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TREATMENT OF THERMALLY MODIFIED WOOD WITH A SUSPENSION OF TITANIA NANOPARTICLES TO IMPROVE ITS SELECTED PROPERTIES

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Abstract:
Results of the research aimed to increase the resistance of beech and thermally modified (TM) beech wood to UV-light and weathering with impregnation with commercially available nano TiO_2 suspensions, originally intended to be used as UV protective coating additives, are presented in this paper. Beech wood and TM beech wood samples were vacuum-pressure impregnated with suspensions, containing around 3% of TiO_2, and in one series, the specimens were at first impregnated and thermally modified afterwards. In general, resistance to UV-light, as demonstrated by visual observation and by colour measurements was increased. Contact angle of water, liquid water absorption, and water vapour sorption also importantly influence on weathering behaviour of wood and it was shown that in general, impregnation with TiO_2 particles has a positive influence with this respect. Anyway, some results of this preliminary research were ambiguous, and subsequent investigations of this novel protection method are needed and planned.

Key words: *beech wood; thermal modification; impregnation with TiO_2; UV-protection.*

INTRODUCTION

Thermal modification (TM) of wood improves its properties, for instance dimensional stability and resistance to wood-decay fungi or wood boring insects. Thermal modification (or thermal treatment) of wood has become commercially important, the number of heat-treating companies is increasing and commercialization of TM wood is growing (Esteves and Pereira 2009). Based on the estimations of Sandberg (2019), there is approximately 500.000m^3 of TM wood produced annually, representing 50% of overall modified wood produced. At University of Ljubljana (Biotechnical Faculty) the method of wood modification in intact vacuum was developed and has been commercialised (Rep *et al.* 2012). TM wood

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retains as a natural product and the grain, original colour variances and characteristics of the wood are still present. However, due to exposure to thermal treatment, wood becomes darker (Bekhta and Niemz 2003, Esteves and Pereira 2009). TM wood is suitable for above ground outdoor applications, however it is well known that it is susceptible to weathering. The problem of photo-degradation of wood in the exterior has been a topic of numerous investigations (George *et al.* 2005, Pandey 2005, Cogulet *et al.* 2018). Similarly to non-modified wood, exposure to UV- and day-light causes greying of TM wood surfaces (Ayadi *et al.* 2003, Huang *et al.* 2012, Srinivas and Pandey 2012). Some studies report that TM wood is more resistant to photo-degradation than untreated wood (Ayadi *et al.* 2003, Nuopponen et al 2004) because heat treatment modifies chromophoric lignin structure that may interfere with the light absorbing process. On the other hand, there are quite many reports saying that TM wood is susceptible to photo-discolourations (Shen *et al.* 2018a, Tomak *et al.* 2018). Protection of wood against weathering has been extensively investigated and the possibilities for anti-weathering improvement are well known. The most common approach is to use wood coatings with UV-protecting additives, and the related reports can be found elsewhere (George *et al.* 2005). With the emerging field of nanotechnology, various nanoscale protection additives have also been intensively investigated (e.g. Saha *et al.* 2013, Rao *et al.* 2018). Among other nano UV protecting particles, nano TiO_2 has attracted considerable attention (Terzi *et al.* 2016, Zanatta *et al.* 2017 etc.). The ways to protect TM wood in exterior exposure to weathering have also been studied, but to a lesser extent than the protection of non-modified wood (Miklečić *et al.* 2017, Shen *et al.* 2018a, Shen *et al.* 2018b). Application of nanoparticles for photo-protection of TM wood is not an exempt, including the utilisation of nano TiO_2 for protection purposes (Miklečić *et al.* 2017, Shen *et al.* 2018a, Shen *et al.* 2018b).

Wood protecting formulations can be delivered to wood by various process, including vacuum pressure impregnation, or just by immersing the specimens in the treatment formulation (Shen *et al.* 2018a). When dealing with TM wood, thermal modification can be carried out prior to impregnation with the protective solution, but the process could be also reversed. If impregnation with anti-weathering substances is performed, it is a question what would be the penetration depth of a protective substance. Because photo-degradation of wood is a surface phenomenon (Pandey 2005), the so called envelope treatment of wood appears to be an interesting option. The envelope treatment is known from the field of wood protection to biological organisms (e.g. De Vetter *et al.* 2011, Kotlarewski *et al.* 2019). Envelope treatment means that only a few mm thick protective envelope is created around the treated wood piece and the rest remains non-impregnated.

Improvement of anti-weathering performance of TM wood by TiO_2 sol or/and paraffin emulsion was considered in the paper of Shen and co-authors (2018a). Their approach was the immersion of TM wood Scots pine wood in a TiO_2 sol and in some cases also the creation of a hydrophobic envelope on the specimens' surfaces. In contrast, our investigations included pre-treatment of wood with TiO_2 nanoparticles prior to thermal modification. We were focused to common beech (*Fagus sylvatica* L.) wood, because its economic importance is increasing (Husmann and Möhring 2017), and because it is expected that its total growing stock may increase in the forests in Central Europe due to the global climate change (Prislan *et al.* 2019).

OBJECTIVE

The main objective of this preliminary research was to investigate if it is possible to increase the resistance of TM beech wood to UV-light by a novel approach of vacuum-pressure impregnation of TM beech wood specimens with a commercial TiO_2 nanoparticle suspension, which is intended to be used as an additive in coating formulations. The research was aimed also to check if the so called envelope protection can be carried out and to evaluate some selected parameters of TM and impregnated wood that are important during exterior applications.

MATERIAL, METHOD, EQUIPMENT

The wood of European beech (*Fagus sylvatica* L.) was used to prepare the specimens of 50mm x 25mm x 15mm (longitudinal (L), semi-radial (SR), SR) for the majority of measurements, and of 100mm x 100mm x 10mm (L, SR, SR) for determination of colour changes during exposure to UV-light. For impregnation, a commercial aqueous dispersion of nano TiO_2 was used (CCR 220 MN, produced by Cinkarna Celje d.d., Celje, Slovenia). The nano particles in the dispersion were modified in order to ensure high protection to UV-light. The original dispersions were diluted with water to 3% final concentration of TiO_2 and the additives in impregnating formulations. Several sets of the specimens were prepared: control - untreated and non-modified wood, TM wood, wood that was impregnated with the TiO_2 dispersion, the specimens that were at first TM and impregnated afterwards, and the samples that were impregnated at first, and TM subsequently. Impregnation was performed by the vacuum pressure process, as follows: 15min in the dispersion, exposed to vacuum (-0.8 bar = -80kPa), to pressure for 1 hour (10 bar = 1MPa), 15min in a vacuum (-0.8 bar), and 30min of immersion at normal pressure. Than the samples were dried for 10 days at normal laboratory conditions. Before further manipulations and measurements, the samples were dried for

24 hours at (103±2)°C. The process of heat treatment in intact vacuum (Rep *et al.* 2012) was applied as the wood modification method: the samples were in vacuum exposed for 30min at 35°C, then at 103°C (30min), followed by exposure at 150°C (20min), at 170°C (20min), at 190°C (20min), and at 210°C for 3 hours. Afterwards the samples were left to cool down to the normal room temperature in one day. Some details of the specimen preparation are given in Table 1.

Table 1

Preparation of beech wood specimens

Sample	Type of TiO_2 and concentration in the formulation (dry matter, in %)	Uptake of the treatment solution (g/cm^3)
Control (C)	/	/
Thermally modified (TM)	/	/
Non-modified – impregnated (IM)	CCR 220 MN, 3 %	0.64
Thermally modified and impregnated (TM-IM)	CCR 220 MN, 3 %	0.68
Impregnated and thermally modified (IM-TM)	CCR 220 MN, 3 %	0.66

The following investigations were performed only after preparation of the specimens and not after exposure to UV-light: SEM observation to investigate the influence of thermal modification on wood structure and to evaluate penetration depth of the TiO_2 particles and their form, size, and positions within the wood structure; moisture content (MC) of the specimens after exposure to almost 100% relative air humidity for various times; uptake of liquid water ("long term water uptake"); capillary uptake of water ("short term water uptake"), and dynamic contact angles CM on semi-radial surfaces. Resistance to UV-light was followed by colour measurements.

SEM investigations: carried out with the FEI Quanta 250 SEM microscope (Hilsboro, Oregon, U.S.A.), equipped with the Energy Dispersive X-ray spectrometer, at a working distance of 10mm (low voltage (10kV), low vacuum (50Pa), a large field (LFD) or a circular backscatter (CBS) detector (for EDAX).

MC of the specimens after exposure to almost 100% relative air humidity: the absolutely dry specimens were put into a chamber with saturated humidity (98-100% relative humidity) for 1, 7, 14, 21, and 28 days. MC of wood was determined after each exposure period. The average values were calculated from 5 individual results per each specimen type.

Uptake of liquid water: the long-term water uptake was determined gravimetrically after immersion of the specimens in water for 1 and 24 hours and for 1 and 2 weeks. 6 parallel measurements per each set of the specimens were carried out and the average values of absorbed water calculated.

The short-term capillary water uptake tests were performed at room temperature (20°C) at a relative humidity of (50 ± 5)%, on a Tensiometer K100MK2 device (Krüss, Hamburg, Germany). The axial surfaces of the specimens (50mm × 25mm × 15mm) were positioned to be in contact with distilled water and their masses were subsequently measured continuously every 2s for 200s. The other parameters used were: velocity before contact 6mm/min, the sensitivity of contact 0.005g and depth of immersion 1mm. 5 parallel measurements were performed in order to get the average values.

Dynamic contact angles of water: the Theta (Optical Tensiometer) CA goniometer from Biolin Scientific Oy, Espoo - Finland was used to determine the CA of distilled water on the surfaces. When the goniometer microscope was focused and adjusted on the image of the drop, it was ensured that the CA on each side of the drop were approximately the same. The CA were measured by means of computer-aided analysis (OneAttension, Version 2.4 (r4931), Biolin Scientific, Young−Laplace CA analysis mode) of shapes of liquid drops, as observed in an optical goniometer and recorded by a digital camera installed in the axial extension of the lens. The drops of about 4−6μL (the volumes were calculated from images of drops) were applied by means of a dispenser. Image recording was set for 60s, and the time when CA started to be calculated (0s) was after detachment of the dispenser tip from the drop, which happened approximately 4s after the first contact of the drop with a substrate. The read-outs were taken for 15 pictures for different drops of water, and the arithmetic mean value was reported. The measurements were taken at a constant temperature of 23°C.

UV irradiation (without exposure to water): the specimens were irradiated in a chamber with one Osram Ultra-Vitalux 300W sunlamp, equipped with UV-A (315nm – 400nm) and UV-B (280nm -315nm) radiation of 13.6W and 3W, respectively, at a distance of approximately 45cm. The irradiation times were 24, 48, and 72 hours, and 7, 14, 21, 28, and 35 days. After each exposure period, the positions of the specimens were changed in order to ensure uniform irradiation.

Colour measurements: the colour of irradiated surfaces of the test samples was measured with an X-Rite SP Series Spectrophotometer (X-Rite Incorporated, S.W. Grandville, Michigan, USA). The reflection spectrum was acquired from a measuring spot of 14mm in 390nm – 710nm regions. Six measurements at precisely defined spots on the irradiated surface of each sample (6 samples per one set of specimens) were carried out after an interval of 50 hours throughout the experimental period. The colour changes were monitored in the intervals listed previously (24, 48, and 72 hours, and 7, 14, 21, 28, and 35 days), with the help of *L*, a** and *b** parameters as per the CIE (Commission Internationale de l'Eclairage) L*a*b* system. Then, the overall colour differences *ΔE** were computed using the following expression (Brock *et al.* 2000),

$$\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (1)$$

where: ΔL^*, Δa^*, and Δb^* are the changes between the initial and the final values of L^*, a^* and b^*, respectively.

RESULTS AND DISCUSSION

Moisture Content of Wood

EMC of untreated control beech wood reached the values even close to 44% (Fig. 1). These are very high values, but similar ones can also be found in literature (Báder and Németh 2017). High variation of EMC of the samples is the consequence of the fact that it is very difficult to condition the samples close to the saturation due to problems with keeping the temperature stable enough to achieve equilibrium in the over-hygroscopic relative humidity (RH) range (98–100% RH) (Thybring *et al.* 2018). Impregnation with the TiO_2 suspension decreased this value to about 28%. As could be expected (Olek *et al.* 2012) thermal modification significantly reduced the EMC of wood, regardless of impregnation with TiO_2, to the values between 17% and 19%.

Uptake of Liquid Water and Capillary Uptake of Water

Uptake of water during the submersion test is in Fig. 2 presented in absolute values, but the results are comparable because the specimens were of the same dimensions. As in the case of water vapour uptake into the samples kept in the water-saturated atmosphere, the uptake of liquid water was the smallest one and comparable into all three types of TM wood (TM, TM-IM, and IM-TM). The uptake was higher in the case of control untreated beech wood and the highest one when beech wood was impregnated with the TiO_2 suspension. Also in the case of the short-term capillary water uptake test, the uptake of water was expectedly the lowest one into the TM specimens (Fig. 3), and only slightly higher into untreated beech wood. However, distinctively higher was the capillary uptake of water into beech wood, impregnated with TiO_2. During the experiment it was clearly seen that the uptake is of water is high and fast: in the region of the capillary uptake, the samples looked wet and became heavily distorted (insert in Fig. 3). At the moment we do not have an explanation for such behaviour, but it might be connected to the TiO_2 deposits in the vessels forming new capillaries influencing short water uptake (see Fig. 5).

Contact Angles of Water

CA of water on untreated wood were around 80° (Fig. 4) and impregnation with TiO_2 as well as thermal modification made the specimens hydrophobic. In addition, impregnated and TM wood were hydrophobic with CA somewhat above 120°. However, due to unexplained reasons, the CA of water were low, only about 40° on the specimens that were TM and afterward impregnated with titania suspension.

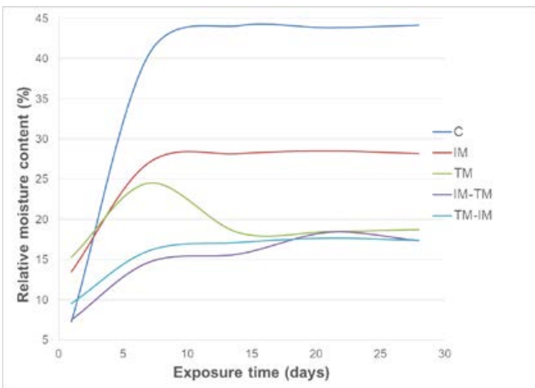

Fig. 1.
Moisture content in control and treated beech wood specimens after exposure to almost 100 % relative air humidity for 1 to 28 days.

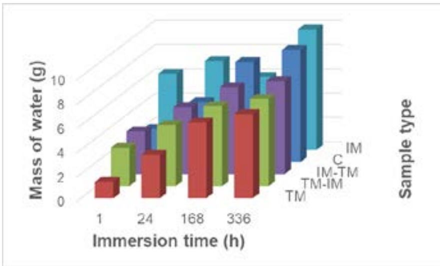

Fig. 2.
Uptake of water during the submersion test.

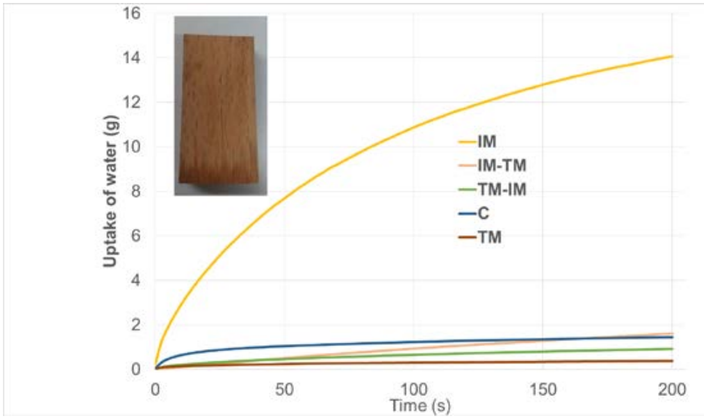

Fig. 3.
Short-term capillary water uptake. In the insert, there is the IM sample immediately after the test.

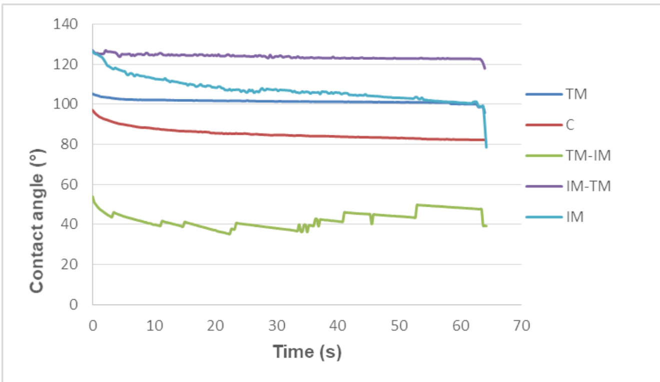

Fig. 4.
Contact angles of water on variously treated specimens.

TiO_2 in Wood

The thickness of the samples was 10mm and the SEM micrographs and EDAX analyses showed (Fig. 5) that the TiO_2 particles were present in impregnated, TM and impregnated, and impregnated and TM samples at all distances from the surface. Therefore, we cannot talk here about envelope treatment. In continuation of the research, it is planned to impregnate larger samples and to change the impregnation parameters with the aim to impregnate only the envelopes. However, it should be considered that beech wood has rather good permeability. At refractory wood species smaller penetration is expected. As can be also seen in Fig. 6, TiO_2 was present in cell lumens in the form of micrometer size scale (roughly between 1μm to 50μm) and not in the nanometer scale. This means that during the impregnation process or later, during drying, agglomeration of the particles occurred in the lumens. Nevertheless, titania retained its UV-protective efficacy, as demonstrated in Figs. 6 and 7.

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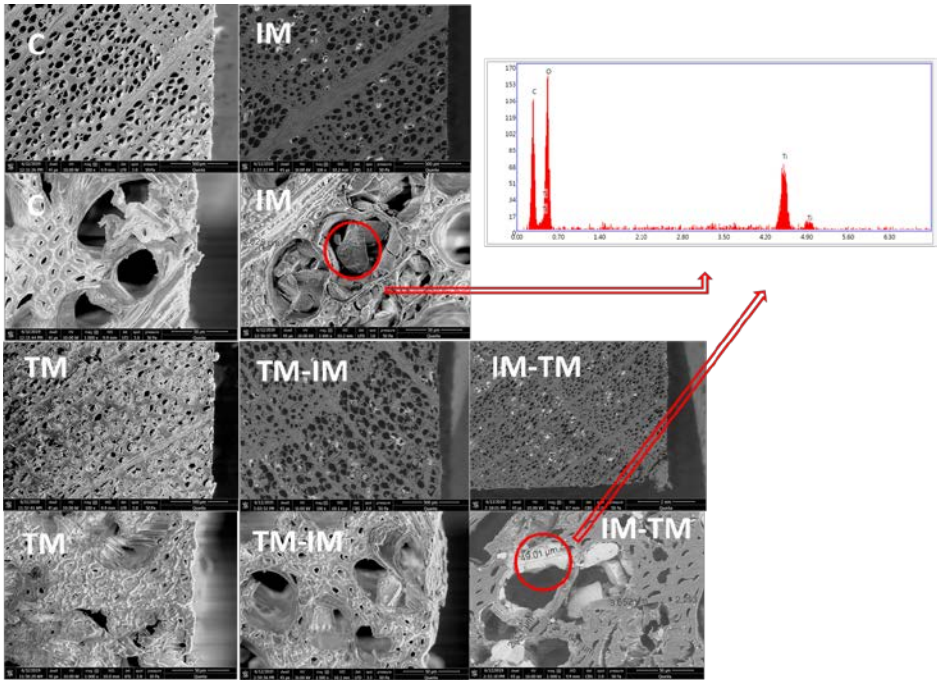

Fig. 5.
SEM micrographs of the specimens. The magnifications were 50x (IM-TM in the third row), 100x (C and IM in the first row, and TM and TM-IM in the third row) and 1000X (all other micrographs). The inserted graph is showing a typical EDX spectrum taken on the particles in the vessels.

Visual Appearance of the Specimens and Colour Changes

Fig. 6.
Visual appearance of beech wood (C), impregnated (IM), thermally modified (TM), impregnated and thermally modified (IM-TM), and thermally modified and impregnated samples (TM-IM) before (upper row) and after 35 days of exposure to UV-light (bottom row).

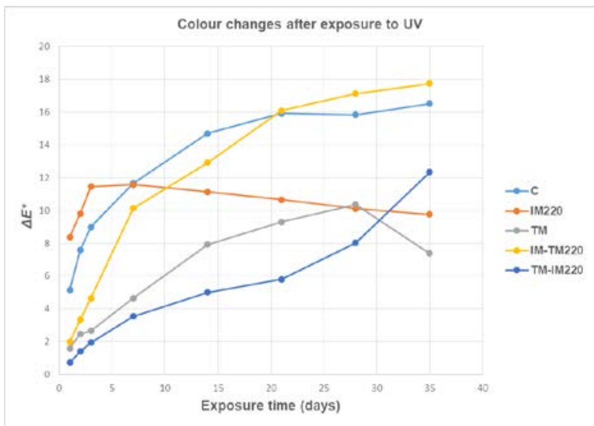

Fig. 7.
Colour changes of variously treated specimens after exposure to UV light for 1 to 35 days.

The visual appearance of all specimen types before exposure to UV-light and after exposure for 35 days is presented in Fig. 6. The specimens were not exposed to water, which can accelerate effects of UV-light (Cogulet *et al.* 2018) and can cause leaching of TiO_2 (Shen *et al.* 2018b). Impregnation of beech wood with the suspension of TiO_2 resulted in its improved resistance to colour changes imposed by UV-light. On the other hand, the situation with TM wood was somewhat different. Impregnation of TM samples with the titania suspension caused the samples to become lighter (last two samples in the upper row), and also exposure to UV-light resulted in their lightness. Anyway, it seems that the samples that were at first TM and afterwards impregnated performed the best among all three types of modified wood. Detailed analysis of colour changes is presented in Fig. 7. The response of the samples in terms of colour changes was pretty much the same in the cases of non-impregnated and non-modified (control) wood and of impregnated and subsequently TM wood. It seems that the process of thermal modification induced the loss of the protective efficacy of TiO_2 and the reasons for such performance should be investigated in further studies. TM wood exhibited the lowest *ΔE** value, similarly as in some other studies (Ayadi *et al.* 2003, Nuopponen *et al.* 2004), reporting that TM wood is more resistant to photo-degradation than untreated wood. The protective effect of TiO_2 on UV resistance of non-modified wood was obvious, and we could not detect any essential negative impacts on UV resistance of TM wood, as was the case at IM-TM220. Finally, it has to be noted, that the values of *ΔE** only are misleading when we think of the visual colour impression. For instance, in Fig. 6 it looks like the sample TM-220 performed better than the TM wood (TM), but the *ΔE** values (Fig. 7) show just the opposite. However, the colour itself is not a sufficient measure of photo-degradation. In order to fully elucidate this phenomenon, other methods (FT-IR, roughness determination,…) will be applied in our future investigations.

CONCLUSIONS

The results of this preliminary research demonstrated that it is possible to impregnate beech and thermally modified beech wood with the TiO_2 nanoparticle suspension, commercially intended to be a UV protective coating additive. In all types of impregnated specimens, TiO_2 formed 1-50μm large agglomerates in cell lumens. TiO_2 in wood exhibited UV protective efficacy. During outdoor exposure, weathering performance of wood is importantly influenced also by the wood – water or wood – water vapour relationships and in general, after treatment, the specimens became more hydrophobic and the liquid water absorption was decreased. It is believed that the results are promising enough to continue our initial research of protection of beech and thermally modified beech wood with vacuum- pressure impregnation with commercial TiO_2 suspensions, currently used as UV-protective additives in coatings.

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