

BONDING ACETYLATED HORNBEAM WOOD (*Carpinus betulus* L.)

Fanni FODOR

Institute of Wood Science, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Arts,
University of Sopron, Bajcsy-Zs. u. 4. Sopron, 9400 Hungary
E-mail: fodor.fanni@phd.uni-sopron.hu

József ÁBRAHÁM

Institute of Wood Science, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Arts,
University of Sopron, Bajcsy-Zs. u. 4. Sopron, 9400 Hungary

Róbert NÉMETH

Institute of Wood Science, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Arts,
University of Sopron, Bajcsy-Zs. u. 4. Sopron, 9400 Hungary

Abstract:

*Hornbeam (*Carpinus betulus* L.) is a subsidiary tree species found in Hungary's hill and mountain forests and it covers about 5.2% of the forest area. It can also be found almost all over Europe, from Sweden to Western Asia. In previous studies, it was found that acetylation – wood modification using acetic anhydride – improves the physical, mechanical, and also the durability properties of this wood species. The aim of this research was to examine the change of the bonding properties of hornbeam wood after acetylation it under industrial conditions. The compatibility of adhesives with modified wood is crucial to investigate, as changes in wood chemistry affect the bonding characteristics greatly, among other product-related properties like surface finishing and joinery with metal fasteners. In order to draw proper conclusions regarding this complex property, several tests were carried out concerning the wettability of the surface, testing the shear strength of solid wood samples and determining the bonding strength of samples glued with different adhesives. For the wettability properties, the surface energy was determined by the Sessile Drop Technique using water (polar) and diiodomethane (nonpolar) as probe liquids. The surface energy was determined for non-treated and acetylated hornbeam, on aged and non-aged surfaces, as well as on tangential and radial surfaces. Conclusions were drawn on the significance of these properties on the wettability of hornbeam. Tests regarding the shear strength of solid wood and the bonding strength were carried out on samples prepared in the same way, according to EN 205:2016. This way, conclusions were drawn regarding the bonding and shear strength change of wood before and after acetylation, as well as in conditioned, water-saturated or cooked state.*

Key words: hornbeam; acetylation; adhesive; bonding; surface energy; shear strength.

INTRODUCTION

Hornbeam (*Carpinus betulus* L.) is a subsidiary tree species found in Hungary's hill and mountain forests and it covers about 5.2% of the forest area in Hungary. It can also be found almost all over Europe, from Sweden to Western Asia. It is available in larger quantities in France, Germany, Ukraine, Slovakia, Croatia, and also in Turkey and Iran. The wood of hornbeam is known for its high density, hardness, toughness and wear-resistance, but its poor durability, small yield and low availability of straight, defect-free trunks hinder the usability of this species. In previous studies, it was found that acetylation – wood modification using acetic anhydride – improves the physical, mechanical, and also the durability properties of this wood species (Fodor et al. 2017a). This process involves the chemical modification of the wood structure by replacing the hydrophilic hydroxyl groups with acetyl groups. It makes the wood extremely stable against changing climate and moisture, and highly resistant to wood-decaying organisms.

Wood is a hygroscopic material, which means that it changes its shape and dimensions when it takes on or gives off water to balance out with its surrounding environment. It is also hydrophilic; the wood surface has good wetting properties, which is important in case of bonding and surface treatment. A greater bonding strength can be achieved if the adhesive can properly wet the surface, in order to achieve better penetration. As the wood modification processes improve the durability and dimensional stability, issues can occur concerning the bonding of modified wood. After modification, the wood surface is less polar and less porous, which reduces the adhesion of the adhesive thus decreases the bonding strength (Hunt et al. 2007).

During acetylation, ester bonds are formed between the acetyl groups and hydroxyl groups, which decrease the polarity and increase the hydrophobicity. Several studies showed an increase in water contact angle after acetylation (Hill 2007, Bryne 2008, Wålinder et al. 2013, Moghaddam et al. 2015). Bryne and

Wålinder (2010) found that the wetting of acetylated samples did not change significantly after conditioning them for 30 days at room temperature.

When bonding wood, the adhesive layer has to be stronger than the joined parts. The bonding strength is tested similarly to the shear strength of solid wood. After acetylation, the mechanical properties such as the shear strength can increase because of the lower moisture content and increased density (Larsson and Tillmann 1989, Bongers and Beckers 2003, Rowell 2006), but the cross section's lower fiber proportion (because of cell wall swelling) and some degradation of chemical components (because of the hydrothermal treatment and acidic medium) can also impair them (Dreher et al. 1964, Tjeerdsmä et al. 1998). As the acetylated surface absorbs less moisture, water-based adhesives and moisture-curing adhesives can wet the surface to a smaller extent, but there is also less tension in the bonded wood material in wet conditions. The pH of hornbeam decreased from 5.11 to 4.73 and the buffering capacity increased from 1.11mg/g to 2.15mg/g after acetylation (Fodor et al. 2017b), which can influence the reaction and curing time of acid-curing adhesives (Bongers et al. 2016). Those adhesives that need hydrogen bonds in wood to cure (e.g. urea-formaldehyde) and dispersive adhesives (e.g. polyvinyl acetate) perform worse in case of acetylated wood, while bonding with polyurethane, emulsion polymer isocyanate and epoxi glues shows promising results (Vick and Rowell 1990, Larsson et al. 1992, Vick et al. 1993, Frihart et al. 2004, Hill 2006, Bongers et al. 2016). In dry conditions, acetylated wood usually had higher strength and the failure occurred mostly in the wood, while in wet conditions, it had lower strength and the rate of failure in wood was greater than in the case of untreated wood (Larsson et al. 1992, Vick et al. 1993). It is not clear how the chemical modification changes the bonding properties of wood as there are many factors which influence the bonding strength: surface preparation, modification parameters, Weight Percentage Gain (WPG), adhesive properties, wood moisture content, wood species, distribution of glue, etc. (Rowell 1996).

The aim of this research was to examine the change of the bonding properties of hornbeam wood after acetylating it under industrial conditions. The compatibility of adhesives with modified wood is crucial to investigate, as changes in wood chemistry affect the bonding characteristics greatly, among other product-related properties like surface finishing and joinery with metal fasteners. In order to draw proper conclusions regarding this complex property, several tests were carried out concerning the wettability of the surface, testing the shear strength of solid wood samples and determining the bonding strength of samples bonded with different adhesives.

MATERIALS AND METHODS

Wood acetylation

Hornbeam boards of 28×160×2500mm³ (thickness × width × length) from the southwest part of Hungary were used in these tests. The air dry density of the wood varied between 750-790kg/m³. Half of the hornbeam boards were acetylated at Accsys Technologies (Netherlands) under industrial conditions. The average WPG of the boards was 15%.

Wettability

For the wettability properties, the surface energy was determined by the Sessile Drop Technique using water (polar) and diiodomethane (DIM) (nonpolar) as probe liquids. The surface energy was determined for non-treated and acetylated hornbeam, on aged (soaking in water, drying at 103±2°C and conditioning at 20°C and 65% relative humidity till constant weight, respectively) as well as non-aged surfaces (conditioning at 20°C and 65% relative humidity till constant weight), and also on tangential and radial surfaces. Before measuring the contact angle, the surfaces were sanded (prepared) the same way as if they were to be bonded with adhesive (EN 205: 2017). For the tests, PGX goniometer was used. The volume of the probe liquids was 5 µl and the contact angles were determined at 0-0.1-1-2-5-10-20-30-40-50 seconds. The surface energy was calculated by the Owens-Wendt equation.

Shear strength

The samples were prepared according to EN 205:2016 and the shear strength was determined according to MSZ 6786-6:1977 (Table 1). The shear strength was tested with Instron 4208 universal testing machine. For cooking the samples, a Memmert WNB 10 water bath was used. One part of the samples were tested at 1 mm/min (MSZ 6786-6:1977), while the other part was tested at 50 mm/min (EN 205:2016) to compare the results at different loading speeds.

Table 1

Hornbeam sample preparation and number of samples in case of shear and bonding strength tests

Preparation	Number of shear strength samples	Number of bonding strength samples
Conditioning for 7 days at 20°C 65%	9	20
Conditioning for 7 days at 20°C 65%	7	20
Water saturation for 4 days at 20°C		
Conditioning for 7 days at 20°C 65%	7	20
Water saturation for 4 days at 20°C		
Conditioning for 7 days at 20°C 65%		
Conditioning for 7 days at 20°C 65%	7	20
Cooking in boiling water for 6 hours		
Water saturation for 2 hours at 20°C		

Bonding strength

The bonding strength was determined according to EN 205:2016 (Table 1). In order to evaluate not just the wood but the adhesives as well, beech samples were also used for the standardized tests. Four adhesives were supplied by a multinational company in Germany, and they were applied according to the manufacturer's instructions (Table 2). The shear strength was tested using Tinius Olsen H10KT universal testing machine. For cooking the samples, a Memmert WNB 10 water bath was used. The durability classes were determined according to EN 204:2016. The solvent adhesive was only used on beech samples. As this glue did not meet the necessary requirements in the standard, it was not tested further on other species.

RESULTS AND DISCUSSION

Wettability

The test results are shown in Table 3.

There is no significant difference between the wettability of the tangential and radial surfaces, thus the large rays do not influence the results significantly.

The higher contact angle of the acetylated samples indicates worse wettability and lower surface energy which is related to the water uptake and better dimensional stability of acetylated hornbeam (Fodor et al. 2017a).

The contact angle of water increased by more than 40% after acetylation, while that of DIM is virtually unchanged. The time of water penetration was prolonged. The polar component of the surface energy was reduced by more than half compared to untreated hornbeam.

The dispersive component makes up a bigger part of the surface energy, which is 75%, 87% and 89% in the case of hornbeam, acetylated hornbeam and acetylated and aged hornbeam, respectively.

There is no significant difference between the wettability of the aged and non-aged acetylated surfaces, therefore it can be stated that no significant amount of extractives is bleached out of acetylated wood during ageing, which could influence the wettability of the wood surface, and the basic characteristics of the cell wall substrate remain unchanged.

Table 2

Description of adhesives for bonding strength tests

Adhesive	Ingredients	Classification	Bonding parameters	Consumption (g/m ²)		
				B	H	AH
"PVAC D3"	Water-based polyvinyl acetate dispersion	One-component Water resistance: D3 Thermoplastic Physicochemical bond Water-based Cures in acidic medium	Open time: 8-10 minutes Application on one side Press pressure: 0.5N/mm ² Pressing time: 1 hour Setting time: 7 days	175-190	175-190	130-150
"PVAC D4 2-C"	Water-based polyvinyl acetate dispersion + Modified aliphatic polyisocyanate (HDI)	Two-component ("PVAC D3" + D4 hardener) Water resistance: D4 Thermoplastic	Open time: 12 minutes Potlife: 8 hours Application on one side Press pressure:	145	150-160	150

		Physicochemical bond Water-based Cures in acidic medium	0.5N/mm2 Pressing time: 1 hour Setting time: 7 days			
"PUR"	Polyurethane prepolymer with free 4,4'-methylenediphenyl diisocyanate (MDI)	One-component Water resistance: D4 Thermoplastic Chemical bond No solvent	Open time: 10 minutes Application on one side Press pressure: 0.5N/mm2 Pressing time: 1 hour Setting time: 3 hours	120	120-145	160
"SOLVENT"	Rubber solvent	One-component Water resistance not known Thermosetting Physical bond Organic solvent	Open time: 10 minutes Application on two sides Press pressure: 0.7N/mm2 Pressing time: half hour Setting time: 48 hours	195	-	-

Notation: B: beech; H: hornbeam; AH: acetylated hornbeam

Table 3

Contact angle and surface energy of untreated and acetylated hornbeam

Wood	Contact angle (°)		Surface energy (mJ/m ²)							
	Water		Diiodomethane		All		Polar component		Dispersive component	
	Tan	Rad	Tan	Rad	Tan	Rad	Tan	Rad	Tan	Rad
Hornbeam Non-aged	44 ± 3	43 ± 2	18 ± 2	19 ± 3	64	64	16	16	48	48
Acetylated hornbeam Non-aged	62 ± 4	61 ± 2	20 ± 3	20 ± 5	55	55	7	7	48	48
Acetylated hornbeam Aged	64 ± 3	64 ± 3	18 ± 3	17 ± 1	54	54	6	6	48	49

Shear strength

The shear strength test results are shown in Table 4. After acetylation, the shear strength decreased by 9% and 32% in case of low and high loading speed, respectively. During the modification process, cell wall degradation can occur because of the increased temperature and pressure, and the presence of acetic acid can also cause damage to the microstructure, which results in the reduction of the shear strength.

As acetylated wood takes up less moisture than natural wood and its rigidity decreases in moist condition, its shear strength was higher after soaking or cooking in water compared to natural hornbeam. Comparing acetylated wood to natural wood, after soaking the samples in water for 4 days, the shear strength increased by 114% and 92% in case of low and high loading speed, respectively. A similar tendency can be found after another 7-day-long conditioning, where the shear strength increased by 17% and 26% in case of low and high loading speed, respectively. After cooking for 6 hours and soaking for 2 hours, the shear strength increased by 81% and 52% in case of low and high loading speed, respectively. These results can help in the evaluation of the bonding strength tests.

Table 4

Shear strength results of hornbeam and acetylated hornbeam at different loading speeds and moisture states

	Loading speed (mm/min)	Moisture content during testing (%)	Shear strength (MPa)
Conditioning for 7 days at 20°C 65%			
Hornbeam	1	12.29 ± 0.14	17.36 ± 1.26
Hornbeam	50		21.16 ± 1.26
Acetylated hornbeam	1	4.68 ± 0.17	15.72 ± 3.91
Acetylated hornbeam	50		14.42 ± 2.97
Conditioning for 7 days at 20°C 65%, water saturation for 4 days at 20°C			
Hornbeam	1	52.97 ± 1.99	7.92 ± 0.50
Hornbeam	50		10.45
Acetylated hornbeam	1	28.39 ± 1.28	16.94 ± 1.21
Acetylated hornbeam	50		20.03
Conditioning for 7 days at 20°C 65%, water saturation for 4 days at 20°C, conditioning for 7 days at 20°C 65%			
Hornbeam	1	16.30 ± 0.18	15.31 ± 0.87
Hornbeam	50		19.87
Acetylated hornbeam	1	7.45 ± 0.62	17.96 ± 2.48
Acetylated hornbeam	50		25.11
Conditioning for 7 days at 20°C 65%, cooking in boiling water for 6 hours, water saturation for 2 hours at 20°C			
Hornbeam	1	79.67 ± 1.14	8.71 ± 0.23
Hornbeam	50		12.03
Acetylated hornbeam	1	48.32 ± 2.91	15.73 ± 1.61
Acetylated hornbeam	50		18.32

Bonding strength

The bonding strength test results of beech samples indicate if the adhesives met the standard requirements.

As the standard limits are determined only for beech, they are not applicable to hornbeam, but the results of hornbeam can be compared to beech, and the change of bonding properties can also be evaluated after acetylation. The results are summarized in Table 5.

"PVAC D3" adhesive (Table 2) met the requirements according to the standard test (Table 5). Even the results of hornbeam and acetylated hornbeam corresponded to the standard limits (Table 5). Although beech and hornbeam are both diffuse-porous hardwood species and easily bonded by adhesives, in these tests, hornbeam had lower strength values in soaked state, which indicates weaker bonding properties and poor penetration. Among the species, hornbeam had the greatest reduction of bonding strength after soaking (-83%) and conditioning afterwards (-19%). Acetylation improved the bonding strength of hornbeam in wet state by 65%. In case of both hornbeam and acetylated hornbeam, there was a high proportion of glue line failure, which means the glue line may have been weaker than the wood itself.

"PVAC D4 2-C" adhesive (Table 2) showed poor performance and did not meet the standard requirements (in the case of beech) in wet state, as most of the samples broke in the bond line. In case of this two-component adhesive, hornbeam had lower bonding strength than beech. Among the species, hornbeam had the lowest reduction of bonding strength after soaking (-62%) but the greatest after cooking (-78%). Acetylation improved the bonding strength of hornbeam in dry (+39%) and cooked state (+153%) as well. Acetylated wood met the requirements in dry and cooked state (Table 5).

Table 5

Bonding strength test results

	Conditioning for 7 days at 20°C 65%			Conditioning for 7 days at 20°C 65%, water saturation for 4 days at 20°C			Conditioning for 7 days at 20°C 65%, water saturation for 4 days at 20°C, conditioning for 7 days at 20°C 65%			Conditioning for 7 days at 20°C 65%, cooking in boiling water for 6 hours, water saturation for 2 hours at 20°C		
EN 204:2016	≥ 10 MPa			≥ 2 MPa (D3) ≥ 4 MPa (D4)			≥ 8 MPa			≥ 4 MPa		
"PVAC D3"	B	H	AH	B	H	AH	B	H	AH	B	H	AH
Bonding strength (MPa)	12.4 (1.1)	12.1 (1.6)	10.4 (1.6)	2.2 (0.4)	2.0 (0.4)	3.3 (0.3)	11.3 (1.3)	9.8 (1.5)	9.0 (1.8)	-	-	-
Wood failure (%)	70	35	15	0	0	0	95	60	0	-	-	-
Conformity (%)	70	60	35	35	20	65	90	45	40	-	-	-
"PVAC D4 2-C"	B	H	AH	B	H	AH	B	H	AH	B	H	AH
Bonding strength (MPa)	11.1 (1.1)	7.6 (3.6)	10.6 (1.4)	3.2 (0.5)	2.9 (0.4)	2.9 (0.2)	-	-	-	3.5 (0.5)	1.7 (0.8)	4.3 (0.3)
Wood failure (%)	50	10	25	0	0	0	-	-	-	0	0	0
Conformity (%)	50	10	30	0	0	0	-	-	-	15	0	50
"PUR"	B	H	AH	B	H	AH	B	H	AH	B	H	AH
Bonding strength (MPa)	11.6 (0.9)	12.9 (1.2)	11.4 (1.7)	4.6 (0.6)	4.4 (0.3)	7.2 (0.4)	-	-	-	4.1 (0.3)	3.5 (0.7)	7.1 (0.5)
Wood failure (%)	65	35	70	0	0	0	-	-	-	0	0	25
Conformity (%)	60	90	40	55	45	80	-	-	-	30	10	70
"SOLVENT"	B	H	AH	B	H	AH	B	H	AH	B	H	AH
Bonding strength (MPa)	6.6 (1.0)	-	-	0.7 (0.2)	-	-	-	-	-	0.0 (0.0)	-	-
Wood failure (%)	0	-	-	0	-	-	-	-	-	0	-	-
Conformity (%)	0	-	-	0	-	-	-	-	-	0	-	-

Notation: B: Beech; H: Hornbeam; AH: Acetylated hornbeam; bonding strength is given as the average value and standard deviation (in brackets) of the top ten samples; wood failure is given as the ratio between the number of samples where wood failed during the test and the maximum number of samples (20 pieces); conformity is given as the ratio between the number of samples which meet the standard limit and the maximum number of samples (20 pieces)

"PUR" adhesive (Table 2) met the standard requirements virtually in case of all species. Hornbeam had similar bonding strength results to beech, although it was weaker in cooked state. Among the species, hornbeam had the greatest reduction of bonding strength after soaking (-66%) and cooking (-73%) while acetylation had the lowest reduction (-37% and -38%, respectively). Acetylation greatly increased the bonding strength of hornbeam in soaked (+63%) and cooked state (+103%) as well (Table 5).

"SOLVENT" adhesive (Table 2) was only tested on beech samples, but it had poor results even in dry state. The bond line was not resistant to water at all and the adhesive did not penetrate the wood properly.

During the shear test, the flexible glue line stretched instead of holding the wood pieces together. Because of the poor results, it was not tested further on hornbeam and acetylated hornbeam.

During the shear tests, it was common for a high proportion of beech and hornbeam samples to be twisted in soaked or cooked state, which greatly influenced the credibility of the test.

Only "PVAC D3" and "PUR" adhesives met the requirements of EN 204:2016 (Table 6). Hornbeam has similar physical and mechanical properties as beech but it had similar or lower bonding strength properties according to these results. After acetylation, the bonding strength was similar or higher than that of natural hornbeam.

Table 6

Classification of adhesives and evaluation of hornbeam and acetylated hornbeam

	Durability (Water resistance) according to EN 204:2016 (beech samples)	Bonding strength of hornbeam compared to beech	Bonding strength of hornbeam after acetylation
"PVAC D3"	D3	Lower	Similar
"PVAC D4 2-C"	Failed D4	Lower	Higher
"PUR"	D4	Similar	Higher
"SOLVENT"	Failed D4	-	-

Although the less polar, less porous modified surface reduces the rate of adhesion, there was no significant reduction in the bonding properties, but the proportion of wood failure was also lower in the case of dry samples. This means that the adhesion, and thus the bond line, was weaker than the wood itself.

The increase in bonding strength is more outstanding in the case of soaked and cooked samples, as there was less tension in the bonded modified wood than in the unmodified wood. The reduction of hydrophilicity and the increase of shear strength and bonding strength in wet state also confirm the stabilization effect of acetylation which was proven in earlier studies.

The shear strength of solid hornbeam and acetylated hornbeam wood in different states is higher than the bonding strength (same moisture state and loading speed), but the tendency is the same. Acetylated hornbeam has somewhat weaker shear strength and similar bonding strength in dry state compared to hornbeam, but it can even achieve double the hornbeam's shear and bonding strength in soaked and cooked state.

According to these results, the bonding of hornbeam wood requires greater caution than beech in order to achieve similar bonding strength. Acetylation did not hinder the bonding ability of hornbeam and its usage can be recommended in wet and humid application areas. It is advised to take the manufacturer's instructions into consideration and to use longer pressing time.

In the future, microscopic studies will be performed concerning the penetration of the adhesive into the modified wood, the fracture caused by shear tests, and conclusions will be drawn on the influence of these changes on the bonding strength of acetylated wood.

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