Bonding Strength of Some Adhesives in Heat-Treated Hornbeam (*Carpinus betulus* L.) Wood Used for Interior and Exterior Decoration

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Heat-treated wood has an ever-expanding market for exterior and interior applications. The objective of this study was to determine the effect of a heat treatment on the bonding strength of hornbeam (*Carpinus betulus* L.) wood that was bonded with melamine formaldehyde (MF), polyurethane (PUR), and polyvinyl acetate (PVAc-D4) adhesives. Hornbeam lamellas were heat treated at 150 °C, 175 °C, 200 °C, and 225 °C for 3 h and then bonded. The bonding strength of the specimens was determined. In addition, the density, weight loss, and pH value of the heat-treated wood were investigated. The results showed that the bonding strengths of the heat-treated wood specimens decreased with the temperature of the heat treatment. The bonding strength of the PUR adhesive was higher than the MF and the PVAc-D4.

Keywords: Heat treatment; Glue; Bonding strength; Hornbeam (Carpinus betulus L.)

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INTRODUCTION

Heat treatment has become an increasingly important commercial application to improve the dimensional stability and bio-durability of wood (Esteves and Pereira 2009). Different thermal treatment methods significantly improve and enhance some properties of wood without the use of chemical additives (Johansson 2008). All of the processes use sawn wood and treatment temperatures between 160 °C and 260 °C. The main differences are in the process conditions (*e.g.*, the process steps, oxygen or nitrogen steaming, wet or dry process, the use of oils, and steering schedules) (Militz 2002).

During heat treatment, a large number of chemical changes occur in the wood components (Pavlo and Niemz 2003). The changes start with the deacetylation of hemicelluloses, followed by depolymerization catalyzed by the released acetic acid. Thermal modification at high temperatures leads to chemical changes of the wood constituents. First the hemicelluloses degrade, due to their low molecular weight and their branching structure, followed by the cellulose and lignin (Fengel and Wegener 1984; Tjeerdsma *et al.* 1998; Sivonen *et al.* 2002; Nuopponen *et al.* 2004).

The strong bonding strength between the wood material and adhesive depends on a number of factors, including the wettability of the wood surface, the roughness of the wood surface, the penetration behavior, the moisture content, the presence of extractives, the hygroscopicity, chemical composition, and the pH of the wood (Sahin Kol *et al.* 2009). The changes in chemical, physical, and structural properties of wood after heat treatment can affect the overall bonding process with an adhesive (Kariz and Sernek 2010). With thermal degradation, wood loses mass to a degree that depends on the heat treatment conditions; at the same time there are significant changes in the surface properties that affect the wood bonding (Gunduz and Aydemir 2009; Kariz and Sernek 2010). Percin and Uzun (2014) examined the effects of a heat treatment on the bonding strength of Scotch pine (*Pinus sylvestris* L.), beech (*Fagus orientalis* L.), oak (*Quercus petraea* L.), and poplar (*Populus nigra* L.). The bonding strength values decreased with increasing treatment temperature. Esen and Ozcan (2012) studied the effects of heat treatment on the shear strength of oak (*Quercus petraea* L.) wood. For this aim, the samples were exposed to heat treatment at 170 °C, 190 °C, and 210 °C, for 2 h, 6 h, and 10 h. The samples were then bonded with phenol formaldehyde (FF), melamine formaldehyde (MF), melamine urea formaldehyde (MUF), and polyurethane (PUR) adhesives. The heat treatment decreased the shear strength values as the treatment temperature and duration increased. The most important factors affecting the shear strength of wood and wood-based material are the wood species, adhesive type, wood density, log temperature during the veneer peeling or clipping, veneer drying temperature, and the relative moisture content of wood (Demirkir *et al.* 2013). Yorur *et al.* (2014) studied the effect of humidity-water-heat tests on the bonding strength of impregnated oak (*Quercus petraea* spp.) bonded with UF, Desmodur-VTKA (Desmodur-Vinil trieketonol acetate), and polyvinyl acetate (PVAc) adhesives. The bonding strengths of impregnated wood samples exposed to humid-resistance test samples decreased, and the bonding strength of VTKA was higher than that of the UF and PVAc adhesives.

Heat-treated wood is mainly utilized in exterior applications such as exterior cladding, window and door joinery, garden furniture, and decking. There are also many indoor applications for heat-treated wood, such as flooring, paneling, kitchen furnishing, and the interiors of bathrooms and saunas. Heat-treated wood might also have potential as a material for construction use, such as for structural elements in the building industry (Korkut 2007; Sernek *et al*. 2008).

Heat-treated wood has a growing market for exterior and interior applications, such as hornbeam (*Carpinus betulus* L.) wood used in the furniture and the woodworking industry. Therefore, knowledge of the bonding strength of heat-treated hornbeam wood may be important for exterior and interior uses. This study evaluated the bonding strength of heat-treated hornbeam wood bonded with exterior and interior structural adhesives including melamine formaldehyde, polyurethane, and polyvinyl acetate, which are used widely in the furniture and woodworking industry.

EXPERIMENTAL

Materials

In this study, hornbeam (*Carpinus betulus* L.) was chosen randomly from a timber supplier in Siteler, Ankara, Turkey. Hornbeam wood has widespread usage in wood working industrial in Turkey. Accordingly, non-deficient, whole, knotless, and normally grown (without zone line, reaction wood, decay, insect or fungal infection) wood materials were selected.

Wood samples with the dimensions of 10 mm × 60 mm × 550 mm were cut from the sapwood parts of air-dried wood planks and conditioned at a temperature of 20 ± 2 °C and 65 ± 5% relative humidity until they reached an equilibrium in moisture distribution.

Melamine formaldehyde (MF), polyurethane (PUR), and polyvinyl acetate (PVAc-D4) were used as the bonding adhesives. PVAc-D4 had the following properties: density, 1.120 g/cm^3; viscosity, 13000 cPs at 20 °C; pH, 7.5 at 20 °C; solid content, 50%; and hardener ratio, 5% (Turbo-Hardener 303,5, was supplied by the Aktif firm, a representative firm of Kleiberit adhesives in Istanbul, Turkey). MF had the following properties: density, 1.215 g/cm^3; viscosity, 60 cPs at 20 °C; pH, 9.3 at 20 °C; and solid content, 53%. PUR had the following properties: density, 1.130 g/cm^3; viscosity, 8000 mPas at 20 °C; pH, 7 at 20 °C; solid content, 100%. For all adhesives, the amount applied was 200 gr/m^2 (Keskin *et al.* 2003; Atar and Ozcifci 2005; Ozcifci *et al.* 2007; Sahin Kol *et al.* 2009).

Heat Treatment of Wood

The samples were subjected to heat treatment at 150 °C, 175 °C, 200 °C, and 225 °C for 3 h in a heat treatment oven which was made by the Atria firm in Istanbul, controlled to within ± 1 °C under steam. The total heat treatment was performed over three continuous stages. The total time of the heat treatment was 45 h, and the duration at this high temperature was 3 h. The heat-treated and control samples were conditioned at a temperature of 20 ± 2 °C and 65 ± 5% relative humidity to the moisture content of about 12%. After the heat treatment, the pH value was evaluated by the extraction method. For this test, 20 g of wood was ground into small particles and soaked in 160 g of distilled water for 24 h. The extract was filtered and analyzed with a portable pH meter (Sernek *et al.* 2008).

Preparation of Test Samples and Test Method

Weight loss

Prior to the heat treatment, samples were dried in a heating oven at 103 ± 2 °C. The oven-dry weight of the samples was determined. After the heat treatment, the oven-dry weight of the same samples was re-measured. The weight loss (WL) of the samples due to the heat treatment was calculated according to the following formula,

$$\mathrm{WL}\ (\%) = 100(W_{\mathrm{BH}} - W_{\mathrm{AH}}) / W_{\mathrm{BH}} \qquad (1)$$

where W_{BH} is the initial oven-dry weight of the sample prior to the heat treatment (g) and W_{AH} is the oven-dry weight of the samples after the heat treatment (g). The equilibrium moisture content (EMC) of the test samples was determined before the tests.

Density

The air-dried density of the samples was determined according to the following formula (ISO 3131 1975 and TS 2472 1976),

$$\mathrm{D}_{12} = M_{12}/V_{12}\ (\mathrm{g/cm^3}) \qquad (2)$$

where M is the weight of the sample (g) and V is the volume of the samples (cm^3).

Bonding strength

The bonding strength was tested according to the BS EN 205 (1991) standard. The samples were cut parallel to grain in the dimensions of 5 mm × 55 mm × 500 mm. The adhesive was applied at the rate of about 200 g/m^2 on a single bonding surface of the veneer, as recommended by the manufacturer, with a cylindrical adhesive spreading apparatus. The wood veneer panels were pressed under 70 kg/cm^2 for 30 min at 120 °C for

MF, and under 70 kg/cm^2 for 120 min at 20 °C and for PUR and PVAc-D4. Test samples were obtained from these panels (Fig. 1).

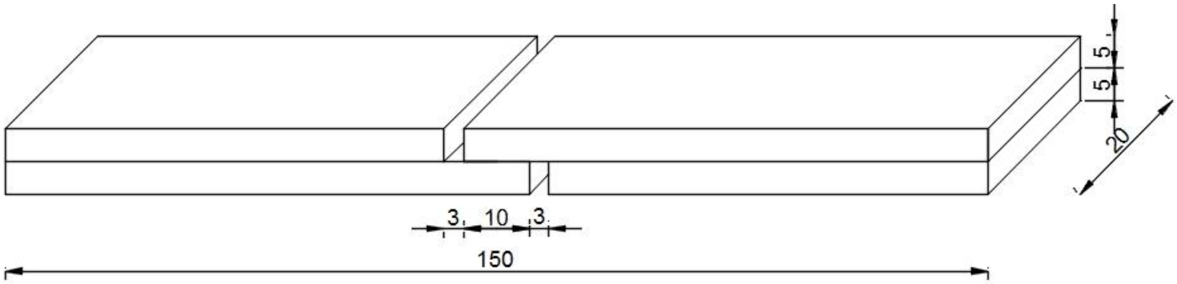

Fig. 1. Bonding strength test sample (mm)

The test samples were conditioned at a temperature of 20 ± 2 °C and 65 ± 5% relative humidity for 30 days. During the tests, force was applied parallel to the glue line, and the loading rate was 2 mm/min. Twenty-two specimens were used for each bonding strength test. The bonding strength (BS) was calculated according to Eq. 3,

$$BS = F_{\max} / a.b \ (\text{N/mm}^2) \tag{3}$$

where $F_{\max}$ is the maximum load (N), a is the width of the glued surface (mm), and b is the length of the glued surface (mm).

RESULTS AND DISCUSSION

The average weight loss due to heat treatment are given in Table 1. As expected, the weight loss varied according to treatment temperature. Weight loss is attributed to the degradation of the wood polymers (hemicelluloses, cellulose, and lignin); in this temperature range, hemicelluloses are the most thermally sensitive wood components (Poncsák *et al.* 2006; Yildiz *et al.* 2006). Gunduz and Aydemir (2009) investigated the weight loss of heat-treated Camiyani black pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood at temperatures of 160 °C, 180 °C, and 200 °C for 2 h and 6 h. Their results showed that increasing the heat treatment temperature increased the weight loss of the test samples. Heat treatment reduced the pH of hornbeam wood from 5.48 to 4.05.

Table 1. Average Weight Loss Values of Test Samples after Heat Treatment

Heat Treatment	Weight Loss (%)	pH
Control	-	5.48
150 °C	0.96	5.02
175 °C	1.71	4.84
200 °C	4.22	4.46
225 °C	6.79	4.05

The average density values of the test samples are shown in Table 2. The air-dried density values of the test samples decreased with an increase in the heat treatment temperature. The wood samples that were heat-treated at 225 °C and bonded with the PUR adhesive had the lowest air-dried density values. The weight of wood material decreases when heat treatment is applied. The changes resulting from heat treatment could be explained by material losses in the cell wall, hemicellulose degradation into volatile products, and the evaporation of extractive substances (Korkut *et al.* 2008).

Table 2. Density Values of Test Samples (g/cm^3)

Heat Treatment	Adhesive					
	MF		PUR		PVAc-D4	
	X	sd	X	sd	X	sd
Control	0.782	0.014	0.768	0.021	0.773	0.017
150 °C	0.768	0.009	0.755	0.016	0.766	0.012
175 °C	0.751	0.016	0.739	0.013	0.748	0.014
200 °C	0.729	0.011	0.712	0.008	0.731	0.015
225 °C	0.702	0.016	0.686	0.009	0.704	0.006

X, average value; sd, standard deviation

Table 3. Equilibrium Moisture Content (EMC) Values of Test Samples (%)

Heat Treatment	Adhesive					
	MF		PUR		PVAc-D4	
	X	sd	X	sd	X	sd
Control	11.8	0.655	11.4	0.557	11.1	0.411
150 °C	10.1	0.422	9.9	0.342	9.7	0.359
175 °C	8.3	0.455	8.1	0.433	7.9	0.467
200 °C	7.2	0.517	7.1	0.359	6.8	0.322
225 °C	5.9	0.405	5.6	0.499	5.7	0.539

X, average value; sd, standard deviation

The average equilibrium moisture content (EMC) values of the test samples are shown in Table 3. Heat treatment may change the wood equilibrium moisture content (EMC). According to Table 3, the EMC values of the heat-treated samples decreased with an increase in the heat treatment temperature. The treatment at 225 °C for wood bonded with the PUR adhesive sample resulted in the lowest EMC values, as previously reported (Jämsä and Viitaniemi 2001; Gosselink *et al.* 2004; Metsä-Kortelainen *et al.* 2006). Epmeier *et al.* (2001) examined the effects of heat treatment on the EMC of spruce wood. The EMC of spruce wood treated at 160 °C changed from 8.7% to 7.0% and from 18.9% to 15.7% at 30% and 90% relative humidity, respectively. The treatment at 190 °C was more effective, decreasing the EMC from 8.1% to 6.5%, and from 18.6% to 14.6% at 30% and 90% relative humidity, respectively.

The average values of the bonding strength obtained for the different adhesives and heat treatment temperatures are given in Table 4. The highest bonding strength was with the control samples, and the bonding strength of the test samples decreased with an increase in the heat treatment temperature. The lowest bonding strength values were obtained from the samples heat-treated at 225 °C (7.75 N/mm^2), where the total loss compared with the control sample was 25.3%. Poncsak *et al.* (2007) studied the effects of heat treatment on the bonding strength of Scotch pine (*Pinus sylvestris*), aspen (*Populus tremuloides*), yellow poplar (*Liriodendron tulipifera*), and jack pine (*Pinus banksiana*) that were bonded with phenol resorcinol formaldehyde (PRF) and polyurethane (PUR) adhesives. The untreated samples had a better bonding strength than the heat-treated samples. Among the three adhesives, the PUR adhesive showed the highest bonding strength (10.12 N/mm^2), followed by MF (8.92, N/mm^2) and PVAc-D4 (8.54 N/mm^2). The bonding strength of PUR was higher than that of MF and PVAc-D4 adhesives by 11.9% and 15.6%, respectively. Percin (2012) investigated the effect of heat treatment on the bonding strength of laminated Scotch pine (*Pinus sylvestris* L.), beech (*Fagus orientalis* L.), oak (*Quercus petreae* L.), and poplar (*Populus nigra* L.) wood. The veneers were bonded with melamine formaldehyde, polyurethane, and polyvinyl acetate adhesives and then heat-treated at 185 °C or 212 °C for 2 h. The bonding strength decreased with increasing heat treatment temperature.

Table 4. Average Bonding Strength According to Adhesives and Temperatures

Variables		Average Bonding Strength (N/mm^2)	HG
Heat Treatment*	Control	10.38	A
	150 °C	9.90	AB
	175 °C	9.30	AB
	200 °C	8.64	BC
	225 °C	7.75	C
Adhesives**	MF	8.92	B
	PUR	10.12	A
	PVAc-D4	8.54	B

*LSD, 1.493; **LSD, 1.156; HG, homogeneous group

Table 5. Multiple Variance Analyses of Treatment Temperature and Adhesive

Source of Variance	Degrees of Freedom	Sum of Square	Means of Square	F- value	Level of significance 5%
Factor A	4	282.075	70.519	3703.6055	0.0000
Factor B	2	149.000	74.500	3912.6988	0.0000
A x B	8	51.826	6.478	340.2362	0.0000
Error	315	5.998	0.019		
Total	329	488.900			

*Note: Factor A is the heat treatment temperature, and Factor B is the type of adhesive

The results of multiple variance analyses tests with regard to the effects of the heat treatment temperatures and the type of adhesives are given in Table 5. The effects of heat treatment temperature and type of adhesive were found to be statistically significant for bonding strength ($P < 0.05$). Table 6 shows the bonding strength and the standard deviations of the untreated and heat-treated samples that were bonded with different adhesives.

The highest bonding strength was obtained in the non-heat-treated and PUR-glued wood (11.06 N/mm^2), and the lowest bonding strength was obtained in the sample containing PVAc-D4 and heat-treated at 225 °C (6.13 N/mm^2). The bonding strength of the heat-treated wood with the PUR adhesive was more satisfactory than that of the wood with the MF and PVAc-D4 adhesives. In addition, the bonding strength of PVAc-D4 was higher than MF in the control and heat-treated samples at 150 °C, while the bonding strength of MF was higher than PVAc-D4 in the heat-treated samples at 175 °C, 200 °C, and 225 °C. The MF adhesive gave more satisfactory results than the PVAc-D4 at high temperatures. As shown in Table 6, the bonding strength of the heat-treated hornbeam wood decreased as the heat treatment temperature increased for the MF, PUR, and PVAc-D4 adhesives. The decreases in bonding strength were 21.7%, 15.3%, and 39.6%, respectively, compared with the control specimens. The reduction of the wood density, the properties of a heat-treated wood surface during a heat treatment, and the characteristics of adhesives used, all potentially influence the adhesive bonding performance of heat-treated hornbeam wood.

Table 6. Average Bonding Strength According to the Interaction of Adhesive and Treatment Temperature

Adhesive	Value	Heat Treatment				
		Control	150 °C	175 °C	200 °C	225 °C
MF	X (N/mm^2)	9.93	9.31	9.01	8.58	7.78
	sd	0.1033	0.1041	0.1392	0.1329	0.1071
	HG	AB	AB	AB	ABC	BC
PUR	X (N/mm^2)	11.06	10.63	10.01	9.53	9.37
	sd	0.1394	0.1024	0.1436	0.1766	0.1451
	HG	A	A	AB	AB	AB
PVAc-D4	X (N/mm^2)	10.15	9.77	8.87	7.81	6.13
	sd	0.1725	0.1172	0.1339	0.1571	0.1462
	HG	AB	AB	AB	BC	C

*LSD, 2.585; X, average bonding strength; HG, homogeneous group; sd, standard deviation

Moreover, the amount of adhesive penetration into a wood surface plays a vital role in the bond performance (Kamke and Lee 2007). Sogutlu and Dongel (2007) reported that the bonding strength of higher density wood is higher than that of lower density wood. The surface wettability is an important factor for good adhesion during wood bonding. Due to the less hygroscopic (hydrophobic) and less polar properties of heat-treated wood, the distribution of the adhesive on the surface and the penetration of the adhesive into the wood structure can be affected (Paul *et al.* 2007; Sernek *et al.* 2007, 2008).

There is evidence supporting the positive relationship between wood wettability and adhesion (Wellons 1977). The wettability of heat-treated spruce and pine wood shows similar behavior, with a decrease in the wettability at treatment temperatures between 100 °C and 160 °C (Hakkou *et al.* 2005). In addition, heat treatment reduces the pH of hornbeam wood due to the production of acetic and formic acids. The pH of wood can influence the hardening of an adhesive, which might either retard or accelerate the adhesive curing, depending on the adhesive used for bonding (Kariz and Sernek 2010).

Dilik and Hiziroglu (2012) investigated the effect of heat treatment and compression on bonding strength of Eastern red cedar (*Juniperus virginiana*) wood. Test samples were exposed to 120 °C, 160 °C, and 190 °C for 6 h before they were compressed using 2.5 MPa pressure for 5 min and bonded with polyvinyl acetate (PVAc) adhesive. Test results showed that the bonding strength of samples decreased with increasing heat treatment temperature. Chow (1971) examined the bonding performance of phenol-formaldehyde glue (PF) bonded heat treated veneers. The results showed that there was a reduction in the bonding strength as the temperature and treatment duration increased. The bonding quality of heat- treated wood changes due to the reduction of surface energy, which affects the wettability of the wood material. Also, bonded heat-treated at 180 °C, 200 °C, and 220 °C aspen, beech, maple, and elm woods with a UF adhesive. There was a decrease in the shear strength of the adhesive line with increasing temperature and duration of treatment (Chang and Keith 1978).

CONCLUSIONS

1. Non-treated hornbeam wood showed highest bonding strength, while the samples bonded with the polyurethane (PUR) adhesive showed a higher bonding strength compared to the others considered.
2. The lowest average reduction values in bonding strength of the test specimens was determined for specimens exposed 150 °C heat treatment.
3. The bonding strength of heat-treated wood specimens decreased after the heat treatment. The highest bonding strengths of the melamine formaldehyde (MF), PUR, and polyvinyl acetate (PVAc-D4) adhesives were observed for the control specimens (9.93, 11.06, and 10.15 N/mm^2, respectively), whereas the lowest strength was observed for the heat-treated specimens at 225 °C (7.78, 9.37, and 6.11 N/mm^2, respectively).
4. Considering the three adhesives used, the PUR adhesive showed the greatest bonding strength, followed by the MF and PVAc-D4 adhesives. Bonding strength of all adhesive types decreased with increasing of the heat treatment temperature. Therefore, the lower heat treatment temperature and surface modification methods should be used in order to reach the highest bonding strength.

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