

Effect of Soil Contact on the Modulus of Elasticity of Beeswax-Impregnated Wood

Róbert Németh,^{a,*} Dimitrios Tsalagkas,^b and Miklós Bak^a

The aims of this study were to use beeswax impregnation as a wood preservative method and to evaluate its suitability to protect wood species with low resistance to decay. Poplar (*Populus x euramericana* cv. Pannonia) and beech (*Fagus sylvatica*) samples were impregnated with beeswax and exposed to soil contact for 18 months. Impregnated samples were separated into three groups, on the basis of their degrees of pore saturation (DPS). With progressing decay, the load-bearing capacity and modulus of elasticity (MOE) of the woods decreased. After one month of soil contact, there was a marked decrease in MOE, which is explained by the increase in the moisture content of the wood. After 18 months, control samples were completely decayed. Nevertheless, impregnated samples showed less decay and a noticeable remaining load-bearing capacity. Impregnation efficiency had a pronounced effect on decay resistance. In both investigated species, samples with higher DPS resulted in less of a decrease in MOE than in samples with lower DPS. Although beeswax is a bio-based material, it showed noticeable decay resistance effects against soft rot. Scanning electron microscopy investigations showed that the impregnation has a barrier effect, mostly in the longitudinal direction, against the spread of the fungi.

Keywords: Beeswax; Soil contact; Decay test; Wood protection; MOE; Beech; Poplar; SEM

Contact information: a: Institute of Wood Science, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, University of West Hungary, Bajcsy-Zsilinszky u. 4., H-9400 Sopron, Hungary;

b: Institute of Wood Based Products and Technologies, Faculty of Wood Sciences, University of West Hungary, Bajcsy-Zsilinszky u. 4., H-9400 Sopron, Hungary;

* Corresponding author: robert.nemeth@skk.nyime.hu

INTRODUCTION

Waxes have been used for wood surface finishing and coating from the ancient times. Since biocides, due to EU regulations, are increasingly restricted, waxes and wax emulsions are becoming some of the most important solutions for non-biocidal wood protection in outdoor applications to improve durability, dimensional stability, and sorption properties. Furthermore, treatments with waxes slow the photodegradation process of wood as well (Lesar *et al.* 2011). The advantage of beeswax is its biological origin and its nontoxic nature. However, natural waxes are generally not biologically stable (Schmidt 2006). They can delay the decay of wood because waxes are water-repellent, and with the impregnation method, the cell lumens can be filled with wax. As a result of the hydrophobic properties of beeswax and the lumen filling, the decay of wood by fungi is slowed (Lesar and Humar 2011).

The presence of free water and the presence of some oxygen is necessary for fungal growth and decay. When the lumens are filled with waxes or oils, it is assumed that the remaining void volume is not suitable to store enough oxygen for respiration (Sailer 2000).

This might be the reason for the improved durability of wax-treated wood using high retentions (Lesar and Humar 2011). On the other hand, during the solidification of wax it has a volume shrinkage; therefore void spaces between the cell walls and wax can be formed. (Scholz *et al.* 2010a). These cracks are pathways for water in liquid or steam state, and hyphae. Thus, wax impregnated wood can be also affected by blue stain fungi (Lesar and Humar 2011).

Waxes have the effect of reducing termite damage as well, but they cannot protect wood completely (Scholz *et al.* 2010b). Another advantage of wax impregnation of wood is the improvement in wood's mechanical properties. For example, the hardness can be increased in beech wood up to 86 to 189% in the longitudinal and lateral directions, respectively.

Impregnation with different waxes can also improve other mechanical properties of wood, such as compression, bending, or impact-bending strength (Scholz *et al.* 2010c). Different waxes, including beeswax, are often used as conservation agents for wooden artifacts (Timar *et al.* 2010, 2011) or wood consolidation (Hutanu *et al.* 2013). This shows that under appropriate conditions, beeswax is suitable for wood protection.

Waxes are natural or synthetic substances depending on their origin. Beeswax is a natural, renewable, first generation, and animal-derived wax. It is a complex, heterogeneous substance, and its chemical composition presents a huge diversity of components because of its lipid nature. Beeswax is mostly composed of a mixture of hydrocarbons (~14%), free fatty acids (~12%), monoesters, diesters, triesters, hydroxy monoesters, hydroxy polyesters, fatty acid polyesters (together ~70%), and some unidentified compounds (~6%). Each class of compounds consists of a series of homologues differing in chain length by two carbon atoms (Abate *et al.* 1970; Tulloch 1971; Endlein and Peleikis 2011; Maia and Nunes 2013). Nevertheless, beeswax, as most of the water repellents, does not exhibit a biocidal effect.

Furthermore, beeswax is partly crystalline, and its melting point can be found between 61°C and 67 °C depending on the geographic origin of the material (Gaillard *et al.* 2011). However beeswax is a naturally water repellent material, and its water vapour permeability is one of the highest among the waxes. This property is attributed to its content of fatty acids, alcohols, and esters (Donhowe and Fennema 1993).

Even more, it was also reported that when beeswax is used as an impregnant, it improves the water vapor barrier properties of chitosan-beeswax bilayer-coated paper, since long-chain fatty acids and waxes are considered to be effective barriers to water vapour (Zhang *et al.* 2014).

During the outdoor utilization of wood, the hazard class of soil contact is ranked very high among the exposure classes (use class 4, according to EN 335 (2013)). For use in soil contact, very effective protection and/or durable wood species are needed. Recently, there have been only a few reports from long-term field tests of wax treated wood above ground (Brischke and Melcher 2015) or in ground soil contact observations (Palanti *et al.* 2011), but these studies investigated empty cell processes. The objective of this study was to evaluate the effectiveness of a full cell process beeswax impregnation against the degradation of less durable wood species (poplar and beech) when are subjected to soil contact for 18 months. Various beeswax impregnation intensities were examined in both poplar and beech.

EXPERIMENTAL

Poplar (*Populus × euramericana* cv. Pannonia) and beech (*Fagus sylvatica*) samples were impregnated with beeswax and exposed to soil contact for 18 months. Unimpregnated samples were used as the control. The two wood species have different physical properties, but both of them have low resistance against decay without protection (Class 5 according to EN 350-2 (1998)).

The sample dimension for all samples was 20 × 20 × 300 mm. Average oven-dry density of the beech samples was 725.51 kg/m³, while the same data for poplar samples was 382.41 kg/m³. Ring orientation was parallel to one sheet of the samples, thus they had clear radial and tangential longitudinal surfaces. The samples of both beech and poplar were cut from planks without red heartwood.

Producer of the beeswax was the Reanal Fine Chemicals Factory. Melting point (dropping point) of the used beeswax was 61-66 °C.

The goal was to completely fill wood pores with wax, or at least get their surface coated with wax as a result of the impregnation, depending on the degree of impregnation. Samples were dried (moisture content: 0%) before impregnation to obtain their dry mass, to be able to calculate the weight of the injected beeswax in the samples. With this data and the density of the beeswax the volume of the injected beeswax (V_{BW}) was calculated for the determination of the degree of pore saturation (DPS). DPS was calculated as a ratio of the theoretical pore volume of wood material and the volume of the beeswax injected into the pores (Eq. 1),

$$DPS = \frac{V_{BW}}{V_{PTh}} \times 100 \quad (1)$$

where DPS is the degree of pore saturation (%), V_{BW} is the volume of the injected beeswax (cm³), and V_{PTh} is the theoretical pore volume of wood (cm³).

Beeswax was melted at 80 °C in a closed chamber, and the dry samples (moisture content: 0%) were put into the melted beeswax. After that, the pressure was decreased in the chamber to 150 mbar for 4 h. Following the vacuum period, the pressure was increased to atmospheric pressure and the temperature of the beeswax (with the samples) was kept at 80 °C for 20 h. Impregnated samples were separated into three groups, on the basis of the DPS (Table 1). All of the separated groups contained 25 samples, so there were 25 untreated samples for the control and 75 impregnated samples.

Table 1. Sample Groups According to the Degree of Pore Saturation (DPS)

| Group | Poplar1 | Poplar2 | Poplar3 | Beech1 | Beech2 | Beech3 |
|---------|---------|---------|---------|--------|--------|--------|
| DPS (%) | 20-40 | 40-55 | 55-70 | 60-75 | 75-90 | 90-100 |

The effect of outdoor exposure with soil contact of the samples was investigated under laboratory conditions, based on the standard ENV 807/2001. The soil was compost, collected in the botanical garden of the University of West Hungary in Sopron. The area is under natural protection, without use of any biocides. Soil was collected in a plastic box. The starting moisture content of the soil was raised up to 95% of its water holding capacity (WHC). The samples were put into this soil to a depth of half their lengths (~15 cm). To retain the moisture content of the soil, the boxes were sealed up with plastic foil,

maintaining a wet climate, which is very favorable for fungal growth (Fig. 1). In addition, the moisture content of the soil was measured with the drying test (at 103 °C) at the beginning and in every month during the test, and if it was necessary, water was added to the soil. The temperature was the room temperature, which was controlled between 20 and 25 °C during the test by common air climatization of the laboratory.



Fig. 1. A plastic box with poplar samples in the soil after opening the foil

Modulus of elasticity (MOE) was determined initially at the absolute dry states of the unimpregnated and impregnated samples before the insertion of the samples into the soil. To determine the MOEs, a standard three-point bending method was used, based on the standard MSZ 6786-15/1984. The MOEs were determined at a defined load to avoid damaging the samples. The different load-bearing capacities of the investigated wood species were taken into consideration, so the loads were 400 N for poplar and 600 N for beech. The second inspection of the MOE of the samples was made after one month of soil contact. The load was the same as that at the initial determination of MOE in the absolute dry state, before soil contact. The third inspection was made after 18 months of soil contact. In this case, the load decreased according to the expected damage of the samples. The load was 300 N for both wood species, but MOE was determined at 150 N as well because, in some cases, the load-bearing capacity of the samples was under 300 N. The inspection after 1 and 18 months in soil contact was carried out without drying the samples, to avoid the sterilization of the samples from the wood destroying organisms. Furthermore, beeswax has a dropping point of 61 to 66 °C; therefore the beeswax would become redistributed in the samples as well, as a result of drying at the necessary temperature (103 °C). During the test, the samples were put into the soil to a depth of half their lengths. Thus, the load during the determination of the MOE was put on the samples at the ground level (between soil-contacted and above soil parts). As this is the weakest point of the wood (Edlund *et al.* 2006) in such a soil contact, it can be stated that predominately the minimum values were measured. Statistical analysis with the software Statistica 12 (ANOVA analysis) was performed with the data to state the validity of the results. The used post-hoc test was the LSD-test.

After 18 months of soil contact, the samples were investigated with scanning electron microscope (SEM) imaging to determine the extent of the decay and the effect of beeswax on the decay. Furthermore, the location and distribution of the beeswax in the structure of the wood was studied. The device used was a Hitachi S3400 scanning electron microscope. Specimens were scanned under vacuum pressure of 70 bar and an accelerating voltage of 25 kV. The surfaces were not gold coated with a sputter-coater machine before the imaging. 30 mm long samples for SEM imaging were cut from the part exposed to soil of the decayed bending samples. The surfaces exposed to soil of these samples were investigated with SEM. Additionally, longitudinal (radial or tangential) sections were also cut and investigated, to have an overview from the location of the beeswax and the longitudinal spreading of the fungi in the wood.

RESULTS AND DISCUSSION

Changes of the MOE during Soil Contact

The protecting effect of beeswax could be observed with visual inspection. The ends of both the untreated beech and poplar samples that were put into the soil were almost completely decayed after 18 months of soil contact (Fig. 2a, 2b). The initial cross sections decreased markedly, and the texture of the wood was disintegrated. The unimpregnated samples lost their load bearing capacity completely until the end of the investigated period, so determination of the MOE was impossible. In spite of that, on the impregnated samples, only surface decay could be observed, and the initial cross sections remained almost unchanged. It was not possible to find differences between the impregnation groups with visual inspection.



Fig. 2. 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. The initially 12,100 MPa MOE of beech increased up to 13,600 to 15,000 MPa, depending on the impregnation efficiency. The initially 6200 MPa MOE of poplar increased up to 8000 to 9200 MPa, depending on the impregnation efficiency. An interesting result was that the impregnation efficiency had an inverse effect on the MOE. Poplar with higher impregnation rates resulted in a higher MOE, but in beech with higher impregnation rates, the opposite could be observed before soil contact and after one month of soil contact (Fig. 3).

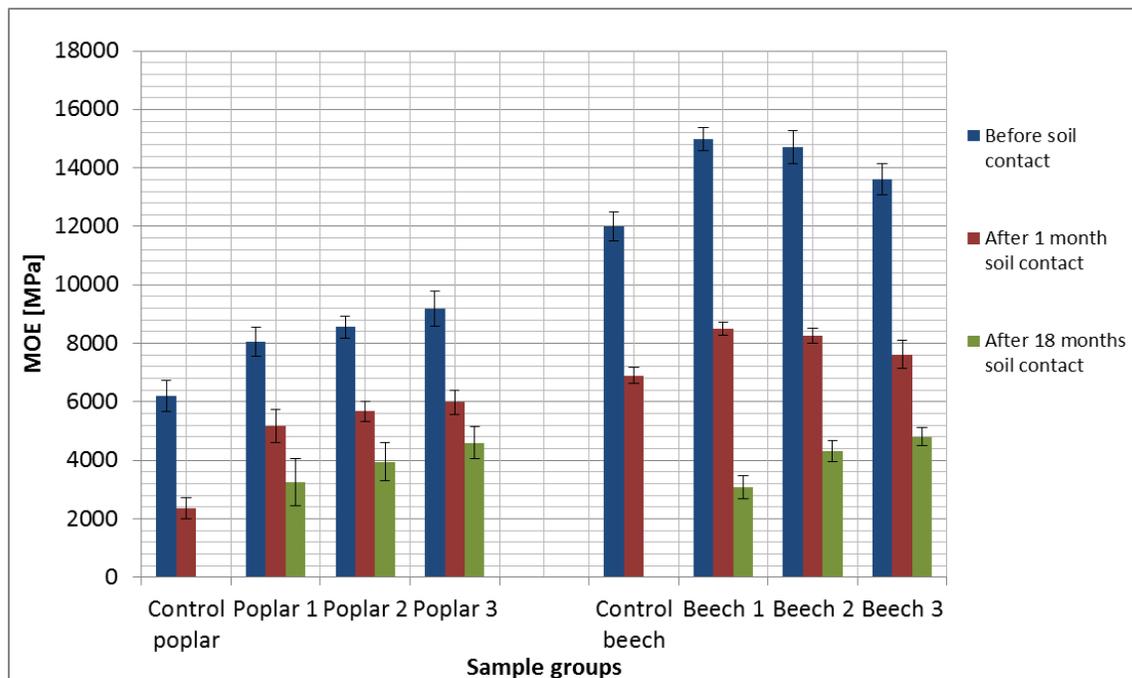


Fig. 3. MOE of poplar and beech samples in the investigation periods

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 to near the fiber saturation point). As no decay could be observed on the samples, and though 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. The values measured at the absolute dry state are close to the theoretical maximum of the investigated samples, while MOE measured after one month of soil contact can be considered the MOE under conditions of utilization. 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 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. Despite this, beech samples showed lower MOE at higher DPS values before soil contact. This result shows clearly that impregnation efficiency is an important factor in wood protection against decay.

The decrease in MOE could be explained also based on visual inspection, because after cutting the samples, the decay of the inner parts could be seen as well as the decay of

the surfaces. This decrease in the solid cross section resulted in a decrease of the load-bearing capacity and the MOE for both wood species. In addition to all of the unimpregnated beech and poplar samples, some impregnated samples were damaged completely as well because their load-bearing capacity was less than 150 N. Some samples had load-bearing capacities of over 300 N, but they had extremely high deformation during the load. This indicates the strong degradation of the cell wall structure. Most of the samples probably could bear much higher loads.

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.

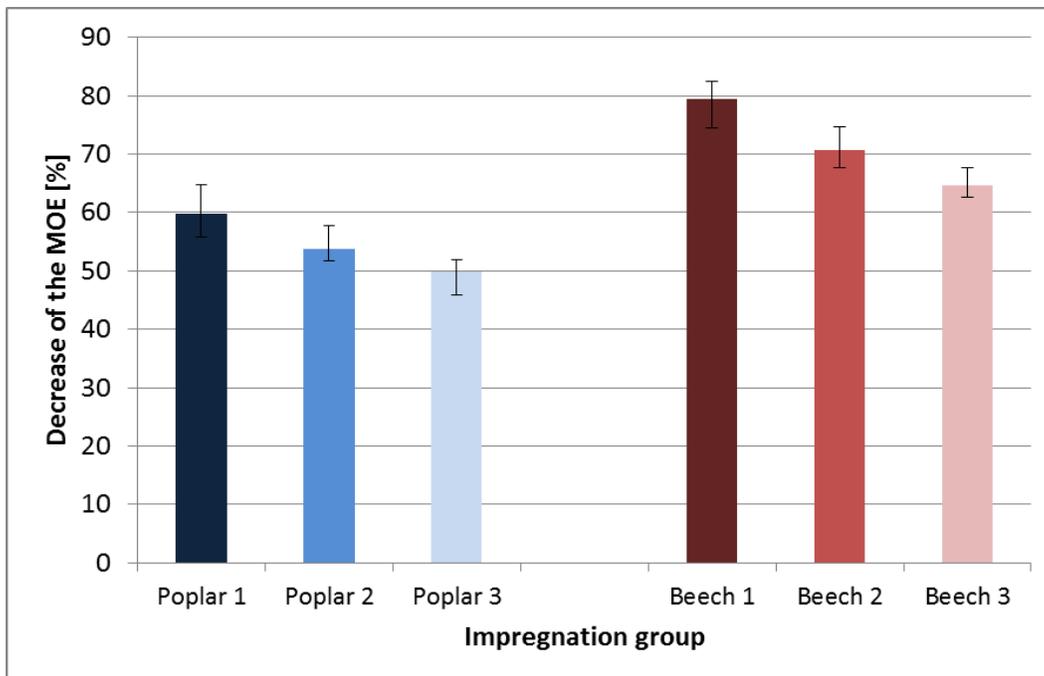


Fig. 4. MOE decrease of beech and poplar samples after 18 months of soil contact

SEM Investigations

The following observations are valid for both beech and poplar wood. The selected pictures represent the most typical observations valid for both wood species impregnated with beeswax. The beeswax could be identified with SEM imaging in the cell lumens, mostly in the vessels. Beeswax filled the whole vessel lumen in most cases (Fig. 5a), but sometimes it only coated the inner surface of the lumen, like a protective layer (Fig. 5b).

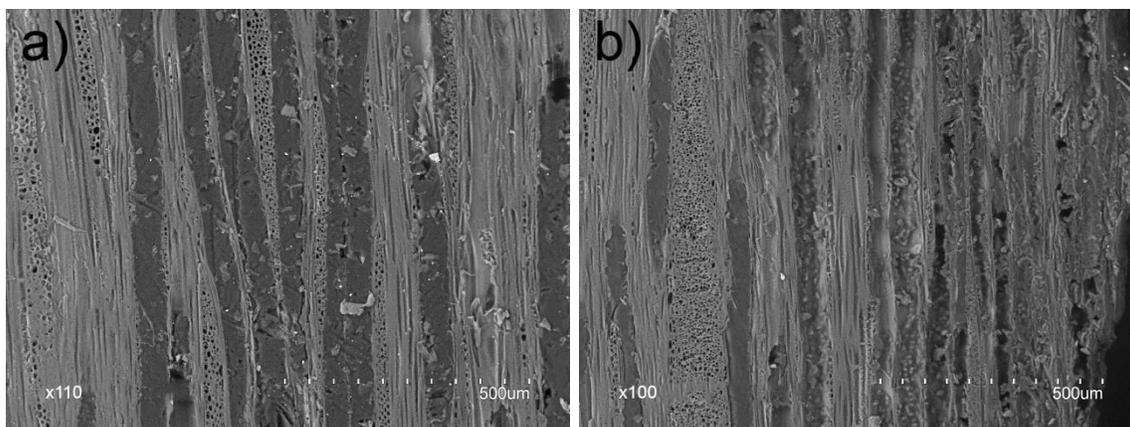


Fig. 5. Beeswax filling the vessels (a) and coating the inner side of vessels (b) of beech wood

Vessels were impregnated best with the beeswax, but filling of voids could be observed in other cell types as well. Ray cells (parenchymal cells) were filled with beeswax as well, but because they have smaller lumen diameter, the ratio of the beeswax filling was lower (Fig. 6a). In several parts of the wood, the filling of the libriform cell lumens could also be observed (Fig. 6b).

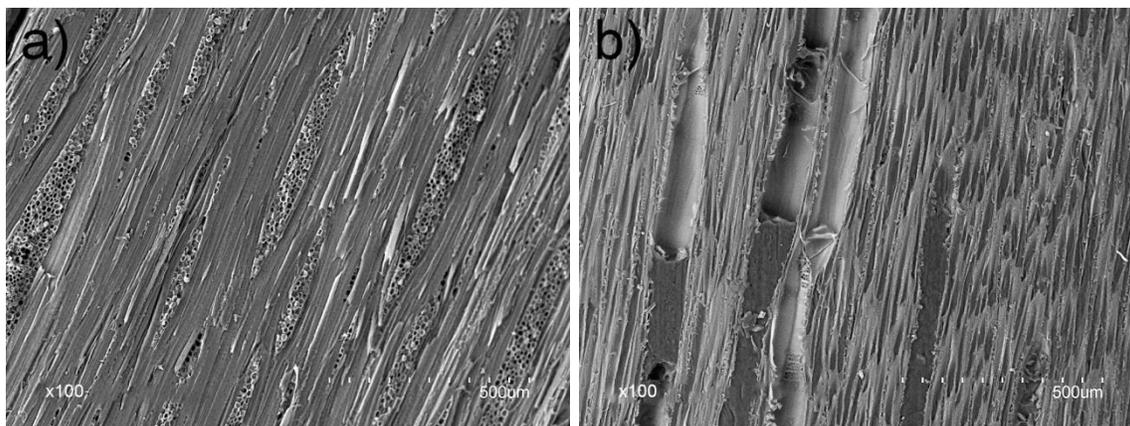


Fig. 6. Beeswax filling the ray cell lumens of beech wood (a) and fibres of poplar wood (b)

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 (Fig. 7). These areas could help the fungus to spread through the wood faster, compared to the totally impregnated wood parts.

Hyphae could be observed in large quantities only on the surfaces of the specimens that had direct soil contact (Fig. 8a). Hyphae in the inner structure of the specimens were rare to find, and only in lumens without any beeswax (Fig. 8b). 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 biocidal 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.

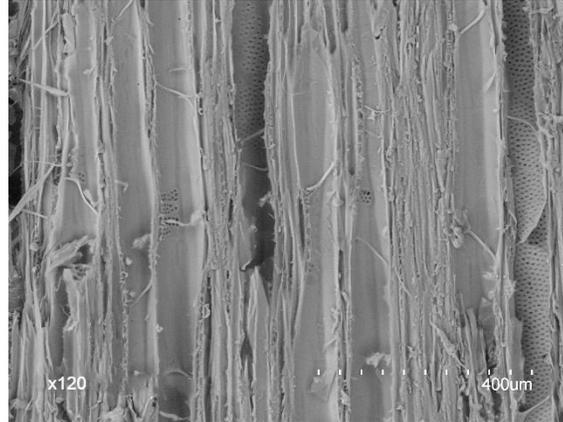


Fig. 7. Poplar wood cells without beeswax

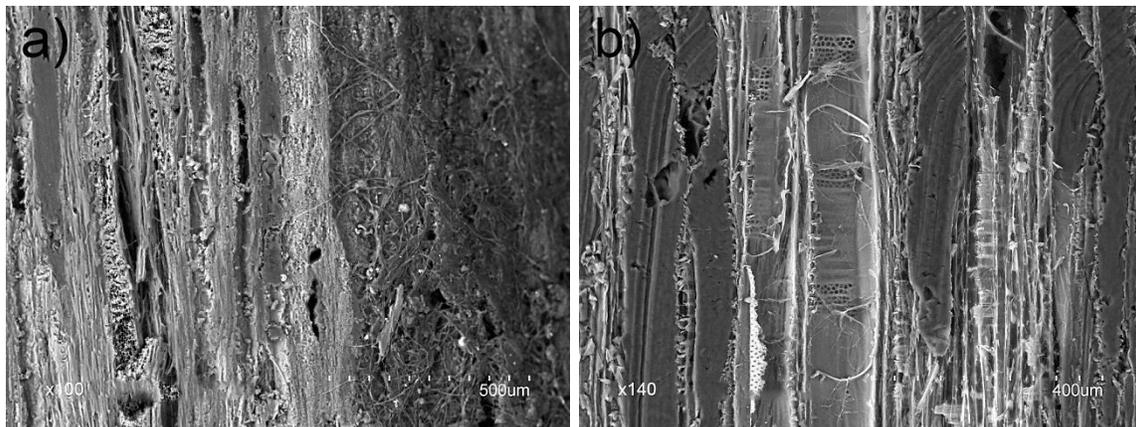


Fig. 8. (a) Hyphae on the surface of a beech sample (b) and in a beeswax free cell lumen of poplar wood

As expected, the mostly decayed area was the surface that had direct soil contact and the surface area of the samples near the soil (Fig. 9a). Decay could be observed mostly in areas that did not contain any beeswax in the cell lumens (Fig. 9b). The decomposition of the wood texture was far gone in these areas.

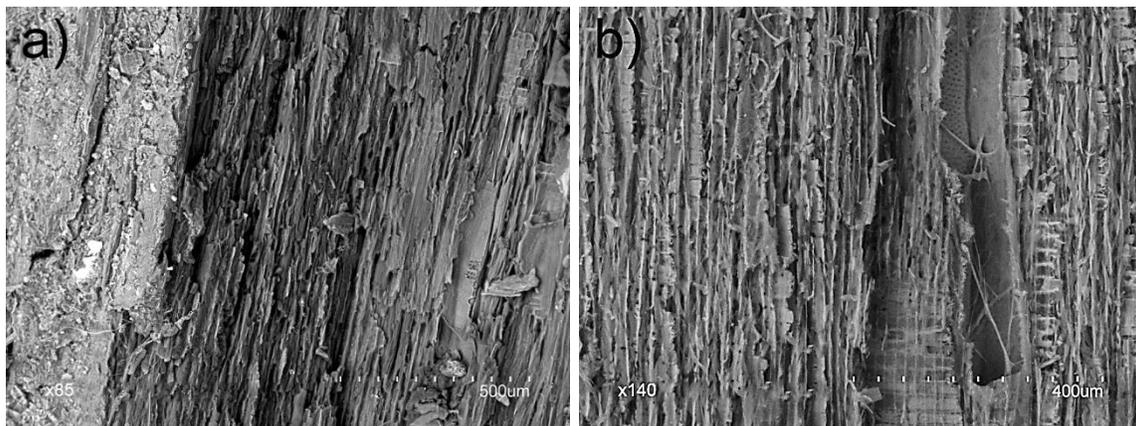


Fig. 9. Decayed surface area (a) and inner part without beeswax (b) of a poplar specimen

Nevertheless, the areas with proper impregnation of the lumens were untouched by decay (Figs. 5 and 6). However, if there was one or more empty vessel near the filled vessels, then the hyphae could have spread through the cell walls in the direction of the impregnated cells and started the decomposition (Fig. 10a,b). Accordingly, the beeswax impregnation presumably slowed much more of the longitudinal spreading of the hyphae than the transversal spreading.

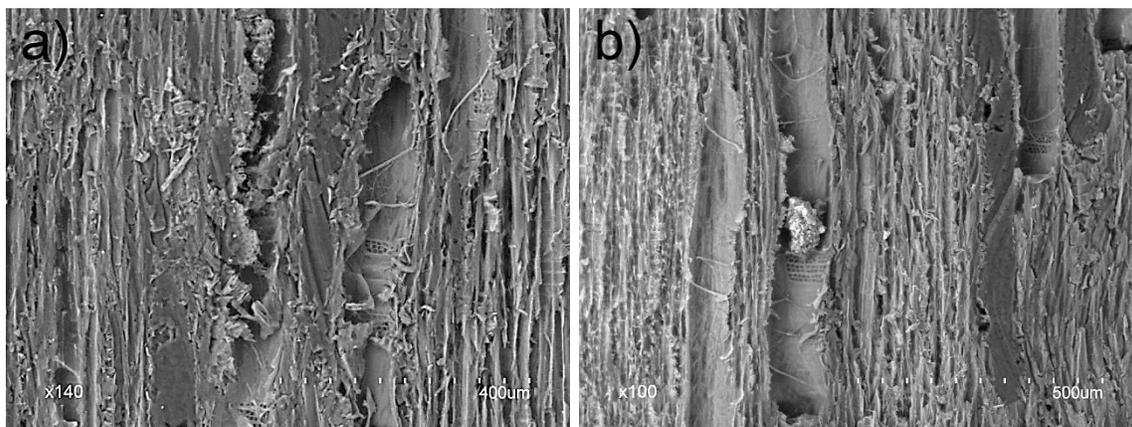


Fig. 10. Spreading of hyphae and start of the decomposition on the borderline of the impregnated and unimpregnated wooden parts of poplar samples (a and b)

In several beech samples, damage in the surface area by insects was observed as well (Fig. 11). This confirms that beeswax has no toxic effect, or that its biocide effect is very low at least. On the other hand, it reveals a problem. Beeswax impregnation has probably no protective effect against insect damage, as it appears that wood-destroying insects can digest beeswax together with wood.



Fig. 11. Insect damage in the surface area of a beeswax impregnated beech sample

CONCLUSIONS

1. Beeswax impregnation increased the MOE of beech and poplar wood. Unimpregnated beech and poplar samples decomposed completely during the 18 months of soil contact. In spite of that, the damage of the impregnated samples was markedly lower. This was confirmed by the MOE measurements, which showed remarkable remaining MOE of the impregnated samples after soil exposure. The impregnation improved the wood's resistance against wood decaying organisms, and higher DPS resulted in less of a decrease in MOE than in samples with lower DPS.
2. SEM imaging showed that beeswax filled the lumens and separated most of the cell walls from the hyphae, which slowed the spreading of the fungi in the wood. This explains the protecting effect of the beeswax, even though it does not contain any "artificial" biocidal agents. The decomposition of cells without beeswax was much more pronounced than that of beeswax filled cells.
3. SEM imaging showed that the beeswax impregnation slowed much more of the longitudinal spreading of the hyphae than the transversal spreading.

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