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SELECTED PROPERTIES OF THERMALLY TREATED ASH WOOD

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ABSTRACT

The paper is focused on the impact of thermal treatment on the changes of selected physical and mechanical properties of ash wood. Effect of the treatment time and treatment temperature on density, equilibrium moisture content, the compression strength in the longitudinal direction and static and dynamic modulus of elasticity was analysed. The samples made of Common Ash (*Fraxinus excelsior* L.) were modified in oxidized atmosphere at the temperatures of 160 °C, 180 °C, and 200 °C. Each temperature treatment lasted for 3, 6, 9 and 12 hours. The decrease in density and equilibrium moisture content was confirmed and important changes in mechanical properties were observed. Following the increase in elastic modulus and apparent strength, a 3-hour treatment at the temperature of 200 °C was considered a reasonable compromise for high temperature treatment of ash wood.

Key words: compressive strength, static modulus of elasticity, dynamic modulus of elasticity, high temperature treatment, ash wood.

INTRODUCTION

In spite of the fact that wood has lots of undeniable advantages and positive features, there are also some negatives. Exposing wood in outdoors may bring a risk, especially of weather conditions, fungi, moulds and biological insects. Thermal modification, which improves the resistance of wood towards weather conditions and partially also resistance against fungi, moulds and biological agents is a possibility how to deal with this problem (HILL 2006, MILITZ 2002, REINPRECHT, VIDHOLDOVÁ 2011). An advantage of thermally modified wood (TMW) is environmental friendliness of its production. Change of color by modification makes possibility to use wood material as aesthetic substitute for some of exotic species. Thanks to these positive improvements, there is ongoing development of several processes of TMW in Europe (MILITZ 2002, KAČÍKOVÁ *et al*. 2013).

TMW is a regulated effect of high temperatures among 150–280 °C, in the interval between 15 minutes up to 14 hours. Thermal treatment can be divided according to the type of heat transfer to the wood/timber and according to the environment in which thermal modification is processed. Nowadays, the heat is transported to wood mainly by air and water vapor, but it is possible to transfer heat by oils too (RAPP 2011).

Under the influence of heat there are changes mainly at the molecular level. It has been demonstrated that thermal treatment decreases the density, changes color or reduces equilibrium moisture content (EMC) that rises dimensional stability (KORKUT *et al*. 2008, ROMAGNOLI *et al*. 2015, AYTIN *et al*. 2015; ANDOR, LAGAŇA 2016). Changes of these properties are caused by degradation of hemicelluloses, increase in crystalline cellulose and

crosslinking of lignin due to polycondensation reactions (BOONSTRA et TJEERDSMA 2006, ČABALOVÁ *et al*. 2016).

Mechanical properties of wood are also changed by thermal modification. The flexural strength as the most affected properties falls down to about 50% (BEKHTA, NIEMZ 2003, BORUVKA *et al*. 2015). Quantity and character of these changes depends on temperature, time and a way of treatment, and wood species (YILDIZ, GÜMÜŞKAYA 2005). Nevertheless, there are cases that the strength of wood increases due to thermal treatment and only at higher temperatures (205 °C for pine) it starts to decrease slowly (CAI J., CAI L. 2012). Although the strength of wood decreases in common, the modulus of elasticity stays at the same level or can increase to some extend (LEKOUNOUGOU, KOCAEFE 2014).

Known correlation of static and dynamic modulus of elasticity may lead to qualitative classification of thermally modified ash wood. For both moduli, it can be assumed that there is a moderate increase in properties and maximum values are reached at certain thermal conditions. The importance of time and temperature impact together with statistical importance of these factors could optimize the process of thermal treatment.

The aims of this study are to quantify an effect of the treatment time and the treatment temperature on selected properties of ash wood and to optimize the process of a thermal treatment in oxidized atmosphere based on changes in density, EMC, compressive strength parallel to the fibres, and the static and dynamic modulus of elasticity.

MATERIAL AND METHODS

Samples of ash wood where prepared from mature wood of one trunk in order to lower variability of properties. The size of samples was 32 × 32 × 120 mm. After sorting out defective samples, they were divided into groups. Each group was designed for a different time and temperature of a thermal treatment. The samples were thermally treated for 3, 6, 9 and 12 hours at the temperatures of 160, 180 and 200 °C. The number of samples of each group varied from 13 to 16 depending upon the intensity of thermal treatment. The samples were oven dried at 103 °C ± 2 °C. After the drying and cooling of the samples, the dimensions and weight were measured, and samples were placed in desiccator over silica gel. For comparing the results with untreated wood, 35 samples were allocated. These samples were placed in a conditioning room at 20°C and a relative humidity (RH) of 65%.

Treated samples were placed into preheated oven for 10 minutes and after that time they were counted down. After the treatment, the samples were placed in a desiccator over silica, where they were cooled down and their dimensions and weight were measured again.

Treated samples were conditioned to EMC in a conditioning room at the temperature of 20 °C and RH = 65 %. At the end of the sample preparation, the final dimensions and weight were determined.

The speed of sound in longitudinal direction was measured for each sample using Fakopp device (Sopron, Hungary). Time of ultrasound signal transition was detected with an accuracy of 0.1 microseconds. Subsequently, dynamic modulus of elasticity "Edyn" was calculated according to the formula:

$$E_{dyn}=c^2.\rho \text{ [MPa]}$$

where c is the speed of sound and ρ is the density of a sample.

Samples were tested in compression using FPZ 100 loading machine (Heckert, Germany). Displacement at the 50 mm gauge length was measured by two potentiometric displacement sensors placed on both sides of a sample and connected to data logger ALMEMO 2690-8 (Holzkirchen, Germany). Sample failure occurred within 60 ± 30 seconds

of loading. Static modulus of elasticity was calculated from the linear portion of a force-deformation curve.

Changes of physical properties were evaluated by comparing the measurements before and after the thermal treatment. The average changes and standard deviation were observed. Two-factor analysis of variance (ANOVA) at the significance level α=0.05 was used for assessing the significance of the treatment time and temperature on static elastic modulus, dynamic modulus and strength. In order to predict elastic properties of thermally treated ash wood, student's paired t-test compared averages of both moduli and correlation coefficients were calculated. Values of static and dynamic modulus of elasticity were compared by Student's paired t-test.

Proper thermal treatment parameters were chosen based on finding the middle ground between shape stability, given by low EMC, and mechanical properties that are at least at the level of untreated wood properties.

RESULTS AND DISCUSSION

The impact of thermal treatment on equilibrium moisture content and density

The density of samples after thermal treatment decreases, in general. Our experiments show that the temperature is more important in changing the density than the time of treatment. A decrease in density at the temperature of 160 °C exceeded by 1% only for 12-hour treatment and a decrease in density at the temperature of 180 °C was between 2.2–3.4 %. The most noticeable change in wood density (12% decrease) happened during 12-hour treatment at the temperature of 200 °C. MOLINSKY *et al*. (2016) recorded similar decline reporting 3.5 % and 9.3% decrease of density of European Ash wood after 2-hour treatment at the temperature of 190 °C and 200 °C treatment, respectively. We consider mainly the degradation of hemicelluloses to volatile substances which evaporated during the thermal treatment. KAČÍKOVÁ *et al*. (2013) stated the decline of hemicellulose content caused by thermal treatment and changes in cellulose and lignin as well.

The EMC is one of the important features determining behaviour of mechanical properties. The EMC of untreated wood in the standard environment was on average 11.5 %. After the thermal treatment at the temperature of 160 °C, EMC dropped to 7.8 % (3 hours) and 7.3 % (12 hours). After treating the samples at the temperature of 180 °C, the EMC decreased under 7% (3 hours) and below 6% after 9-hour treatment. The most significant decrease was recorded for 200 °C treatment, when EMC reached the value 5.2 % for each duration of the treatment. We can assume from the results that EMC as well as the density depends more on temperature by which the wood was treated than the time of duration of the treatment (Figure 2). BORUVKA *et al*. (2015) stated declining of EMC from 11.5 % in unmodified samples of Douglas pine wood to 7.7 % after a 3-hour treatment at the temperature of 165 °C and 6.2 % at 210 °C, respectively. NEMETH *et al*. (2016) mentioned EMC decrease of poplar wood to 5 % after thermal treatment at the temperature of 200 °C. KÚDELA *et al*. (2005) stated that steaming beech wood even at the temperature of 140 °C for 1 hour leads to decrease in sorption properties of wood. This decrease can be explained similarly as the change in wood density by the change in chemical structure of wood. The main reason is a reduction of sorption places in cell wall, which happens due to hemicellulose content decrease and the increase of cellulose crystallinity. Lignin also undergoes some polycondensation reactions leading to crosslinking of chains. Lignin incrusts fibrils as well, thus it prevents access to molecules of water (BOONSTRA, TJEERDSMA 2006, BOONSTRA *et al*. 2007).

The impact of thermal treatment on compressive strength parallel to the fibres

The strength of Ash wood in the fibre direction of reference samples was 49.26 MPa. This is slightly lower than an average value of 55 MPa mentioned by NIEMZ *et al*. (2014). This variation can be caused by variability of mechanical properties within the species.

The compressive strength of Ash wood changed only slightly after the treatment at the temperature of 160 °C and mainly in samples treated for 9 hours and more. The noticeable increase in strength was reached at 180 °C even after a 3-hour treatment. The maximum was reached after 9-hour treatment at the temperature of 180 °C with the overall increase by 9 MPa. At the temperature of 200 °C after 3-hour treatment, the increase was up to 4 MPa, nevertheless, the longer treatment caused decrease in strength when compared to the strength of untreated samples. Thermal treatment increased fragility, what was observed as characteristic failure of the samples (Figure 4 and 5). Splitting of samples was hardly observed, but at higher temperatures it was visible more often. We can conclude that thermal treatment increases the fragility of wood above 200 °C after a 6-hour treatment period noticeably. The similar increase in compressive strength in the fibre direction was described by more authors (BOONSTRA *et al*. 2007, ZAWADSKI *et al*. 2013, LAGANA *et al*. 2015). This increase is mainly caused by different EMC of samples. When the moisture increases, the compressive strength in direction of fibres decreases, resulting in 4 % decline for 1 % moisture content increase (REGINÁČ *et al*. 1978).

During the thermal treatment, crystallization process of amorphous cellulose takes place, which may have a positive impact on the compressive strength (Boonstra et TJEERDSMA 2006). Volume of amorphous cellulose increases during being exposed to the temperature ranged from 160–220 °C in shorter intervals of heating but for longer period it decreases (BHUIYAN *et al*. 2000). This corresponds to decrease in strength for a longer treatment. Polycondensation reactions of lignin have also impact on strength and they lead to its crossing. Crossed molecules of lignin become less elastic (HILL 2006) and are used as stiffener between fibrils which incrust and so they have positive impact on the strength in the fibre direction. Due to high amount of lignin in the middle lamella its crossing leads to the increase in strength of middle lamella and cell wall as well. It also prevents the movements between cell walls in the fibre direction (BOONSTRA *et al*. 2007).

When evaluating the results, we should consider also the variability of strength. There is a decrease in results after the treatment at the temperature of 200 °C for a period longer than 3 hours. With respect to significantly lower moisture content, this decrease is probably more important. At this stage of treatment, the decrease in strength is caused by depolymerisation reactions in wood and decline of hemicellulose, which are the least stable component of wood. The changes of mechanical properties are not only affected by amounts of main components of wood but also by changes in their relations. Some portion on change in mechanical properties is caused by a change in morphological structure (KAČÍKOVÁ, KAČÍK 2011; KAČÍKOVÁ *et al*. 2013; LAGAŇA *et al*. 2015). ANOVA showed that the factor of treatment time has negligible impact on compressive strength parallel to the fibres (Table 1). The temperature showed to be a significant factor.

The impact of thermal treatment on the modulus of elasticity

The average static modulus of elasticity of untreated ash wood samples was 9,629 MPa, which is below the average value mentioned in the literature. NIEMZ *et al*. (2014) stated the modulus of elasticity of 12,110 MPa. The average dynamic modulus of elasticity was 11,522 MPa.

The static and dynamic moduli of elasticity were higher than untreated ones up to 3-hour treatment at 200 °C. However, the most significant increase did not happen at identical time and the temperature of treatment. The static modulus of elasticity reached its maximum at the temperature of 180 °C after 3-hour treatment. The dynamic modulus was the largest at the

temperature of 160 °C and the treatment time of 9 hours. When we applied thermal treatment at the temperature of 200 °C, the increase in both moduli did not occur (Figure 6 and 7). The most important decrease was recorded after a 6-hour treatment at the temperature of 200 °C for static modulus (–10.65%), and a 9-hour treatment for dynamic modulus (–13.1%). The fact that the modulus of elasticity at the temperature of 200 °C declined, supported a statement that Ash wood is not suitable for thermal treatment for a longer period of time.

Similarly to the compressive strength, the modulus of elasticity is to some extend affected by different EMC, but this impact is not so significant. Results obtained during different loading modes showed a decrease in MOE, especially in bending. The strength in bending, as many studies have proved, decreases significantly and this decline may reach up to 50 % based on the method of thermal modification (BORUVKA *et al.* 2015; CAI J., CAI L., 2012; BOONSTRA *et al*. 2007). This important decrease is related to decline in tensile strength while the compressive strength decreases less markedly (KORKUT *et al*. 2007). The increase of MOE due to heat treatment was proved also by ZAWADSKI, RADOMSKI (2013) who stated its increase for Scots pine treated at the temperatures of 160 °C and 200 °C during 2, 5 and 10 hours.

ANOVA showed statistical significance of both factors, the treatment time and the treatment temperature, respectively (Table 1). The Duncan test divided the treatments into two distinguished groups, the first one with the reference samples and the samples treated at 200 °C and the other group with samples treated at 160 and 200 °C.

For each temperature and time of treatment, the t–test proved minimum dependency of static and dynamic modulus of elasticity measured using identical samples. However, similarly to ROHANOVÁ, LAGAŇA (2014), the correlation coefficient confirmed significant degree of dependence between static and dynamic modulus of elasticity in all cases (Table 2).

Tab. 1 Results of ANOVA with two factors, the treated time and the treated temperature. Tested properties: density (ρ), equilibrium moisture content (EMC), compressive strength parallel to the fibres (σ), static modulus of elasticity (E_{stat}), dynamic modulus of elasticity (E_{dyn}). Values in bold show significant differences.

		ρ		EMC		σ		E_{stat}		E_{dyn}	
	DOF	**F**	**p**	**F**	**p**	**F**	**p**	**F**	**p**	**F**	**p**
Abs. Value	**1**	**107,506**	**0.000**	**199,528**	**0.000**	**23,917**	**0.000**	**8,594**	**0.000**	**16,504**	**0.000**
Time	**3**	**60.20**	**0.000**	**9,109**	**0.000**	1.380	0.250	**3.418**	**0.018**	**2.810**	**0.039**
Temperature	**3**	**3.600**	**0.014**	**25.20**	**0.000**	**25.93**	**0.000**	**18.10**	**0.000**	**30.26**	**0.000**
Time x Temp.	**9**	1.600	0.127	**12.50**	**0.000**	**3.230**	**0.001**	1.113	0.353	**1.920**	**0.049**

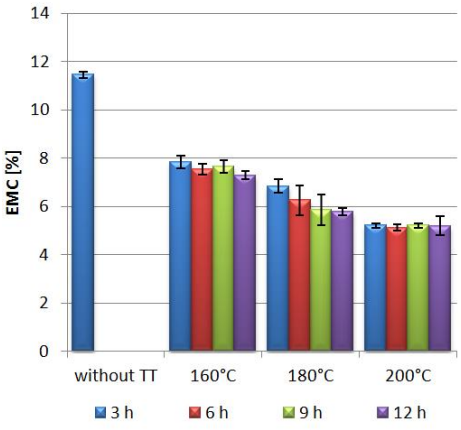

Fig. 1 The equilibrium moisture content of samples after the thermal modification. Bars stand for standard error.

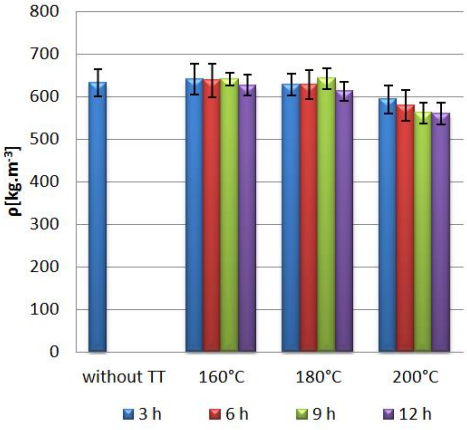

Fig. 2 The density of samples after the thermal modification. Bars stand for standard error.

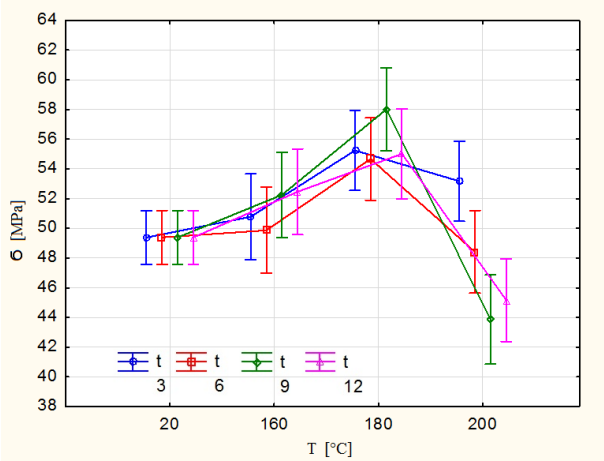

Fig. 3 Compressive strength parallel to the fibre depending upon the treatment time and the temperature, Bars stand for standard error.

Fig. 4 The failure of samples treated by the temperature of 200 °C for 9 hours.

Fig. 5 The failure of samples treated by the temperature of 200 °C for 12 hours.

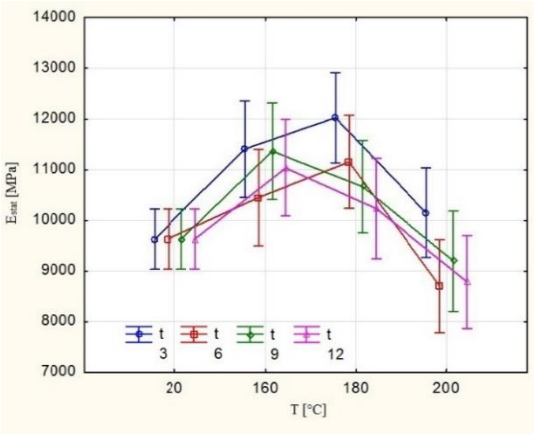

Fig. 6 Static modulus of elasticity for each treatment time and temperature. Bars stand for standard error.

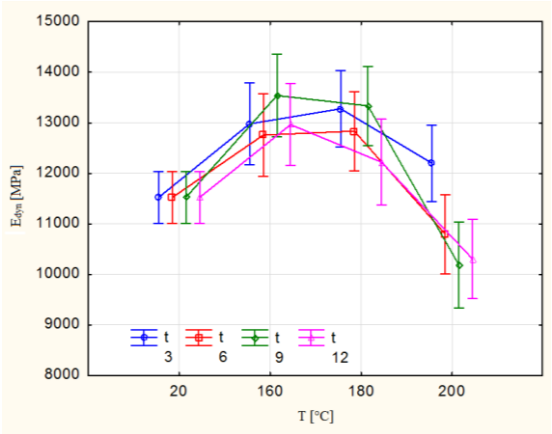

Fig. 7 Dynamic modulus of elasticity for each treatment time and temperature. Bars stand for standard error.

Tab. 2 The average values and standard deviations (STD) of density (ρ), EMC, the compressive strength parallel to the fibres (σ), the static modulus of elasticity (Estat) and dynamic modulus of elasticity (Edyn). Mean values followed by the same letters within the same column across treatments are not significantly different at p<0.05. The last column r, shows coefficient of correlation between static and dynamic modulus of elasticity. Values in bold are statistically significant.

TU	ρ	STD	EMC	STD	σ	STD	E_{stat}	STD	E_{dyn}	STD	r
Ref.	632.3	31.67	11.46	0.137	49.26 c,d	5.739	9,629 a,b,c,d,e	1,844	11,520 b,c	1,542	0.49
160/3	640.3	36.21	7.851	0.265	50.78 c,d,e	3.577	11,460 f,g	1,633	12,980 d,e	1,259	0.35
160/6	637.7	40.28	7.537	0.230	49.87 c,d	3.586	10,450 c,d,e,f	1,821	12,760 d,e	1,440	**0.55**
160/9	641.3	14.26	7.651	0.265	52.25 c,d,e,f	3.639	11,360 e,f	1,345	13,540 e	1,288	**0.70**
160/12	626.8	25.15	7.300	0.180	49.67 c,d,e,f	3.576	9,925 e,f,g	1,431	12,960 d,e	1,567	0.46
180/3	628.7	25.38	6.853	0.286	55.24 f,g	3.082	12,020 g	1,409	13,280 d,e	1,150	0.36
180/6	627.9	33.90	6.258	0.608	54.67 e,f,g	5.776	11,150 f,g	2,145	12,840 d,e	1,946	**0.83**
180/9	642.3	24.70	5.870	0.646	58.01 g	2.218	10,660 d,e,f,g	1,865	13,340 d,e	1,246	**0.76**
180/12	612.7	22.66	5.791	0.141	55.01 f,g	3.881	10,230 c,d,e,f	1,398	12,220 c,d	1,470	**0.72**
200/3	593.3	33.64	5.213	0.110	53.17 d,e,f	5.290	10,150 b,c,d,e,f	1,791	12,200 c,d	1,726	**0.60**
200/6	579.8	35.96	5.134	0.124	48.41 b,c	6.023	8,701 a	1,298	10,850 a,b	1,336	**0.68**
200/9	562.0	24.34	5.211	0.083	43.86 a	7.895	9,196 a,b,c	2,060	10,180 a	1,383	**0.48**
200/12	560.5	24.87	5.205	0.384	45.16 a,b	5.925	8,783 b,c	1,594	10,300 a	1,338	**0.76**

CONCLUSION

Results of mechanical properties of European ash wood thermally treated in the oxidized atmosphere at the temperatures of 160, 180 and 200 °C could be summarized in the following conclusions:

1. The density decreases due to thermal treatment. Density of wood treated at the temperature of 160 and 180°C slightly declines, but when wood is treated at 200 °C (12 hours) it drops by 12%. The change in density depends on the temperature of a thermal treatment.
2. The equilibrium moisture content of wood decreases because of thermal treatment. Similar to the density, the EMC mainly depends on the temperature of the thermal treatment. After treatment at the temperature of 200°C, the equilibrium moisture in the standard conditions dropped to 5%.
3. The compressive strength parallel to the fibres increases apparently due to the thermal treatment, reaching the peak at 180°C. This increase, compared to untreated wood, was caused mainly by declining of EMC and it was recorded up to 3-hour treatment at 200 °C. For a longer time of the treatment, the strength did not reach reference strength values.
4. The static and dynamic modulus of elasticity due to thermal treatment increased firstly but after being treated at 200 °C they declined. The significant impact of treatment time and temperature was observed.
5. Prediction of static modulus of elasticity using dynamic modulus of elasticity is reliable and could be used for grading purposes of thermally treated wood.

Based on the results of this study, we can state that thermal modification of European Ash wood is more suitable for shorter thermal treatments (up to 3 hours). Concerning the low density and low EMC, longer thermal treatment at the higher temperature is more suitable, but it is limited by a change in mechanical properties. The 3-hour treatment in the oxidized atmosphere up to 200°C gives compression strength and modulus of elasticity higher than those of untreated wood. Moreover, this treatment guarantees good shape stability given by low EMC.

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