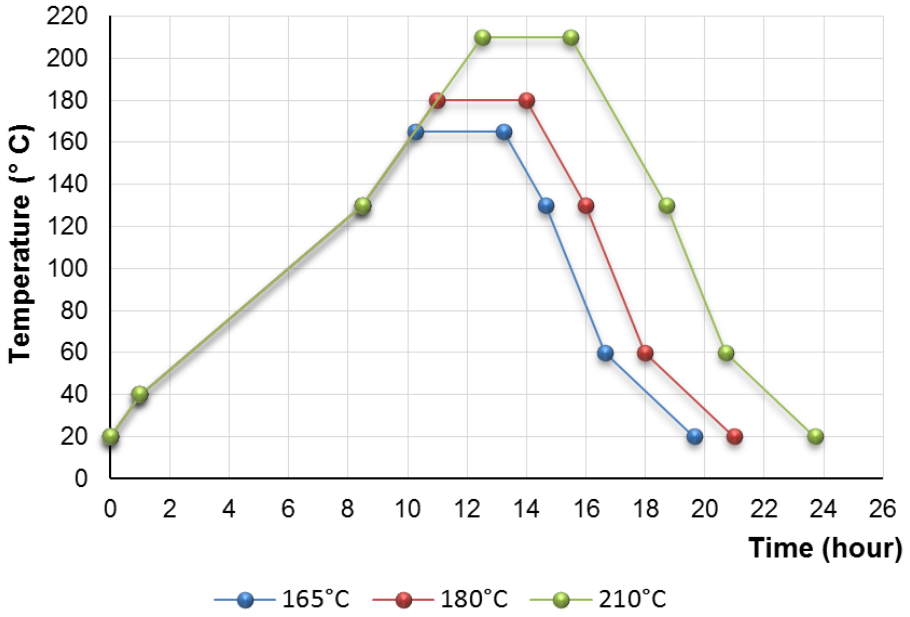

Fig. 2. Thermal treatment phases

The second stage of the thermal treatment began at 130 °C with a set temperature increase rate of 20 °C/h until the desired temperature was reached. When this temperature was reached, the temperature was maintained in the chamber for 3 h. This time was of equal duration for all treatment types. The third stage consisted of cooling and relaxing the material. It began by cooling the temperature to 130 °C at a set rate of 25 °C/h, and then the temperature was subsequently reduced to 60 °C at a rate of 35 °C/h. The humidity was also gradually increased in the chamber, so that the samples did not suffer temperature and humidity stress upon their removal from the chamber.

The thermally modified samples were then conditioned (ϕ = 65 ± 3% and T = 20 ± 2 °C) for 3 weeks. After this, the native and thermally modified samples were prepared for measurements.

Methods

Non-destructive ultrasound measurement

Ultrasound measuring is a method of determining the dynamic MOE of wood by measuring the sound wave passage time through a material. Two types of MOE measurements were carried out in the longitudinal direction using the Ultrasonic Timer ultrasound apparatus (Fakopp Enterprise Bt., Agfalva, Hungary) with a frequency range of 15 to 300 kHz and two 45-kHz triangle-shaped piezoelectric sensors. The measurement was performed with a sensor distance of 300 mm on the tangential and radial surfaces. This type of measurement was performed both before and after the thermal treatment on the same samples. The difference between the resulting moduli showed the effect of the various thermal treatment temperatures on the dynamic MOE.

The second measuring method was carried out on a radial surface, with sensor distances of 60, 100, 140, 500, and 615 mm, with the measurement center in the middle

of the samples (Fig. 3). The values of the dynamic MOE after the thermal treatment were compared with a group of values measured from the untreated (reference) samples.

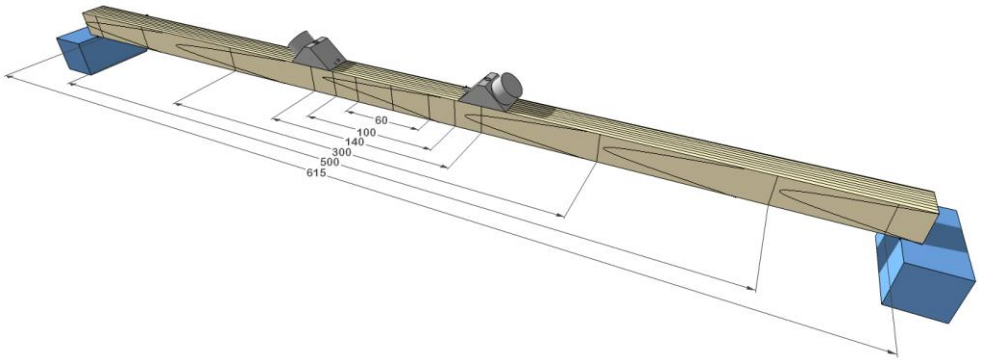

Fig. 3. Principle of the ultrasound MOE measurement

The measuring was performed in accordance with the instructions of the Ultrasonic Timer measuring instrument. The sample was supported on each end by polyurethane foam supports so as to not interfere with the position of the sensor or to affect the passage of the wave through the material. The sensors were gradually attached to the prepared guidelines at various distances and constantly pressed against the material. The device recorded the wave passage time through the material in μs. The sound wave velocity was calculated according to Eq. 1,

$$v = \frac{l}{(t - K)} \tag{1}$$

where v is the sound wave velocity (m/s), l is the distance between the transducers (m), t is the measured sound wave transit time (s), and K is the time correction (s).

Determining the time correction was very important because it greatly influenced the test results. The time correction was determined empirically. The data measured on one sample was placed in a graph and translated into a linear function. The resulting regression equation indicated the correction time of that particular sample (Fig. 4). This was the time required for the wave passage with no distance between the sensors.

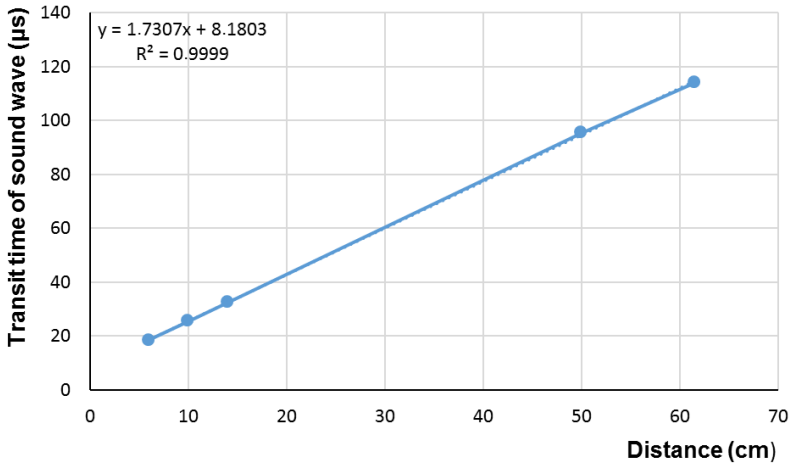

Fig. 4. Principle of the time correction determination

The resulting correction could be introduced into the equation to calculate the speed, or the average correction value for the given group could be determined. In the case of this study, a specific time correction value for each sample was substituted.

The resulting sound wave velocity was substituted into the equation for the dynamic MOE according to Moavenzadeh (1990) and Eq. 2,

$$MOE_{dyn} = v^2 \rho \quad (2)$$

where MOE_{dyn} is the dynamic MOE (MPa), v is the sound wave velocity (m/s), and ρ is the wood density (kg/m^3).

Non-destructive resonance measurement

The MOE was determined using the resonance (stress) wave velocity, where the frequency was induced by hitting the sample with an impact hammer. The samples had rubber supports at a span of $L \times 0.22$, *i.e.*, 143 mm, due to the regular wave propagation. The hammer used to induce the vibrations weighed between 0.5% and 5% of the weight of the sample.

When measuring in the transverse direction (bending dynamic modulus – $MOE_{T_{dyn}}$), first an ECM8000 microphone (Behringer Spezielle Studiotechnik GmbH, Willich, Germany) with an UR22 amplifier (Steinberg GmbH, Hamburg, Germany) was placed approximately 2 mm from the surface of the sample. The hammer hit the sample next to the microphone on the radial surface, and the microphone recorded the frequency of the response (Fig. 5), which was transferred to a PC with software that contained Fast Fourier Transform (FFT) analysis.

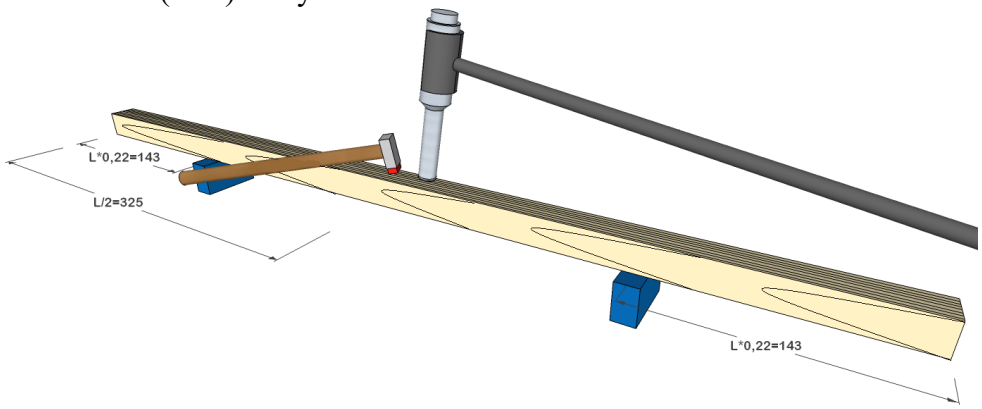

Fig. 5. Principle of the resonance MOE measurement in the transverse direction

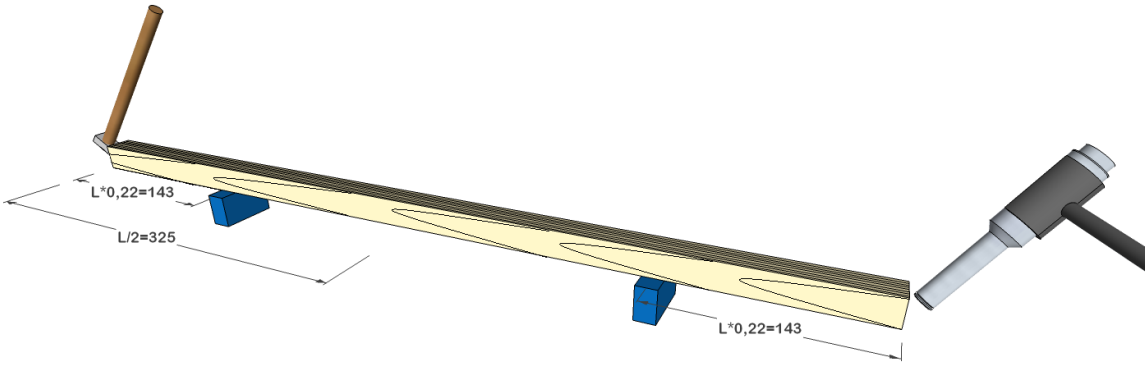

Fig. 6. Principle of the resonance MOE measurement in the longitudinal direction

In the longitudinal direction (longitudinal dynamic modulus – MOE_{Ldyn}), the microphone was placed on one end of the sample, and the hammer hit the sample on the other side on the cross-section (end) of the sample (Fig. 6). The microphone on the other end recorded the frequency and transmitted it directly to the PC.

The dynamic MOE in the transverse direction was calculated according to Eq. 3,

$$MOE_{T\,dyn} = \left(\frac{2 f_n}{\gamma\ \pi}\right)\frac{m l^3}{I} \quad (3)$$

where MOE_{Tdyn} is the dynamic MOE in the transverse direction (MPa), f_n is the frequency of the transverse vibration (Hz), γ is the correction coefficient for the transverse direction, m is the weight of the sample (kg), l is the length of the sample (m), and I is the moment of inertia (m^4).

The dynamic MOE in the longitudinal direction was calculated according to Moavenzadeh (1990) and Eq. 4,

$$MOE_{L\,dyn} = v^2 \rho \quad (4)$$

where MOE_{Ldyn} is the dynamic MOE in the longitudinal direction (MPa), ρ is the wood density (kg/m^3), and v is the resonance wave velocity (m/s).

The resonance wave velocity was calculated according to Ilic (2003) and Eq. 5,

$$v = 2 l f_n \quad (5)$$

where v is the resonance wave velocity (m/s), l is the length of the sample (m), and f_n is the frequency of the longitudinal vibration (Hz).

Destructive static measurement

The static bending test was performed on a universal testing machine UTS (TIRA GmbH, Schalkau, Germany) at 50 kN. The test was performed in accordance with EN 310 (1993) with a support span of 240 mm (12 times the thickness of the sample) and one force was applied at the center of the supports (Fig. 7). The sample was loaded in the tangential direction, *i.e.*, the force was acting on the radial surface. The static bending test was performed on the same samples that were used to measure the dynamic MOE with the ultrasound and resonance methods to make the comparison of each MOE as accurate as possible.

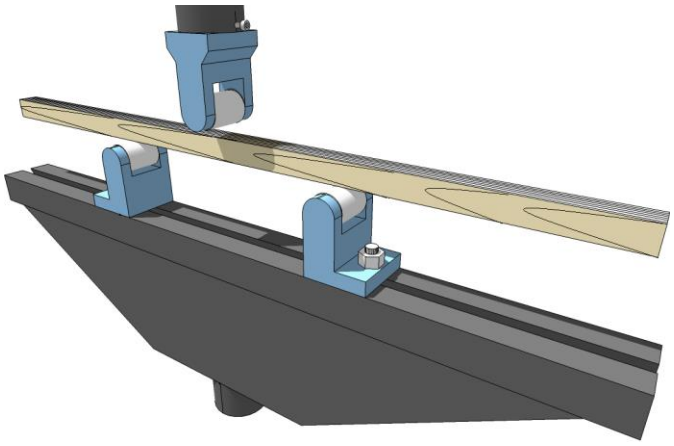

Fig. 7. Principle of the static MOE measurement

With one force acting in the center of the supports, the static MOE was calculated according to EN 310 (1993) and Eq. 6,

$$MOE_{stat} = \frac{1}{4} \cdot \frac{\Delta F l_0^3}{b h^3 \Delta y} \quad (6)$$

where MOE_{stat} is the static MOE (MPa), ΔF is the increment of load on the straight line portion of the load-deflection curve (N), l_0 is the distance between the centers of the supports (mm), b is the width of the sample (mm), h is the thickness of the sample (mm), and Δy is the increment of deflection at the mid-length of the sample corresponding to ΔF (mm).

Evaluation and calculation

The influence of factors on the MOE was statistically evaluated using MANOVA, mainly by the Fisher's F-test and Duncan's test, with STATISTICA 13 software (Statsoft Inc., Tulsa, OK, USA). The results were evaluated using a 95% confidence interval, which reflected a significance level of 0.05 ($P < 0.05$).

The density was calculated according to Eq. 7 from ISO 13061-2 (2014),

$$\rho_w = \frac{m_w}{V_w} \quad (7)$$

where ρ_w is the density of the sample at a certain moisture content w (kg/m^3), m_w is the mass (weight) of the sample at a certain moisture content w (kg), and V_w is the volume of the sample at a certain moisture content w (m^3).

The moisture content of the samples was determined according to ISO 13061-1 (2014) and Eq. 8,

$$w = \frac{m_w - m_0}{m_0} * 100 \quad (8)$$

where w is the moisture content of the sample (%), m_w is the mass (weight) of the sample at a certain moisture content w (kg), and m_0 is the mass (weight) of the oven-dried sample (kg).

RESULTS AND DISCUSSION

Density

Wood density depends on the thermal treatment temperature and generally decreases when the thermal treatment temperature increases. This effect was confirmed by experimental data; there were no statistically significant differences in the density at various thermal treatment temperatures (Table 2, Fig. 8).

The untreated spruce wood had an average density of 467 kg/m^3. As the thermal treatment temperature increased, this density gradually decreased. The decrease in the density was caused not only by a rapid decrease in the moisture content created by the reduced weight and size but also by the chemical changes in the wood caused by the elevated temperatures. Several studies have shown that the thermal treatment of spruce wood increases the proportion of lignin, slightly decreases the degree of polymerization

of the cellulose, and substantially reduces the proportion of hemicellulose (Windeisen and Wegener 2008; Gonzáles-Peña *et al*. 2009; Rousset *et al*. 2009; Belleville *et al*. 2013; Heigenmoser *et al*. 2013). These changes are most significant above 220 °C.

Table 2. Effect of the Thermal Treatment on the Density

Monitored Factor	Sum of Squares	Degrees of Freedom	Variance	Fisher's F - Test	Significance Level P
Intercept	23,213,421.7	1	23,213,421.7	6,201.8	0.000
Thermal treatment	5,688.5	3	1,896.2	0.507	0.679
Error	404,247.7	108	3743.0		

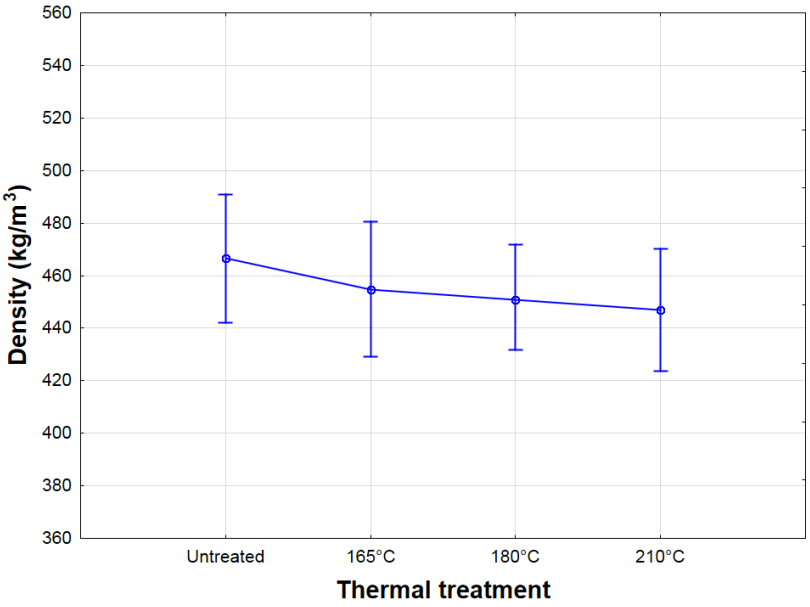

Fig. 8. Influence of thermal treatment on density

Ultrasound Method

The dynamic MOE on the tangential and radial surfaces was measured with the ultrasound method at a constant sensor distance of 300 mm. Table 3 contains the statistical evaluation of the effect of the thermal treatment and the surface of the wood (anatomical direction) on the dynamic MOE. Both factors, as well as their interaction, were statistically significant ($P < 0.05$).

Table 3. Effect of Individual Factors on the MOE_{dyn}

Monitored Factor	Sum of Squares	Degrees of Freedom	Variance	Fisher's F - Test	Significance Level P
Intercept	107,720307.0	1	107,720307.0	56.308	0.000
Thermal treatment	308,006843.0	2	154,003421.0	80.501	0.000
Wood surface	14,249828.9	1	14,249828.9	7.449	0.007
Thermal treatment × Wood surface	12,525476.5	2	6,262738.3	3.274	0.040
Error	309,916334.0	162	1,913063.79		

The effect of thermal treatment was evaluated using the difference of two values before and after the thermal treatment. This method allowed the accurate monitoring of one specific factor without the interference of additional variables, which can occur when comparing reference samples.

Figure 9 shows that the effect of thermal treatment was significant for all temperatures. The individual curves in the graph show a slight increase in the MOE_{dyn} at a treatment temperature of 165 °C; MOE_{dyn} increased by 3.8% on the tangential surface and 5.3% on the radial surface. This increase was due to the lower moisture content of the wood after thermal treatment, which had a more significant effect than the actual chemical changes in the wood. At 180 °C, there was a visible decrease in the MOE of 1.1% on the radial surface and 3% on the tangential surface. This decrease was due to the greater chemical changes in the wood, which began to disrupt its integrity. The highest temperature of 210 °C showed a significant decrease in the MOE_{dyn}, which was 13.9% on the radial surface and 21.6% on the tangential surface.

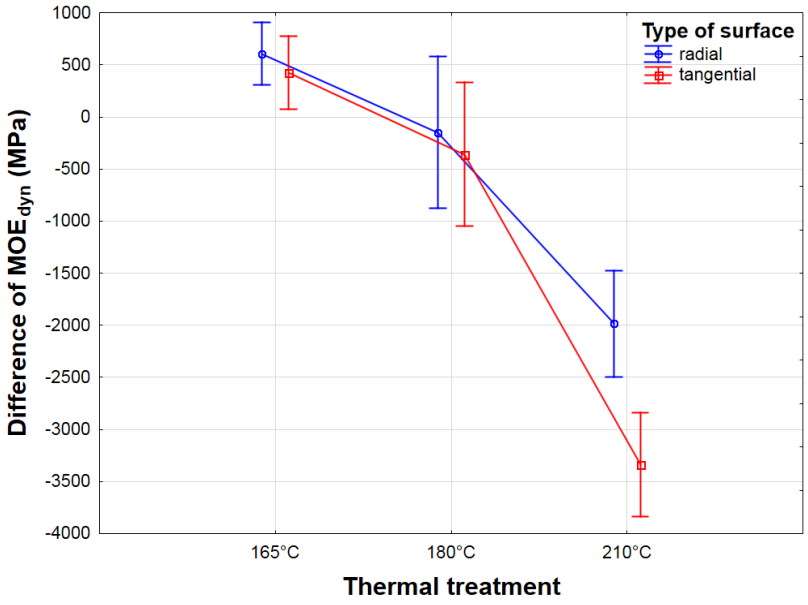

Fig. 9. Difference of MOE_{dyn} before and after the thermal treatment for different surfaces

Table 4. Duncan's Multiple Range Test of Difference in MOE_{dyn} before and after the Thermal Treatment for the Radial and Tangential Surfaces

Thermal Treatment Temperature	Wood Surface	165 °C Radial	165 °C Tangential	180 °C Radial	180 °C Tangential	210 °C Radial	210 °C Tangential
Mean of MOE_{dyn} difference (MPa)		606.2	424.8	-148.7	-360.2	-1,986.0	-3,341.0
165 °C	Radial		0.624	0.052	0.015	0.000	0.000
165 °C	Tangential	0.624		0.121	0.043	0.000	0.000
180 °C	Radial	0.052	0.121		0.567	0.000	0.000
180 °C	Tangential	0.015	0.043	0.567		0.000	0.000
210 °C	Radial	0.000	0.000	0.000	0.000		0.000
210 °C	Tangential	0.000	0.000	0.000	0.000	0.000	

The negative values represent a decrease in the MOE_{dyn}.

The Duncan's test (Table 4) showed that at 165 °C and 180 °C there was no significant difference between the MOE_{dyn} on the tangential and radial surfaces. However, there was a significant difference at 210 °C. Measurements on the tangential surface showed substantial changes that increased as the temperature increased.

Effect of thermal treatment on wood is strictly dependent on resistance of its basic components. Hemicelluloses are the weakest components that are degraded first at temperatures up to 200 °C, because of its low degree of polymerisation, higher branching, different composition, and amorphous structure. The degradation of the hemicelluloses is characterized by the formation of organic acids (Byrne and Nagle 1997; Weiland and Guyonnet 2003). Amorphous cellulose is also changing quite rapidly, similarly to hemicelluloses. Chemical modifications of polysaccharides lead to the loss of the hydroxyl groups and the weak the ability to react with water. Crystalline cellulose is resistant, such that temperatures up to 300 °C influence it to a small extent (Kim *et al*. 2005). Lignin, which is the most durable component of wood, is a phenolic polymer with a heterogeneous structure and content of different types of linkage. It reacts to thermal treatment in various ways within the temperature range. After the initial depolymerisation of lignin, there is a thermo-assisted acidolytic cleavage of ether bonds, which causes condensation and elimination reactions (Yildiz *et al*. 2006; Brodin *et al*. 2010).

Table 5. Effect of Individual Factors on the MOE_{dyn}

Monitored Factor	Sum of Squares	Degrees of Freedom	Variance	Fisher's F - Test	Significance Level P
Intercept	1.486020E+11	1	1.486020E+11	13,369.47	0.000
Thermal treatment	9.777202E+07	3	3.259067E+07	2.93	0.033
Type of MOE	4.566193E+09	5	9.132386E+08	82.16	0.000
Thermal treatment × Type of MOE	3.666962E+07	15	2.444641E+06	0.22	0.999
Error	7.202539E+09	648	1.111503E+07		

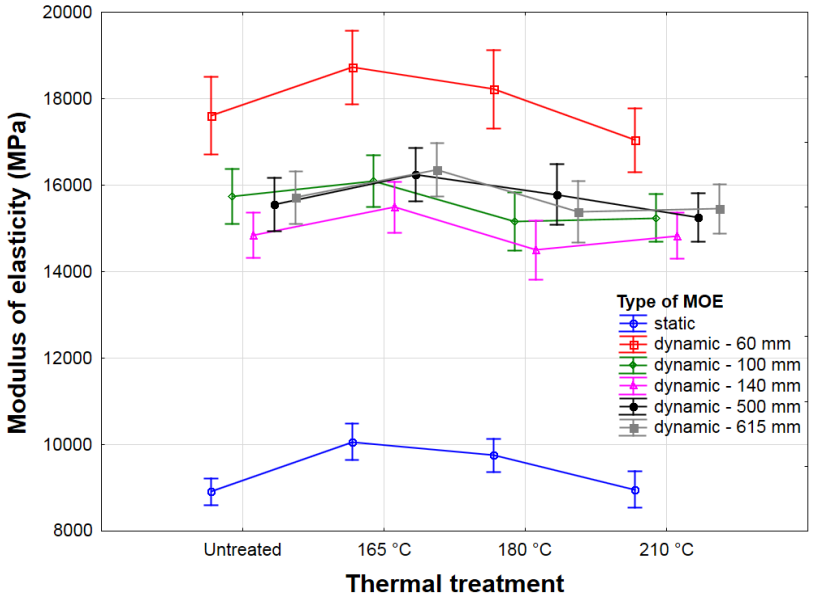

Fig. 10. Influence of the thermal treatment and measuring distance on the MOE_{dyn} and MOE_{stat}

The second experiment determined the MOE_{dyn} on the radial surface with sensor distances of 60, 100, 140, 500, and 615 mm. The thermal treatment and the type of MOE were statistically significant ($P < 0.05$), but their interaction was not (Table 5; Fig. 10).

In the thermally modified wood at 165 °C and 180 °C, slightly higher MOE values were found in comparison to the untreated wood. A similar trend was observed at a measuring distance of 60 mm. For all other distances, the highest values were only found for the wood treated at 165 °C. In all cases, the MOE_{dyn} reached higher values than the MOE_{stat}, by approximately 78.4% in the untreated wood and 66.7% in the thermally treated wood.

To compare the correlation of the dynamic MOE measured by ultrasound at various differences and the static MOE, the relationship between these two variables was charted in a correlation matrix (Table 6). A higher correlation coefficient indicated a greater the dependency was between them. If one of the variables changed, the other one changed correspondingly and *vice versa*.

Table 6. Correlation between MOE_{stat} and MOE_{dyn} for Various Measuring Distances

	Thermal Treatment	Measuring Distance				
		60 (mm)	100 (mm)	140 (mm)	500 (mm)	615 (mm)
Untreated	-	0.786	0.879	0.915	0.891	0.907
Thermally treated	165 °C	0.851	0.892	0.893	0.915	0.915
	180 °C	0.682	0.716	0.732	0.656	0.746
	210 °C	0.662	0.630	0.630	0.689	0.676

The most suitable distance for measuring the MOE_{dyn} in the untreated spruce wood was 140 mm between the sensors (Fig. 11). As the thermal treatment temperature increased, the correlation rapidly decreased (Table 6). In general, the distance of 615 mm was most suitable for the ultrasound measuring, and the measured values had the greatest correlation with the MOE_{stat}.

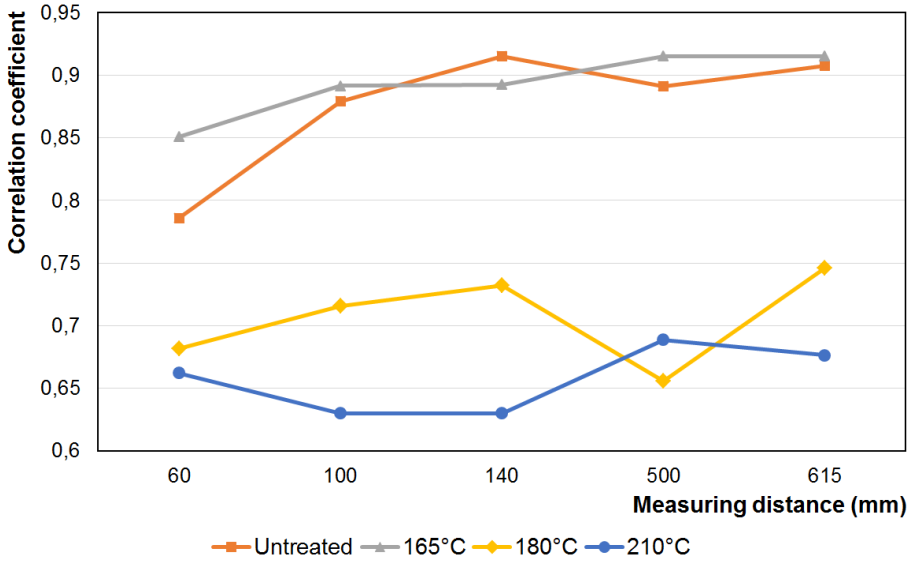

Fig. 11. Correlation between the MOE_{dyn} and MOE_{stat}

In contrast, the distance of 60 mm was the least suitable for both the thermally treated wood and reference samples. This result was probably due to the different types of sound wave propagation. Sound may propagate as longitudinal waves, transverse waves, surface waves, and circular waves in thin materials. With a small distance between the sensors, the surface wave propagation may have affected the passage lengthwise, which skewed the final passage time of the wave through the material. As the distance between the sensors increased, the surface wave propagation disappeared, which decreased the measurement error.

Table 7 contains the specific values of the dynamic and static MOE for the different thermal treatment groups and reference samples. The measuring distance had no clear effect on the MOE. In contrast, as the measuring distance increased, the differences between the MOE values for the different thermal treatment temperatures balanced out.

Table 7. MOE_{dyn} and MOE_{stat} Values for Various Measuring Distances

			Before Thermal Treatment	After Thermal Treatment
Thermal Treatment	Type of MOE	Measuring Distance (mm)	Mean MOE_{dyn}/MOE_{stat} (MPa)	
Untreated (reference)	static (MOE_{stat})	-	8,907.6 (18.8)	-
	dynamic (MOE_{dyn})	60	17,606.3 (27.1)	-
	dynamic (MOE_{dyn})	100	15,734.8 (21.4)	-
	dynamic (MOE_{dyn})	140	14,843.2 (18.8)	-
	dynamic (MOE_{dyn})	500	15,552.1 (21.0)	-
	dynamic (MOE_{dyn})	615	15,717.0 (20.4)	-
165 °C	static (MOE_{stat})	-	-	10,065.3 (22.2)
	dynamic (MOE_{dyn})	60	-	18,725.5 (24.0)
	dynamic (MOE_{dyn})	100	-	16,090.9 (19.8)
	dynamic (MOE_{dyn})	140	-	15,495.1 (20.1)
	dynamic (MOE_{dyn})	500	-	16,240.2 (20.1)
	dynamic (MOE_{dyn})	615	-	16,349.2 (20.0)
180 °C	static (MOE_{stat})	-	-	9,750.4 (20.4)
	dynamic (MOE_{dyn})	60	-	18,216.5 (26.3)
	dynamic (MOE_{dyn})	100	-	15,157.4 (23.5)
	dynamic (MOE_{dyn})	140	-	14,499.5 (24.9)
	dynamic (MOE_{dyn})	500	-	15,783.7 (23.3)
	dynamic (MOE_{dyn})	615	-	15,388.7 (24.3)
210 °C	static (MOE_{stat})	-	-	8,959.3 (24.8)
	dynamic (MOE_{dyn})	60	-	17,043.4 (23.0)
	dynamic (MOE_{dyn})	100	-	15,240.0 (19.0)
	dynamic (MOE_{dyn})	140	-	14,826.8 (18.9)
	dynamic (MOE_{dyn})	500	-	15,250.6 (19.6)
	dynamic (MOE_{dyn})	615	-	15,450.4 (19.4)

The values in parentheses are the coefficients of variation (CV) in %.

Resonance Method

The multi-factor ANOVA showed that only the anatomical direction had a statistically significant effect on the MOE_{dyn} ($P < 0.05$). The thermal treatment did not result in significant differences (Table 8).

Table 8. Effect of Individual Factors on the MOE_{dyn}

Monitored Factor	Sum of Squares	Degrees of Freedom	Variance	Fisher's F - Test	Significance Level P
Intercept	1.319571E+11	1	1.319571E+11	7,079.287	0.000
Thermal treatment	8.397164E+07	3	2.799055E+07	1.502	0.215
Direction	1.934464E+10	1	1.934464E+10	1,037.809	0.000
Thermal treatment × Direction	1.015442E+08	3	3.384806E+07	1.816	0.145
Error	4.026214E+09	216	1.863988E+07		

The difference in the MOE for the transverse and longitudinal directions was significant. The MOE_{dyn} in the longitudinal direction reached approximately 15,000 MPa, while in the transverse direction it reached about 33,000 MPa. In contrast, the effect of thermal treatment showed less effect than that determined by the ultrasound method. Because the same samples were used for both measuring methods, it was assumed that the resonance method is less accurate for thermally modified wood. The resonance method did not sufficiently recognize the effect of the individual temperatures, and the MOE_{dyn} results were very similar, whereas the real difference between them was greater.

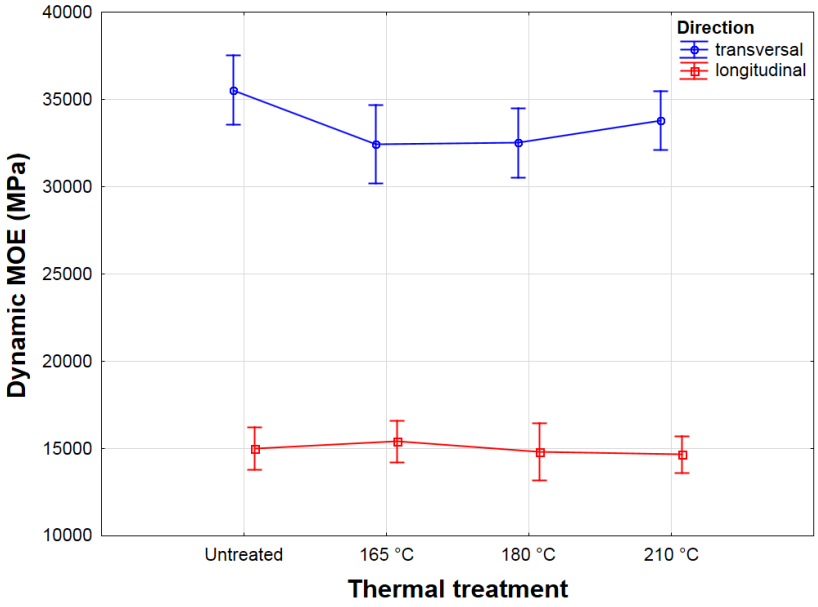

Fig. 12. Influence of thermal treatment on the MOE_{dyn} in the transverse and longitudinal directions

The evaluation of each MOE using Duncan's test (Table 9) showed that there was a significant difference in the transverse direction between the reference samples and the samples thermally treated at 165 °C and 180 °C.

In other cases, the differences were too small. In terms of the dynamic MOE values, this difference was too small to have any practical significance.

Practical measuring with this method may result in measuring errors that are larger than the difference obtained in this study, so this change cannot be attributed to the factor. The same effect often occurs due to the variability of the wood characteristics, which is caused by the differences in the structure of the wood itself.

Table 9. Duncan's Multiple Range Test of MOE_{dyn} for the Transverse and Longitudinal Directions

Direction	Thermal Treatment Temperature	Transverse				Longitudinal			
		Untreated	165°C	180°C	210°C	Untreated	165°C	180°C	210°C
Mean value of MOE_{dyn} (MPa)		35,537	32,440	32,510	33,771	15,016	15,414	14,820	14,663
Transverse	Untreated		0.012	0.012	0.126	0.000	0.000	0.000	0.000
	165°C	0.012		0.952	0.280	0.000	0.000	0.000	0.000
	180°C	0.012	0.952		0.274	0.000	0.000	0.000	0.000
	210°C	0.126	0.280	0.274		0.000	0.000	0.000	0.000
Longitudinal	Untreated	0.000	0.000	0.000	0.000		0.730	0.865	0.776
	165°C	0.000	0.000	0.000	0.000	0.730		0.632	0.561
	180°C	0.000	0.000	0.000	0.000	0.865	0.632		0.892
	210°C	0.000	0.000	0.000	0.000	0.776	0.561	0.892	

The MOE_{dyn} and MOE_{stat} measured in the transverse and longitudinal directions for the thermally modified and referential samples are shown in Table 10. In the untreated wood, the measured MOE_{stat} values were approximately 40.7% lower than the MOE_{dyn} values measured. The thermal treatment gradually reduced this difference to 34.7%, 34.2%, and 38.9% for 165 °C, 180 °C, and 210 °C, respectively.

Table 10. MOE_{dyn} and MOE_{stat} Values for the Transverse and Longitudinal Directions

		Before Thermal Treatment	After Thermal Treatment
Thermal Treatment	Type of MOE	Mean MOE_{dyn}/MOE_{stat} (MPa)	
Untreated (reference)	static (MOE_{Lstat})	8,907.6 (18.8)	-
	dynamic longitudinal (MOE_{Ldyn})	15,015.9 (21.0)	-
	dynamic transverse (MOE_{Tdyn})	35,536.9 (14.3)	-
165 °C	static (MOE_{Lstat})	-	10,065.3 (22.2)
	dynamic longitudinal (MOE_{Ldyn})	-	15,413.6 (20.1)
	dynamic transverse (MOE_{Tdyn})	-	32,439.9 (18.0)
180 °C	static (MOE_{Lstat})	-	9,750.4 (20.4)
	dynamic longitudinal (MOE_{Ldyn})	-	14,820.3 (28.2)
	dynamic transverse (MOE_{Tdyn})	-	32,509.5 (15.7)
210 °C	static (MOE_{Lstat})	-	8,959.3 (24.8)
	dynamic longitudinal (MOE_{Ldyn})	-	14,663.2 (18.7)
	dynamic transverse (MOE_{Tdyn})	-	33,770.8 (12.8)

The values in parentheses are the coefficients of variation (CV) in %.

The difference between the dynamic and static MOE values were mainly due to the fact that wood is a viscoelastic material (Halabe *et al.* 1997; Divós and Tanaka 1997).

This was most apparent in the static test, which involved a longer period than the non-destructive (NDT) tests. The MOE_{dyn} values were higher compared to the MOE_{stat} values, which were confirmed by almost all other similar studies. Only the value of the difference between the MOE_{dyn} and MOE_{stat} values varied. Divós and Tanaka (2005) reported that the MOE_{dyn} values were generally 10% higher than the MOE_{stat} values. De Oliveira *et al*. (2002) found 17% higher MOE_{dyn} values compared to the MOE_{stat} values.

The measuring methods themselves often led to differences between the dynamic and static MOE values. In general, the highest dynamic MOE values were determined using the ultrasound method rather than the static method. The resonance method resulted in slightly lower MOE values than the ultrasound method, but they were still higher than the static MOE values. These findings were confirmed by many other studies, such as Haines and Leban (1997), Liang and Fu (2007), and Unterwieser and Schickhofer (2011). The wood moisture content was the main property that affected the ultrasound and resonance methods because it directly affected the sound wave velocity. The sound wave velocity is the highest in dry wood, and it gradually decreases as the moisture content increases (Saadat-Nia *et al*. 2011; Unterwieser and Schickhofer 2011).

The thermal treatment strongly affects the physical and mechanical properties of the wood, depending on the temperature used. The effect of the thermal treatment on the MOE may vary. The thermal treatment with temperatures ranging between 180 °C to 200 °C significantly affects the structure of the wood, but it also reduces the moisture content so much that the MOE is only slightly reduced, or it may even increase slightly. In this research, a slight increase in the MOE was also found, which was most noticeable in the static MOE. Other studies on the effect of thermal treatment on the mechanical properties of wood reported a similar effect. Navickas *et al*. (2015) found a 1.5% increase in the MOE after 3 h of thermal treatment of spruce. This fact can be explained by a decrease in the moisture content, which increased the wood density and therefore improved the mechanical properties, too (Zhang 1995). In contrast, Metsä-Kortelainen and Viitanen (2010), who examined the effect of thermal treatment on the MOE and MOR of spruce, found a decrease in the MOE in the range of 8.6% to 18.3% for sapwood and 0.4% to 23.4% for heartwood.

CONCLUSIONS

1. A negative effect from the thermal treatment on the MOE_{dyn} was found using the ultrasound method. A decrease of 21.6% was measured on the tangential surface, and a 13.9% decrease was measured on the radial surface. The verification of the effect of the sensor distance confirmed that the highest correlation (0.91 to 0.92) between the MOE_{dyn} and MOE_{stat} occurred between distances ranging from 140 to 615 mm. The correlation increased as the distance increased, but gradually decreased as the temperature increased. As the sensor distance increased, the effect of the increasing temperature on the MOE_{dyn} and MOE_{stat} values decreased. Compared to the MOE_{stat} values, the MOE_{dyn} was 78.4% higher in the untreated wood and 66.7% higher in the thermally treated wood.
2. With the resonance method, the MOE_{dyn} values were twice as high in the transverse direction as in the longitudinal direction. The thermal treatment only resulted in a significant decrease in the MOE_{dyn} values in the transverse direction. In the

longitudinal direction, the differences in the MOE_{dyn} values of wood treated at different temperatures were minimal, whereas the ultrasound method found greater differences. The resonance method also found higher MOE_{dyn} values than MOE_{stat} values. The MOE_{dyn} values were 68.6% higher for the untreated wood and were 66% higher for the thermally treated wood.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Internal Grant Agency (IGA) of the Faculty of Forestry and Wood Science at Czech University of Life Sciences Prague, Project No. A 30/16.

REFERENCES CITED

Allegretti, O., Cividinir, R., and Tessadri, B. (2008). "Thermal treatment in saturated vapour pressure for spruce," in: *Proceedings of the Conference COST Action E53*, Delft, Netherlands, pp. 125-134.

Ates, S., Akyildiz, M. H., and Ozdemir, H. (2009). "Effects of heat treatment on calabrian pine (*Pinus brutia* Ten.) wood," *BioResources* 4(3), 1032-1043. DOI: 10.15376/biores.4.3.1032-1043

Ayrilmis, N., Buyuksari, U., and As, N. (2010). "Bending strength and modulus of elasticity of wood-based panels at cold and moderate temperatures," *Cold Reg. Sci. Technol.* 63(1-2), 40-43. DOI: 10.1016/j.coldregions.2010.05.004

Baillères, H., Hopewell, G., Boughton, G., and Brancheriau, L. (2012). "Strength and stiffness assessment technologies for improving grading effectiveness of radiata pine wood," *BioResources* 7(1), 1264-1282. DOI: 10.15376/biores.7.1.1264-1282

Bal, B. C., and Bektaş, İ. (2012). "The effects of some factors on the impact bending strength of laminated veneer lumber," *BioResources* 7(4), 5855-5863. DOI: 10.15376/biores.7.4.5855-5863

Belleville, B., Stevanovic, T., Cloutier, A., Pizzi, A., Prado, M., Erakovic, S., Diouf, P. N., and Royer, M. (2013). "An investigation of thermochemical changes in Canadian hardwood species during wood welding," *Eur. J. Wood Prod.* 71(2), 245-257. DOI: 10.1007/s00107-013-0671-x

Borrega, M., and Kärenlampi, P. P. (2008). "Mechanical behavior of heat-treated spruce (*Picea abies*) wood at constant moisture content and ambient humidity," *Holz Roh. Werkst.* 66(1), 63-69. DOI: 10.1007/s00107-007-0207-3

Borůvka, V., Gaff, M., Babiak, M., and Marko, M. (2015). "Dimensional changes of veneer layered materials after cold pressing," *BioResources* 10(4), 6663-6675. DOI: 10.15376/biores.10.4.6663-6675

Brodin, I., Sjöholm, E., and Gellerstedt, G. (2010). "The behavior of kraft lignin during thermal treatment," *Journal of Analytical and Applied Pyrolysis* 87(1), 70-77. DOI: 10.1016/j.jaap.2009.10.005

Bučar, D. G., and Bučar, B. (2011). "Strength grading of structural timber using the single mode transverse damped vibration method," *Wood Research* 56(1), 67-76.

Byrne, C. E., and Nagle, M. (1997). "Carbonization of wood for advanced materials applications," *Carbon* 35(2), 259-266. DOI: 10.1016/S0008-6223(96)00136-4

de Oliveira, F. G. R., de Campos, J. A. O., and Sales, A. (2002). "Ultrasonic measurements in Brazilian hardwood," *Materials Research* 5(1), 51-55. DOI: 10.1590/S1516-14392002000100009

de Oliveira, F. G. R., and Sales, A. (2006). "Relationship between density and ultrasonic velocity in Brazilian tropical woods," *Bioresource Technol.* 97(18), 2443-2446. DOI: 10.1016/j.biortech.2005.04.050

Divós, F., and Tanaka, T. (1997). "Effect of creep on modulus of elasticity determination of wood," *J. Vib. Acoust.* 122(1), 90-92. DOI: 10.1115/1.568440

Divós, F., and Tanaka, T. (2005). "Relation between static and dynamic modulus of elasticity of wood," *Acta Silvatica & Lignaria Hungarica* 1, 105-110.

EN 310 (1993). "Wood-based panels: Determination of modulus of elasticity in bending and of bending strength," European Committee for Standardization, Brussels, Belgium.

Gaff, M., and Gáborík, J. (2014). "Evaluation of wood surface quality after 3D molding of wood by pressing," *BioResources* 9(3), 4468-4476. DOI: 10.15376/biores.9.3.4468-4476

Gonzáles-Peña, M. M., Curling, S. F., and Hale, M. D. C. (2009). "On the effect of heat on the chemical composition and dimensions of thermally modified wood," *Polym. Degrad. Stabil.* 94(12), 2184-2193. DOI: 10.1016/j.polymdegradstab.2009.09.003

Haines, D. W., and Leban, J.-M. (1997). "Evaluation of the MOE of Norway spruce by the resonance flexure method," *Forest Prod. J.* 47(10), 91-93.

Halabe, U. B., GangaRao, H. V. S., and Solomon, C. E. (1993). "Nondestructive evaluation of wood using ultrasonic dry-coupled transducers," in: *Review of Progress in Quantitative Nondestructive Evaluation, Volumes 12A and 12B*, D. O. Thompson and D. E. Chimenti (eds.), Plenum Press, New York, NY, pp. 2251-2256.

Halabe, U. B., Bidigalu, G. M., GangaRao, H. V. S., and Ross, R. J. (1997). "Nondestructive evaluation of green wood using stress wave and transverse vibration techniques," *Mater. Eval.* 55(9), 1013-1018.

Heigenmoser, A., Liebner, F., Windeisen, E., and Richter, K. (2013). "Investigation of thermally treated beech (*Fagus sylvatica*) and spruce (*Picea abies*) by means of multifunctional analytical pyrolysis-GC/MS," *J. Anal. Appl. Pyrol.* 100, 117-126. DOI: 10.1016/j.jaap.2012.12.005

Ilic, J. (2003). "Dynamic MOE of 55 species using small wood beams," *Holz Roh. Werkst.* 61(3), 167-172. DOI: 10.1007/s00107-003-0367-8

ISO 13061-1 (2014). "Physical and mechanical properties of wood -- Test methods for small clear wood samples -- Part 1: Determination of moisture content for physical and mechanical tests," International Organization for Standardization, Geneva, Switzerland.

ISO 13061-2 (2014). "Physical and mechanical properties of wood -- Test methods for small clear wood samples -- Part 2: Determination of density for physical and mechanical tests," International Organization for Standardization, Geneva, Switzerland.

Kačík, F., Veľková, V., Šmíra, P., Nasswettrová, A., Kačíková, D., and Reinprecht, L. (2012). "Release of terpenes from fir wood during its long-term use and in thermal treatment," *Molecules* 17(8), 9990-9999. DOI: 10.3390/molecules17089990

Karsulovic, J. T., León, L. A., and Gaete, L., (2000). "Ultrasonic detection of knots and annual ring orientation in *Pinus radiata* lumber," *Wood Fiber Sci.* 32(3), 278-286.

Kim, D. Y., Nishiyama, Y., Wada, M., Kuga, S., and Okano, T. (2005). "Thermal decomposition of cellulose crystallites in wood," *Holzforschung* 55(5), 521-524. DOI: 10.1515/HF.2001.084

Kretschmann, D. E. (2010). "Mechanical properties of wood," in: *Wood Handbook: Wood as an Engineering Material*, U.S. Department of Agriculture, Forest Products Laboratory, Madison, WI, USA, pp. 1-46.

Kubš, J., Gaff, M., and Barcík, Š. (2016). "Factors affecting the consumption of energy during the milling of thermally modified and unmodified beech wood," *BioResources* 11(1), 736-747. DOI: 10.15376/biores.11.1.736-747

Liang, S.-Q., and Fu, F. (2007). "Comparative study on three dynamic modulus of elasticity and static modulus of elasticity for lodgepole pine lumber," *Journal of Forestry Research* 18(4), 309-312. DOI: 10.1007/s11676-007-0062-4

Liu, Z., Liu, Y., Yu, H., and Yuan, J. (2006). "Measurement of the dynamic modulus of elasticity of wood panels," *Frontiers of Forestry in China* 1(4), 425-430. DOI: 10.1007/s11461-006-0048-y

Liu, Y., Gong, M., Li, L., and Chui, Y. H. (2014). "Width effect on the modulus of elasticity of hardwood lumber measured by non-destructive evaluation techniques," *Constr. Build. Mater.* 50, 276-280. DOI: 10.1016/j.conbuildmat.2013.09.029

Martinka, J., Hroncová, E., Chrebet, T., and Balog, K. (2014). "The influence of spruce wood heat treatment on its thermal stability and burning process," *Eur. J. Wood Prod.* 72(4), 477-486. DOI: 10.1007/s00107-014-0805-9

Metsä-Kortelainen, S., and Viitanen, H. (2010). "Effect of fungal exposure on the strength of thermally modified Norway spruce and Scots pine," *Wood Material Science and Engineering* 5(1), 13-23. DOI: 10.1080/17480271003786738

Mishiro, A. (1996). "Effect of density on ultrasonic velocity in wood," *Mokuzai Gakkaishi* 42(9), 887-894.

Moavenzadeh, F. (1990). *Concise Encyclopedia of Building and Construction Materials*, M.I.T. Press, Cambridge, MA, USA.

Murata, K., Watanabe, Y., and Nakano, T. (2013). "Effect of thermal treatment on fracture properties and adsorption properties of spruce wood," *Materials* 6(9), 4186-4197. DOI: 10.3390/ma6094186

Navickas, P., Karpavičiūtė, S., and Albrektas, D. (2015). "Effect of heat treatment on wettability and modulus of elasticity of pine and spruce wood," *Mater. Sci.* 21(3), 400-404. DOI: 10.5755/j01.ms.21.3.7304

Niemz, P., Hofmann, T., and Rétfalvi, T. (2010). "Investigation of chemical changes in the structure of thermally modified wood," *Maderas Ciencia y Tecnología* 12(2), 69-78. DOI: 10.4067/S0718-221X2010000200002

Rousset, P., Lapierre, C., Pollet, B., Quirino, W., and Perre, P. (2009). "Effect of severe thermal treatment on spruce and beech wood lignins," *Ann. For. Sci.* 66(1), 110-110. DOI: 10.1051/forest/2008078

Saadat-Nia, M. A., Brancheriau, L., Gallet, P., Enayati, A. A., Pourtahmasi, K., and Honarvar, F. (2011). "Ultrasonic wave parameter changes during propagation through poplar and spruce reaction wood," *BioResources* 6(2), 1172-1185. DOI: 10.15376/biores.6.2.1172-1185

Sales, P., Candian, M., and de Salles Cardin, V. (2011). "Evaluation of the mechanical properties of Brazilian lumber (*Goupia glabra*) by nondestructive techniques," *Constr. Build. Mater.* 25(3), 1450-1454. DOI: 10.1016/j.conbuildmat.2010.09.020

Unterwieser, H., and Schickhofer, G. (2011). "Influence of moisture content of wood on sound velocity and dynamic MOE of natural frequency- and ultrasonic runtime measurement," *Eur. J. Wood Prod.* 69(2), 171-181. DOI: 10.1007/s00107-010-0417-y

Vafai, A., Farshad, M., and Ahmadieh, A. (1976). "Determination of modulus of elasticity of wood from vibrating reed measurements," *Fibre Sci. Technol.* 9(1), 1-10. DOI: 10.1016/0015-0568(76)90019-1

Weiland, J. J., and Guyonnet, R. (2003). "Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy," *Holz Roh. Werkst.* 61(3), 216-220. DOI: 10.1007/s00107-003-0364-y

Windeisen, E., and Wegener, G. (2008). "Behavior of lignin during thermal treatments of wood," *Ind. Crop. Prod.* 27(2), 157-162. DOI: 10.1016/j.indcrop.2007.07.015

Yildiz, S., Gezer, E. D., and Yildiz, U. C. (2006). "Mechanical and chemical behavior of spruce wood modified by heat," *Building and Environment* 41(12), 1762-1766. DOI: 10.1016/j.buildenv.2005.07.017

Zhang, Y. S. (1995). "Effect of growth rate on wood specific gravity and selected mechanical properties in individual species from distinct wood categories," *Wood Sci. Technol.* 29(6), 451-465. DOI: 10.1007/BF00194204

Zhang, H. J., Cui, Y. Y., Wang, C. Z., and Su, J. (2009). "Determining modulus of elasticity of structural lumber with vibration methods," *Key Eng. Mat.* 407-408, 521-527. DOI: 10.4028/www.scientific.net/KEM.407-408.521

Article submitted: September 26, 2016; Peer review completed: November 20, 2016; Revised version received and accepted: November 28, 2016; Published: December 6, 2016.
DOI: 10.15376/biores.12.1.819-838