28th ICWST International Conference

IMPLEMENTATION OF WOOD SCIENCE IN WOODWORKING SECTOR

PROCEEDINGS

Zagreb, 7th - 8th of December 2017

University of Zagreb - Faculty of Forestry • Biotechnical Faculty, University of Ljubljana • Faculty of Forestry and Wood Sciences, Czech University of Life Sciences Prague • Forest Products Society • InnovaWood

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FOREWORD

Continuous changes on international market open up new horizons and opportunities, and the new strategies adopted by Europe and the world bring new concepts that need to be adapted and followed. This concept seeks increased social cohesion, striking with the harmful effects of climate change, nature preservation and the creation of a healthy environment. At the same time, creative potentials are open to new knowledge and innovative processes whose primary objective is to adapt to the needs of customers and the environment.

One of the activities carried out in recent years in order to preserve and stimulate rational utilization of raw material is certainly the traditional international scientific conference AMBIENTA. During its continuous sequence in the last 27 years it has become a platform for meeting and networking among scientists, teachers, researchers, students and professionals. In the year 2015 this conference has grown into an international conference on wood science and technology (ICWST), and from last year it became a two-day event, hosted by the Faculty of Forestry of the University in Zagreb.

This year's conference, the second in a row held under the title "*The implementation of science in the woodworking sector*" aims to ensure a multidisciplinary forum where all the participants have the opportunity to present and discuss innovations, trends and practical challenges they have faced in the world of wood science and technology, but also in relation to other materials, technologies, design and other related topics whose aim is to upgrade the wood industry.

We hope that this year's conference will contribute to awareness raising about the significance of wood as an irreplaceable natural raw material, and that the application of scientific research has a positive impact on the wood sector as well as any user of wood.

Assistant Professor Ivica Župčić, PhD

Effect of Water Repellents on the Surface Characterisation of Scots Pine (*Pinus sylvestris*) Exposed to Accelerated and Outdoor Weathering

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ABSTRACT

Numerous chemicals have been investigated to inhibit photodegradation of wood and to protect it long term against weathering in-service. However, most of these chemicals cannot provide a long-term protection on wooden surface. Therefore, water repellent materials are needed to stabilize the wood surfaces and make it permanent against photodegradation. In this study, the influence of Poly Ethylene Glycol (PEG 600), N,N-(1,8-Naphthalene) hydroxylamine (NHA-H) and Methyl Hydrogen Silicon (MHS) were investigated against UV light. For this purpose, wood samples were impregnated with these chemicals according to full cell process (pressure or vacuum). After impregnation, the samples were exposed to accelerated weathering (500 hours) and to outdoor weathering for 6 and 12 months respectively. Optical and chemical changes on the surface of treated and untreated wood samples were studied by colour changes, glossiness and Fourier Transform Infrared Spectroscopy (FTIR) analysis. When the results regarding total colour change were examined, the best results were obtained in the samples impregnated with MHS both in the accelerated and outdoor weathering test. In the control specimens, new peaks were formed at 2927 cm⁻¹ and 1697 cm⁻¹, while the chemical structure maintained its integrity in samples impregnated with MHS.

Key words: Methyl Hydrogen Silicon, N,N-(1,8-Naphthalene) hydroxylamine, Poly Ethylene Glycol, Weathering

2. INTRODUCTION

Wood is treated by either biocidal or non-biocidal chemicals to protect it against biotic and abiotic causes of degradation. Wood impregnated with CCA (copper/chromium/arsenic) displays leaching which is less than preservatives free of chromium. However, due to the presence of arsenic in the CCA, its use in today has been restricted and replaced by new generation wood preservatives. The main problem of the preservatives substitution with CCA is its susceptibility to leaching from the wood material after the impregnation and corrosiveness.

Despite the use of plastic, metal, aluminium, concrete and cement products instead of wood with the advancing technology, wood is always preferred in many places because of its appearance, insulation and easy processing. Impregnated wood is considered to be a building material because it is resistant to biotic and abiotic agents, due to the aesthetic appearance and economic aspect. Wood is used in several parts in the buildings such as roofing materials, joinery and covering materials, scaffoldings, and decorative materials. Apart from such materials, wood provides services in the following places; telecommunication poles, railway sleepers, water cooling towers, marine support posts, joinery and exterior facades, roofing materials, fence poles (Kartal, 1998).

Water-borne wood preservatives do not cause odour problem, moreover surface treatments can be applied to wood after impregnation, and offer a safer material for transportation and use.

Unprotected wood surfaces exposed outdoors are affected by photodegradation, leaching, wood hydrolysis and water swelling, discoloration and decay fungi. Chemical, colour, microscopic and physical changes occur on wood surface as a consequence of exposure to biotic and abiotic factors. Wood degrading factors due to the weathering are as follows;

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Light. It was reported that when the wood surface was exposed the weathering, 60% of the wood components are affected by the UV radiation in the wavelength range of 200-400 nm; in particular, lignin was degraded as a result of chemical reaction series. Hence, the colour of the wood exposed to sunlight turns into yellowish or brownish, and then greying. These changes are superficial, and occur only at a depth of only 0.05-0.5 mm, and in particular UV radiation initiates photodegradation by changing the chemical composition of the lignin on the wood surface.

Water: It increases the formation of free radicals by enlarging the gaps in the wood and by allowing light to penetrate deeper into wood.

 O_2 : Oxygen molecules react with free radicals to form polymeric epoxy radicals and peroxides. Thus, oxygen provides the formation of free radicals and plays an important role on the weathering by light effect.

Temperature: The increase in temperature is accompanied by an increase in photochemical reactions in wood in addition to crack formation.

To protect wood surface against outdoor weathering, paints are used to either form a film layer on the wood surface or penetrate into wood without forming a coating layer.

Protection methods against weathering should ensure the following (Temiz, 2005);

- prevention of UV radiation
- modification of light absorption units being in wood components
- elimination of the components causing colour change
- removal of oxygen or reduction of O₂ reaction
- removal of free radicals generated from the light effect.

Water repellents are used to protect wood material against moisture uptake and decay fungi. Such materials reduce water absorption and delay the development of decay fungi. Although they are not classified as paints, preservatives protect wood from outdoor weathering and decay without the use of paint. Wood impregnated with preservatives under pressure generally shows high resistance to exterior conditions (Feist and Ross, 1995).

The use of biocides in wood protection for outdoor use poses an environmental risk in the case of leaching. Therefore, environmentally friendly chemicals are needed. Many traditional preservatives are used to protect wood against outdoor conditions. However, these chemicals are not effective for long-term protection on wood surface. For this reason, water repellent chemicals which should be permanent are required for protection on wood surface. In this study, Poly Ethylene Glycol (PEG 600), N, N- (1,8-Naphthalene) hydroxylamine (NHA-NA) and Methyl Hydrogen Silicone (MHS) were investigated to get insight into their effectiveness against UV radiation by recording colour changes and FTIR-ATR analysis.

2. MATERIALS AND METHODS

Sapwood samples of Scots pine free of defects and discoloration or decay were prepared with the dimensions of 1,5x7,5x15 cm (Radial x Tangential x Longitudinal) in this research.

PEG 1600 (Merck) was a clear transparent liquid at 23 °C, NHA-H ($C_{12}H_7NO_3$) having following properties: melting point 283-286 °C, molecular weight 213,19, pale yellow colour, insoluble in water was provided from Alfa Aesar Methyl Hydrogen Methyl Hydrogen Silicon (MHS) was purchased from RUI Chem.

PEG was prepared as 20% concentration, whereas MHS was diluted with water for 5%. NHA-H was prepared with methanol at the concentration of 0.1%.

After the kiln dry weight was recorded, the wood samples were impregnated with water repellents under vacuum (30 min) and pressure (6 bar for 1 h). Afterwards, wet weight of the samples was done, then left to drying at 80 °C for 2 days. Weight percent gain (WPG%) values of the samples was calculated according to equation 1.

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 $WPG(\%) = ((My-M_0)/M_0)*100$

2.1. Outdoor weathering

The outdoor weathering test was carried out for 6 and 12 months in the province of Bartin which is located 41° 38' 4" N, 32° 20' 15" E in the Western Black Sea region.



Figure 1. Weathering rack in Bartin province

2.2. Accelerated weathering

Accelerated weathering test was carried out in the device of Q-Panel Lab Products, in the laboratory of the Industrial Forest Engineering department of Bartin University. Test parameters in the device are as follows; water spray for 15 min, UV (340 nm, wavelength) at 0,85 W/m² for 8 h and conditioning at 50 °C for 3.45 h. Overall, this cycle was operated for 500 h totally (ASTM-G53, 1998).

2.3. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR, Shimadzu) instrument was used to measure the chemical analysis of the test samples before outdoor weathering. In accordance with the FTIR device, ATR Diamond/ZnSe kit (PIKE) was used to make solid measurements on the surface without causing any damage in the samples. The spectra of the samples were obtained at 700-4000 cm⁻¹ wavelength, with a resolution of 4 cm⁻¹ with 30 scans.

2.4. Colour measurement

Colour measurement of the test samples before and after the outdoor and accelerated weathering were carried out in accordance with ISO 7724 standards by Konica Minolta CD-600 colour meter. On the wood samples, the colour measurements from 3 different points were measured and their mean value were calculated for three replicates in each variation (ISO-7724, 1984).

The CIELab (Commission Internationale de l'Eclairage) system consists of three variants (ISO 7724). L* refers to Light stability, a* and b* chromatographic coordinates (+a* indicates red, -a* green, +b* yellow, -b* blue). The values of L*, a* and b* were measured on the samples and the color changes were determined according to the following formulas

	0	0	0	
$\Delta L^*=Lf^*-Li^*$				(3)
∆a*=af*-ai*				(4)
$\Delta b^* = bf^* - bi^*$				(5)
$\Delta E^* = (\Delta L^{*2} + \Delta a)$	$a^{*2} + \Delta b^{*2})^{1/2}$			(6)

3. RESULTS AND DISCUSSION

The weight percent gain (WPG) values in the wood samples after impregnation were given in *Table 1*. The WPG results of the samples exposed to accelerated and natural weathering respectively were 134.68%-130.99% with PEG impregnation, 115.68%-111.91% with MHS and 62.15-60.64% with NHA.

Table 1. Weight percent gain (WPG %) of the samples

	Accelerated weathering	Outdoor weathering
PEG	134,68 (4,64)	130,99 (7,36)
MHS	115,68 (5,49)	111,91 (12,95)
NHA	62,15 (3,92)	60,64 (18,81)

*Values in parenthesis indicate the standard deviation

Under outdoor conditions, UV light plays the most important role in the degradation of organic material with the energy of UV rays by breaking the chemical bonds in the wood. This shortens the using period of the samples in service.

The results regarding colour change after the accelerated weathering were given in *Figure* 2, and after outdoor weathering in *Figures 3 and 4*.



Figure 2. Colour change after accelerated weathering (ΔL^* - first set of columns, Δa^* - second set of columns, Δb^* - third set of columns, ΔE^* - fourth set of columns)



Figure 3. Colour change after six months outdoor weathering (ΔL^* - first set of columns, Δa^* - second set of columns, ΔE^* - third set of columns, ΔE^* - fourth set of columns)



Figure 4. Colour change after twelve months outdoor weathering (ΔL^* - first set of columns, Δa^* - second set of columns, Δb^* - third set of columns, ΔE^* - fourth set of columns)

When ΔL^* light stability was examined, all samples including treated and untreated showed a tendency to negative direction. The darkening was observed on the surface of the samples exposed to both accelerated and outdoor weathering.

Besides, increasing darkening on the surface accompanied with the increase in the duration of outdoor weathering. In contrast to outdoor weathering lower ΔL^* values were obtained by accelerated weathering.

The light stability gave better results in test samples than control. It means that both accelerated and outdoor weathering test samples have lower ΔL^* values compared to the control samples. The samples impregnated with MHS were found to be the most stable with respect to light stability in the accelerated test, whereas PEG was the best in outdoor weathering involving both 6 and 12 months.

When Δa^* (red-green) values were examined, tendency of a^* value was positive in the accelerated test, whereas a^* was negative in the outdoor test. In the case of accelerated aging test, it was considered that there was a tendency towards red colour on the surface of the samples due to the UV effect.

In the outdoor weathering test, the mold formation induced to green colour on the samples surfaces within 6 months. When the samples exposed to 12 months, the trend towards green colour was more apparent as the mold growth increased on the surface over time. The least change was in samples impregnated with MHS, but the most change was in samples impregnated with NHA-H in terms of colour.

The colour of all samples tended to be negative in Δb^* (yellow-blue) colour scale. In the accelerated test, the control samples showed a tendency towards the yellow colour, while tended to blue colour in the outdoor test. In the outdoor test, the trend towards blue colour increased with the increasing in time.

When the total colour change (ΔE^*) in the samples was examined, there was less colour change in the test samples than in the control or equal to the control. The highest colour change was observed with control samples in the accelerated test, but with NHA-H in the outdoor weathering test.

However, the almost same colour change values were obtained in all variations. The total colour change values obtained in the samples exposed to the accelerated test were less than those in the outdoor weathering test. The reason is that the accelerated aging test applied for 500 hours might correspond to less than 6 months compared to the outdoor weathering test, or the light intensity (0.85W/m2) applied in the accelerated test might be lower than outdoor. This means that the 500-hour accelerated aging test is equivalent to less than 6 months in the outdoor

weathering. It is thought that the depolymerization of lignin and other compounds by UV effect in aging takes place and the characteristic colour changes are caused by the o- and p-quinonoid units formed by the lignin fragmentation (Feist and Hon, 1984; Hon and Shiraishi, 2001). The lowering of colour change resulted from the chemicals in the accelerated weathering test can be explained by the modification of lignin and other substance (Castellan and Davidson, 1994).

Changes in colour that we observe on the surface are the consequence of chemical changes. *Figures 4-8* show chemical changes of the wood surface analyzed by the FTIR method.

In the present study, outstanding peaks at 900, 1025, 1030 and 1050 cm⁻¹ were originated by the modification of chemical bonds such as O-H, C-H and C-O-C being in cellulose due to the outdoor weathering, and their relationship with polysaccharides was mentioned in the literature (Stark *et al.*, 2004; Stark and Matuana; 2007; Zhang *et al.*, 2009; Scholz *et al.*, 2010; Castro *et al.*, 2011).

These peak values showed a decrease in the control samples. In the impregnated samples, there was an increase in the peak values after the accelerated weathering, whereas it was decreased after outdoor weathering except for MHS. In the samples impregnated with PEG, three different peaks were formed by dividing the peak at 1114 cm⁻¹. The changes occurring at 1230-1267 cm⁻¹ absorption peaks are directly related to lignin delignification and modification of aromatic rings. The change in the peaks at 1230-1270 cm⁻¹ band shows the CO tension in lignin and hemicellulose and vibration in the guaiacyl ring.

The disappearance of the peak in this band gap is an indication of delignification and degradation of the aromatic rings. There was a decrease in the peaks in all variations except MHS after the outdoor weathering, on the other hand, these peak values increased after accelerated weathering.



Figure 5. FTIR spectrum of control samples exposed to accelerated and outdoor weathering

It was reported that the peak of 1630-1660 cm⁻¹ was related with of -C=O bonded aryl ketones in lignin which was associated with the unbound C = C and water band as well (Temiz *et al.*, 2006). The present study also revealed that the control samples showed an increase in the peak after the accelerated aging, but these peak values decreased after the outdoor weathering. In addition, the effect of UV light on the lignin was confirmed in this research. After the aging test, these peak values decreased and then disappeared.



Figure 6. FTIR spectrum of the samples treated with PEG after accelerated and outdoor weathering



Figure 7. FTIR spectrum of the samples treated with MHS after accelerated and outdoor weathering

New peaks were observed at 2162 cm⁻¹ in the samples treated with MHS. This is due to the structure of the chemical used in the experiments, not related with the weathering. In the samples impregnated with PEG, peak formation was observed at this point after the outdoor weathering. The 2161 cm⁻¹ bond which is extremely strong and shows the -S-C=N bond in the aromatic rings (Socrates, 2004; Hesse *et al.*, 2005).



Figure 8. FTIR spectrum of the samples treated with NHA-H after accelerated and outdoor weathering

The peak at 2916 cm⁻¹ refers the C-H bond in methyl and methylene groups. There was no change in the samples impregnated with MHS, while increasing with NHA and control, and decreasing with PEG after accelerated weathering. After the outdoor weathering test, decreasing occurred in the samples treated with control and NHA and then disappeared (Socrates, 2004; Hesse *et al.*, 2005).

4. CONCLUSION

MHS was found to be effective in reducing the total colour changes. Colour changes of the samples exposed to outdoor weathering were similar or higher then the control samples. At the end of 12 months, the highest colour change was obtained in the samples impregnated with NHA-H. This might be due to the lower concentration or UV blocking behaviour of NHA-H.

According to the results of chemical analysis, the most effective chemical was MHS, because there was no change in chemical structure of wood both in accelerated and in outdoor weathering test. As a result, lignin which is responsible for the colour change in wood was blocked by MHS, resulted in the protection to the UV rays.

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