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WOOD MODIFICATION RELATED RESEARCH AT THE UNIVERSITY OF WEST HUNGARY

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ABSTRACT: Wood modification in different ways dates back from decades at the University of West Hungary (Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, Institute of Wood Science). First investigations were high pressure and temperature steaming, and ammonia treatment of black locust. Complex investigations were made in terms of Hungarian hardwoods' heat treatment in the project "Wood preservation without chemicals". Due to this project, also industrial developments were established in Hungary. Wood modification indicates a lot of research objective in our institute. Wood modification processes indicate continuously new challenges. During the last years, special attention was given to heat treatment processes in vegetable oils and paraffin, acetylation and some impregnation processes. The application possibility of nano-scale materials in wood industry was also investigated.

KEYWORDS: Wood modification, Heat treatment, Acetylation, THM treatment, Nano-zinc, beeswax

1 INTRODUCTION

Wood is recognised as the most important of the renewable base materials with the added advantage of being recyclable and CO_2-neutral. It is a highly versatile material and as such has been utilized in building and construction. The demand for timber is continually increasing, especially in slower growing hardwood and tropical species. Such species offer a greater durability and higher aesthetic qualities than many of the faster growing softwood species. It is well known that there are grave ecological and environmental concerns over current 'virgin timber' demands, and various attempts are underway to prevent the demise of many of the biologically diverse regions where these timbers originate. A greater emphasis is now being placed in sustainable harvesting of timber species, though the slow growth of many species means a slow turnover in materials and profits. Thus it is necessary to encourage the use of faster growing timbers which may be readily gained from such sustainable plantations. But wood is a biodegradable material. Degradation leads to the reduction in strength and the increased risk in structural decay through fungal infestation. Many traditional protection treatments currently exist to prevent these deteriorations, but often they are based on toxic materials. Apart from the risks involved in using such materials for treatments, there is increasing concern over the problems arising in the disposal of the timbers after the end of their commercial lifetime. Whilst the timber sector recognizes these increasing safety requirements, it also realizes a lot of research and development will be required in overcoming these problems. Wood modification is a generic term describing the application of chemical, physical, or biological methods to alter the properties of the material. The aim is to get better performance from the wood, resulting in improvements in dimensional stability, decay resistance, weathering resistance, etc. It is essential that the modified wood is non-toxic in service and the disposal at the end of life does not result in the generation of any toxic residues [1].

2 HEAT TREATMENT IN GASEOUS ATMOSPHERES

The institute possesses a programmable heat treatment chamber (Figure 1) in which it is possible the treatment of maximum 60 cm long samples. In this electric heated, with ventilators and control panels equipped device heat treatment in normal atmospheric air can be realized. Due

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to the elaborated schedules and experience the good quality of heat treated wood is secured.

In 2010 a combined heat treatment-steaming equipment with 0,5 m^3 capacity were purchased (Figure 2). This autoclave is suitable for heat treatments up to 250°C temperature in vacuum, inert gases and steam. Investigated wood species so far: oak, turkey oak, black locust, poplar, hornbeam, beech, maple, pine and spruce.

Figure 1: *Experimental heat treatment chamber*

Figure 2: *Combined treatment autoclave*

As a result of the treatments durability was improved remarkably and swelling decreased as well (Figure 3).

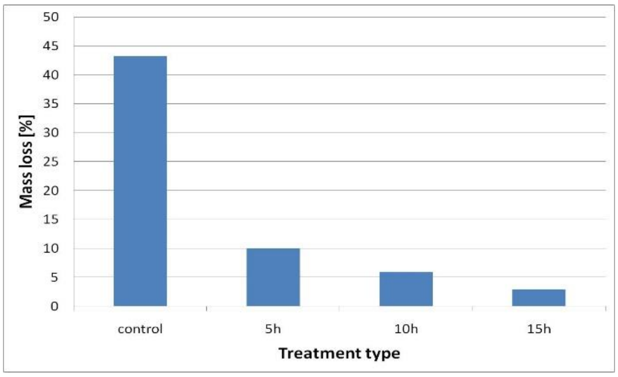

Figure 3: *Improvement in durability of poplar wood against Coriolus versicolor at different heating times (treatment temperature: 200°C) [2]*

By means of heat treatments exotic and homogeneous colour can be achieved in whole cross-section of the wood. This property was very useful by producing of flooring elements from the heat treated material (Figure 4). Beside of the favourable properties the bending, tensile (20-40%) and impact bending strength (30-70%) was decreased considerably. However, hardness and compressing strength increased slightly. These increased properties are also especially important during the production of flooring elements.

Figure 4: *Flooring elements with heat treated oak, turkey oak, beech and ash top-layer (left to right)*

Comparing different heat treatment methods, the influence of the heat treatment medium and parameters on the physical and mechanical properties was shown significantly (Figures 5-8).

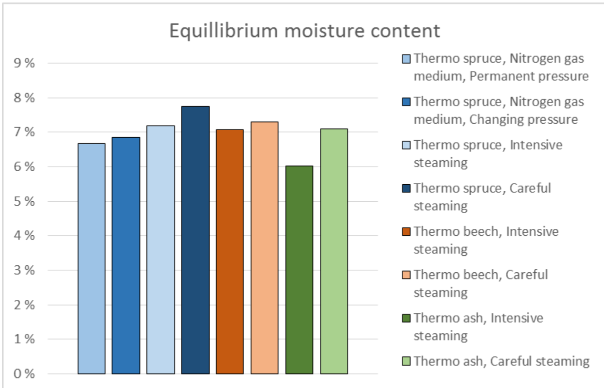

Figure 5: *Effect of treatment parameters on the equilibrium moisture content of spruce and beech wood (rh. = 65%, t = 20°C)*

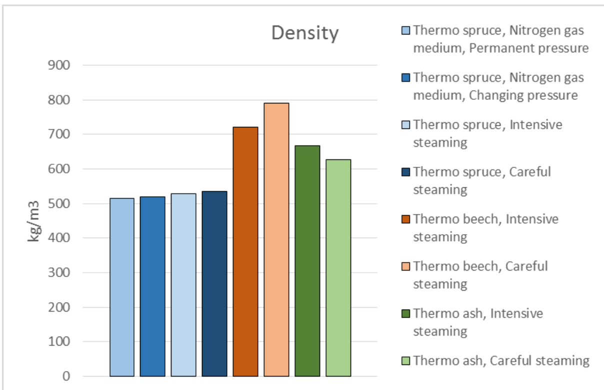

Figure 6: *Effect of treatment parameters on the density of spruce and beech wood (rh. = 65%, t = 20°C)*

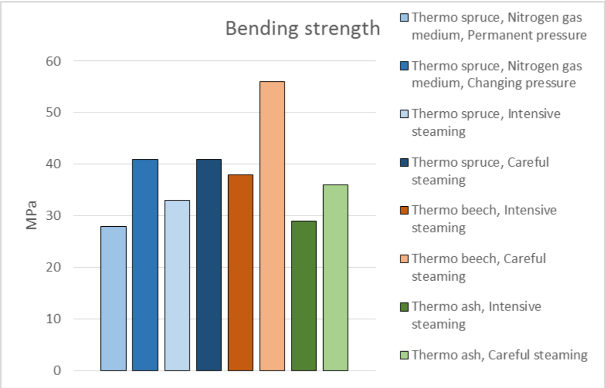

Figure 7: *Effect of treatment parameters on the bending strength of spruce and beech wood (rh. = 65%, t = 20°C)*

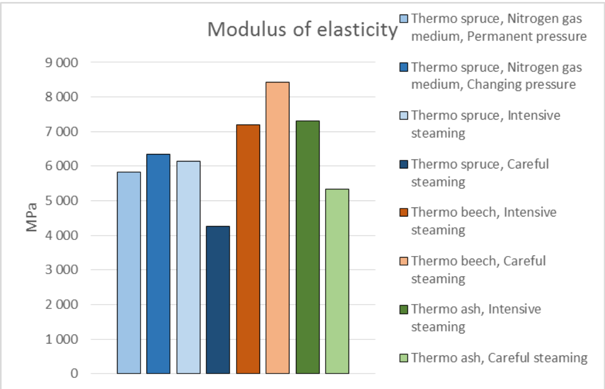

Figure 8: *Effect of treatment parameters on the modulus of elasticity of spruce and beech wood (rh. = 65%, t = 20°C)*

We analysed the effect of dry heat treatment on abrasive resistance and hardness of wood species most frequently used in flooring panels. (Figure 9). Although early- and latewood proportion has influence on the abrasive resistance, the average mass loss of heat treated beech wood caused by sandblasting showed a near correlation to the result of Taber test method.

Figure 9: *Heat treated beech sample after sandblasting*

3 HEAT TREATMENT IN DIFFERENT FLUIDS

Efficiency of heat treatment processes depends on the rate and regularity of the heat growth in the wood, and on the reducing of oxidative processes in the interest of avoid unreasonable decomposition. Heat treatment in vegetable oils (OHT) can be a solution for these problems. Wood was heat treated in rapeseed-, linseed- and sunflower oil at 160-200°C. Swelling properties decreased 20-60% and strength decreased less than by heat treatment in a gaseous atmosphere.

With applying paraffin (PHT) as heat treatment medium instead of vegetable oils, similar results can be achieved as well as moisture uptake decreased further because of the thin paraffin layer on the surface [3]. During moisture uptake, the change in moisture content of OHT wood was similar to that of the untreated wood. After 48 hours the moisture content increased only slightly, and all specimens were close to their EMC (Figure 10). Similar to the observations of Pfriem et al. [4] by another heat treatment method it can be stated that OHT treatment reduces the moisture uptake rate because heat-treated samples adsorb less moisture during the same amount of time than untreated samples. But in spite of the mentioned authors, the saturation of the OHT and untreated wood specimens occurs in the same duration.

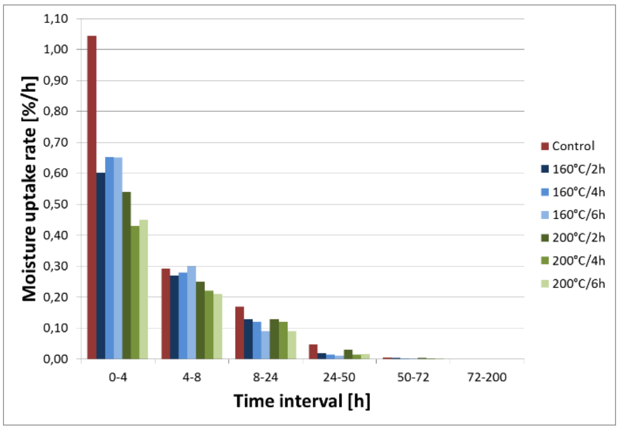

Figure 10: *Moisture uptake rates at normal climate (t= 20°C, rh= 65%) for the several investigated periods (Treatment medium: linseed oil*

In spite of that, the change in moisture content of PHT wood is significantly slower than that of the natural and OHT wood (Figure 11). Natural and OHT wood reached EMC already after 100 hours but PHT wood only after 200 hours. It is well known that the amount of sites which are able to adsorb bound water (-OH groups) reduces due to chemical changes during heat treatment [5]. The water storage capacity of the wood therefore decreases. Considering that saturation occurs during the same time by natural and OHT wood, it is revealed that a decrease in the moisture uptake rate in OHT wood is due to the decrease in water storage capacity and the thin oil layer on the surface of the wood does not have any effect on the moisture uptake. However, the thin paraffin layer on the surface of the PHT wood has some barrier effect against moisture uptake.

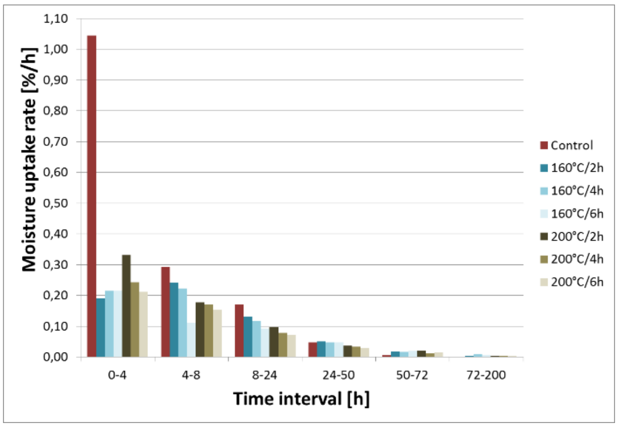

Figure 11: *Moisture uptake rates at normal climate (T= 20°C, φ= 65%) for the several investigated periods (Treatment medium: paraffin*

OHT has a significant influence on the moisture diffusion properties as well. Moisture transport through the untreated wood was significantly higher compared to the heat treated samples in both radial and tangential direction (Figure 12). As a result of heat treatments, diffusion decreased ~65% in tangential and ~80% in radial direction. Diffusion of untreated material was lower in tangential direction compared to the radial direction. In case of heat treated samples these differences were diminished or turned to the opposite between the anatomical directions. The effect of treatment medium was not clear, this means that the oil uptake and the thin oil layer on the surface does not have significant effect on the diffusion of heat treated wood.

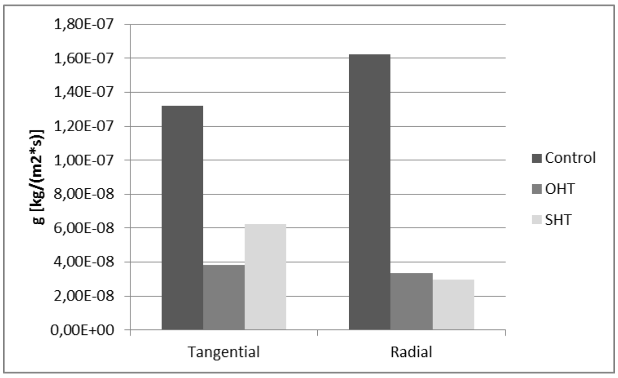

Figure 12: *Density of water vapour flow rate for untreated, oil-heat-treated (OHT) and steam-heat-treated (SHT) wood material in tangential and radial directions*

Figure 13: *Colur change of poplar wood due to different heat treatment schedules in linseed oil*

Colour changes due to OHT were similar than by heat treatments in a gaseous atmosphere (Figure 13). Initial colour of OHT and PHT wood is almost similar, however yellow hue (b*) of PHT wood is significantly higher than by OHT wood. During weathering, both of OHT and PHT samples darkened after 1 year of outdoor exposure, but higher treatment temperature and longer durations resulted in less darkening. Lightness of OHT wood decreased more than lightness of PHT wood during weathering. The lightness of the untreated samples decreased permanently, but it remained squarely higher as the lightness of the treated samples.

The red hue (a*) of the heat treated samples decreased continuously during the whole period. Red hue of OHT wood decreased more, than red hue of PHT wood. The yellow hue (b*) of the treated samples declined remarkably and continuously. At the end of the 12th month differences were diminished between treated and untreated samples in terms of yellow hue. The decrease in b* values of OHT and PHT wood at 160°C were similar, but heat treatment at 200°C resulted in different change of the yellowish character in case of oil and paraffin. In case of 200°C treatment yellow hue of PHT wood decreased more pronounced compared to OHT wood.

The ΔE* values of all samples increased continuously during the investigated period (Figures 14 and 15). Because of the lower decrease in lightness and red hue, total colour change of PHT wood is smaller by the 160°C treatments. But in case of the treatments at 200°C, total colour change of OHT wood is smaller, because of the minor decrease in yellow hue. The material treated at lower (160°C) temperature underwent more sever total colour changes compared to the higher temperature (200°C) by both treatments.

The colour changes of the treated samples did only exceed the changes of the untreated materials in case of 160°C treatment after one year weathering. The colour stability of treated material is depending on the treatment parameters, mainly on the temperature. Higher treatment temperature is favourable for outdoor application however on long term colour protection is needed for aesthetical applications. By 160°C treatments PHT suffered less colour change during weathering, but by 200°C treatments OHT resulted in better colour protection. This result shows the importance of the treatment medium well.

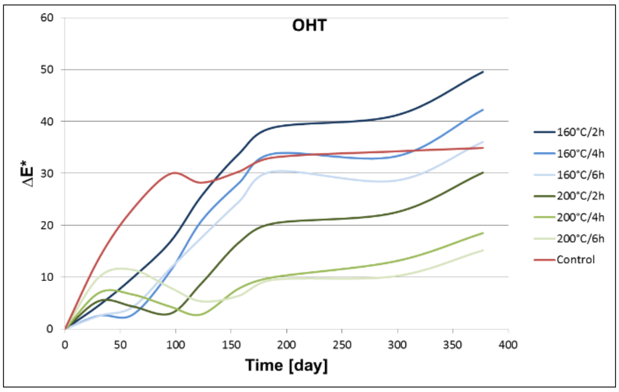

Figure 14: *Total colour change (ΔE*) of OHT wood during 1 year of weathering*

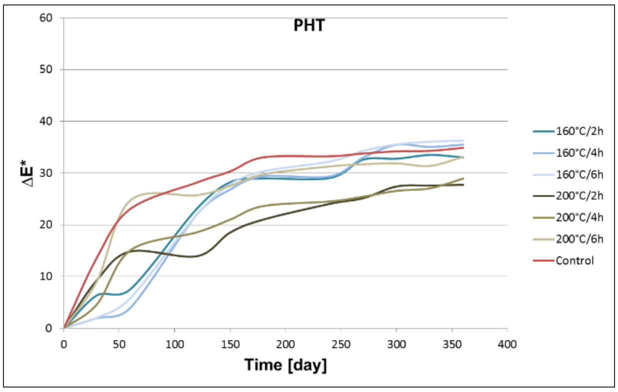

Figure 15: *Total colour change (ΔE*) of PHT wood during 1 year of weathering*

Further advantage of a heat treatment in vegetable oils is the short treatment time (by a 25mm thick poplar board, up to 6 hours including drying). However, it has to be noted, that for example by black locust, which has closed pore structure (thyloses), longer treatment times are needed to avoid cracks and deformations [6].

4 THERMO-HIGRO-MECHANICAL (THM) TREATMENT OF WOOD

In terms of a product often only one major property is determining the usability of the wood species. In terms of poplar woods indoor use surface hardness is the property which limits the utilization. The goal in this case was to produce a material with low density and high surface hardness with using compression and heat on wood (Figure 16).

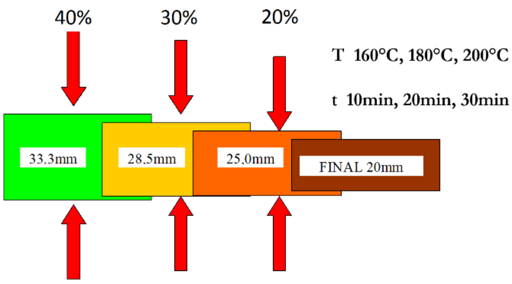

Figure 16: *Parameters of the different THM process schedules*

With Thermo-Hygro-Mechanical treatment – using heat, steam and compressing on wood – hardness of poplar wood can be increased from the very low 10 N/mm^2 to 22 N/mm^2. With 30% compression of poplar wood hardness increases by 120% and reaches hardness of maple wood, which is a popular wood species of flooring element construction (Figure 17). Beside of the improved surface hardness wood colour became brown in 2-3 mm depth [7]. The result of the treatment is the controlled cell wall collapse in the surface region of the material (Figure 18). As cell lumens are compressed perpendicular to the grain and porosity decreases, it will result in increased surface hardness and abrasion resistance of the material. It means that it is possible to widen the utilization field of the initially soft poplar wood material.

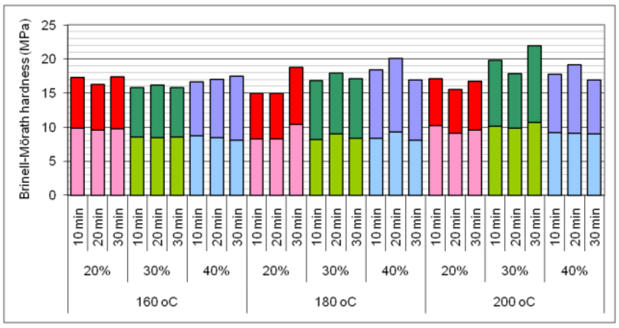

Figure 17: *Brinell-Mörath hardness of poplar wood perpendicular to grain as a result of different THM process schedules*

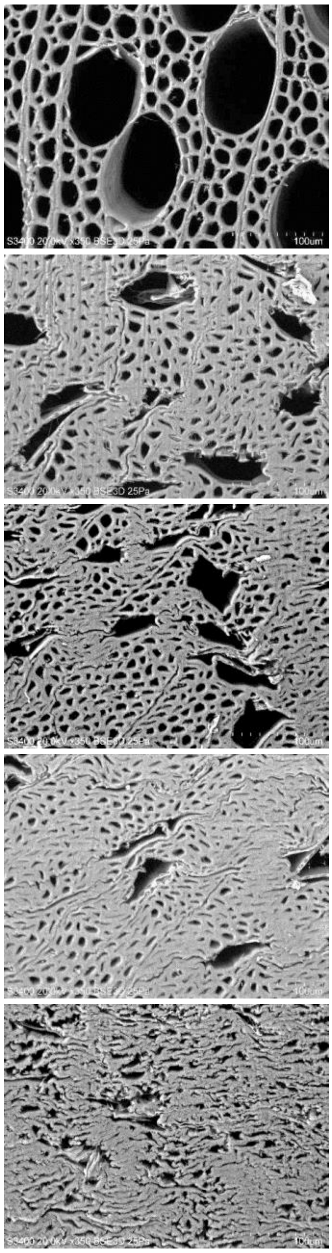

Figure 18: *SEM micrographs of the changes in the anatomical structure of poplar wood as a result of THM treatments with different intensities at 350× magnification (from upside to down: untreated, 10%, 20%, 30% and 40% compression)*

Beside the advantages, there are unfortunately some lacks of the process as well. The process is very sensitive on the material quality, mainly on the presence of cracks and knots. Another important factor during the treatment is the orientation of the annual rings. The annual rings have to be oriented nearly perpendicular or parallel to the direction of the compression force. Otherwise the result will be often deformation or failures in the material structures (Figure 19). The reason for the failures can be too low density, not optimal moisture content of the material, or the presence of the so called “wet pockets”, with locally higher moisture content in the poplar wood material. Failures occur mainly at the annual ring border as the split-up of the annual rings. The reason for that is the different behaviour of the earlywood and latewood under compression.

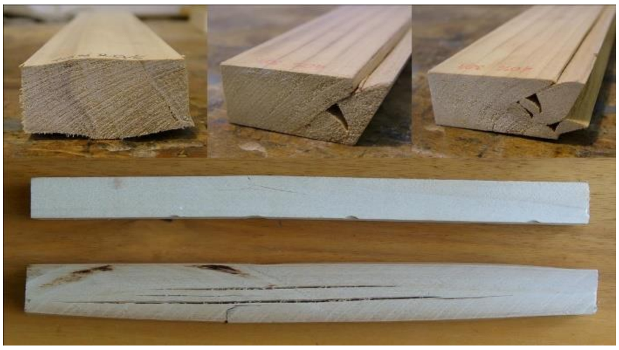

Figure 19: *Typical failures of poplar wood as a result of THM treatment*

5 ACETYLATION OF WOOD

One of the most common chemical modification processes is acetylation, which changes –OH groups in wood to acetyl-groups. In the centre of our first investigations were black locust (*Robinia pseudoacacia* L) and poplar (*Populus × euramericana* cv. Pannonia). The swelling of poplar wood decreased by 70%, beside that the mechanical properties remained unchanged. Black locust cannot be effectively treated due to the small penetration deepness caused by its tyloses. However, as veneer or flake good result can be achieved (e.g. production of weather resistant panels). Treated material typically loses colour but with appropriate surface finishing it can be brightened up. Acetylated wood has pungent smell for a long time (evaporation of acetic acid), furthermore by the application of hinges increased corrosion have to be taken into account.

As a next step hornbeam (*Carpinus betulus* L.) wood was acetylated in cooperation with Accsys Technologies (the Netherlands). The results are promising, as the equilibrium moisture content and fibre saturation point decreased by 58% and 33% respectively, beside a slight increase in the density (4-15%, depending on the moisture content state). As a result of that, shrinkage decreased remarkably as well. The decrease was ~80% in radial and tangential directions, and ~60% in longitudinal direction. Weight loss by decaying fungi decreased by 95-98% as a result of acetylation (Figure 20), this means that the weight loss by three types of fungal decay was below 1%.

Moisture content	
20°C/65%	- 58%
FSP	- 33%
Density	
dry	15%
20°C/65%	10%
saturated	4%
Shrinkage	
radial	- 82%
tangential	- 81%
longitudinal	- 60%
Max. water uptake (49 days, tan.)	- 17%
Weightloss by fungal decay (16 weeks)	- 95-98%

Figure 20: *Changes of the physical properties and decay resistance of acetylated hornbeam wood compared to the untreated material*

Hardness increased in case of conditioned samples (20°C/65%) by 50-70%, depending on the orientation. However, in case of water-saturated material (over fibre saturation point) the increase of the hardness was 110-160%, depending on the anatomical orientation. In dry state the MOR and MOE did not change significantly but after water saturation the MOR and MOE of acetylated hornbeam decreased less than in untreated form. The compression strength and impact bending strength increased by 43% and 69% due to acetylation, respectively (Figure 21).

Janka hardness	
tangential (dry)	56%
radial (dry)	55%
tangential (saturated)	111%
radial (saturated)	154%
Brinell hardness	
tangential (dry)	49%
radial (dry)	68%
end grain (dry)	51%
tangential (saturated)	124%
radial (saturated)	163%
end grain (saturated)	145%
Compression strength ‖ to grain	43%
MOE	
dry	0%
saturated	36%
MOR	
dry	20%
saturated	93%
Impact bending	69%

Figure 21: *Changes of the mechanical properties of acetylated hornbeam wood compared to the untreated material*

Usually acetylation has a slight effect on wood colour as well, which can be darkening and lightening as well. It depends usually on the initial colour and the WPG, that means light coloured woods became slightly darker, while dark coloured woods, slightly lighter [8]. Similar results were found for acetylated hornbeam as well, as a slight darkening was observed as a result of acetylation (Figure 22).

Figure 22: *Colour change as a result of acetylation on hornbeam wood (left: untreated; right: acetylated)*

6 OTHER WOOD MODIFICATION PROCESSES

In addition to the modification processes above, some other processes were investigated too. First of all, impregnation processes with beeswax or nanozinc-particles can be highlighted. Both treatments have the goal to improve fungal resistance of wood.

Table 1. shows the mean percentage mass loss of the specimen test at higher (0.220%, m/m), lower (0.055%, m/m) nano-zinc concentration and at control. There were prominent differences in percentage mass loss based on wood species and on preservative retention. Zinc-nanoparticles improved durability very effective because already very low concentrations (0,22 and 0,055m/m%) resulted in significant resistance against decay [9]. At nano-zinc concentration of 0.220% (m/m) pine, spruce, beech and poplar test specimens showed 2.48, 3.26, 13.1 and 12.35% of percentage mass loss respectively. Then at nano-zinc concentration of 0.055% (m/m) pine, spruce, beech and poplar test specimens showed 3.27, 4.62, 14.93 and 8.91% of percentage mass loss respectively. All the specimens except poplar showed a decreasing decay as a result of increasing nano-zinc concentration. Only pine and spruce specimens exhibited a percentage mass loss lower than 5% for both preservative concentrations.

Table 1: *Percentage mass loss about the decay specimens*

Treatment group	Percentage mass loss		
	nano-zinc concentration		
	High concentration 0.220% (m/m)	Low concentration 0.055% (m/m)	Control
pine	2.48	3.27	23.05
spruce	3.26	4.62	38.49
beech	13.10	14.93	23.01
poplar	12.35	8.91	22.55

Impregnation with beeswax has a positive result that the process decreases moisture uptake of wood significantly (10-40%) and it increases the durability in short term applications, thus it can be a natural based preservative for wood without any chemicals. The advantage of beeswax is its biological origin and its nontoxic nature. It is in general not biologically stable. As a result of the hydrophobic properties and the lumen filling, fungi can decay wood only slower. To show this effect, untreated and impregnated beech and poplar samples were put in fertile compost soil for 18 months. The modulus of elasticity (MOE) was determined after 1 and 18 months of soil contact.

The protecting effect of beeswax could be observed with visual inspection. The parts of both the untreated beech and poplar samples that were put into the soil were almost completely decayed after 18 months of soil contact (Figure 23).

Figure 23: *Unimpregnated (a) and impregnated (b) beech samples after 18 months of soil contact*

As a result of beeswax impregnation, MOE at the absolute dry state increased markedly, depending on the impregnation efficiency, by 30 to 50% and 15 to 25% in beech and poplar samples, respectively, when compared to the control samples (Figure 24).

MOE decreased markedly during the exposure to soil (Fig. 3). A strong decrease (30 to 60%) could be observed in the MOE after one month of soil contact, but this is mostly explained by the suspected increase in the moisture content of the samples. The initial MOE was determined for the samples at absolute dry states; however, after one month of soil contact, the moisture content of the samples should be increased considerably (probably close to the fibre saturation point). As no decay could be observed on the samples, and however the moisture content of the samples was not determined, a high moisture content increase should be the reason for the strong decrease of the MOE. After one month of soil contact, no pronounced decay was expected, but it could have had a slight effect on the elastic properties of the wood.

During the next 17 months of soil contact, the MOE decreased markedly. Untreated beech and poplar specimens lost their load-bearing capacity completely because wood destroying microorganisms (fungi and/or probably some bacteria) destroyed their texture. Accordingly, their MOE was 0 MPa because of the heavy decay. Nevertheless, some load-bearing capacity of the impregnated beech and poplar specimens remained. Therefore, their MOE was measurable. Only a few impregnated samples decayed to such a large extent that the load-bearing capacity was under the investigation load (150 N). The degree of pore saturation (DPS) had a noticeable effect on the decay. Higher DPS for both beech and poplar specimens resulted in a higher MOE after 18 months of soil compared to specimens with lower DPS.

Compared to the absolute dry state, after 18 months of soil contact exposure, the MOE of beech and poplar wood decreased from 65 to 80% and from 50 to 60%, respectively (Fig. 4). Impregnation efficiency had a marked effect on decay resistance, as higher DPS resulted in a smaller decrease in the MOE of both beech and poplar samples. The advantage of the beeswax impregnation is that when the beech and poplar wood had higher DPS, it resulted in a higher MOE at the wood’s absolute dry state (before soil contact), and the higher MOE decreased less during soil contact than it did for wood (beech and poplar) with lower DPS. The decrease in the MOE was lower in the case of the poplar in comparison with the beech samples; thus, beeswax impregnation was more effective in poplar wood for the prevention of a MOE decrease during soil contact.

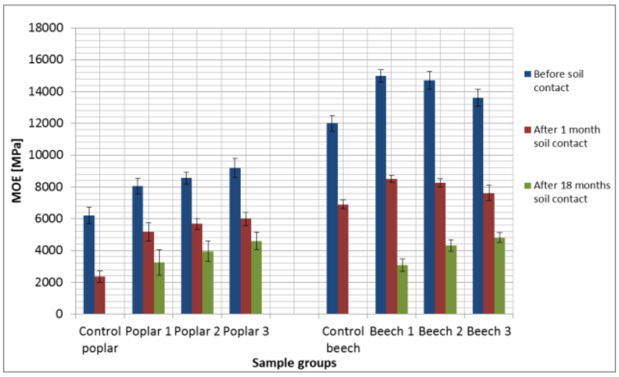

Figure 24: *MOE of poplar and beech samples (control and with 3 different DPS) in the investigation periods*

The beeswax could be well identified with SEM imaging in the cell lumens, mostly in the vessels. Beeswax filled the whole vessel lumen in most cases (Figure 25a), but sometimes it only coated the inner surface of the lumen, like a protective layer (Figure 25b). Vessels were impregnated best with the beeswax, but it could be observed in other cell types as well. Ray cells (parenchymatic cells) were filled with beeswax as well, but because they have smaller lumen diameter, the ratio of the beeswax filling was lower. In several parts of the wood, the filling of the libriform cell lumens could also be observed. However, after the beeswax impregnation of the samples, the filling of the pores was, in most cases, not complete. Vessels without beeswax could be observed as well, mostly in the inner parts of the samples. These areas could help the fungus to spread through the wood faster, compared to the totally impregnated wood parts.

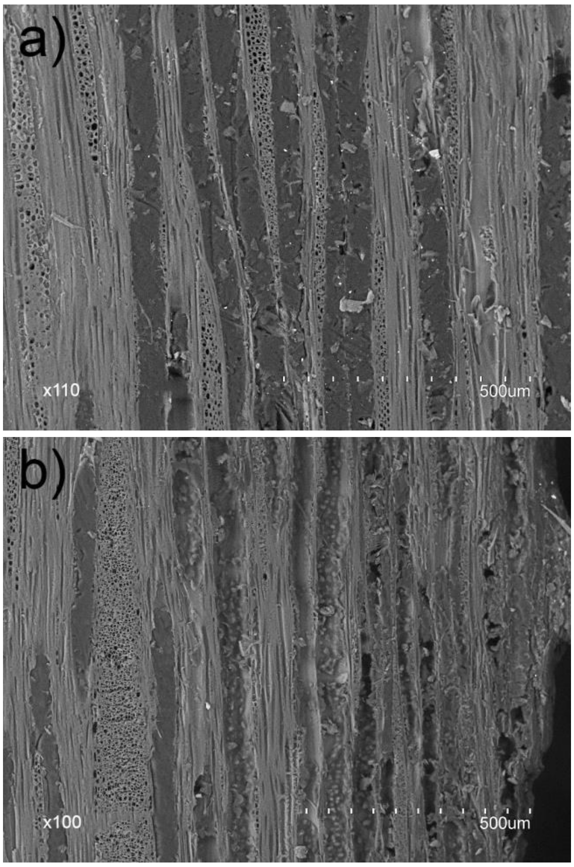

Figure 25: *Beeswax filling the vessels (a) and coating the inner side of vessels (b) of beech wood*

Hyphae could be observed in large quantities only on the surfaces of the specimens that had direct soil contact (Figure 26a). Hyphae in the inner structure of the specimens were rare to find, and only in lumens without any beeswax (Figure 26b). The spreading of the hyphae was physically inhibited by the presence of the beeswax in the lumens, which slowed the progression of the fungi in the wood. As beeswax has no biocide effect, only the physical barrier effect can be the reason for the lower decomposition of the impregnated samples than in the control samples. This can explain the slower decay of the impregnated samples. A higher ratio of filled lumens better inhibits the spreading of hyphae, which can explain the higher remaining MOE values of samples with higher DPS

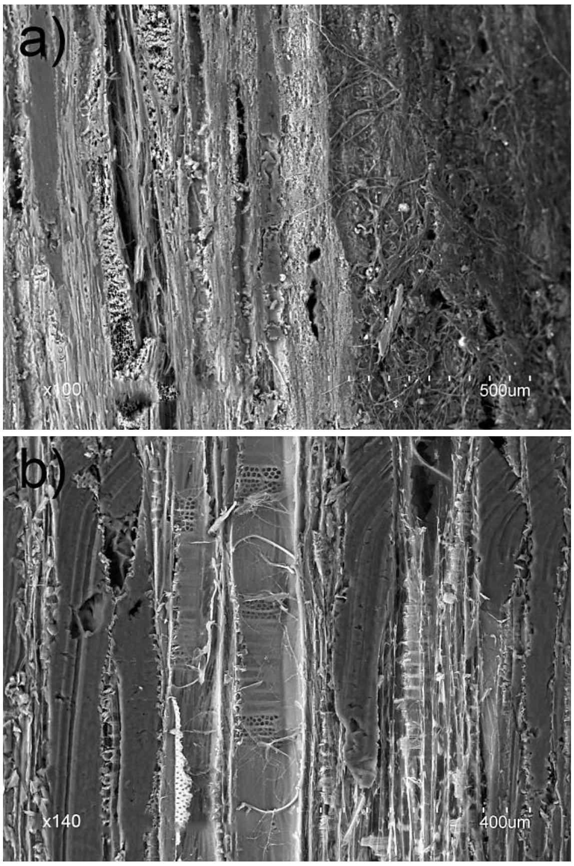

Figure 26: *Hyphae on the surface of a beeswax impregnated beech sample (a) and in a beeswax free cell lumen of poplar wood (b)*

7 CONCLUSIONS

At the University of West Hungary (Institute of Wood Science) important research activity were executed in the last 15 years in terms of wood modification. In course of that, effects of numerous modification processes were investigated on wood. The main topic was investigation of different heat treatments (heat treatment in different gaseous atmospheres or liquids and with compressing), but in terms of acetylation and in development of environmental friendly wood preservatives (beeswax, nano-zinc particles) too [10].

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