PhD Theses

Acetylation of European hornbeam (*Carpinus betulus* L.) wood for outdoor applications

by

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INTRODUCTION

In the past decades, there has been an increased awareness of the fragility of the environment. Sustainable forestry, using native wood species and minimizing transportation are just part of the solutions for reducing greenhouse gas emissions. Many European wood species, including hornbeam (*Carpinus betulus* L.), have low natural durability which makes them unable to be used for exterior applications without additional protection. For decades, this has been done using biocide preservatives which are toxic for both environment and humanity. Today, non-toxic methods are preferred for wood preservation, among which are the wood modification methods, including acetylation (Pozsgayné Fodor 2016, 2017).

The reaction of acetic anhydride with wood results in esterification of the accessible hydroxyl groups in the cell wall, with the formation of the by-product acetic acid. During acetylation, acetyl groups replace some hydroxyl groups on the cell wall polymers, which reduces the hygroscopicity or the water uptake of the wood cell wall. This results in lower equilibrium moisture content, fiber saturation point, and flatter sorption isotherm. Although the mechanical properties are not markedly changed after acetylation, they are considerably higher in a wet state compared to untreated wood. It is also very durable against microorganisms and weathering (Rowell, 2006).

Presently, acetylated wood is industrially produced by Accsys Technologies in Arnhem, the Netherlands. It is marketed today under the commercial name Accoya®, utilizing predominantly radiata pine wood (*Pinus radiata* D. Don), and to a lesser extent, beech (*Fagus sylvatica* L.) and alder (*Alnus* sp.), having a 20% acetyl weight gain on the average. In the Accoya® process, the wood is dried at high temperature and then conditioned to a moisture content of 5-7% before the acetylation. The wood is impregnated in a pressurized autoclave, so that the reagent penetrates through the wood and into the cell walls. It is heated approximately at 120-140°C allowing the reagent to react with hydroxyl groups in the wood (Larsson-Brelid, 2013). As the value of weight percentage gain (WPG) cannot be precisely calculated from its dry weight for industrially treated wood, the maximum degree of acetylation and acetyl content is determined with chemical analysis methods.

Nowadays, Accoya® wood is available worldwide, and its main uses are typically in the outdoors, above or in ground contact. Because of its cost, it is mainly used in value-added products in transportation, sports equipment, military, and construction. It has been utilized as exterior doors and windows, residential decking, sidings, outdoor furniture, musical instruments, wet rooms, greenhouses, bridges, outdoor sculptures, etc (Mantanis, 2017).

OBJECTIVES

At the beginning, an extensive literature research was thoroughly carried out on the availability and future of European hornbeam wood, from both forestry and wood industry point of view. A distribution map was drawn according to standing stock information of hornbeam in the world.

Acetylation-induced chemical transformations and anatomical alterations in the wood structure were analyzed by chemical analytical methods, Fourier-transform infrared spectroscopy, bright-field microscopy and scanning electron microscopy.

Its product-related properties were also targeted in order to verify the conformity to utilization, like durability in soil, photostability exposed to UV irradiation, and compatibility with surface finishes and adhesives.

Based on the results, possible product groups and use classes were proposed, product characterization was carried out and comprehensive SWOT analysis was made.

MATERIAL

European hornbeam (*Carpinus betulus* L.) can be found all over Europe, except for the Mediterranean. Besides Hungary, hornbeam can be found in fair amounts in various countries: Slovakia, Ukraine, Germany, Poland, Croatia, Bulgaria, Serbia, Slovenia and Austria. There are many hornbeam forests found in France, Romania, and Iran. It accounts for 5% of the Hungarian forest cover (97,231 ha) (NFK, 2021). Hornbeam trees are of medium height (15-25m) and usually grow in hill and mountain forests as there are favorable climatic conditions for it. If there is optimal climate, it can also grow on planes. The stems are often crooked, buttressed, fluted, and twisted.

There are also populations with smooth, cylindrical, straight trunks, but these require more planning and work. They have diameters around 35–50 cm, rarely up to 90 cm. Due to climate change, its territory will shift to northern areas, or from plains to higher altitudes, where soil conditions do not limit its spread (Varol et al., 2022). Its potential habitat will decrease in southern territories of its area, including Hungary. On the other hand, its standing stock can still increase in the short-term depending on forest political decisions, forest management changes, and unexpected events, like new wood pests. In the future, hornbeam will be a good option as mix species in regions where its area will grow. It will appear in the areas of beech, spruce and fir forests, which will need to be exchanged to oak hornbeam mixed forests due to climate change-induced drought and dry climate (Sikkema et al., 2016).

It is a diffuse-porous wood species, with dull white to grayish color, often with cross-grain or wavy figure. It has the highest density among all industrial wood species in Hungary. It has moderately high tendency to warping, moderately fast moisture uptake, and high fiber saturation point. Based on its physical and mechanical properties, hornbeam is a very dense, hard, wear-resistant, and strong wood. The natural durability of hornbeam is Durability Class 5 (not durable). Its high density, compression strength, bending strength, Brinell hardness and modulus of elasticity are perfect for applications where hardness and wear-resistance are key factors. The utilization of hornbeam is problematic as the wood defects make it difficult to manufacture, and the low dimensional stability and durability narrow the possible fields of use to indoors and fuel (Molnár and Bariska, 2002).

Wood modification could improve the disadvantages of hornbeam wood which hinder its utilization. The thermal modification of hornbeam was the subject of several research projects in Hungary and abroad, where all the papers identified change in mechanical properties, reduction of physical properties, mass loss, color darkening and improvement of durability. If high-quality raw material is selected and proper processing parameters are chosen during the modification treatment, hornbeam can have a bigger share of the wood market (Fodor et al., 2016).

Therefore, it could be a perfect feedstock for manufacturing parquet flooring, wood sidings, garden furniture, gardening utensils, playground elements, gates, fencing, decking, cladding, panelling, railroad crossties, heavy construction, pulley wheel, and wood-based panels for engineered wood products.

In the past century, various wood species have been subjected to acetylation, both in laboratory as well as on a (semi)commercial scale. Those wood species may be successfully acetylated, which are easy to dry, to impregnate, and which have a low to intermediate density (300 to 700 kg/m^3) (Bongers et al., 2008).

In a previous work (Fodor and Németh 2016, Fodor et al. 2017a), European hornbeam wood was industrially acetylated in order to improve its properties and widen its usage. It had an average of 15.3% WPG. There were promising results concerning its physical, mechanical and durability properties. This investigation about improving the properties of hornbeam by industrial acetylation has been a unique form of research since 2014 (Pozsgayné Fodor, 2015).

METHODS

Chemical analysis

Here, the aim was to assess the changes in chemical properties of hornbeam wood after acetylation and how these are related to the change in physical and mechanical properties. Cellulose content (Kürschner-Hoffer method), lignin content (Klason method), extractive content (Soxhlet extraction) and ash content (EN 15403:2011) were measured and compared in untreated and acetylated wood. The hemicellulose content was given by subtracting these chemical components from 100%. As percentage changes show only the relative increase or decrease of compounds compared to each other, the special markers (formic acid, acetic acid, levulinic acid, furfural) were determined by HPLC-PDA-ESI-MS/MS. In order to monitor the possible breakdown products of the structural carbohydrates and lignin, the total soluble carbohydrate content (Dubois method) and the total phenolic content (Folin-Ciocâlteu method) were measured. Changes in pH (pH meter) and buffering capacity (titration) were also determined.

Fourier-transform infrared spectroscopy analysis

DRIFT and ATR spectroscopy were also used during the research. The chemical changes in wood were evaluated observing the difference spectra, where the absorption increase was represented by positive band while absorption decrease was represented by negative band. The band assignments were made using the difference spectra.

FTIR analysis was used to evaluate the change in untreated hornbeam after acetylation, in the isolated Klason lignin of hornbeam after acetylation, in the acetylated stake after durability test, and in untreated and acetylated hornbeam samples after mercury-vapor-lamp irradiation.

Microscopic studies

Untreated and acetylated hornbeam samples, sound and decayed specimens, and PVAc and PUR-bonded lap joint samples were analyzed by bright-field microscopy and scanning electron microscopy (SEM).

For bright-field microscopy, sections were cut with a sledge microtome after softening. They were stained with safranin and astra blue or potassium permanganate (1% solution) for two minutes and embedded in Euparal. Staining enabled the chemical changes to be seen after acetylation and fungal decay. Safranin was used to indicate lignin, and astra blue to indicate polysaccharides.

For SEM, the sections were given a smooth surface by hand cutting with a razor blade after softening. Cell-wall thickness of untreated and acetylated hornbeam was measured to see if it changed after acetylation. Data set for each material contained 50 measurements.

Field test

The aim was to see if acetylated hornbeam could be utilized as an outdoor product in real-field conditions, with many different microorganisms that can attack the wood separately or cooperatively. The test was conducted according to EN 252, with sample dimensions $20 \times 50 \times 300$ mm^3. There were 12 stakes of each type: untreated hornbeam and industrially acetylated hornbeam, supplemented with beech and Scots pine sapwood according to standard.

The stakes were buried in the outdoor exposure testing field at the University of Sopron (47°40′41.4″ N 16°34′32.6″ E) in April 2016, and it is ongoing since then. As the rate of degradation is greatly influenced by the soil and climate, the soil characteristics (skeletal grain content, pH, calcium carbonate content, humus content, particle content) and weather parameters (average temperature, maximum temperature, monthly precipitation, number of days with precipitation above 0.25 mm, sunshine duration, solar irradiance, relative humidity, Scheffer climate index) were determined. The difference in the rate of degradation was determined visually and by calculating density and mass loss.

Wetting, shear strength and bonding strength assessment

The Sessile Drop Technique was used with water (polar) and diiodomethane (DIM) (nonpolar) as test liquids. Before measuring the contact angle, the surfaces were sanded in the same way as if they were to be bonded with adhesive (EN 205: 2016). A PGX goniometer was used for the test. 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, and 50 s. The surface energy was calculated by the Owens–Wendt equation. Tangential and radial surfaces of untreated and acetylated hornbeam samples were tested as well.

The shear strength was determined according to MSZ 6786-6:1977 in the conditioning sequences (CS) defined by EN 204:2016. The shear strength was tested with an Instron 4208 universal testing machine, and a Memmert WNB 7-45 water bath was used to heat the samples. The testing speed was 1 mm/min.

Bonding strength was determined according to EN 205:2016, using beech, hornbeam, and acetylated hornbeam standard lap joint samples. The adhesives were a PVAc D3 adhesive (‘Ponal Super 3’), a PVAc D4 adhesive (‘Ponal Super 3’ + 5% ‘Ponal D4 Hardener’), and a one-component PUR adhesive (‘Ponal Pur-Leim’), all supplied by Henkel Magyarország Ltd. They were applied on the tangential surface, where 0.5 N/mm^2 pressure was applied for at least 1 h at 20 °C and 65% relative humidity. The bonding strength was tested using a Tinius Olsen H10KT universal testing machine. A Memmert WNB 7-45 water bath was used to heat the lap joint samples. The durability classes were determined according to EN 204:2016.

There were 20 samples of each species for each conditioning sequence (CS 1, 3, 4, and 5). The testing speed was 50 mm/min. The microscopic images were examined manually in AutoCAD 2019 software to determine the effective penetration (total area of adhesive detected in the interphase region of the bondline divided by the width of the bondline) and the maximum penetration (average penetration distance of the five most distant adhesive objects detected within the field of view).

Color and photostability

Untreated and acetylated hornbeam samples were exposed to weather on a metal stand tilted at 45 degrees in the Outdoor Exposure Testing Field (47°40'41.4"N 16°34'32.6"E) between July 2016 and January 2018. In another test, they were exposed on a wooden stand tilted at 45 degrees on the roof of The Central Library of the University of Sopron (47°40’54.3”N 16°34’40.6”E) between April 2018 and April 2020. In every case, the weather parameters (average temperature, maximum temperature, monthly precipitation, number of days with precipitation above 0.25 mm, sunshine duration, solar irradiance, relative humidity, Scheffer climate index) were taken into consideration.

The UV irradiation test was performed in an artificial ageing chamber. In one test, there were two mercury-vapor lamps used (800 Watt, 76 W/m^2), in the other test, two xenon lamps (482 W/m^2) were used. The lamps were 64 cm above the samples and the equipment temperature was set to a maximum of 50 °C.

The color was expressed in CIE L*a*b* color space with a portable colorimeter. Here, L* defines lightness (0 is black and 100 is white), a* denotes red/green hue (positive values for red and negative values for green), and b* denotes yellow/blue hue (positive values for yellow and negative for blue). The colorimeter’s sensor head was 8 mm. The color was measured and calculated based on the D65 illuminant and 10° standard observer. Color was measured after 0-5-10-20-30-60-120-200 hours of irradiation, and also during weathering tests in the first weeks, then after months. The color coordinates were calculated according to ISO 11664:2019. Photos and scans were taken of the samples in their current state to compare visual appearance.

Coating of acetylated hornbeam to improve photostability

There was a preliminary photostability test with boiled linseed oil to test the color change and coating properties of acetylated hornbeam. It was applied two times according to the manufacturer's instructions.

Later, another photostability test was conducted with various oils and stains: Oli-Natura oil-colorless (OO0), Auro stain-colorless (AS0), -umbra (ASU), Biopin stain-colorless (BS0). -Swedish red (BSR), -palisander (BSP), Auro oil-teak (AOT), Biopin oil-teak (BOT), and -red (BOR). OO0, AS0, AOT and ASU coatings were linseed oil-based coatings; the rest were mainly polymer made from natural oils, fatty acids and resins. The finishes were applied using a paintbrush according to the manufacturer's instructions. Weathering and irradiation tests were conducted as well.

Statistical analysis

Statistical analysis was performed using the Dell Statistica software. Correlation tests, t-tests and factorial analyses of variances (ANOVA) combined with the Fisher's LSD test was conducted, and the differences were considered significant at $p < 0.05$.

RESULTS AND MAIN CONCLUSIONS OF THE RESEARCH

Thesis 1. The chemical structure of hornbeam after acetylation

I concluded that the proportional increase of the hemicellulose content (+11.10%) was compensated by the decrease of cellulose (-8.55%) and of lignin (-2.86%) contents. I proved that the extractive content increased to 1.91% and the pH decreased to 4.73.

I concluded that the slightly darker, walnut-like color of acetylated hornbeam was due to the alterations in the structure of lignin (oxidation, reaction with furfural) as well as the increase of the extractive content.

By HPLC analysis, I concluded that during the acetylation of hornbeam wood it is the hemicellulose fraction that gets acetylated and which is primarily responsible for the WPG, while a slight degradation also occurs to it (Fodor et al. 2016, 2018b).

Thesis 2. FTIR studies of chemical changes after acetylation, and after UV irradiation

I proved that the increase of absorption of methine, methylene and methyl groups at 2970 cm^{-1} is due to the exchange of OH groups into acetyl groups, which corresponds to the decrease of moisture sorption in acetylated hornbeam.

I proved that the hemicellulose fraction was acetylated by the higher absorption of the C=O (carbonyl) groups at 1769 cm^{-1} and of the C-O stretching at 1270 cm^{-1} in xylan. The absorption of the conjugated carbonyl groups at 1597 cm^{-1} decreased slightly in the acetylated wood, probably due to a minor degradation of xylans in acidic medium.

I proved that lignin degraded during acetylation by decrease of the aromatic skeletal vibration in syringyl at 1597 cm^{-1} and guaiacyl lignin at 1505 cm^{-1}, of the asymmetric C-H deformation in lignin at 1464 and 1432 cm^{-1}, and of the aromatic C-H skeletal vibration at 1136 cm^{-1}.

I proved that as the total phenolic content decreased, the real decrease of the lignin content is only minor, and changes in the FTIR spectra only indicate internal structural changes of the lignin matrix. FTIR studies of lignin samples also confirmed this finding (Fodor et al. 2016, 2018b).

I concluded that during UV irradiation of acetylated hornbeam, the absorption of functional groups in lignin at ~1600, 1500, 1470, 1270, 1150 cm^{-1} decreased while that of methane, methylene, methyl and carbonyl groups increased at ~2900 cm^{-1}. The rate of degradation and structural changes were the highest in case of acetylated samples, but the strengthening polymers did not degrade significantly (Fodor and Németh 2017, 2018b, Fodor et al. 2017b, 2017d).

Thesis 3. Microscopic studies of acetylated hornbeam

I concluded that the cutting of smooth, undamaged sections of acetylated hornbeam was only possible after swelling and softening in acetone, not water as in case of untreated wood. I proved that native hornbeam wood was sufficiently acetylated due to the minimal staining effect of safranin. I concluded that acetylation did not damage the wood microstructure and increased the cell-wall thickness by 7%.

The pits of acetylated hornbeam were much narrower, due to permanent cell-wall swelling. I found new brown deposits in parenchyma cells of acetylated hornbeam. Each cell contained a small amount of an insoluble substance, which led to the hypothesis that a natural component of the living cells (e.g. lignans) became insoluble during acetylation (Rousek and Fodor 2017, Rousek et al. 2022).

Thesis 4. Durability of acetylated hornbeam

In a long-term field test, acetylated hornbeam exhibited high resistance against fungal decay, mold, insects, and moisture, while control samples of hornbeam, beech and Scots pine failed after 0.5-6 years with 23-39% of their mass lost. The remaining acetylated hornbeam stakes are still in the soil for further testing, without any signs of decay or insect damage up to this point (Fodor et al. 2017c, 2022a, b).

Microscopic studies showed that fungi were able to colonize acetylated cell lumina in field test studies and in laboratory studies against *Coniophora puteana*, *Rhodonia (Poria) placenta*, and *Trametes (Coriolus) versicolor* (Rousek and Fodor 2017, Rousek et al. 2022).

Thesis 5. Wetting, shear strength and bonding strength of acetylated hornbeam

I proved that acetylated hornbeam is less wettable, its surface energy decreased from 64 to 55 mJ/m^2, due to the decrease of the polar component. The dispersive component did not change. The water contact angle increased significantly by 18° after acetylation. I proved that there is no significant difference between the wettability of radial and tangential surfaces.

I proved that the shear strength of conditioned samples decreased after acetylation, but due to its lower water uptake, acetylated hornbeam exhibited significantly enhanced shear strength after soaking in cold and boiling water, compared to untreated hornbeam.

I proved that acetylated hornbeam retained its bonding strength after soaking in cold and boiling water, unlike untreated hornbeam. I concluded that PUR adhesive had better results than PVAc adhesives, and it gave better results when used with acetylated hornbeam.

I proved during my microscopic studies that the higher PUR bonding strength of acetylated hornbeam wood was due to the lack of overpenetration, while it had lower PVAc bonding strength due to overpenetration. With overpenetration, high maximum penetration values are obtained, while the effective penetration values (which affect bonding strength more) are lower (Fodor et al. 2018a, 2018b, 2023, Pozsgayné Fodor et al. 2018).

Thesis 6. Color stability of acetylated hornbeam exposed to UV irradiation

I proved that acetylated hornbeam undergoes surface brightening during UV irradiation, and eventual graying outdoors. I proved that during graying of acetylated hornbeam, the lightness (L*) increased, while red (a*) and yellow (b*) hue decreased.

I concluded that it was due to transformation of extractives and degradation of lignin. These breakdown products were leached out during weathering.

I proved that acetylated hornbeam resists dimensional changes caused by changing climate and precipitation, but it is susceptible to wasp stripping and can be stained by mold during weather exposure, but to a lower extent than untreated wood.

The color resulted in 200 hours of aging was achieved in one month or less when exposed to weather (Fodor et al. 2017b, 2017d, 2022c, Fodor and Németh 2017, 2018a, 2018b).

Thesis 7. Coating of acetylated hornbeam to improve color stability

I proved that the coating absorbance of hornbeam decreased after acetylation due to its lower water uptake. I proved that its photostability can be largely improved by coating it with dark stains like Auro Wood stain No. 160 or Biopin Weather Protection Stain (for at least two years).

On the other hand, colorless stains, and colorless or colored oils did not have a long-lasting effect on its photostability. Oli-Natura colorless oil and Auro colorless stain had no significant effect on the color stability under tested conditions (Fodor et al. 2022c, Fodor and Németh 2018a).

PRODUCT CHARACTERIZATION

According to these results, acetylated hornbeam was characterized further. The most optimal countries (price, amount and distance from acetylation plant) to acquire 3 or 4-sides-clear hornbeam boards could be France, Germany, Poland and Romania.

The price (range) of acetylated hornbeam wood would be the same as Accoya®, because the raw material is similar or cheaper than the raw material used for Accoya® radiata pine, beech or alder production. Hornbeam has similar density and pore-structure to beech, it can be optimized to similar acetylation settings. The properties of acetylated hornbeam are similar to acetylated beech, and in some cases, even better.

Product groups of acetylated hornbeam may include: indoor furniture (P1), floor and non-structural interior uses (P2), exterior joinery (P3), decking (P5), fencing (P6). It could possibly be used for outdoor furniture (P7), and in-ground timber (P9), but less likely for cladding (P4) and some construction elements (P8) due to its high density. Construction elements of acetylated hornbeam can be steps, stairs, flooring, and thresholds, etc. On the other hand, acetylated wood is less promoted for indoor utilization due to its smell of acetic acid, especially when uncoated.

SWOT analysis was established which is elaborated in the dissertation. Its main product competitors were identified, like thermo hardwood, furfurylated wood, tropical hardwood, WPC, composites, etc. It has comparable properties to other commonly used materials like aluminum, concrete, porcelain, PVC or rubber, e.g. low thermal conductivity and high strength to density ratio. Its advantages can be emphasized, e.g. long service life which can be extended by regular maintenance cycles, sustainability, it can be reused, recycled, composted, burned at the end of its service life.

Modified wood in general needs to find its niche market, where it is appreciated, and recognized as a superior product. It can even enter new non-wood markets as well. It is important to keep in mind that modified wood is not a replacement for preservative-treated wood; it is ideally a replacement for non-timber materials.

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