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INVESTIGATION OF ELASTIC CONSTANTS FOR BLACK PINE WOOD SUBJECTED TO HEAT TREATMENT

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Abstract:
Effects of heat treatment on elastic constants of Black pine (Pinus nigra A.) wood were investigated. Specimens were exposed to heat under atmospheric pressure at two different temperatures (180 and 210°C) and three different time levels (2, 5, 8 hours). Three modulus of elasticity in the principal anatomical directions, six Poisson's ratios and three Shear modulus values associated with the main directions were evaluated by compression tests. Compression strength of the samples in three principal directions was also determined. All of the properties of the specimens tested were altered by heat treatment. The degree of alteration is correlated with the temperature as well as duration applied. Results indicate that E_L and compression strength in L direction were not significantly influenced, compression strength in R direction significantly decreased, E_R, E_T and compression strength in T direction were increased for shorter periods, then dropped for 8-hour application of 180ºC. E_R was not significantly affected, compression strength in R direction and E_L was significantly decreased, E_T and compression strength in T direction were increased for shorter periods, then decreased for 8-hour application of 210ºC. Shear modulus of the samples were decreased with application of treatment combinations. Most of the Poisson's ratios were not affected by heat treatment.

Key words*: black pine; elastic constants; heat treatment.*

INTRODUCTION

Demands for the architectural use of the heat-treated wood is growing in the design for outdoor and indoor applications (Kuzman *et al.* 2015). The factors contributing to the increase in this demand are decrease of durable timber, deforestation, the increase in the demand for sustainable building materials and some restrictive regulations (Boonstra 2008) Heat treated wood offers superior durability and dimensional stability comparing to untreated wood. Heat treatment is also environmentally friendly process comparing to chemical treatments (Cao *et al.* 2022). In the heat-treatment process, the temperature which wood is exposed can exceed 200°C depending on the species used and the desired material properties (Kocaefe *et al.* 2008). Along with the improvement in many properties, some losses in mechanical properties are also observed, which may limit the potential use of thermally treated wood in structural applications (Fajdiga *et al.* 2016), due to the degradation processes of the cell-wall components, especially the hemicelluloses, through the thermal treatment (Fengel and Wegener 2003; Windeisen *et al.* 2009).

Different industrial processes appeared due to increase of demand for heat treated wood in different countries during the last decades. Increase in demand also reflected in the number of investigations conducted on different wood species. While selected properties for heat treated wood species can be found in Esteves and Pereira (2009), in general, color change, equilibrium moisture content (EMC), dimensional stability, bending properties and compression strength were studied in most investigations. Studies investigating elastic constant of heat treated wood is limited to a few references. A study by Gomez-Royuela *et al.* 2021 focused on the elastic constants of heat treated beach wood and revealed that elastic modulus is more sensitive to heat treatment than the Poisson ratios.

Pinus nigra known as European black pine or black pine, is a fast-growing tree which has a scattered distribution across Europe and Asia Minor (Enescu *et al.* 2016). Previous studies on the mechanical properties of heat-treated black pine wood have mainly focused on modulus of elasticity, bending strength and compression strength properties. Reported properties emphasized that heat treated black pine wood had lower bending properties (Akyıldız *et al.* 2009; Kol 2010; Dündar *et al.* 2012; Bal 2014; Akyürek *et al.* 2021) and lower compression strength (Gündüz et al. 2008; Gündüz and Aydemir 2009; Akyürek *et al.* 2021). Contrary to those Kol (2010), Kamperidou and Barboutis (2017) reported that heat treatment resulted an increase in the compression strength of black pine wood which is similar that of stated by Thermowood handbook (2003).

Elastic constants are important in the structural modeling and design. While elastic properties in L direction for heat treated wood species have been studied extensively, the available information on the perpendicular directions is scarce.

OBJECTIVE

Elastic and strength properties based on the three dimensional approach are essential input parameters required for advanced computational models such as finite elements used in engineering analysis. In this study, elastic constants for heat treated Black pine are determined using compression tests under constant moisture conditions. Compression strength in three orthotropic directions was also evaluated.

METHOD

Small clear wood samples were prepared from Black pine logs which harvested from Bucak Forest District in Turkey. 25 x 100mm in cross section radial or tangential planks were prepared for heat treatment. Before testing and after heat treatment process which were applied in a temperature-controlled oven under atmospheric pressure, specimens were conditioned in climatic chambers at 65± % relative humidity (RH) at a temperature of 20±2°C. Specimens were exposed to heat under atmospheric pressure at two different temperatures (180, 210°C) and three different time levels (2, 5, 8 hours). Wood MC was determined by the oven-drying method. Apparent densities of the samples were calculated using stereo-metric method which based on measurements of the sample volume and mass.

Compression tests (Fig. 1) were conducted using a bi-axial extensometer with a loading speed of 2mm/minute on 20 x 20 x 60mm samples (Fig. 2). The following elastic constants were determined: modulus of elasticity in longitudinal (E_L) radial (E_R) and tangential (E_T) directions, shear modulus in three principal planes (G_{LR}, G_{LT}, G_{RT}) and six Poisson's ratios (ν_{LR}, ν_{LT}, ν_{RL}, ν_{RT}, ν_{TL}, ν_{TR}). Compression strength (CS) in three principal directions was also determined. The stress-strain curves obtained were used in order to evaluate elastic constants of the specimens. Modulus of elasticity was calculated from the ratio of the stress σ to the strain ε measured in the linear elastic range:

$$E_i = \frac{\Delta\sigma_i}{\Delta\varepsilon_i} = \frac{\sigma_{i,2}-\sigma_{i,1}}{\varepsilon_{i,2}-\varepsilon_{i,1}} \quad i \in R, L, T \tag{1}$$

The Poisson's ratio is defined as:

$$\nu_{ij} = -\frac{\varepsilon_j}{\varepsilon_i}, \quad i, j \in R, L, T \text{ and } i \neq j \tag{2}$$

where: ε_i represents the active strain component in the load direction and ε_j is the passive (lateral) strain component, which was determined in the linear elastic range from the linear regression of the passive-active strain diagram. Since the strength behavior of wood in R and T directions is obscure, maximum compression strength was calculated using 0.2% yield values using following formula.

$$CS = P_{max}/A \tag{3}$$

where: CS represents compression strength, P_{max} is the yield load and A is the cross-sectional area of the specimen.

Shear modulus of the specimens with 45 ° angle in planes LR, LT and RT was determined using the following:

$$G_{LR}=\frac{\tau_{LR}}{\gamma_{LR}}=\frac{\sigma_V}{2\,(\varepsilon_H-\varepsilon_V)} \tag{4}$$

$$G_{LT}=\frac{\tau_{LT}}{\gamma_{LT}}=\frac{\sigma_V}{2\,(\varepsilon_H-\varepsilon_V)} \tag{5}$$

$$G_{RT}=\frac{\tau_{RT}}{\gamma_{RT}}=\frac{\sigma_V}{2\,(\varepsilon_H-\varepsilon_V)} \tag{6}$$

where: σ_V = average vertical stress, ε_H = average horizontal strain, ε_V= average vertical strain. More detailed information on calculation of shear modulus from angled specimens in compression tests can be found in Aira *et al*. (2014).

Fig. 1.
Compression test using bi-axial extensometer.

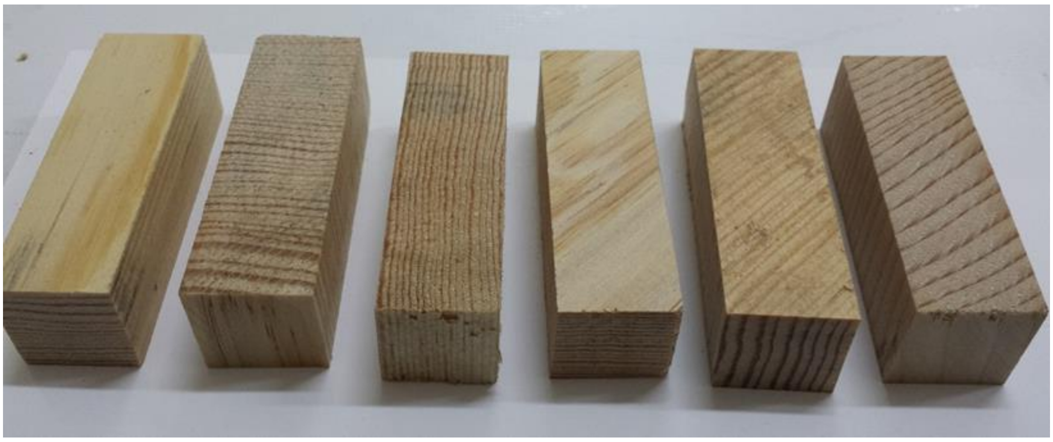

Fig. 2.
Compression test samples (Control samples).

RESULTS AND DISCUSSION

Average values for modulus of elasticity and compression strength in principal directions as affected by heat treatment are presented Table 1-3. Modulus of elasticity and compression strength of the samples tested in this study were significantly modified by heat treatment. In general, the degree of modification is associated with the fiber direction as well as the treatment applied. When 180ºC heat was applied, the changes in the modulus of elasticity and compression strength in L direction are not significant. Insignificant effects of heat treatment up to 200ºC on bending MOE of Black pine were also reported by Bal (2014). On the contrary, significant decrease in bending MOE of Rowan wood when exposed to heat treatment up to 180ºC was presented by Korkut and Budakçı (2009). According to Boonstra *et al*. (2007) degradation of the hemicelluloses and increase of the relative amount of crystalline cellulose could contribute to the increase of stiffness. When 210ºC heat was applied, the modulus of elasticity in L directions significantly decreased up to % 16. Compression strength in L direction is also seemed to increase, but the differences are insignificant. Effect of heat treatment on compression strength is controversial in the literature. Some authors (Ünsal and Ayrılmış, 2005; Korkut *et al.* 2008a; Korkut *et al.* 2008b) found some degree of decrease in L direction by heat treatment, while others (Boonstra *et al.* 2007; Boonstra and Blomberg 2007; Altınok *et al.* 2010; Kamperidou *et al.* 2014; Hannouz *et al.* 2015) reported an increase with heat treatment. The conflict may be a result of lower MC, thus slight increase in compression strength, but this increase is superseded by the degradation of the chemical components and lower densities.

The decrease in the compression strength of R direction is significant for all treatment combinations. When 180ºC heat was applied, the modulus of elasticity in the R direction increased up to 5 hours, then decreased for 8 hours. When 210ºC applied, although there is a decreasing tendency for the shorter periods, the differences are not significant. For the T direction, heat treatment significantly increases both the modulus of elasticity and compression strength for the most of treatments except when 210ºC was applied for 8 hours. Kubojima *et al.* (1998) observed that the modulus of elasticity in L and R directions increased in the first two hours of treatment and remained constant afterwards for *Picea sitchensis* treated at 120, 160 and 200°C. At 200°C, the modulus of elasticity increased in the beginning, decrease after that. Boonstra *et al.* (2007) reported a decrease of 43% in radial compressive strength and a slight increase (8%) of tangential compressive strength for Scots pine wood when subjected to commercial heat treatment less than 200ºC.

Table 1

Modulus of elasticity and compression strength influenced by heat treatment in L direction

T (°C)	D (Hour)	d (gr/cm^3)	MC (%)	E_L (N/mm^2)	% Change	Compression Strength (N/mm^2)	% Change
180	0	0.50	12.1	7286 (9)*	0	32.58 (7)	0
	2	0.47	9.2	7470 (10)	2.53	33.89 (7)	4.02
	5	0.45	8	7207 (9)	-1.08	35.3 (6)	8.35
	8	0.44	6.15	6389(12)	-12.31	33.51(5)	2.85
210	0	0.49	11.9	9464 (10)	0	36.3(8)	0
	2	0.45	7.1	8886 (11)	-6.11	39.2 (10)	7.99
	5	0.43	5.8	8070 (14)	-14.73	38.77 (11)	6.80
	8	0.41	4.6	6640 (11)	-29.84	36.55 (13)	0.69

*Values in parenthesis are coefficient of variations, T = temperature, D = duration, MC= Moisture content

Table 2

Modulus of elasticity and compression strength influenced by heat treatment in R direction

T (°C)	D (Hour)	d (gr/cm^3)	MC (%)	E_R (N/mm^2)	% Change	Compression Strength (N/mm^2)	% Change
180	0	0.51	12.15	1263 (25)	0.00	5.84 (10)	0.00
	2	0.46	7.5	1278 (25)	1.19	5.36 (11)	-8.22
	5	0.43	6.5	1450 (17)	14.81	5.14 (5)	-11.99
	8	0.40	6.1	530 (43)	-58.04	4.86 (6)	-16.78
210	0	0.55	11.8	1084 (29)	0.00	6.48 (9)	0
	2	0.52	7.2	853 (30)	-21.31	5.51 (7)	-14.97
	5	0.49	4.9	904 (12)	-16.61	5.06 (6)	-21.91
	8	0.47	4.5	1019 (27)	-6.00	3.78 (6)	-41.67

* Values in parenthesis are coefficient of variations, T = temperature, D = duration, MC= Moisture content

Table 3

Modulus of elasticity and compression strength influenced by heat treatment in T direction

T (°C)	D (Hour)	d (gr/cm^3)	MC (%)	E_T (N/mm^2)	% Change	Compression Strength (N/mm^2)	% Change
180	0	0.53	11.9	509 (11)	0	5.67 (6)	0
	2	0.50	6.98	500 (9)	-1.77	5.91 (6)	4.23
	5	0.47	6.5	740 (9)	45.38	6.88 (7)	21.34
	8	0.43	6.2	558 (9)	9.63	6.44 (10)	14.11
210	0	0.52	12.2	487 (6)	0.00	5.33 (5)	0.00
	2	0.50	7.1	609 (7)	25.05	6.67 (9)	25.14
	5	0.49	5.6	537(9)	10.27	7.13 (6)	33.77
	8	0.44	4.4	374 (8)	-23.20	4.66(8)	-12.57

* Values in parenthesis are coefficient of variations, T = temperature, D = duration, MC= Moisture content

Hannouz *et al.* (2015) reported that heat treatment increases the elastic modulus in the perpendicular direction for ash wood when exposed to 210ºC for 2 hour during commercial heat treatment procedure. According to Bergander and Salmén (2002) hemicellulose in the S1 and S3 layers controls the radial mechanical properties of wood and cellulose microfibrils mainly govern the mechanical properties in L directions. Most investigations (Hillis 1984; Kotilainen 2000; Esteves *et al.* 2008; Windeisen *et al.* 2009) agree that changes or loss of hemicelluloses play key roles in the strength properties of wood heated at high-temperatures. Loss of hemicelluloses results an increase in the degree of crystallinity of wood, besides those changes related to degradation/rearrangement of the amorphous cellulose content (Hill 2006). Increase in the degree of crystallinity and in the amount of lignin may be responsible some improvements shown by tested properties.

The effects of heat treatment on shear modulus are presented in Table 4. Shear modulus of heat treated wood is widely unknown. Available literature is limited to mostly shear strength which is greatly reduced by heat treatment. According to Hannouz *et al.* (2015) with a decrease of 74%, shear strength is the most negatively impacted mechanical property by heat treatment. Reduction of the shear strength can be explained by the degradation of the hemicelluloses in the middle lamella, thus reducing the load sharing capacity between cellulose microfibrils/fibrils (Stamm 1964). On the other hand, an increased cross linking within the lignin polymer network could have a positive effect on the shear strength. Radial cracks developed during the thermal treatment leads to lower shear strength (Boonstra *et al.* 2007).

In general, shear modulus of wood is positively affected by density and negatively affected by moisture content, temperature and loading direction (Brandner *et al.* 2007). Since densities of the heat treated wood tends to decline, lower shear modulus values are expected for heat treated wood. Test results indicated that heat treatment significantly reduces the values of shear modulus, but the decrease is more apparent in the RT plane. Final shear modulus values of treated samples were up to % 62 lower than control samples. A study by Kubojima *et al.* (1998) indicated that the shear modulus in longitudinal and radial directions increased for softer treatments and decreased for severe treatments. According to Esteves and Pereira (2009) heat treatment may cause some micro-cracks between the S1 and S2 layers, in the middle lamella or more open structures of wood. Some other anatomical changes due to the heat treatment such as increase of pores or porosity can also be expected. Extreme decrease of shear modulus for Black pine wood in can be due to these anatomical changes and reduced load carrying capacity of degraded hemicelluloses in the middle lamella.

Table 4

Shear modulus for heat treated Black pine wood

T ° C	D (hour)	d (g/cm^3)	G_{TR} (N/mm^2)	% change	G_{LT} (N/mm^2)	% change	G_{LR} (N/mm^2)	% change
180	0	0.53	122 (14)	0	810 (16)	0	1085(13)	0
	2	0.50	105 (15)	-14	670 (16)	-17	1150 (11)	-6
	5	0.49	78 (29)	-36	616 (18)	-24	785 (23)	-27
	8	0.47	68 (15)	-44	547 (24)	- 32	740 (25)	-32
210	0	0.55	164 (18)	0	1130 (14)	0	1280 (14)	0
	2	0.52	135 (17)	-18	905 (10)	-17	900(19)	-30
	5	0.49	91(26)	-44	770 (27)	-31	810 (16)	-37
	8	0.47	62 (15)	-62	690 (31)	-39	725 (25)	-43

* Values in parentheses are coefficient of variations

The effects of heat treatment on Poisson's ratios are presented in Table 5. According to Kretschmann (2010) Poisson's ratios vary within and between species and are affected by moisture content and specific gravity. Since heat treatment lowers the density and moisture content of the samples some changes in the Poisson's ratios might be expected. Statistical analysis indicates that the effects of heat treatment on most of the Poisson's ratios are not significant for Black pine wood samples. Only υ_{RT} and υ_{RL} when 180ºC applied and υ_{TL} when 210ºC applied were significantly increased. The effects of heat treatment on samples can be confined by high coefficient of variations, lower densities and moisture content of the samples. Higher coefficient of variation in the Poisson's ratios of wood was also presented by Kretschmann and Green (1996), Hering *et al.* (2012), Ozyhar *et al.* (2013), Jeong *et al.* (2010), Mizutani and Ando (2015). It appears that no explanation has been presented regarding the effects of heat treatment on Poisson's ratios for wood materials.

Table 5

Poisson's ratios for heat treated Black pine wood

Temp (°C)	Duration (Hour)	υ_{TR}	υ_{TL}	υ_{RL}	υ_{RT}	υ_{LR}	υ_{LT}
180	0	0.52 (28)	0.079 (47)	0.119 (30)	0.58 (33)	0.49 (43)	0.53 (18)
	2	0.59 (22)	0.078 (38)	0.156 (42)	0.82 (10)	0.57 (30)	0.55 (21)
	5	0.61 (32)	0.118 (40)	0.275 (29)	0.84 (10)	0.54 (23)	0.57 (16)
	8	0.51 (43)	0.074 (17)	0.152 (40)	0.74 (12)	0.46 (27)	0.54 (21)
210	0	0.6 (15)	0.08 (43)	0.148 (48)	0.75 (9)	0.5 (24)	0.58 (20)
	2	0.71 (21)	0.072 (29)	0.162 (39)	0.71 (17)	0.7 (12)	0.8 (11)
	5	0.56 (40)	0.063 (55)	0.186 (22)	0.77 (17)	0.7 (10)	0.89 (7)
	8	0.58 (30)	0.077 (65)	0.156 (29)	0.84 (6)	0.69 (16)	0.87 (5)

* Values in parentheses are coefficient of variations

According to Taniguchi and Ando (2010) the mechanism of Poisson's effect in wood is greatly dominated by the microscopic/macroscopic tissue structure of wood. Morooko *et al.* (1979) and Ohgama (1982) have shown that the porous structure of wood affects mainly the Poisson's ratios (υ_{RT}, υ_{TR}), according to the results of numerical analysis using a model.

CONCLUSIONS

The results of the study show that heat treatment does not proceed to a simple degradation with the same level on all elastic properties tested: some properties are more affected than others by the heat treatment. The most significant change occurred for the shear modulus of the samples tested, which was drastic decrease of the values for all treatment combinations. The modules of elasticity values were responded differently to the heat treatment. While softer treatments did not change or increased the modulus of elasticity, severe treatments decreased it. Compression strength in L direction did not significantly influenced by heat treatment. While compression strength in R direction severely reduced, compression strength in T direction was increased for softer treatments. Poisson's ratios seem to be less sensitive to heat treatment process. Results of the study also show that heat treatment did not change the stiffness order of in which $E_L>E_R>G_{LR}>G_{LT}>E_T>G_{RT}$.

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