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INVESTIGATION OF BENDING STRENGTH OF TANNIN IMPREGNATED WOODEN BEAMS AFTER HEAT TREATMENT

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ABSTRACT

In this study, the changes in bending strength were investigated by applying heat-treatment to laminated beams modified with acorn tannin to improve the mechanical properties of wooden load-bearing structural members. For this purpose, acorn tannin was impregnated on samples prepared from Scotch pine (*Pinus sylvestris* L.), oak (*Quercus petraea* L.), and chestnut (*Castanea sativa* Mill.) woods. Heat treatment was applied to the samples impregnated with acorn tannin at 150ºC for 3 hours. Untreated, heat-treated, and tannin-modified samples were conditioned until they reached constant weight at 20ºC at 65% relative humidity (RH), 40ºC at 35% RH, and 10ºC at 50% RH. Bending resistance tests were applied to the elements that are conditioned in outdoor conditions according to ISO 13061-3. The results of bilateral interaction between tree species and treatment type were compared, the highest bending strength increase was found in Scotch pine samples by 5% compared to control samples.

KEYWORDS: Heat treatment, wood modification, acorn tannin, bending strength, wooden beams.

INTRODUCTION

In wooden buildings, the combined effects of insects, fungi, weather conditions, and ultraviolet radiation from the sun can lead to wood decomposition and a reduction in its physical and mechanical strength over time (Esteves 2009). Throughout history, wood has been an essential source of raw material for humanity. However, the gradual depletion of forest resources has necessitated more efficient processing methods to ensure its prolonged use (Kurtoglu 2000). The use of wood as a structural material in buildings began in the late 18th century, enabling greater architectural freedom. Furthermore, innovative techniques and adhesives have facilitated the production of various wood-based products, including "glue-laminated constructions" (Kayakiran et al. 2019). As technology progresses, impregnation techniques are increasingly employed to protect wood from harmful factors and prevent deterioration. The present-day trend towards modifying wood materials using environmentally friendly and non-hazardous methods has been growing, with heat treatment being one of the popular approaches in this field (Bourgois et al. 1989). Heat treatment, known as "ThermoWood" in the woodworking industry, is implemented in various countries worldwide, but under different names and techniques. For instance, Finland's "ThermoWood" method utilizes hot steam for heating wood materials, while the Plato method in the Netherlands employs a combination of hot air and steam. In France, a method called "rectification" uses inert gas, and in Germany, "oil heat treatment" is conducted using hot oil (Ayata et al. 2017). Through the application of different wood modification techniques, it is possible to enhance the biological resistance, weather resistance, and dimensional stability of wood materials, thereby expanding their range of applications (Németh et al. 2020).

The dimensional stability of the heat-treated wood material increases and its dehumidification properties decrease in both the radial and tangential directions. In addition, heat treatment causes some improvements in the physical properties of the material. (Azis et al. 2020). It was observed that the mechanical properties decreased in the samples exposed to high temperatures and long-term thermal modification (Xie et al. 2020). When examining the mass losses of heat-treated wood materials, it is observed that the losses increase as the process temperature and application time are elevated. This suggests a positive correlation between the level of mass loss and the severity of the heat treatment process. (Hillis 1984). The effect of heat treatment applied between 160°C and 180°C on the mechanical properties of bamboo wood was investigated. It provided an average of 8% increase in the modulus of elasticity (MOE) of the samples kept at 160°C and for a short time. (Zhang et al. 2019). It was determined that both MOE and MOR values increased by prolonging the processing time in bamboo wood (*Phyllostachys pubescens*, 4 years old) treated with saturated steam at 140°C (Wang et al. 2020). When its temperature or time exceeds a certain value, a significant decrease in the mechanical properties of bamboo can be observed. For example, there was a 12.3% decrease in MOR when bamboo wood heat-treated at 180°C for 4 h. It showed a further decrease when the temperature exceeded 200°C (Zhang et al. 2013). Dimensional stabilization and biological strength are improved at heat treatment temperatures above 150°C, but with the increase in temperature, mechanical properties decrease and the chemical properties of the wood change (Bekhta end Niemz 2003). This unfavorable situation has limited some uses of heat-treated wood (Aytekin et al. 2009).

Heat treatment applied at a temperature of 160°C can lead to a reduction in the moisture content and size of wood materials, as well as an improvement in certain mechanical properties. This may contribute to greater stability and resistance to deformation, such as rotting, warping, or bending. However, as the process temperature exceeds 200°C, there is a rapid decline in both mechanical properties and mass of the material. Heat treatment was observed that both MOE and MOR values increased when Keruing and Light red meranti hardwood lumber were heat-treated to 150°C, 170°C and 190°C temperature levels for 1 h. It was observed that the values decreased in the heat treatment at 210°C (Noh et al. 2017). The adsorption ability and mechanical properties of the rubber tree (*Hevea brasiliensis*) were investigated by heat treatment at 140°C, 150°C, 160°C with hot steam for 1, 2 and 3 h. Pressure resistance values were higher in samples treated at 140°C and for 3 h. It was observed that the equilibrium moisture content decreased at all temperatures (Patcharawijit et al. 2018). Samples prepared from the poplar tree (*Populus alba* L.) were heat-treated at 120°C, 150°C, 180°C and 210°C for 2 and 4 h. There were improvements in the swelling and shrinkage properties of the wood, but reductions in bending strength and modulus of elasticity occurred. The best heat treatment results in mechanical properties were obtained at 120°C for 2 h at low temperatures. Heat treatment applications above 150°C or over 4 h are not recommended because they cause a decrease in mechanical properties (Kaymakci et al. 2021). Oriental beech (*Fagus orientalis* L.) samples impregnated with acorn tannin were heat-treated at 150°C, 175°C and 200°C for 2 and 5 h. The bending strength and compressive strength parallel to the fibers increased slightly at low temperatures, but these resistances decreased at high temperatures (Percin et al. 2016).

Scotch pine, which was impregnated with a mixture of tannin compounds, was subjected to heat treatment at 190°C for 4 h and kept in outdoor conditions for specified periods. It has been found that wood samples treated with tannin show effective results against rotting organisms (Lopes et al. 2020). White oak (*Quercus alba* L.) specimens, which were conditioned at 20°C and 65% RH, were heat-treated at 160°C, 180°C and 200°C for 3, 6 and 9 h. It was determined that the MOR values decreased as the temperature values increased. The MOR value of the untreated samples was 203.85 N/mm^2, the minimum decrease was 202.36 N/mm^2 in the samples treated at 160°C for 3 h, and the maximum decrease was 169.28 N/mm^2 in the samples treated at 200°C for 9 h (Xu et al. 2019). Samples made of chestnut wood were glued and laminated with PVAc-D4 glue. After the samples with and without tannins were brought to 103°C ± 2°C and 65 ± 5% RH (12% equilibrium humidity) in an oven, heat treatment was applied at 180°C and 2 h. The highest bending strength was obtained in the samples without tannin-heat treatment, and the lowest in the samples with no tannin-heat treatment. The compressive strength was 51.2 N/mm^2 in the control samples, the highest at 58.22 N/mm^2 in the tannin-treated samples and 55.12 N/mm^2 in the tannin-treated samples (Gunduz et al. 2011). Higher values of compressive strength and modulus of elasticity were obtained in fir wood, which was heat-treated at low temperatures, compared to the control samples (Ozkan et al. 2017, Kol 2010, Korkut 2008).

When previous studies were examined, it was understood that the mechanical properties could be improved by heat treatment under 160°C after impregnation with various chemicals. This study was carried out in the following order. Firstly, Scotch pine (*Pinus sylvestris* L.), oak (*Quercus petraea* L.) and chestnut (*Castanea sativa* M.) wood, which are widely used in wooden structures, were impregnated with tannin. Secondly, samples with tannin impregnation were subjected to low-temperature heat treatment. Third, the treated samples were laminated and conditioned in the conditioning cabinet. Bending strength tests were applied to the beam samples, and it was determined that mechanical properties could be improved after tannin impregnation and heat treatment.

MATERIAL AND METHODS

Material

Scotch pine (*Pinus sylvestris* L.), oak (*Quercus petraea* L.) and chestnut (*Castanea sativa* M.) woods were used in this study due to their widespread use in structural applications. Timbers with air-dry moisture (approximately 12%) were obtained from Turkey-Ankara-Siteler district by random selection method. Care has been taken to ensure that the timbers are straight, fibrous, knot-free, and sapwood.

Tannin solution

Acorn tannin is an environmentally friendly and natural material obtained by extraction method from acorn, a fruit of the oak tree, which is abundant in forest regions. Tannin is most commonly used in the leather industry in Turkey and in the world. The acorn tannin used in the experiments was supplied from the manufacturer in powder form. It was prepared by dissolving 10% in distilled water heated up to 80°C to make it a solution.

Preparation of experimental samples

The samples to be used in the experiments were prepared according to ISO 3129 standards from randomly selected 1st class trees, without cracks, knots, gaps, smooth fibers, density and color differences. The dimensions of the test samples were reduced to a 1/5 scale and were prepared in the dimensions of 32 x 64 x 880 mm.

Methods

Determination of air-dry density was provided according to Eq. 1. Samples with dimensions of 20 x 20 x 30 mm were prepared in accordance with ISO 13061-2 standards, and the samples were kept at 20 ± 2°C and 65 ± 5% RH climate conditions until they reached a constant weight. Sample weights were weighed on a scale with a sensitivity of 0.01 g and their dimensions were measured with a digital caliper with a sensitivity of 0.01 mm.

$$\delta_{12} = \frac{M_{12}}{V_{12}} g/cm^3 \quad (1)$$

where: M_{12} - air-dry weight (g), V_{12} - air-dry volume (cm^3).

The samples were immersed in 10% acorn tannin solution so that all of them remained in solution and kept for 24 h. The solution residues on the samples removed from the immersion vessel after waiting were cleaned and made ready for applying heat treatment. After impregnation, the samples were kept in an environment with air circulation until they became air-dry. Retention amounts and rates were determined by weighing their weights on a digital scale with a sensitivity of 0.001 g.

The first stage of heat treatment is high temperature drying. The humidity in the wood material before the heat treatment has no effect on the heat treatment. No conditioning was done before the heat treatment. Heat treatment can be applied to freshly cut wood or any dry wood. The most time-consuming phase in the heat treatment process is the drying process. As seen in Fig. 1, the temperature was increased to 100°C in 6 h by sending superheated steam at 3 atm pressure into the oven, and then the temperature was increased to 130°C in 12 h and high temperature drying was carried out. In this way, the humidity of the wood has been reduced to 0%. After the high temperature drying (1st stage) was completed, the oven temperature was increased to 150°C in 6 h (2nd stage). It was kept at these temperatures for 3 h and the cooling phase (3rd phase) was started. In the cooling part, which is the last stage of the heat treatment, the temperature inside the furnace was reduced to 50°C in 12 h.

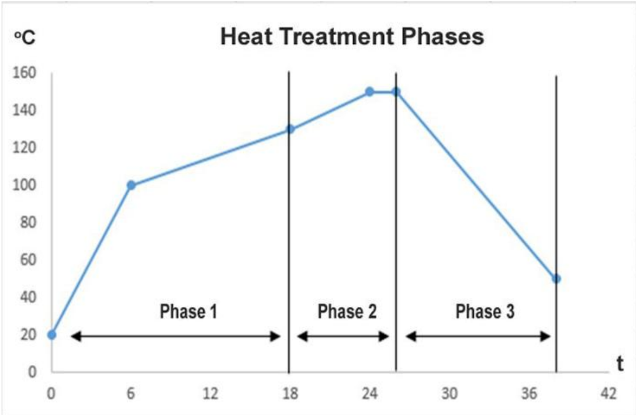

Fig. 1: Heat treatment temperature-time graph.

After heat treatment at 150°C for 2 h, two pieces of 32 x 32 x 880 mm moisture-cured D4-specific polyurethane glue (PUR) in EN 204 standards are adhered under the press in the radial direction, with 32 x 64 x 880 mm dimensions according to DIN EN 386 laminated. Laminated samples were conditioned, respectively, until they reached constant weight at 20ºC and 65% RH, 40ºC and 35% RH, and 10℃ and 50% RH.

Bending strength and modulus of elasticity in bending tests were carried out at Gazi University, Faculty of Technology, Woodworking Industrial Engineering Department, Wood Technology Laboratory. The samples (Fig. 2) were placed in the universal test device in accordance with the principles of bending strength according to ISO 13061-3 and the forces obtained from the tests were calculated by putting them in their places in Eqs. 2 and 3:

$$\sigma_e = (3 \cdot P_{max} \cdot l) \,/\, (2 \cdot b \cdot h^2) \quad (2)$$
$$E_m = (\Delta F \cdot l^3) \,/\, (4 \cdot b \cdot h^3 \cdot \Delta f) \quad (3)$$

where: P_{max} - the maximum force applied at the time of fracture (N), P - load equal to the difference between the arithmetic mean of the lower and upper limits of the loading (N), ΔF - force difference in the region of elasticity (N), l - spacing between support points (mm), b - width of test specimen (mm), h - height of the test specimen (mm), f - deflection in the net bending area (mm).

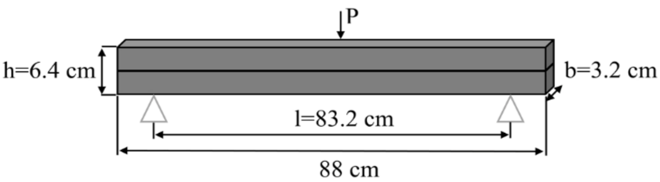

Fig. 2: Bending strength test.

RESULTS AND ANALYSIS

In order to determine the effect of modification processes on the bending strength in laminated wood materials, multiple variance analysis was performed at $p < 0.05$ significance level. Duncan test was applied to determine the homogeneity groups when the effect of tree species, treatment type and climatic conditions on the tested properties was found to be significant. The air-dry density averages of the test samples are given in Tab. 1. and the analysis of variance (ANOVA) values are given in Tab. 2.

Tab. 1: Air-dry density averages (g/cm^3).

Tree species	Number of samples	Total	X	s	Min	Max
Scotch pine	10	4.415	0.442	0.01	0.427	0.453
Oak	10	8.353	0.835	0.01	0.846	0.821
Chestnut	10	4.357	0.436	0.01	0.426	0.445
Total	30	17.125	0.571	0.19		

X - average, s - standard deviation.

Tab. 2: Air dry (ANOVA) analysis of variance.

Variance source	Degrees of freedom	Sum of squares	Mean squares	F-value	Significance level p < 0,05
Between groups	2	1.049	0.525	6686.000	0.000
In-group	27	0.002	0.000		
Total	29	1.051			

Air-dry density values of wood species were found to be significant at $p < 0.05$ significance level. According to Duncan test results, the highest air dry density values were found in oak wood (0.835 g/cm^3), Scots pine wood (0.442 g/cm^3) and chestnut wood (0.436 g/cm^3) respectively.

Retention amount and rates

Scotch pine (*Pinus sylvestris* L.), oak (*Quercus petraea* L.) and chestnut (*Castanea sativa* M.) samples, which were determined by random method, were dipped in 10% acorn tannin solution and kept for 24 h to be impregnated. The amounts, ratios and densities of natural wood preservatives attached to the samples were determined by the weighing method and given in Tab. 3.

Tab. 3: Density values, retention amount and rates of the samples.

Tree type	Scotch pine	Oak	Chestnut
Air dried (g/cm^3)	0.5112	0.7337	0.5174
Tannin air dried (g/cm^3)	0.4862	0.7109	0.4881
Amount of retention (g)	2.76	1.23	1.87
Retention (%)	9.04	5.03	7.32

After the treatment of the impregnated samples, the amount of tannin adsorption penetrating into the samples was calculated in Eq. 4 and the percent retention rates were calculated according to Eq. 5:

$$R = [(T2 - T1) \cdot C) \; / \; V] \cdot 10 \tag{4}$$
$$R(\%) = [(Moes - Moeo) \; / \; Moeo] \cdot 100] \tag{5}$$

where: *T2* - sample weight after impregnation (g), *T1* - sample weight before impregnation (g), *Moes* - full dry weight after impregnation (g), *Moeo* - sample full dry weight before impregnation (g), *V* - sample volume (cm^3), *C* - solution concentration (%).

Bending strength

Bending strength values of tree species (A), treatment type (B), tree species - treatment type (AxB) binary interaction were found significant at $p < 0.05$ significance level (Tab. 4).

Tab. 4: Multiple variance analysis of bending strength.

Variance source	Degrees of freedom	Sum of squares	Mean squares	F-value	Significance p < 0,05
Tree species (A)	2	12977.804	6488.902	27.9743	0.0000
Treatment type (B)	2	4788.844	2394.422	10.3226	0.0001
AxB	4	6214.342	1553.585	6.6976	0.0000
Error	126	29226.937	231.960		
Total	134	53207.927			

The homogeneity groups of tree species are shown in Tab. 5 and the homogeneity groups of the treatment type are shown in Tab. 6. Among the tree species, the bending strength changes were highest (89.04 N/mm^2) in oak wood, then scotch pine (80.76 N/mm^2), and the lowest (65.38 N/mm^2) in chestnut wood (Fig. 3a). The bending strength of the heat-treated samples was lower than the control samples (Hao et al. 2021). Based on the bending strength of the control samples, heat treatment reduced the bending strength by 14.9%. Tannin + heat treatment application, on the other hand, increased the bending strength slightly (74.52 N/mm^2) compared to the heat-treated samples, reducing the degrading effect of the heat treatment (Fig. 3b).

Tab. 5: Homogeneity groups of tree species.

Tree Species	X (N/mm^2)	HG
Scotch Pine	80.76	B
Oak	89.04	A
Chestnut	65.38	C
LSD value = 6.350		

Tab. 6: Homogeneity groups of the treatment.

Treatment	X (N/mm^2)	HG
Control	86.81	A
Heat treatment	73.89	C
Tannin + Heat treatment	74.48	B
LSD value = 6.350		

X- average, HG- homogeneity group, LSD- smallest significant difference.

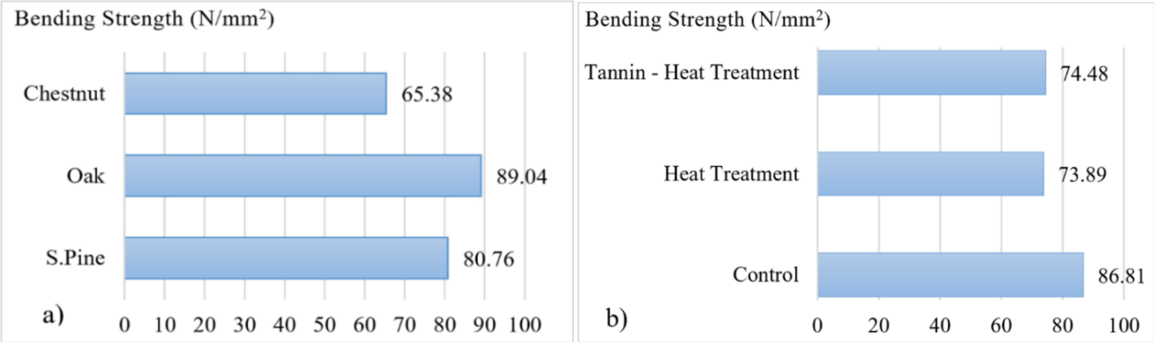

Fig. 3: a) Bending strength of tree species, b) bending strength according to treatment type.

Tree species - homogeneity groups of the treatment type are shown in Tab. 7. It was determined that the bending strength of the tannin-impregnated heat-treated samples increased in Scotch pine samples.

Tab. 7: Tree species - treatment type homogeneity groups.

Tree species +Treatment	X (N/mm^2)	HG
Scotch pine + Control	80.04	BC
Scotch pine + Heat treatment	78.13	BC
Scotch pine + Tannin + Heat treatment	84.09	B
Oak + Control	109.5	A
Oak + Heat treatment	83.02	B
Oak + Tannin + Heat treatment	74.65	CD
Chestnut + Control	70.93	DE
Chestnut + Heat treatment	60.51	F
Chestnut + Tannin + Heat treatment	64.69	EF
LSD value = 6.350		

Compared to the chestnut control samples, there was a decrease in the bending strength of the heat-treated samples, and a slight increase was observed in the samples with tannin supplementation. In trees such as oak and chestnut, the trachea can be clogged with tulle or foreign materials. This may prevent liquid substances from entering the wood (Bozkurt et al. 1997). It has been determined that this process is not suitable for oak, since the tannin solution cannot enter the walls by free dipping method due to veiling in oak wood (Fig. 4).

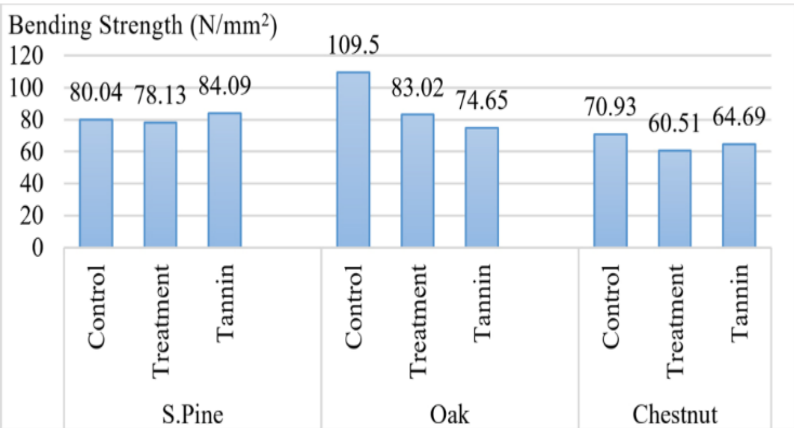

Fig. 4: The effects of wood type - treatment type binary interaction on the bending strength change.

It can be said that tannin-impregnated heat-treated Scotch pine and chestnut wood reduces and delays the degradation of cellulose and lignin components, and therefore reduces the loss of mechanical properties of the wood.

CONCLUSIONS

Among the wood species used in the experiment, oak had the highest bending strength, while chestnut had the lowest. The maximum decrease in bending strength after heat treatment was 24% for oak samples. Tannin-modified and heat-treated samples showed an increase in bending strength values (except for oak) only when compared to the heat-treated samples. This increase was mostly detected in Scots pine wood. Scots pine wood soaked in tannin solution for 24 hours had the highest retention rate at 9.04% and the highest retention amount at 2.76g. Scots pine wood, impregnated with tannin and heat-treated at 160°C for 2 hours, provided a 5% increase in bending strength compared to the control samples and a 7.6% increase compared to the heat-treated samples. Tannin-impregnated and heat-treated Scots pine beam samples may offer advantages by exhibiting higher bending strength performance under outdoor conditions.

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