

Department of Materials and Environmental Technology

# IMPREGNATED WOOD PERFORMANCE IN OUTDOOR ENVIRONMENT

IMMUTATUD PUIDU VASTUPIDAVUS VÄLISKESKKONNAS

MASTER THESIS

Student Peeter Tubli

Student code 132779KVEMM

Supervisors Heikko Kallakas, Jaan Kers

Tallinn 2017

### AUTHOR'S DECLARATION

Hereby I declare, that I have written this thesis independently.

No academic degree has been applied for based on this material.

All works, major viewpoints and data of the other authors used in this thesis have been referenced.

Author: Peeter Tubli.....

/signature /

Thesis is in accordance with terms and requirements

Supervisors: Heikko Kallakas...../Jaan Kers.....

/signature/

Accepted for defence

Chairman of theses defence commission: .....

/name and signature/

## Table of contents

List of Figures	5
List of Tables	7
Introduction	8
1 Literature overview	9
1.1 Wood performance in outdoor environment	10
1.2 Impregnation of wood	12
1.3 Impregnation impact on wood properties	
1.4 Impregnated wood performance in outdoor environment	19
2 Materials and methods	24
2.1 Materials	24
2.1.1 Preservative Impralit KDS	24
2.1.2 Impregnated Spruce specimens	25
2.1.3 Performance table	
2.1.4 Fasteners	
2.2 Methods	
2.2.1 COST Action FP 1303 – Cooperative Performance Test	
2.2.2 Exposure conditions	29
2.2.3 Decay	
2.2.4 Discoloration	
2.2.5 Surface disfigurement due to mold and staining fungi	
2.2.6 Corrosion	
2.2.7 Crack formation	
2.2.8 Moisture content and temperature	
2.2.9 Color change under UV irradiation	

2.2.10 Swelling test and water uptake
3 Results and discussion
3.1.1 Decay
3.1.2 Discoloration
3.1.3 Surface disfigurement due to mold and staining fungi
3.1.4 Corrosion
3.1.5 Crack formation
3.1.6 Moisture content and temperature
3.1.7 Color change under UV irradiation
3.1.8 Swelling test and water uptake
Conclusion
References
Acknowledgements71
Abstract
Kokkuvõte73

## List of Figures

Figure 1. Stressing factors, influencing factors and weathering effects that contribute to
wood/finish performance [1]12
Figure 2. Bethell process description [4]14
Figure 3. AS Imprest products exiting from auroclave after impregnation [9]17
Figure 4. Folding table for performance test [20]28
Figure 5. Performance table set up at the exposure site on 8 <sup>th</sup> of April 201631
Figure 6. North side surroundings of the performance test table
Figure 7. South side surroundings of the performance test table
Figure 8. East side surroundings of the performance test table
Figure 9. West side surroundings of the performance test table
Figure 10. Placement of specimens, screws and color measurement points on the performance table
Figure 11. Position for measuring L*a*b color values [20] and measuring points on the specimens
Figure 12. Crack measuring gauge [20]
Figure 13. Isolated fasteners were used for fixing electrodes on wood specimens38
Figure 14. Thermofox datalogger and gigamodule [20]
Figure 15. Spectrum of the UVA-351 type fluorescent lamp (PHILIPS TL-D 36W/08 T8 blacklight) [26]
Figure 16. Under (left) and top (right) of the specimens on 5 <sup>th</sup> of May 2017 for evaluation decay of the specimens
Figure 17. Topside of performance table on 8 <sup>th</sup> of April 2016 (left) and on 5 <sup>th</sup> of May 2017 (right)
Figure 18. Total color change of the specimen groups from 8 <sup>th</sup> of April 2016 until 5 <sup>th</sup> of May 2017

Figure 19. Color change of every specimen and average color change by specimen group from 8 <sup>th</sup> of April 2016 until 5 <sup>th</sup> of May 201746
Figure 20.Total color change of specimens alongside with UV-index values from 8 <sup>th</sup> of April 2016 until 5 <sup>th</sup> of May 2017
Figure 21. Performance table on 9 <sup>th</sup> of November 2016
Figure 22. Galvanized screws removed from performance table on 29 <sup>th</sup> of April 2016 for inspection
Figure 23. Galvanized screws removed from performance table on 5 <sup>th</sup> of May 2017 for inspection
Figure 24. Total crack length and moisture contents of specimen groups
Figure 25. Number of cracks and moisture contents of the specimen groups
Figure 26. Mean crack width and moisture contents of specimen groups
Figure 27. Wood and air moisture content from 8 <sup>th</sup> of April 2016 until 21 <sup>st</sup> of October 2016
Figure 28. Wood and air moisture content from 21 <sup>st</sup> of October 2016 until 5 <sup>th</sup> of May 2017
Figure 29. Test specimens before (left) and after (right) 336 hours of artificial UV irradiation
Figure 30. Color change of specimens after UV irradiation of 336 hours61
Figure 31. Submerged swelling test specimens
Figure 32. Average water uptake during swelling test
Figure 33. Longitudial, radial and tangential percentage swelling of the laboratory specimens provided by AS Imprest during the swelling test

## List of Tables

Table 1. Information about the specimens provided by Imprest AS for	.26
Table 2. Information about impregnation cycles of specimens	.27
Table 3. Information about impregnation cycles of specimens	.27
Table 4. Assessment schedule for performance test [20]	.29
Table 5. Decay rating scale [20; 21]	.34
Table 6. Fungal disfigurement rating scale [20]	.36
Table 7. Assessment of corrosion attack [24]	.36
Table 8. Initial color measurements of the specimens	.44
Table 9. Initial weight of the fasteners used in the performance test	.50
Table 10. Evaluation values of corrosion attack	.51
Table 11. Results of crack formation measurements from 8 <sup>th</sup> of April 2016 until 5 <sup>th</sup>	<sup>h</sup> of
May 2017	.53
Table 12. Results of initial color measurements before UV irradiation	.60
Table 13. Moisture contents before and after the UV irradiation	.61
Table 14. Average water uptake of the laboratory specimens	.63
Table 15. Longitudial, radial and tangential percentage	.64

## Introduction

Wood is natural material that has been highly valued for its versatility and for its durability and structural properties. Therefore, it has been attractive material for engineering. Wood can maintain its protective qualities for very long time if favorable environment and sufficient service are provided. Wood is a biological material and thus a perishable material. This drawback has often considered of being its greatest disadvantages if compared to other building materials. Wood preservation can be interpreted to mean protection from fire, chemical degradation, mechanical wear, weathering, as well as biological attack.

This master thesis is inspired of a COST Action FP 1303 "Performance of bio-based building materials" which has successfully started in October 2013. A Cooperative Performance Test has been organized in the frame of this action as decided during the first workshop in Paris. The idea was to distribute a fairly simple test set up among as many places in Europe as possible to collect performance data under the full range of climatic conditions to be expected.

Performance data during outdoor exposure conditions is gathered, weather data from respective test site is required to establish relationships between climate conditions and the measurands. Wood decay, discoloration, development of mold and other staining fungi, corrosion of screws, formation of cracks and moisture performance are to be measured in this test and the results are compared to see the difference between different test specimens.

The aim of this master's thesis was to gather and to examine the performance data of Norway spruce specimens with different impregnation cycles in outdoor environment. This thesis is divided into three parts. The first part of the thesis is literature overview about the topic and relevant researches. The second part of the thesis consist of descriptions of used materials and methods and in the third part, research results are presented and discussed.

## **1** Literature overview

Wood is natural material that has been evaluated for its versatility and it has been attractive material for engineering for its durability and structural properties [1]. In a favorable environment and with sufficient service wood can maintain its protective qualities for many centuries.

Exposed wood in outdoor environment, as a biological material, is prone to environmental degradation as a complex combination of chemical, mechanical and ultraviolet (UV) radiation factors contribute to process described as weathering. [1] Weathering can be unacceptable in certain application thus the weathering has to be taken into consideration when outdoor wood and wood structures preservation is an issue. Wood decay on the other hand needs excessive moisture and air for prolonged period of time and is a result of living organisms known as fungi and it is not consider as a wood weathering [1]. Wood decay can damage wood rapidly if suitable conditions are granted and the results are very different from natural outdoor weathering.

Wood used in indoors can be protected for extensive time periods as wood properties does not affect indoor finishes as greatly as they do outdoors. Outdoor finishes durability depends significantly on the wood properties and some wood finishes can endure only few years in power of UV irradiation and water exposure. Moisture content, density and texture, resin and oil content, width and orientation of growth rings, and defects such as knots, reaction wood and diseased wood are considered as important aspects affecting durability of finishing. Also nature and quality of used finish, technique, pre-treatment and time between refinishing are important factors for durability of finish alongside with weather conditions and if the surface is sheltered or not. [1]

The primary function of any outdoor wood finish is to protect the wood surface from weathering elements and to ensure desired appearance. If appearance is not important wood can be used also unfinished and weather naturally and that wood will often offer enough protection needed. [1] Different finishes give varying degrees of protection from the weather. Generally, the greater the pigment concentration, the greater the protection; paints give the most protection, transparent varnishes the least.

The weather resistance of the bonding agents of the finish will affect any protection that surface treatments provide against light and water. These bonding agents are subject to photo degradation to some extent. [1] Wood exposed outdoors can be protected from the effects of weather with different finishes, design and engineering solutions.

#### 1.1 Wood performance in outdoor environment

The sensitivity of the wood to degradation is one of its greatest weaknesses in outdoor usage. The factors that are generally considered to cause changes in wood surfaces on weathering are sunlight (UV, visible and infrared radiation), moisture (dew, rain, snow), temperature and oxygen. [2] Because of the limited ability of light to penetrate into wood, the effect of the weathering is limited to a 2.5 mm thick surface layer and the erosion is slow, 5-12 mm per 100 years [2].

First sign of the weathering of untreated wood during outdoor exposure is color change of the wood surface due to visible light and UV irradiation which alters the color depending on the specific type of wood. Although after a long period of outdoor use, all types of wood develop a greyish appearance, as water-soluble decomposition products are removed and the more or less delignified fibres are exposed [2; 3].

The photochemical degradation is a very slow process which during a decade degrades only a few millimetres of the wood surface and leaves the underlying wood practically unaffected. The combined effect of water and sunlight degrades the main components of the wood and transforms the wood surface into a network of weakly connected cellulose fibrils which are strongly contaminated by spores from microorganisms. [2]

Micro cracks are forming during the drying of the wood, photochemical reactions or moisture induced stress fields which during outdoor exposure grow into visible cracks on the wood surface. Wood for outdoor use should have vertical annual rings, which should minimize the risk of cracks because of anisotropic moisture movements. [2; 3] Cracks in the radial surface are also smaller than in the corresponding tangential surfaces [2].

Original smooth wood surfaces become rough during the outdoor weathering as the grain raises and checks grow into large cracks. Also wood can change as the grain loosens, boards cup, warp and pull away from fasteners, the tainted surface lose its appearance as it lose its color and starts gathering dirt, also mold may affect the desired

appearance. Completeness of wood disappears and splinters and fragments may break off the surface and all this is the result of natural weathering. [1]

Cracking of wood surface is affected greatly on annual ring orientation and to avoid cracks occurring on wood used outdoors, annual ring orientation should be perpendicular to the exposed wood surface. This becomes especially important when wood is used outdoors without any finish or the surface is exposed to strong wind and weather. [2] Density of the wood nor type of wood is not with such importance than annual ring orientation in the wood surface exposed oudoors without a finish when considering crack development of pine and spruce wood. Impregantion treatment with a chromate copper arsenate (CCA) - agent or surface treatment with linseed oil has only marginal influence on the crack formation. [1; 2]

On a microscopic level, it is also possible to see clear differences in degradation between radial and tangential surfaces. Tangential surfaces have more and deeper cracks than radial surfaces. The cracks on the tangential surfaces occur frequently in both early and latewood. On radial surfaces, cracks occur primarily at the annual ring border, but to a certain extent also in the earlywood. On an ultrastructural level, decomposition of the pits is the clearest difference between radial and tangential surfaces. In both radial and tangential surfaces, degradation of the cell wall takes place. Cracks arise which follow the fibril orientation in the S2 cell wall layer. Delamination in the middle lamella is especially clear in the latewood on tangential surfaces, but it also occurs in the earlywood. The microfibrils are the parts of the tracheid which are most resistant to weathering. [1 - 3]

There is two main types of treatments being used to protect wood surfaces during outdoor exposure: firstly those finishes that form a film, layer, or coating on the wood surface, and secondly those finishes which penetrate the wood surface and leave no distinct layer or coating on the surface. [1] Stressing factors, influence factors and weathering effects that contribute to wood-finish performance and weathering, are summarized in Figure 1.

#### Stressing elements



Weathering effects

Figure 1. Stressing factors, influencing factors and weathering effects that contribute to wood/finish performance [1]

#### **1.2 Impregnation of wood**

Wood preservation can be interpreted to mean protection from fire, chemical degradation, mechanical wear, weathering, as well as biological attack. Wood is biological material and thus a perishable material and the non-durability of it has been often considered of being its greatest disadvantages when compared to other building materials. The premature degradation of solid timber and wood-based composite products is very costly but it can be avoided. The first important aspect is the right use of construction techniques that minimize the exposure of wood to conditions that favor weathering. Where such constructions are not practical, possible or cost effective, wood preservation techniques can prolong the service life of wood. [3]

Wood impregnation with preservative chemicals and impregnated wood has been criticized due to health and environmental concerns although health and safety concerns are constantly being relieved as less toxic chemicals are taken into use. Extended durability of impregnated wood products balances environmental concerns as it allows conservation of forest resources. Although there is different construction materials capable of substituting wood in many applications, these materials are generally more expensive and require more energy to produce. [3]

Heartwood of many tree species exhibits some resistance to attack by decay fungi and insects. This natural durability can be attributed to a combination of toxic extractives present in the wood and low inherent permeability. Sometime naturally durable wood is being considered as more environment friendly and preferable to chemically treated wood. As some of these species have an attractive appearance and excellent strength properties, these attributes have led to increasing interest of using these naturally durable species from tropical countries for construction North America and Europe. Growing stock of these naturally durable species is rather low compared to the demand for durable wood products, this aspect limits the use of naturally durable species and exporting these species raises concerns about exploitation of developing countries. [3]

Historically chromate copper arsenate (CCA) has been mostly used for impregnating wood products. CCA is a mixture of chromic acid, cupric oxide and arsenic pentoxide. Advantages of CCA are strong fixation to wood and excellent protection in different environments. Main drawback of CCA is considerable human health concerns associated with arsenic and hexavalent chromium, which are both considered carcinogenic and CCA is not available for use in many countries due to this reason. In Europe, the production of CCA has been prohibited since September 2006. Chromium and arsenic free alternatives have been developed which rely still on copper as primary biocide alongside with other components to replace chrome and arsenic. Nowadays even copper is under supervision considering environmental aspects and there is noteworthy interest for developing wood preservatives that do not contain copper or other heavy metals. [3; 4]

As most impregnation chemicals are classified toxic and dangerous to the environment because they are not or only slowly biodegradable. The preservation solution consists of the preservative itself, but also of anti-mold agents, anti-slime build-up agents and coloring agents. All of these chemicals, except coloring agents, are toxic and dangerous to the environment. Therefore replacing hazardous chemicals with less hazardous ones when technically and economically possible is a key target in the wood impregnation industry. Wood preservation chemicals can be divided into water-borne, oil-borne and organic solvent-based preservatives. During the last decade, waterborne preservatives have become more widely used. Today the most commonly used preservatives are copper compounds (typically containing ammoniacal copper quaternary compounds or copper azole, whereas sometimes also other copper compounds). Some of the preservatives also contain chromium, boric acid and/or water-based micro emulsions such as azoles or quaternary ammonium compounds. The advantages of water-borne preservatives include dry and paintable surface of treated wood and no odour. [4]

The most typical wood preservation techniques in use in the Nordic countries are the Bethell process and its modifications used mainly in impregnation with metal oxides. It is a treatment process where an aqueous solution of impregnant is applied using a vacuum and pressure cycle. The process is also called the full-cell method in which the purpose is to fill the wood cells with the impregnation agent in order to receive the highest possible amount of impregnant in the wood. The penetration of the impregnation agent is based on the vacuum created before the process. The Bethell process begins with a preliminary vacuum, which creates a vacuum inside the wood also. The vacuum is maintained and injection of the impregnation agent begins. When the pressure cylinder is full of the impregnation agent the vacuum is removed and overpressure is created. The overpressure makes the impregnation agent penetrate the wood. The time of overpressure varies from minutes to hours. After the overpressure period, normal pressure is restored. Finally a short vacuum is created to remove all excess impregnation agents from the wood. This prevents dripping when the wood packages are stored. The process is illustrated in Figure 2. [4]



Figure 2. Bethell process description [4]

Norway spruce (*Picea abies*) is considered one of the most important European wood species, as it has excellent ratio between mechanical properties and density which is the reason why it is continuously used in construction applications. However, spruce wood is classified as less durable material thus it needs impregnation with wood preservatives if used for load-bearing outdoor applications. This brings us to next problem with Norway spruce, which is the fact that it is extremely hard to impregnate. Proper

selection of preservative solution, proper selection of impregnation procedure and proper treatment of wood prior impregnation help overcome the issue of hard impregnability. [5; 6]

One solution for improving impregnability of spruce is incising which increases the amount of transverse area exposed to potential preservative flow. The longitudinal pathways exposed on these transverse faces are far more receptive to preservative flow than are the radial or tangential surfaces. Therefore incising improves preservative penetration to the depth of the incision. Mechanical incising results in damaged surface of the treated wood as deteriorated visual appearance and deteriorated mechanical properties. The remaining holes in the wood after incising are also water traps and represent potential pathway for fungal colonization. [5]

In order to improve impregnability of the refractory wood species, wood swelling agents (WSA) are combined with a commercial copper-ethanolamine (Cu-EA) based preservative solution. The purpose of those chemicals is to swell wood and thus enable better penetration of preservative solutions to wood. [5]

It has been reported that the maximum swelling of wood is mainly influenced by three wood swelling agents properties, the solvent basicity, the molar volume and the hydrogen bonding capability and it is believed that the cellulose is the major polymer responsible for wood swelling. During swelling, the voids in the wood structure opens, and this mechanism enables better penetration of wood preservatives to wood. New voids are new capillaries, which improves liquid penetration to wood by capillarity mechanism. Water itself is wood swelling agent, therefore WSA agents that causes more prominent swelling than water shall be used. The purpose of WSA is not to reopen pits but to increase capillarity of the wood. [5]

It has been reported that some wood swelling agents like ammonia, formic acid and triethanolamine have positive impact on the preservative uptake to vacuum-pressure impregnated spruce wood, while other agents like dimethyl sulfoxide and ethylene glycol have negative impact on the uptake. The highest uptake was determined at spruce wood specimens impregnated with Cu-EA in combination with 10 % of formic acid and 2.5 % of triethanolamine. There is tight correlation determined between penetration and uptake of preservative solution at vacuum-pressure treated spruce wood. It is reported that best penetration has determined at spruce wood impregnated with Cu-EA in combination with Cu-EA in combination with triethanolamine and formic acid. [5]

Wood swelling agents influence copper fixation, some have positive aspect on the fixation like ammonia or certain concentrations of DMSO. Addition of formic acid, ethylene glycol and triethanolamine considerably increased copper leaching from impregnated spruce wood. The highest leaching has been determined at spruce wood impregnated with combination of formic acid or triethanolamine which had an average around 30 % of retained copper was leached. [5]

The applicable EU standards (335-1, 351-1 and 599-1) provide guidance on use of impregnating agents for solid timber and wood-based products in defined situations. The standards define five Use Classes that shall be used as the basis for specifying preservative treatments for particular products in order to guarantee the durability of the products in the defined situations:

- Class 1 is to be used in a situation in which the wood or wood-based product is under cover, not exposed to the weather and soaking.
- Class 2 is to be used in a situation in which the wood or wood-based product is under cover and not exposed to the weather but where high environmental humidity can lead to occasional, but not persistent wetting.
- Class 3 is to be used in a situation in which the wood or wood-based product is not under cover and not in contact with the ground. It is either continually exposed to the weather or is protected from the weather but subject to wetting.
- Class 4 is to be used in a situation in which the wood or wood-based product is in contact with the ground or fresh water and thus is permanently exposed to wetting.
- Class 5 is to be used in a situation in which the wood or wood-based product is permanently exposed to salt water. [4; 7; 8]

The European wood preserving industry produces around 6.5 million m<sup>3</sup> of pressuretreated wood per year. 44 % of the production is used as garden timber, 21 % as construction timber, 15 % as small roundwood and 6 % as sleepers. 71 % of this wood is treated with water-borne products, 11 % with creosote, mainly poles and sleepers and 18 % with solvent based products, mainly construction timber such as window and door joinery. [4]

The Nordic wood preserving industry produced around 2.1 million m<sup>3</sup> of pressuretreated wood in 2012. This amount comprises about one third of the total European supply of pressure-treated wood. In the Nordic countries, Sweden is the biggest producer of pressure-treated wood, followed by Norway, Finland and Denmark. The highest season in sales and use is typically from March to August, whereas in the other seasons the plants produce varying amounts to stock and to export. In Sweden about 92 %, in Norway about 88 % and in Finland about 76 % of industrially pressure-treated wood is sawn and planed timber, which is significantly more than elsewhere in Europe. Poles comprise about 21 % in Finland, but in Sweden and Norway only about 3 % of industrially impregnated wood. Similarly as elsewhere in Europe, wood is mostly treated with waterborne preservatives also in the Nordic countries. [4]

In the Baltic countries AS Imprest is the leading manufacturer of machine rounded timber products. They started operating in the year 2000 and today they export 93% of their output to 24 European countries and more than 250 trade customers. On Estonian market they supply all major retail chains with gardening and construction materials. All Imprest AS products are treated using a vacuum-pressure-vacuum method and they also offer timber treatment as a separate service. They have two treatment tanks (one treatment tank is presented on Figure 3) and they use water-borne, chromium free wood preservative – Impralit-KDS. Impralit-KDS ensures the necessary protection depending on the hazard class. For different products, markets and customers they are using different treatment processes but the majority of their timber is being treated according to European Standard EN 351-1 to meet the use classes described earlier. [9]



Figure 3. AS Imprest products exiting from auroclave after impregnation [9]

#### **1.3 Impregnation impact on wood properties**

When used correctly, no exposure to biological or chemical hazards, most timbers will maintain its integrity beyond the planned lifetime of almost any structure as deterioration of wood due to age is almost non-existent. Sadly in real world where biological and chemical hazards exist, treatment with suitable preservative is essential for both sapwood and heartwood. As described earlier, with refractory species, as spruce, it is often very difficult to obtain more than about 3-6 mm lateral penetration in sapwood, which is known to be several times more permeablt than heartwood. This is essential with natural rounds where well-treated wood is very important. [10]

Obtaining satisfactory treatment of refractory species of wood is a problem the timber preservation industry is facing. The amount of moisture in wood has a profound influence on the quality of preservative treatments and on this basis the need to dry before impregnation is well known, but there is almost no information on optimum moisture levels for individual wood species. [10]

It has been shown that the drying process significantly improves the percentage of void volume filled with preservative in the case of porosity related to wood density at respective moisture contents. For example Caucasian fir requires drying to below the fiber saturation point (FSP) to allow the most adequate preservative uptake. [10]

Studies have shown that the effect of moisture content confirm that the degree of increase in mean preservative uptake due to treatment varies among the moisture content levels. The experimental result show that preservative uptake is limited to the level of moisture content in the amount of void volume in wood, thus improvement in the maximum volume of preservative which could be absorbed by wood would be achieved by reducing the moisture content due to drying. The experimental studies also indicate that moisture contents below the fiber saturation point result in greater preservative uptake although drying to a very low moisture content does not appear to improve impregnation and is probably uneconomical. Therefore the moisture content of refractory wood species before impregnation with full-cell treatment process should be below the fiber saturation point for adequate preservative uptake which may also influence the depth penetration. [10]

Mechanical properties of treated wood depend mainly on the properties of the wood and treatment does not cause any drastic changes to the mechanical properties. The cross-

linking between the agent and wood improves several of the strength properties of wood, excluding the impact strength. The impact strength decreases after treatment, because cross-linking between cell wall and agent reduces the mobility of the cell wall components. Waterborne solutions tend to cause loss in wood strength, because treatment with aqueous solutions increases the rate of hydrolysis in the wood. Metallic oxides are widely used in waterborne preservatives and they react with cell wall components, thereby reducing the strength of wood that is basically known as fixation. [11]

Lignin modification has a significant role in weathering as it is considered the most important wood component in weathering as it is very sensitive to ultraviolet light. Different modifiers can protect wood from weathering but chemical treatment seems to be the most suitable solution. Water resistance of wood improves remarkably when the preservative is cross linked by a double bond with wood or when it has blocked the hydroxyl groups in wood. The dimensional stability has been improved by artificial swelling when there has not been space anymore for water in wood cells. The dimensional stability can also improve the chemical reaction between the OH groups of cellulose and the OH groups of solution. Lower moisture content is considered to improve wood properties in many aspects, for example, it improves the dimensional stability of wood and resistance against fungi. Chemical treatment can change the structure of wood cells, which is the most important aspect of reducing moisture in wood and thus the chemical treatment of wood is the best way to control and stabilize the moisture content in wood. [11]

#### 1.4 Impregnated wood performance in outdoor environment

Studies have shown that spruce wood treated with preservative solution based on boric acid and quaternary ammonium compounds has its lifecycle prolonged only for a few years, but after leaching of the boric based ingredients from wood and potential bacterial degradation of quaternary ammonium compounds, decay started. Same regularity appeared when copper was leached out from wood treated with copper sulphate. Wood is protected from decay and fungi until there is at least low copper contents present in wood but when copper is leached the decay proceeds. Studies have shown that lower concentration of copper does not prevent but even promote fungal growth and even if copper is combined with ethanolamine, it does not ensure complete protection if retention is too low (retention = 2,1 kg/m3 of CuEa or 1,3 kg/m3 of CuEaO). Also if wood protection with copper ethanolamine preservatives is desired copper should be combined with additional co-biocides like quaternary ammonium compounds in order to assure protection. With good combination of biocides the preservative gives adequate protection to wood even at low solution concentrations. In use class 3 usually service life of Norway spruce is between 4 and 6 years but if leachable biocides are used or if biocides in too low retentions are applied, decay will not be prevented but only delayed for 1 to 3 years. [12]

Copper-tolerant wood-decay fungi express an ability to degrade copper treated wood this ability is associated primarily with oxalic acid excretion. Oxalic acid produced by copper-tolerant fungi reacts with copper in wood to form insoluble, and therefore bio-unavailable, inert forms. Excreted acids react with copper, but these organisms also depolymerize cellulose, hemicelluloses and lignin, thus there is matter of mechanical properties of wood impregnated with copper, which is decayed by copper-tolerant wood decay fungi and how the acids influence the properties. Studies have shown that spruce wood impregnated with copper-chromium based preservative prevented decay by copper tolerant fungi (*A. Vaillantii*), while impregnation of spruce wood with copper-ethanolamine did not. [13]

Color change of wood surface is one of the first and clearest sign of weathering of the wood during outdoor exposure. Visible light and UV-radiation change the color of the wood to a darker or lighter shade, depending on the type of wood. All types of untreated wood develop a greyish appearance after long outdoor use due to the fact that water-soluble decomposition products are removed and the more or less delignified fibers are exposed. [2]

Wood is affected by direct or indirect solar radiation in several outdoor and indoor enduses. The chemical transformation of a compound into smaller compounds caused by the absorption of ultraviolet, visible, or infrared radiation is called photodegradation. When studying the ultraviolet-visible light-infrared part of the spectrum it is the UV part (wavelength approx. 1–400nm) that mainly affects the color of wood. The shortest wavelengths of the UV part of spectrum are being absorbed in the atmosphere, causing that only the UV-A part (approx. 320–400nm) of the entire UV radiation can reach the earth's surface. This is the part of UV radiation that affects materials in outdoor uses. That part of UV radiation can be simulated, for instance, by using UVA-351 type (peak wavelength 350nm) fluorescent lamp apparatus. Impregnation with weak aqueous solutions of chromium (VI) trioxide and Fe(III) nitrate have proved ineffective in photo stabilizing the wood surface, in much the same way as the film of clear stain offers no protection from UV-light. Photochemical degradation is a very slow process which during a decade degrades only a few millimeters of the wood surface and wood material under the surface stays practically unaffected. The combined effect of water and sunlight degrades the main components of the wood and transforms the wood surface into a network of weakly connected cellulose fibrils, which are strongly contaminated by spores from microorganisms. [2; 14; 15]

Wood annual ring orientation is considered the most important aspect considering crack development during weathering and other aspects like type of wood, impregnation treatment and surface treatment are considered to have only marginal effect on the crack development. Studies have not found any relations between the density of the wood and the crack development. Tangential surfaces tend to have more cracks, the cracks are also wider and longer than corresponding radial surfaces, although tangential and radial surfaces show the same color change on the surface as a result of weathering. [2]

On the micro-level, tangential surfaces have more and deeper cracks than radial surfaces. On tangential surfaces cracks occur in both earlywood and latewood. On radial surfaces, cracks occur primarily at the annual ring borders, but to a certain extent also in the earlywood. The radial cell wall of the earlywood has a large number of pits which are degraded at an early stage. Decomposition of the cell wall takes place on both radial and tangential surfaces. Cracks arise which follow the S2 fibril orientation in the cell-wall. Delamination in the middle lamella is especially noticeable in the latewood on tangential surfaces. [2]

During weathering, wood is exposed to moisture variations, in cell walls and between cells stresses develop which lead to damage and to the propagation of existing cracks. The photochemical reactions happening in the wood surface during weathering accelerate this process, but this is not main reason for the great difference in number of cracks between radial and tangential wood surfaces. Crack length ratio between tangential and radial surfaces was studied, between exposed surfaces and non-exposed surfaces, it was established that this ratio was smaller for the exposed surfaces, which implies that the difference in crack development between radial and tangential surfaces is not only a consequence of photochemical degradation. Strong exposure to sunlight and rain means that the size and frequency of cracks increase strongly. Radial and tangential surfaces sensibility in relation to crack formation the main reason are the stresses, which arise in the wood, caused by anisotropic moisture movements of the wood material and moisture gradients between the surface of the wood. [2]

The shrinkage and swelling in the tangential direction are about twice as large as the radial moisture movement. Tangential surfaces thus move more than radial surfaces. This means that the stresses become higher in the tangential direction than in the radial direction when the moisture movement in the surface layer is limited by underlying wood which does not have the same moisture content as the wood in the surface layer. This relationship applies in the case of rapid moisture changes in the surface, e.g. when the surface is alternately exposed to rain and dried out in strong sunlight. Moisture gradients then arise between the surface area and the underlying wood material. This results in the formation of more cracks on tangential surfaces than on radial surfaces. Visible cracks arise in the wood surface during outdoor exposure because of the growth of microcracks formed during the drying of the wood, photochemical reactions or moisture-induced stress fields. Wood for outdoor use should have vertical annual rings which minimizes the risk of cracks as a consequence of anisotropic moisture movements. [2]

Corrosion of metals in wood is similar to a dry cell that generates voltage, simply because materials at a higher energy state tend to revert to materials of a lower energy state. The zinc electrode in the dry cell (anode) gives up electrons, making it the negative electrode. During a similar process within a metal fastener, an excess of electrons occurs at the anode (embedded fastener shank) and a lack of electrons is created at the cathode (fastener head). Mainly copper-based preservatives are used and primary cathode reaction in these preservatives is the reduction of cupric ions. Studies show that metals anodic to copper are more susceptible to corrosion and therefore fastener materials that are cathodic to copper should be chosen. [16]

Corrosion of embedded metal fasteners is very different from atmospheric corrosion as it is more dependent wood moisture content than relative humidity of air. Metal fasteners will not corrode without the presence of an electrolyte (water). Wood moisture content between 15% to 20% is considered the critical level where corrosion of metal fasteners starts to noticeably occur and air humidity of 60% to 75% is needed to start the process of corrosion. [16] Metals in wood will corrode if necessary moisture contents are present because most wood species are naturally acidic, even though heartwood are generally more acidic than sapwoods of the same species, it is considered to ecological way to use more heartwood to reduce corrosion. Also the permeability and density of the wood should be consider regarding corrosion, as less permeable and denser woods provide longer service life than more permeable and less dense wood. Source of wood should also be consider regarding corrosion, because it is known that logs that have been in salt water tend to corrode metal more rapidly. Almost all studies show that CCA treated wood will corrode metals faster than untreated wood – approximately 2 times more corrosive than untreated wood. [16]

## 2 Materials and methods

Methods used in current research are inspired of a COST Action FP 1303 "Performance of bio-based building materials", where fairly simple test set up was distributed among as many places in Europe as possible to collect performance data under climatically different conditions. Weather data from respective test site is used to establish relationships between climate conditions and the measurands. Wood decay, discoloration, development of mold and other staining fungi, corrosion of screws, formation of cracks and moisture performance are to be measured and evaluated in this test according to the COST Action FP 1303. In current outdoor performance test nine test specimens are used made from Norway spruce (*Picea abies*) impregnated with Impralit KDS, with three different impregnation cycles. The results of the performance test are analyzed and used to determine how different impregnation cycles change the Norway spruce wood performance and resistance to weathering.

## **2.1 Materials**

Imprest AS manufactured the specimens for the performance test made of Norway spruce and impregnated with chromium free Impralit KDS. Folding table alongside with equipment for recording specimen moisture content and temperature were obtained through COST Action FP 1303. Different fasteners are used and evaluated regarding to corrosion during the performance test.

#### 2.1.1 Preservative Impralit KDS

Impralit KDS is chromium free wood preservative for the prevention of attack by insects (including termites), soft rot and fungal decay. Impralit KDS is only to be used for treating wood according to the requirements of use class 1, 2, 3 and 4 as stipulated in EN 335-1. Impralit KDS is only suitable for vacuum-pressure impregnation and it may easily be washed out of the wood immediately after application. For the efficacy of the wood preservative it is essential that the treated wood is stored protected from weathering for at least 2 days, with temperatures under 5 °C for at least 7 days. [24]

Active ingredients of Impralit KDS are 205.3 g/kg (20.53 %) cupric carbonate – cupric hydroxide (1:1), 100.0 g/kg (10.00 %) didecylpolyoxethylammoniumborate, technical, 80.0 g/kg (8.00 %) boric acid. The wood preservative contains biocides protecting

structural timber against fungal decay and/or insect attack. Therefore, apply only if stipulated or necessary in specific cases. Misuse may also be harmful to health and to the environment. [17]

Didecyl polyxyethyl ammonium borate (DPAB), also known as Polymeric Betaine, was developed as a co-biocide for chromium-free copper based preservatives in Europe in the 1980's. Impralit KDS contains copper, DPAB and boric acid. KDS was commercialized in Europe in 1992. The Impralit KDS-B formulation is identical to KDS except no additional boric acid is added. KDS-B still has borates counter ion for DPAB. In recent years other formulations based on DPAB has been developed. One of these formulations is Impralit TSK10 which contains DPAB and Fenoxycarb. [18]

The betaine nature of DPAB presents unique properties as a wood preservative. Since DPAB exists mostly in the betaine dimer form at work solution strength, it does not behave as a typical cationic molecule and therefore does not have strong interactions with wood components during the wood treating process. Upon fixation, however, DPAB behaves as a cationic molecule and has excellent fixation properties in the treated wood. It is believed that the hydroxyl groups of DPAB also allow interaction with wood through hydrogen bonding. Typical AACs do not have hydroxyl groups and their fixation in wood does not involve hydrogen bonding. [18]

Studies strongly support the excellent fixation of DPAB in the treated wood and Impralit KDS has shown excellent efficacy in comparison with CCA. In two separate 10-year field tests in Norway and Sweden, KDS showed similar or better performance than CCA. The solution corrosion characteristics of Impralit KDS and KDS-B (no additional boric acid) have also been evaluated and both KDS formulations are non-corrosive compare to CCA. The low corrosion of KDS treated wood and KDS formulations is expectable as DPAB can be used as corrosion inhibitor for certain applications. [18; 19]

#### 2.1.2 Impregnated Spruce specimens

Imprest AS provided nine specimens made from spruce with approximate dimensions of 490x24x49mm (longitudinal, radial, tangential). Specimens were treated with Impralit KDS and three different impregnation cycles were used and information regarding the specimens was also provided, this information is presented in Table 1 and information about the impregnation cycles is presented in Table 2. For evaluating the amount of

preservative in wood, specimens were measured by the company before and after the impregnation.

Specimens made from board number 1 are made with rather long impregnation cycle and the amount of preservative is about 10 times more than required by the preservative manufacturer and theoretically these specimens should be the most resistant to outdoor environment. Specimens made from board number 2 dried lighter than they were before impregnation, so it is very hard to evaluate the amount of preservative in the wood. As the cycle of impregnation was also short it is possible that very little amount of preservative was forced inside the wood. Specimens made from board number 3 are similar to the Imprest AS products and amount of preservative is same as usually used by the company.

Number of specimen	Moisture content; %	Weight; g	Weight after impregnation; g	Impregnants; kg	Impregnants in wood; kg/m3
1/1	26.7	322.0	400.0	0.078	127.3
1/2	28.4	314.0	400.0	0.086	140.4
1/3	24.5	314.0	390.0	0.076	124.1
2/1	34.0	376.0	370.0	-0.006	-9.8
2/2	39.8	480.0	472.0	-0.008	-13.1
2/3	38.7	376.0	396.0	0.020	32.7
3/1	36.8	338.0	344.0	0.006	9.8
3/2	35.0	336.0	350.0	0.014	22.9
3/3	34.0	328.0	334.0	0.006	9.8

 Table 1. Information about the specimens provided by Imprest AS for

 performance table

In April 2017, AS Imprest produced 40 new specimens made of spruce with approximate dimensions of 490x24x49mm (longitudinal, radial, tangential) for laboratory testing, 4 different specimen groups were produced. Specimen group 0 consist of untreated specimens and groups 2, 3 and 4 are impregnated with Impralit KDS and 3 different impregnation cycles were used. Information about impregnation cycles of the specimen groups is presented in Table 3.

Unfortunately AS Imprest was not able to provide us credible weight and moisture content data about the specimens as the specimens were waterlogged when impregnated

and after impregnation measurements were done many days after impregnation. Unfortunately impregnation cycles and markings of specimen groups produced for laboratory testing were not exactly the same as they were on initial specimens produced for performance table. Specimen group 1 of laboratory testing specimens is similar to initial specimen group 3. Specimen group 2 of laboratory testing specimens is similar to initial specimen group 2. Specimen group 3 of laboratory testing specimens is similar to initial specimen group 1.

Impregnation cycle 12.8 bar Number of final Use primary vacuum; pressure; vacuum; specimen min min min class 1/11/230 240 15 HC 4 1/32/110 2/230 10 HC 3 2/33/1 15 HC 4 3/260 15 3/3

Table 2. Information about impregnation cycles of specimensprovided by Imprest AS for outdoor performance test

Table 3. Information about impregnation cycles of specimens

provided by Imprest AS for laboratory testing

	Impregnation cycle 12.8 bar			
Specimen group	primary vacuum; min	pressure; min	final vacuum; min	Use class
0	0	0	0	-
1	15	60	10	HC4
2	15	45	10	HC3
3	30	180	10	HC4

#### 2.1.3 Performance table

A folding table for fixing the spruce specimens was obtained through COST Action FP 1303 and principle concept can be seen on Figure 4. The specimens are fixed onto the table with screws, which are evaluated regarding of corrosion. Folding table has a plastic insulator strips to avoid moisture build up between the table and the specimens, this way the behavior of the specimens is more inherent.



Figure 4. Folding table for performance test [20]

#### 2.1.4 Fasteners

Two different types of fasteners are used in the performance test, galvanized and stainless steel screws. In parallel with the visual assessment of fungal decay, the fasteners will be inspected regarding corrosion. All fasteners on the upper surface will be removed and inspected visually according to the rating scheme explained in following methodology part. [20]

#### **2.2 Methods**

#### 2.2.1 COST Action FP 1303 – Cooperative Performance Test

Using weather data from weather station nearest to the respective exposure site, allows to establish relationships between climate conditions and the following measurands, which are evaluated regularly:

- Decay
- Discoloration
- Development of mold and other staining fungi
- Corrosion
- Formation of cracks
- Moisture performance

The schedule for measurements and assessment is given in Table 4. [20]

Table 4. Assessment schedule for	or performance test [20]
----------------------------------	--------------------------

Test/Assessment	Period	Interval
Decay	Full	Every 6 months
Mold/stain	First 6 months	Every 4 weeks
	From month 7 on	Every 6 months
Crack formation	Full	Every 3 months
Color measurements	First 4 weeks	Every week
	First 6 months	Every 4 weeks
	From months 7 on	Every 6 months
Corrosion visual	Full	Every 6 months
Corrosion final	After 3 years	
MC recordings	Full	Every 3 months

#### **2.2.2 Exposure conditions**

An exposure location for performance table should meet the following requirements:

- Typical free exposure (to avoid shading elements)
- Using standard test site to allow for further comparison with other running tests
- Performance table has to be safe
- The table should be fixed to the ground
- Dataloggers has to be in safe and dry place

The following information need to be provided about the exposure site:

- Geographical position
- Height above sea level

Photographs showing the performance table and the surrounding, showing the four compass directions

For interpretation of the results from performance table detailed information about the respective exposure conditions are needed. In particular the weather parameters corresponding to the exposure period are requested. Daily data for the following parameters need to be obtained from nearest weather station:

- Precipitation
- Average air temperature
- Minimum air temperature
- Maximum air temperature
- Average relative humidity [20]

Performance table of performance test was positioned for exposure on to the roof of U06 study building at Tallinn University of Technology. Height from the ground is 9.75 meters and from the sea level 43.75 meters. Coordinates of the exposure site are 59°23'38.76"N; 24 °40'10.588"W. Wood moisture, temperature and color change are measured and formation of cracks, mold, fungi and decay are observed. Weather data is obtained from nearest weather station located at Harku which is approximately 5 km away from the exposure site.

Performance table was set up on the roof of U06 study building at Tallinn University of Technology on 8<sup>th</sup> of April 2016 and the performance table at the exposure site is shown on Figure 5. After setting up the performance test table pictures of surroundings were taken to directions of North, South, East and West. North view is shown on Figure 6, South view on Figure 7, East view on Figure 8 and West view on Figure 9.

8 electrodes for measuring moisture content were mounted into the specimens, specimen 3/3 is without an electrode and it will be evaluated only visually. Temperature sensors were mounted into specimens 1/2 and 3/2.

On Figure 10 it is shown how the specimens, fasteners and measuring points situate on the table if we watch them from side where numbers of specimen are written, which is South-East side of the performance table.



Figure 5. Performance table set up at the exposure site on 8<sup>th</sup> of April 2016



Figure 6. North side surroundings of the performance test table



Figure 7. South side surroundings of the performance test table



Figure 8. East side surroundings of the performance test table



Figure 9. West side surroundings of the performance test table



Figure 10. Placement of specimens, screws and color measurement points on the performance table

#### 2.2.3 Decay

The specimens should be inspected once a year regarding the onset and progress of decay. Therefore the screws should be loosened and the specimens removed from the rig to allow inspection from all sides. [20]

Decay shall be assessed by visual inspection combined with a pick-test using a pointed knife. The knife should be pricked into the specimens and pulled out again to inspect the surface strength as well as the fracture depth and splinter characteristics for the rating

pursuant to EN 252 (1989). The rating scheme according to EN 252 (1989) is shown in Table 5. [20; 21]

Rating	Classification	Definition
0	No attack	No change perceptible by the means at the disposal of the inspector in the field. If only a change of color is observed, It shall be rated 0.
1	Slight attack	Perceptible changes, but very limited in their intensity and their position or distribution: changes which only reveal them-selves externally by superficial degradation, softening of the wood being the most common symptom.
2	Moderate attack	Clear changes: softening of the wood to a depth of at least 2 mm over a wide surface (covering at least 10 square centimetres) or by softening to a depth of at least 5 mm over a limited surface area (covering less than 1 square centimetre).
3	Severe attack	Severe changes : marked decay in the wood to a depth of at least 3 mm over a wider surface (covering at least 25 square centimetres) or by softening to a depth of at least 10 mm over a more limited surface area.
4	Failure	Impact failure of the stake in the field.

 Table 5. Decay rating scale [20; 21]

Additionally, on the basis of visible characteristics the type of decay should be identified. Information how to distinguish the main decay types (brown rot, white rot, soft rot and tunnelling bacteria) can be found in CEN/TS 15083-2 (2005) – Annex D.[22]

### 2.2.4 Discoloration

Color measurements are recorded at three points on the upper surface of each specimen with a colorimeter Minolta Chroma Meter CR-121. The measuring points are marked on each specimen through cross lines. The positions of measuring points are centrally between the long sides of the specimens and 30 mm from the end-grain and in the middle of the specimen as shown on Figure 11. [20]

The measurements are taken in  $L^*a^*b$  coordinates, as established by the Commision Internationale de Enluminure (CIE) in 1976, where  $L^*$  determines the lightness,  $a^*$  and  $b^*$  determine the chromatic coordinates on the green-red and blue-yellow axis, respectively. [20] The measurements should be conducted every week during the first four weeks of exposure, every 4 weeks during the first six months, and later on twice a year. Measurements should be determined on the dry surface, at least 2 hours after the last rainfall. In case that there is snow or ice on the table, measurements should be postponed. To determine color changes over time the distance in color space  $\Delta E$  shall be determined according to Equation 1:



Figure 11. Position for measuring L\*a\*b color values [20] and measuring points on the specimens Equation 1. Distance in color space  $\Delta E$ .

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$
(1)  
where:  $\Delta E$  = distance in color space [-]

 $L^* = \text{lightness}[-]$ 

 $a^*$  = chromatic coordinate of the red-green axis []

 $b^*$  = chromatic coordinate of the yellow-blue axis [] [20]

#### 2.2.5 Surface disfigurement due to mold and staining fungi

Examine the upper surface of the test specimens visually for the presence of mold and staining fungi. Evaluate the upper and lower surface (underside) of the samples as shown in Table 6. For easy evaluation of surface disfigurement due to fungal growth use the following criteria adapted from EN 152:2011 (Determination of the protective effectiveness of a preservative treatment against blue stain in wood in service – Laboratory method), 8.5.2 Assessment of the test specimen surface, with some modification to even accommodate the evaluation of disfigurement caused by mold fungi. [20; 23]

Rating	Classification	Definition
0	No disfigurement	No surface disfigurement can be detected visually on the surface.
1	Slight disfigurement	The surface exhibits only a few individual small colonies none larger than 1.5 mm in width and 4 mm in length.
2	Moderate disfigurement	The surface is colonized up to a maximum of one third of the total area.
3	Severe disfigurement	More than one third of the surface area is colonized.

 Table 6. Fungal disfigurement rating scale [20]

#### 2.2.6 Corrosion

In parallel with the visual assessment of fungal decay the fasteners will be inspected regarding corrosion. All fasteners on the upper surface should be removed and inspected visually according to the rating scheme presented in Table 7. Each fastener is washed in ethanol and weighed before being fixed in the wood sample. There will be two types of fasteners used, galvanized and stainless steel screws. [20; 24]

After 3 years of exposure, fasteners should be replaced with new ones. The old ones should be analysed according to the following procedure. The metal loss is calculated and expressed as metal loss (%) and as depth of corrosion (mm). In order to determine the metal loss and depth of corrosion the corrosion products had to be eliminated. Thus the fasteners will be pickled, cleaned and then weighed. [20; 24]

Rating	Description	Definition
0	No attack	
1	Insignificant attack	<5 % of surface attacked
2	Slight attack	5-50 % of surface attacked
3	Serious attack	50-95 % of surface attacked
4	Completely attacked	>95 % of surface attacked

Table 7. Assessment of corrosion attack [24]

#### **2.2.7 Crack formation**

The formation of cracks on the upper surface of the specimens shall be determined. Therefore the following three measures need to be recorded:

- Total crack length (total length of cracks with length of more than 5 mm)
- Number of cracks (longer than 5 mm)
#### Mean maximum crack width

•

The length of cracks can be easily determined using a ruler. The maximum width of each single crack (longer than 5 mm) shall be recorded to determine the mean maximum crack width. All the crack formation measurements were done with crack measuring gauge which is basically a ruler with extra section for measuring crack width, which you can see on Figure 12. Specimens shall be inspected regarding the formation of cracks before exposure (initial state) and then every 3 months. [20]



Figure 12. Crack measuring gauge [20]

#### 2.2.8 Moisture content and temperature

The performance table is equipped with moisture content and temperature sensors, which consist of Scanntronik Mugrauer GmbH Thermofox Universal data logger and Thermofox Material Moisture Gigamodule (extension module) for electrical resistance measurements and recordings. Data logger is connected with the electrodes which are fixed to the specimens with insulated fasteners as shown in Figure 13. Therefore, the moisture content is recorded only in the central part of the specimen. Black cables are mounted for moisture content measurements; red cables are used for temperature measurements as shown on Figure 14. There are 8 electrodes and 2 temperature sensors mounted into the specimens. The data logger is recording electrical resistance in 10logOhm and temperature, which can be translated to wood moisture content with Equation 2. [20]

Equation 2. Moisture content from electrical resistance and temperature.

$$MC(R;T) = (a \times T) + EXP((c \times T + d) \times R) + (e \times T + f) + (g \times R^{2}) + (h \times T) + i;$$
(2)

where MC – wood moisture content; %

- R electrical resistance (10logOhm)
- T wood temperature; °C
- *a*, *b*, *c*, *d*, *e*, *f*, *g*, *h*, *i* variables. [20]

Setting up the performance table and the moisture logging devices requires two persons in order to avoid potential damage of the cables. Prior to exposure cables should be checked and also their position to the data logger and gigamodule. Black cables should be mounted to gigamodule, while red ones should be mounted to Thermofox. Gigamodule and Thermofox were positioned indoors near the performance table and outdoor cables were protected against mechanical damage with conduit. [20]



Figure 13. Isolated fasteners were used for fixing electrodes on wood specimens



Figure 14. Thermofox datalogger and gigamodule [20]

#### 2.2.9 Color change under UV irradiation

Specimens with approximately dimensions of 490x49x24mm (longitudinal, radial, tangential) in UV radiation test. Specimens were stored in dark storage at indoor conditions (+25°C, RH 50%) before test procedure. Before treating specimens with UV radiation color and moisture content measurements of the specimens are made. Color measurements are recorded at three points on the upper surface of each specimen with a colorimeter Minolta Chroma Meter CR-121, according to L\*a\*b coordinates described earlier in method of evaluating discoloration of the specimens. The measuring points are marked on each specimen through cross lines corresponding to Figure 11. The positions are centrally between the long sides of the specimens and 30 mm from the end-grain and in the middle of the specimen. Moisture content measurements are made with moisture meter Hydromette HT 85T by GANN which working principle is based on electrical resistance. Test specimens are placed under UV irradiation alternately by specimen group.

The tests were executed in a UV chamber under conditions with a temperature of +45°C and relative humidity of approximately 5%. UV chamber was equipped with six fluorescent tubes which were evenly positioned over the chamber bottom surface at height of 400mm over the specimens top surface. Specimens were irradiated with UVA-351 type fluorescent lamps (PHILIPS TL-D 36W/08 T8 blacklight) and the peak wavelength of the tube was 351nm as seen on Figure 15. According to EN ISO 4892-1 the UV irradiation was in every position of the bottom surface of the UV-chamber a minimum of at least 70% of the maximum irradiation value obtained from the central area of the UV-chamber bottom surface. [25]

For irradiation of the specimens one continuous cycle was used and a total irradiation time was 336 hours. During the UV irradiation test intensities of irradiation at a wavelength of 350nm were recorded with a radiometer Delta OHM HD 2102.2 and with lux-meter probe SICRAM, UVA.

After the UV irradiation of 336 hours color measurements of every specimen were done and difference between initial and final lightness and chroma coordinate measurements was evaluated. Total color difference between initial and final color was calculated for each specimen according to previously described method of evaluating the discoloration of the specimens.



Figure 15. Spectrum of the UVA-351 type fluorescent lamp (PHILIPS TL-D 36W/08 T8 blacklight) [26]

#### 2.2.10 Swelling test and water uptake

Swelling test and water uptake procedure are adapted from G. I. Mantanis research of Swelling of wood Part 1. Swelling in water [28], which observes the rate and maximum swelling of several North American wood species in water. In current swelling test specimens with approximate dimensions of 490x24x49mm (longitudinal, radial, tangential) were used. 3 specimens from every specimen group obtained from Imprest AS for laboratory testing were used in swelling test. In addition to water uptake od the specimens, longitudinal, radial and tangential percentage swelling were measured. Specimens were oven-dried with laboratory dryer Ecocell 222 from MMM Medcenter Einrichtungen GmbH at 103 °C.

The cooled oven-dry specimens were weighed, dimensions and MC measured at indoor conditions as soon as possible. Weights were measured with KERN precision balance PLJ, dimensions were measured with jaw-type vernier caliper and MC measurements were done with moisture meter Hydromette HT 85T by GANN which working principle is based on electrical resistance.

The specimens were placed into a plastic container which was filled with water and specimens were left submerged for 44 hours. The soaked specimens were again weighed, dimensions and MC measured at indoor conditions as soon as possible similarly as done before the soaking.

Obtained information was used to calculate oven dry weight of the wood, which was compared with the weight of wood after being submerged and percentage swelling of longitudinal, radial and tangential was calculated dimensions according to Equation 3 and Equation 4 respectively. [27; 28]

Equation 3. Oven dry weight of wood [27]

$$W_0 = \frac{W_g}{(1 + (\frac{mc}{100}))}$$
(3)

Where:  $W_0$  = oven dry weight of the

 $W_g$  = green weight of the wood

mc = moisture content.

Equation 4. Percentage swelling. [28]

$$Percentage \ swelling = \frac{(Swellen \ dimension - Oven \ dry \ dimension)}{(Oven \ dry \ dimension)} \times 100\%$$
(4)

# **3** Results and discussion

The performance test has been running since 8th of April 2016 and in current master thesis results from performance test until 5th of May 2017 are to be observed and analyzed. The performance test itself is still ongoing and in the future more comprehensive conclusions can be made.

### 3.1.1 Decay

On 1st of December and 5th of May specimens were evaluated regarding decay and visually no progress of decay was found. On Figure 16, both sides of the specimens are shown during the last evaluation of decay on 5<sup>th</sup> of May 2017 which is approximately 13 months from exposure of the performance table.



Figure 16. Under (left) and top (right) of the specimens on 5<sup>th</sup> of May 2017 for evaluation decay of the specimens

As protective mechanism of modified timber is based on its hydrophobic character and improved moisture performance and different studies have shown that wood moisture content has significant impact to funcal decay [29]. If moisture content is kept below favorable conditions for decaying organisms the wood is sufficiently durable for outdoor applications.

Critical moisture levels are material-specific characteristics and need to be considered when estimating the resulting moisture and temperature induced risk for decay of wood-based materials. In current research Norway spruce specimens are used and according previous research the moisture content for the fibre saturation point of spruce species is about 35 - 37% [29]. It has been indicated that wood decay fungi require wood moisture contents in excess of fiber saturation to propagate, also that development below fiber

saturation is greatly retarded and even that below 20% wood moisture content their development is completely inhibited [30, 31].

Decay fungi should be able to digest wood cell wall material only if there is some free water present which is needed as a diffusion medium for the extracellular digestive enzymes. When decay is occurring, but the wood is exposed to atmospheric conditions that would induce drying, generation of metabolic water may prolong conditions under which decay can progress. If moisture conditions fall much below the fibre saturation the fungi can go to so call hibernation state and revive itself when moisture conditions again reach levels around fiber saturation. Most wood decay fungi will develop only between 15° and 40°C. Studies have shown that optimum temperatures for most wood-decay fungi are between 21° and 32°C and that no fungi would grow at 12°C and that most would not grow at 40°C. Cold temperatures are not lethal to decay fungi and the fungi will revive itself when temepratures are again suitable for growth, while high temperatures are lehtal to wood decay fungi, but the tolerance for elevated temperture vary between decay fungi species. Temperature level, length of exposure, and moisture content during exposure are parameters that influence high-temperature survival of decay fungi [31].

As pointed earlier spruce species fibre saturation point is considered to be 35 - 37% and during the performance test all the test specimens had an average moisture content under 30%, ofcourse there were short periods of time where relative humidity, wood moisture content and temperature were suitable for wood decay fungi to grow but the suitable conditions did not last long enough to promote fungi to grow. Taking into consideration other reasearches and data obtained from the current performance test with impregnated spruce specimens, it can be said that result of no wood decay due to fungi after 13 months of exposure is rather expected. As spruce specimens impregnated with Impralit KDS have maintained during 13 months average moisture contents under 30% (specimen group 1 - 21.4%, group 2 - 20.4% and group 3 - 29.1%) it can be said that so far all three impregnation cycles have ensured wood moisture content which does not offer suitable conditions for wood decay fungi to grow.

#### **3.1.2 Discoloration**

Before setting up the performance table color measurements of the specimens were made with colorimeter Minolta Chroma Meter CR-121 at three points on the upper surface of each specimen with a colorimeter. The measuring points were marked on each specimen through cross lines and all the results from the initial color measurements are given in Table 8. On Figure 17, performance table is presented at exposure site on 8<sup>th</sup> of April 2016 and on final evaluation 5<sup>th</sup> of May 2017.

Specimen	Measurment point	L	a	b	Specimen	Measurement point	L	a	b
	1	48.7	-0.7	13.2		1	50.4	-0.9	12.5
	2	47.7	-0.1	11.6		2	49.1	-0.5	13.3
1/1	3	47	-1.6	11.5	2/1	3	49.3	-1.1	14.6
	1	46.2	-0.6	8.6		1	52.5	0.3	18.9
	2	46.7	-0.2	10.8		2	56	-0.8	18.3
1/2	3	48	-0.9	12.1	2/2	3	51.2	-0.3	16.8
	1	52.6	-0.6	13.6		1	51.9	0.9	19
	2	52.7	-2.8	13		2	52.1	-1.5	17.4
1/3	3	52.6	-1.8	11.7	2/3	3	51.2	-1	16.3
	1	47.7	-1.7	11.4					
	2	55.4	-2.4	13.8					
3/1	3	52.2	-2.9	15					
	1	49.3	-2.9	11.5					
	2	48.6	-2.5	13					
3/2	3	44.9	-0.1	10.7					
	1	52.3	-3.2	13.1					
	2	51.3	-1.1	16.3					
3/3	3	52	-1.1	14					

Table 8. Initial color measurements of the specimens

Until 5th of May color measurements have been done 10 times (including initial measurements), the time period is about 13 months and total color change is given on Figure 18. Trough out the test discoloration of the specimen group 2 has been slightly different and smaller than others but measurements made on 1<sup>st</sup> of December showed that after 8 months total color change of the specimens was very similar. Next 2

measurements conducted after 1st of December again show somewhat opposite behavior for specimen group 2 regarding specimen groups 1 and 3.



Figure 17. Topside of performance table on 8<sup>th</sup> of April 2016 (left) and on 5<sup>th</sup> of May 2017 (right)

On Figure 19 total color change of every specimen from exposure to 5th of May is given, also average value of total color change by specimen group is presented. On Figure 20, the total color change of the specimens is given alongside with the UV-indexes in this time period, UV-indexes are presented as an average UV-index in one hour, as a maximum UV-index in one hour and total UV-indexes during 24 hours.



Figure 18. Total color change of the specimen groups from 8<sup>th</sup> of April 2016 until 5<sup>th</sup> of May 2017



Figure 19. Color change of every specimen and average color change by specimen group from  $8^{th}$  of April 2016 until  $5^{th}$  of May 2017



Figure 20.Total color change of specimens alongside with UV-index values from  $8^{th}$  of April 2016 until  $5^{th}$  of May 2017

As said before color change of wood surface is one of the first and clearest sign of weathering of the wood during outdoor exposure. Visible light and UV-radiation change the color of the wood due to the fact that water-soluble decomposition products are removed and delignified fibers are exposed. Wood is affected by direct or indirect solar radiation and the chemical transformation of a compound into smaller compounds caused by the absorption of ultraviolet, visible, or infrared radiation is called photochemical degradation, which transforms the wood surface into a network of weakly connected cellulose fibrils which may be contaminated by spores from microorganisms. The photochemical degradation is a very slow process which during a decade degrades only a few millimetres of the wood surface [2].

In current performance test Norway spruce specimens have been exposed for 13 months (performance test is ongoing) in use class 3 conditions (EN 335), which mean that specimens are exposed to weather, not in contact with ground but subject to wetting [32]. It can be said that after exposure on 8<sup>th</sup> of April 2016 it took specimens two weeks to reach the discoloration level that has been similar the rest of 13 months. During the first months of the performance test the specimen group 2 specimens tend to lose less color and to behave slightly different compared to specimen groups 1 and 3 but as the test proceeded all the specimen groups discoloration has evened out. After 13 months all three specimen groups discoloration values were about 5 times smaller than untreated Norway spruce discoloration in 12 months of exposure at same exposure site.

While impregnated spruce specimens tend to lose less color than untreated spruce specimens, in photo stabilizing of the wood surface point of view Impralit KDS have proven itself to be ineffective and if apperance of the wood is essential in certain applications other solutions should be considered. Wood impregnated with Impralit KDS is considered to be overpaintable which gives some extra opportunities if the wood color has to be maintained for longer time periods. It has to be mentioned that as clear stain film does not offer protection from UV-radiation the film used for maintaining the color should be opaque.

Due to time constraints leaching of the preservative during wetting of the wood due to rain and snow have not been considered in current research, which has certainly effect on discoloration of the specimens as well as diminishing the protection of the preservative. As the performance test is still ongoing the leaching aspect should be thoroughly investigated in future.

#### 3.1.3 Surface disfigurement due to mold and staining fungi

On 29<sup>th</sup> of April and on every measuring day after that evaluation of surface disfigurement due to mold and staining fungi have been done and visually no surface disfigurements has been detected. On Figure 21, the performance table is presented in winter conditions to show the wide range of conditions the specimens were subject to.

Mold and staining fungi affects the surface of the wood as opposed to wood decay which affects wood also from inside. Similarly to wood decay fungi mold and staining fungi need high relative humidity for growth, although many mold fungi can tolerate lower relative humidity than wood decay fungi. Lower limit of tolerance for growth of mold fungi is considered to be at 75% to 80% relative humidity, which is due to fact that mold fungi have greater moisture holding capacity. Staining fungi starts to develop near or at the fiber saturation point, moisture content around 22% and relative humidity around 93% are needed for growth [32]. Conditions for growth of mold and staining fungi are very similar to wood decay fungi, but they need slightly lower relative humidity and wood moisture for growth. As pointed earlier spruce species fibre saturation point is considered to be 35 - 37% and during the performance test all the test specimens had an average moisture content under 30%, ofcourse there were short periods of time where relative humidity, wood moisture content and temperature were suitable for mold and staining fungi to grow but the suitable conditions did not last long enough to promote fungi to grow and no surface disfigurement was detected [31]. Norway spruce specimens impregnated with Impralit KDS have maintained during 13 months of exposure average moisture contents under 30% (specimen group 1 - 21.4%, group 2 - 20.4% and group 3 - 29.1%).

Preservative Impralit KDS manufacturer has promised temporary protection against blue stain and mold, and they have not specified the term temporary [17]. During 13 months of exposure no surface disfigurement due to mold and staining fungi was detected on specimens impregnated with Impralit KDS and all three impregnation cycles have ensured wood moisture content and protection which did not offer suitable conditions for mold and staining fungi to grow.



Figure 21. Performance table on 9<sup>th</sup> of November 2016

#### 3.1.4 Corrosion

Stainless steel and galvanized screws were used as fasteners to attach the specimens to the performance table. Before exposure each fastener was washed in ethanol and weighed before being fixed in the wood sample and the results are given in Table 9, after 3 years of exposure the fasteners should be analyzed metal loss will be calculated and expressed as metal loss and as depth of corrosion [20].

Evaluation of corrosion was done four times and results from all the evaluations are given in Table 10, on Figure 22 and Figure 23 galvanized screws used in the test are presented as they were respectively on 29<sup>th</sup> of April 2016, 3 weeks after exposure and on 5<sup>th</sup> of May 2017, 13months after exposure.

The surface of Stainless Steel screws was intact on all the times and all the screws got a rating of "0" which means that there was no surface attack. The surface of galvanized screws was visibly attacked and all the screws got a rating of "2" on 29<sup>th</sup> of April, which means 5-50% of surface was attacked. On 16<sup>th</sup> of September most of the galvanized screws got a rating of "4" which means over 95% of surface was attacked and some

galvanized screws got a rating of "3" which means 50-95% of surface was attacked. Already on  $10^{\text{th}}$  of March all the galvanized screws got a rating of "4".

Number of screw	Weight of Stainless steel screw; g	Weight of galvanized screw, g
1	2.83	2.56
2	2.89	2.57
3	2.82	2.57
4	2.82	2.57
5	2.81	2.59
6	2.83	2.58
7	2.84	2.58
8	2.81	2.57
9	2.81	2.58

Table 9. Initial weight of the fastenersused in the performance test

These results are in correlation with the wood moisture content, which was most of the time higher than the considered level needed for fasteners corrosion which is 15-20 percents, and as the galvanized steel is anodic to copper galvanized screws are more prone for corrosion [18]. Impralit KDS formulation is considered non-corrosive compare to CCA. However in current performance test galvanized screws are corroded therefore statement that Impralit KDS is non-corrosive needs further investigation.

As described earlier corrosion of metals in wood is similar to a dry cell that generates voltage, simply because materials at a higher energy state tend to revert to materials of a lower energy state. Metal fasteners corrosion in wood very dependent of wood moisture content than relative humidity of air which makes it very different from atmospheric corrosion. When copper based preservatives are used the reduction of cupric ions is the primary cathode reaction occurring in these preservatives. As most wood species are naturally acidic the metal fasteners will corrode if suitable moisture contents are provided. Studies have shown that wood impregnated with CCA are approximately 2 times more corrosive than untreated wood [16].

In current performance test where Norway spruce specimens impregnated with Impralit KDS, which is considered non-corrosive compared to CCA, after 13 months of

exposure stainless steel screws had no corrosion and galvanized screws were corroded already after two weeks. As metal fasteners need presence of water (electrolyte) to corrode, wood moisture content between 15% to 20% is considered to be the critical level where corrosion of metal fasteners starts and relative air humidity of 60% to 75% is needed to start the process of corrosion.

Corrosion ratings							
Galvanized screws; #	29.04.2016	16.09.2016	10.03.2017	5.05.2017			
1	2	3	4	4			
2	2	4	4	4			
3	2	4	4	4			
4	2	4	4	4			
5	2	3	4	4			
6	2	3	4	4			
7	2	4	4	4			
8	2	4	4	4			
9	2	4	4	4			
Stainless steel screws; #							
1	0	0	0	0			
2	0	0	0	0			
3	0	0	0	0			
4	0	0	0	0			
5	0	0	0	0			
6	0	0	0	0			
7	0	0	0	0			
8	0	0	0	0			
9	0	0	0	0			

Table 10. Evaluation values of corrosion attack

Conditions for corrosion of metal fasteners to start were fulfilled in current performance test during 13 months as all the specimen groups had an average moisture content over 20% and an average relative air humidity of this period was approximately 80% [16]. Corrosion of galvanized screws happed already with two weeks which seems to be in correlation with statements that CCA impregnated wood is two times more corrosive than untreated wood, because in similar performance test with similar conditions untreated Norway spruce was used and it took four weeks for the corrosion of metal fasteners to occur[33]. After 13 months of exposure it can be said that in current performance test Impralit KDS seems to be as corrosive as CCA and different impregnation cycles have not influenced the corrosion rate of the specimens. More accurate conclusion can be made after three years of exposure as the performance test is still ongoing.



Figure 22. Galvanized screws removed from performance table on 29<sup>th</sup> of April 2016 for inspection



Figure 23. Galvanized screws removed from performance table on 5<sup>th</sup> of May 2017 for inspection

#### **3.1.5 Crack formation**

The formation of cracks on the upper surface of the specimens was determined 5 times after exposure with crack measuring gauge and the results are presented in Table 11. On Figure 24, Figure 25 and Figure 26 respectively total crack length, number of cracks and mean crack width changes are presented alongside with corresponding moisture contents of the specimen groups.

	08.04.2016	29.04.2016	27.05.2016	01.12.2016	10.03.2017	05.05.2017		
Total crack length; mm								
Imp. Spruce 1	0.00	0.75	250.00	430.33	481.33	571.33		
Imp. Spruce 2	0.00	139.67	360.67	647.67	679.33	744.00		
Imp. Spruce 3	0.00	48.67	275.00	515.33	594.00	723.67		
MC Imp. Spruce 1	30.08	27.86	13.49	24.33	18.19	13.18		
MC Imp. Spruce 2	28.07	24.24	13.26	22.90	20.48	13.99		
MC Imp. Spruce 3	35.55	38.69	11.23	35.46	30.99	17.28		
Number of cracks;	#	<u>.</u>						
Imp. Spruce 1	0.00	2.67	11.00	7.00	8.67	8.33		
Imp. Spruce 2	0.00	6.00	13.33	8.67	9.33	9.33		
Imp. Spruce 3	0.00	2.67	12.00	9.33	11.00	13.33		
Mean crack width; mm								
Imp. Spruce 1	0.00	0.19	0.25	0.37	0.37	0.55		
Imp. Spruce 2	0.00	0.24	0.34	0.32	0.38	0.67		
Imp. Spruce 3	0.00	0.33	0.38	0.32	0.38	0.56		

Table 11. Results of crack formation measurements from 8 <sup>th</sup>	<sup>th</sup> of April 2016 until 5 <sup>tl</sup>	' of May 2017
--	---	---------------

During drying of the wood, photochemical reaction, moisture induced stress fields in the cell walls and between cells, micro cracks form which grow to visible cracks during outdoor exposure. During drying of the wood, photochemical reaction, moisture induced stress fields in the cell walls and between cells, micro cracks form and grow to visible cracks during outdoor exposure [2].

During drying of the wood, photochemical reaction, moisture induced stress fields in the cell walls and between cells, micro cracks form which grow to visible cracks during outdoor exposure. Exposure to sunlight and rain will propagate frequency and size of the cracks, but photochemical reactions on wood surface accelerate the process but they

are not the main reason of crack formation. Most important aspect of crack formation during weathering is considered to be wood annual ring orientation; type of wood, impregnation treatment and surface treatment are considered to have only marginal effect on crack formation. Also difference between linseed oil treatment, impregnation or type of wood have not been observed [2].



Figure 24. Total crack length and moisture contents of specimen groups

Crack length ratio between tangential and radial surfaces has been studied, between exposed surfaces and non-exposed surfaces, it has established that this ratio is smaller for the exposed surfaces, which implied that the difference in crack development between radial and tangential surfaces is not only a consequence of photochemical degradation. From it follows that shrinkage and swelling in the tangential direction are about twice as large as the radial moisture movement thus tangential surfaces move more than radial surfaces. Stresses in wood are higher in tangential direction than in radial direction when the moisture movement in the surface layer is limited by underlying wood which does not have the same moisture content as the surface layer of the wood. With rapid moisture changes in the surface moisture gradients arise between the surface area and the underlying wood material which results in the formation of more cracks on tangential surfaces than on radial surfaces. [2]



Figure 25. Number of cracks and moisture contents of the specimen groups



Figure 26. Mean crack width and moisture contents of specimen groups

After 13 months of exposure the crack formation of the specimen groups is rather similar as all the specimen groups have similar number of cracks with similar width and total crack length. During the performance test only specimen group 1 have stand out from other specimen groups as it has slightly smaller number of cracks and the cracks are slightly shorter and narrower. During 13 months average moisture content of specimen group 1 has been approximately 21%, specimen group 2 20% and specimen group 3 29%, there is no clear relationship between the specimen group 1 slightly better crack development performance and average moisture content. As previous studies have shown the impregnation and in this case different impregnation cycles seem not have any effect on crack development and the statement that most important aspect considering crack development is wood annual growth ring orientation is correct and should be consider when using wood in outdoor applications [2].

#### **3.1.6 Moisture content and temperature**

Specimen moisture contents and temperatures were measured constantly on every hour from 8<sup>th</sup> of April 2016 until 5<sup>th</sup> of May 2017 (performance test is ongoing). Wood moisture content changes according to the air moisture content, but not so rapidly and also the change happens with little delay, wood moisture content fluctuated between 3.8% to 46% and relative humidity fluctuated between 25% to 100%.. Specimens and air moisture contents from 8th of April 2016 until 5th of May 2017 are presented on Figure 27 and on Figure 28.

When wood moisture content did not respond so rapidly to changes of air moisture content then wood temperatures are in direct relation to air temperature. Slight variation of temperatures are probably caused by occasional direct sunshine and also by the fact that the Harku weather station is located slightly off the exposure site and the air temperatures can vary between these locations.

As protective mechanism of modified timber is based on its hydrophobic character and improved moisture performance, specimen groups 1 and 2 stand out as specimens with best moisture performance as specimen group 1 had during 13 months of exposure an average moisture content of 21% and specimen group had an average moisture content of 20% which compared to specimen group 3 is considerably better moisture performance which specimens had an average moisture content of 29% during the performance test. The wood capability to take up moisture is considered to be a component of wood resistance which means that improving wood moisture performance is essential for preserving the wood [34].



Figure 27. Wood and air moisture content from 8<sup>th</sup> of April 2016 until 21<sup>st</sup> of October 2016



Figure 28. Wood and air moisture content from 21<sup>st</sup> of October 2016 until 5<sup>th</sup> of May 2017

During the current performance test where three different impregnation cycles were used to evaluate their influence on resistance to weathering of Norway spruce specimens, we can say that specimen groups 1 and 2 have better moisture performance compared to specimen group 3, which could be the result of impregnation cycle and the amount of preservative forced inside the specimens.

#### 3.1.7 Color change under UV irradiation

One continuous cycle was used for irradiation of the specimens and a total irradiation time was 336 hours. Test specimens were placed under UV irradiation alternately by specimen group and during the UV irradiation test intensities of irradiation at a wavelength of 350nm were checked with a radiometer Delta OHM HD 2102.2. On Figure 29, upper sides of test specimens are presented before and after the artificial UV irradiation.



Figure 29. Test specimens before (left) and after (right) 336 hours of artificial UV irradiation

Before and after treating the specimens with UV irradiation color measurements were done with colorimeter Minolta Chroma Meter CR-121 and difference between initial and final lightness and chroma coordinate measurements was evaluated. Total color difference between initial and final color was calculated and total color change of specimens after 336 hours of UV irradiation is presented on Figure 30. Results from initial color measurements are given in Table 12. Moisture content measurements were done before and after the UV irradiation with moisture meter Hydromette HT 85T and the results are given in Table 13.

Changes in wood color reflect chemical changes during irradiation and the appearance of new chromophoric structures. When natural weathering tests can last very long, they provide most accurate and reproducible data, advantage of artificial weathering is its relatively short duration. Artificial weathering is considered to be 5 to 20 times faster than natural weathering depending on exposure conditions chosen. [35] Neither natural weathering nor artificial weathering with UV lamp offer a precise way to measure the energy of radiation on the wood sample surface, even more if photo degradation is influenced by other factors like moisture, temperature, visible light and infrared radiation [36].

Lignin makes up 29 to 33% of softwood and is responsible for absorbing 80 to 95% of the total UV light absorbed by wood, carbohydrates 5 to 20% and extractives about 2%. Formation of free radicals by UV irradiation triggers the photo degradation process and the lignin content relative to cellulose content will decrease on the weather wood surface. [36]

During 336 hours of continuous artificial UV irradiation at wavelength of 350nm with measured intensities ranging between 4.40 W/m2 and 5.46 W/m2 all impregnated specimens total color change was very similar and no remarkable variations due to different impregnation cycles were observed. However untreated spruce specimens total color change was almost 3 times greater than on impregnated specimens, which expectable result as untreated spruce tends to lose more color. According to similar outdoor performance test with similar exposure conditions untreated spruce specimens total color change during one year was over five times greater than impregnated spruce specimens used in current performance test. This confirms that total color change ratios obtained from current artificial UV irradiation test are rather correct.

Although current UV irradiation intensities and test conditions have proven to be insufficient to accelerate the photo degradation process compared to natural weathering in outdoor conditions as same total color change values were obtained in outdoor performance test for untreated spruce with two weeks and for impregnated spruce specimens with one week [33]. Of course in outdoor testing the UV intensities fluctuate during different time of year but in general it can be said that with current artificial UV test set up no photo degradation acceleration was accomplish compared to outdoor weathering. According to the results it seems that different impregnated with Impralit KDS.

Specimen	Measurment point	L	a	b	Specimen	Measurment point	L	a	b
	1	70.3	1.3	14.0		1	50.0	0.3	10.3
	2	6.5	1.3	15.6		2	57.1	0.6	14.8
0/3	3	73.0	3.7	21.7	1/3	3	55.1	-0.4	12.0
	1	73.0	1.4	11.9		1	54.6	0.7	13.2
	2	69.0	3.9	18.6		2	57.2	0.0	15.8
0/6	3	70.3	0.8	11.6	1/6	3	52.7	0.4	13.6
	1	80.0	0.0	14.9		1	54.6	0.4	12.1
	2	71.3	1.2	14.1		2	53.7	0.5	12.1
0/9	3	74.0	0.7	13.4	1/9	3	52.7	-0.2	11.9
	1	55.3	0.0	12.1		1	50.4	0.0	11.4
	2	57.1	0.0	15.6		2	50.8	0.2	12.0
2/3	3	54.8	1.2	17.3	3/3	3	50.1	-0.1	9.8
	1	47.7	2.6	15.0		1	50.6	1.8	16.5
	2	54.7	0.8	11.7		2	53.1	0.0	13.2
2/6	3	56.3	0.5	14.1	3/6	3	50.0	1.1	15.3
	1	51.4	0.1	12.0		1	52.3	0.1	12.5
	2	54.4	0.3	12.6		2	48.6	-0.5	10.5
2/9	3	50.9	0.0	12.9	3/9	3	53.5	0.0	11.9

Table 12. Results of initial color measurements before UV irradiation



Figure 30. Color change of specimens after UV irradiation of 336 hours

Specimen	MC before UV irradiation; %	MC after UV irradiation; %
0/3	13.8	0.2
0/6	12.4	1.0
0/9	11.7	0.4
1/3	23.6	0.3
1/6	22.8	0.4
1/9	20.1	0.3
2/3	17.0	0.7
2/6	19.2	0.6
2/9	38.3	0.3
3/3	17.6	0.7
3/6	22.7	1.1
3/9	19.6	1.0

Table 13. Moisture contents before and after the UV irradiation

#### 3.1.8 Swelling test and water uptake

Water uptake, longitudinal, radial and tangential percentage swelling were evaluated during the swelling test. Specimens were oven-dried at 103 °C and cooled oven-dry specimens were weighed, dimensions and MC measured at indoor conditions as soon as possible. The specimens were placed into a plastic container which was filled with water as seen on Figure 31 and specimens were submerged for 44 hours. The soaked specimens were again weighed, dimensions and MC measured at indoor conditions as soon as possible similarly as done before the soaking. Obtained information was used to calculate the amount of water absorbed by the specimens and percentage swelling of longitudinal, radial and tangential dimensions. Water uptake results and amounts are given in Table 14 and presented on Figure 32, percentage swelling results are given in Table 15 and presented on Figure 33.

Result from the swelling test show and correlate with statements described earlier in chapter of crack formation, that the tangential swelling was approximately 2 to 3 times greater than the radial swelling, which also is the reason why more stresses form in tangential surfaces and result in greater crack formation.



Figure 31. Submerged swelling test specimens

Table 14. Average water uptake of the laboratory specimens

provided by	AS Im	prest during	the swelling	test
provided by	110 111	prese auring	the swenning '	<i>coc</i>

Specimen #	W0 calculated after drying; g	MC after swelling 44h; %	Weight after swelling 44h; g	Water uptake; g	Average water uptake by specimen group; g
0/2	154.34	40.50	322.02	167.68	
0/5	158.22	32.60	325.03	166.81	170.13
0/8	171.83	35.00	347.71	175.88	
1/2	166.70	42.30	343.84	177.14	
1/5	171.63	36.30	345.03	173.40	172.53
1/8	161.59	34.50	328.64	167.05	
2/2	164.07	32.30	331.23	167.16	
2/5	157.31	38.80	326.77	169.46	170.77
2/8	166.99	35.50	342.68	175.69	
3/2	155.24	37.70	316.00	160.76	
3/5	139.31	37.70	293.58	154.27	159.29
3/8	169.25	33.00	332.10	162.85	



Figure 32. Average water uptake during swelling test



Figure 33. Longitudial, radial and tangential percentage swelling of the laboratory specimens provided by AS Imprest during the swelling test

#### Table 15. Longitudial, radial and tangential percentage

swelling of the laboratory specimens provided by

AS Imprest during the swelling test

Specimen group	MC after swelling 44h; %	Longitudial percentage swelling; %	Radial percentage swelling; %	Tangential percentage swelling; %
0	36.03	0.16	2.67	8.02
1	37.70	0.28	2.85	4.89
2	35.53	0.14	1.91	6.05
3	36.13	0.11	1.71	4.95

During swelling test also water uptake of the impregnated specimens was measured and the results show that specimen groups 3 (according laboratory testing groups, see Table 3) had smallest amount of water absorbed during the test. Specimen group 3 had also longest impregnation cycle which means that greater amount of preservative was forced inside wood, which means there less space for water in wood cell. Artificial swelling of preservative helps to improve dimensional stability of wood, water resistance of wood improves remarkably when the preservative is cross linked with wood or when it has blocked the hydroxyl groups in wood [11]. Low moisture content of wood helps to

improve many of its properties, also it provides more protection against different mold and fungi.

## Conclusion

The objective of this master's thesis was to gather and to examine the performance data of Norway spruce, impregnated with wood preservative Impralit KDS, in outdoor environment and make conclusions about the impregnated spruce resistance to weathering and how different impregnation cycles change the wood resistance to weathering. The test has been running for about 13 months but impregnated wood products are designed to withstand outdoor environment for much longer time than 13 months and the performance test is still ongoing which mean that current conclusions are not yet fundamental and current research should be continued.

In the study, following conclusions were made:

- Preservative Impralit KDS and all three impregnation cycles ensured better moisture performance of Norway spruce preventing the growth of mold and different fungi.
- Impralit KDS have proven itself to be ineffective in photo stabilizing of the wood surface. Different impregnation cycles did not influence the total color change of spruce specimens impregnated with Impralit KDS.
- Long impregnation cycle and low moisture content before impregnation ensure better moisture performance which is essential for preserving the wood.
- Good moisture performance also results in smaller tangential swelling percentage thus a better crack development performance.
- Different impregnation cycles do not have an effect on crack development nor corrosion rating of the metal fasteners.
- Corrosion of galvanized screws happened already with two weeks which seems to be in correlation with statements that impregnated wood is two times more corrosive than untreated wood.

Good performance of Norway spruce, with longer impregnation cycles and low moisture content before impregnation, could have expected as consequently these specimens had the highest preservative uptake during Impregnation. Drying of refractory wood species moisture content under the fiber saturation point before impregnation, alongside with long impregnation cycle, is very important aspect to ensure better preservative uptake and thus a better protection against weathering.

### References

- Feist, W. C. Outdoor wood weathering and protection. American Chemical Society. 1990. Researchgate (29.05.2017)
- Sandberg, D. Weathering of radial and tangential wood surfaces of pine and spruce. – *Holzforschung*. 1999, 53(4), 355-364. Researchgate (29.05.2017)
- Walker, J. C. F. Primary wood processing Principles and practice. 2<sup>nd</sup> edition. Dordrecht : Springer, 2006.
- 4. Salminen, E., Valo, R., Korhonen, M., Jernlas, R. Wood preservation with chemicals. Copenhagen : Nordic Council of Ministers, 2014.
- Thaler, N., Humar, M. Performance of selected copper amine based wood preservative supplemented with wood swelling agents. – *Wood Research*. 2012, 57 (3), 453-462. Researchgate (29.05.2017)
- Durability of wood and wood-based products. Natural durability of solid wood. Part 2: Guide to natural durability and treatability of selected wood species of importance in Europe. EN 350-2:1995.
- Durability of wood and derived materials Definition of use classes of biological attack – Part 1 General. EN 335-1:2006.
- Durability of wood and wood-based products Performance of preservatives as determined by biological tests Specification according to hazard class. EN 599-1:1997.
- 9. AS Imprest official webpage. http://www.imprest.ee/EN/services/ (09.04.2016)
- 10. Usta, I. The effect of moisture content and wood density on the preservative uptake of Caucasian fir treated with CCA. *Journal of agriculture*. 2004, 28 (1-7). Researchgate (29.05.2017)
- Lahtela, V., Hämäläinen, K., Kärki, T. The effects of preservatives on the properties of wood after modification. – *Baltic Forestry*. 2014, 20 (1), 189-203. Researchgate (29.05.2017)

- 12. Humar, M., Thaler, N. Performance of the copper treated, and naturally durable wood in laboratory and outdoor conditions. Wood is good with knowledge and technology to a competitive forestry and wood technology sector: International conference, Zagreb, 12<sup>th</sup> of October 2012. Researchgate (29.05.2017)
- Humar, M., Pohleven, F. Brown-rot decay of copper-impregnated wood. *International Biodeterioration & Biodegradation*. 2006, 58, 9-14. Researchgate (29.05.2017)
- 14. Grekin M., Lukkarinen A., Verkasalo E. Color change of Nordic Scots pine wood under UV radiation. – A laboratory approach. - 2005. http://www.metla.fi/metinfo/northernpine/docs/WP5.01.04-Grekin-et-al.pdf (29.05.2017)
- 15. Jirous-Rajkovic, V., Turkulin, H., Miller, E. R. Depth profile of UV-induced wood surface degradation. *Surface coatings international part B: Coatings transactions*. 2004, 87 (B4),235-xxx. Researchgate (29.05.2017)
- 16. Narciso, W., Eng, P. Wood preservation and its corrosive effects on metal fasteners. http://www.jrsengineering.com/documents/Articles\_Papers/Wood%20Preservati on-Metal%20Corrosion%20Paper%20-%20Wesley%20Narciso.pdf (29.05.2017)
- 17. Impralit KDS Technical Leaflet. Edition 2012.04.10.
- Härtner, H., Schmitt, S., Cui, F., Barnes, H. M. Polymeric betaine as a wood preservative. – 2008 IRG Americas Meeting Playa Flamingo, Guanacaste, Costa Rica. Researchgate (29.05.2017)
- Härtner, H., Schmitt, S., Cui, F. Impralit KDS and KDS-B. American Wood Protection Association. 2009, 105, 128-135. Reasearchgate (29.05.2017)
- Brischke, C., Humar, M., Meyer, L., Bardage, S., Van den Bulcke, J. COST Action FP 1303 – Cooperative performance test. – *Instructions for participants*. Researchgate (29.05.2017)

- 21. Field test method for determining the relative protective effectiveness of a wood preservative in ground contact. EN 252:2012.
- 22. Durability of wood and wood-based products Determination of the natural durability of solid wood against wood-destroying fungi, test methods Part 2: Soft rotting micro-fungi. CEN/TS 15083-2:2005.
- 23. Wood preservatives. Determination of the protective effectiveness of a preservative treatment against blue stain in wood in service. Laboratory method. EN 152:2011.
- 24. Jermer, J., Andersson, B.-L. Corrosion of fasteners in heat-treated wood progress report after two years exposure outdoors. - *The International Research Group on Wood Protection*. 2005, IRG/WP 05-40296.
- Plastics Methods of exposure to laboratory light sources. Part 1: General guidance. EN ISO 4892-1:1999.
- 26. Datasheet Phillips Blacklight TL-D 36W BLB 1SL/25. Edition 21.03.2017.
- 27. Eckelman, C. A. Wood moisture calculations. https://www.extension.purdue.edu/extmedia/FNR/FNR-156.pdf (29.05.2017)
- Mantanis, G. I., Young, R. A., Rowell, R. M. Swelling of wood. Part 1. Swelling in water. - *Wood science and technology*. 1994, 28, 119-134. Researchgate (29.05.2017)
- Meyer, L., Brischke, C., Treu, A., Larsson-Brelid, Pia. Critical moisture conditions for fungal decay of modified wood by basidiomycetes as detected by pile tests. – *Holzforschung*. 2015. Reasearchgate (29.05.2017)
- 30. Rosner, S., Karlsson, B., Konnerth, J., Hansmann, C. Acoustic emissions related to the dehydration stress behavior of green Norway spruce wood. http://www.ndt.net/article/jae/papers/26-014.pdf (29.05.2017)
- Carll, C. G., Highley, T. L. Decay of wood and wood-based products above ground in buildings. - Journal of testing and evaluation. 1999, 207, 150-158. Researchgate (29.05.2017)

- 32. Anagnost, S. E. Wood decay, fungi, stain and mold. New England kiln drying association spring 2011 meeting. http://www.esf.edu/nekda/documents/WoodDecay-nagnostsmallversion\_000.pdf (29.05.2017)
- Truusa L. Erinevatest puiduliikidest valmistatud laua vastupidavus väliskeskkonnas : bachelor thesis. Tallinn : Talinn University of Technology, 2015.
- 34. Brischke, C., Welzbacher, C. R., Meyer, L., Bornemann, T., Larsson-Brelid, P., Pilgard, A., Frühwald Hansson, E., Westin, M., Rapp, A. O., Thelandersson, S., Jermer, J. Service life prediction of wooden components – Part 3: Approaching a comprehensive test methodology. – *IRG 42<sup>nd</sup> Annual Meeting Queenstown, New Zealand, 2011.* Researchgate (29.05.2017)
- Cogulet, A., Blanchet, P., Landry, V. Wood degradation under UV irradiation: A lignin characterization. – *Journal of Photochemistry & Photobiology, B: Biology.* 2016, 158, 184-191. Researchgate (29.05.2017)
- 36. Teaca C.-A., Rosu D., Bodirlau R., Rosu L. Structural changes in wood under artificial UV light irradiation determined by FTIR spectroscopy and color measurements – A brief review. – *Bioresources*. 2013, 8, 1478-1507. Researchgate (29.05.2017)

# Acknowledgements

I would like to express my sincere gratitude to my supervisors Jaan Kers and Heikko Kallakas for their guidance and support during my master studies.

I would also like to acknowledge AS Imprest and Tõnis Tagel for providing the impregnated specimens for the performance test.

Last, but not least, many thanks to my loving family and friends for their kind words and support.

### Abstract

Wood is a biological material and thus a perishable material. This drawback has often considered of being its greatest disadvantages if compared to other building materials. Protective mechanism of modified timber is based on its hydrophobic character and improved moisture performance. The wood capability to take up moisture is considered to be a component of wood resistance which means that improving wood moisture performance is essential for preserving the wood. In current outdoor performance test specimens made from Norway spruce (*Picea abies*) impregnated with chromium free preservative Impralit KDS are used, which are impregnated with three different impregnation cycles. The aim of this Master's thesis was to gather and to evaluate the performance data of Norway spruce specimens with different impregnation cycles in outdoor environment. Wood decay, discoloration, development of mold and other staining fungi, corrosion of screws, formation of cracks and moisture performance were measured and evaluated.

Specimens performance data was collected at exposure site alongside with weather data from respective test site to establish relationships between climate conditions and the measurands. The test has been running for about 13 months but impregnated wood products are designed to withstand outdoor environment for much longer time than 13 months and the performance test is still ongoing which mean that current conclusions are not yet fundamental and current research should be continued.

The results showed that different impregnation cycles do not influence the total color change of spruce specimens impregnated with Impralit KDS. Specimen groups with longest impregnation cycles have better moisture performance, which is the result of greater amount of preservative forced inside the specimens. Good moisture performance also results in the smallest tangential swelling percentage thus a better crack development performance. Corrosion of galvanized screws happened already with two weeks which seems to be in correlation with statements that impregnated wood is two times more corrosive than untreated wood. Drying of refractory wood species moisture content under the fiber saturation point before impregnation, alongside with long impregnation cycle, is very important aspect to ensure better preservative uptake and thus a better protection against weathering.
## Kokkuvõte

Puit on naturaalne materjal, mis on kõrgelt hinnatud tänu oma mitmekesistele omadustele ning vastupidavusele. Sobivas keskkonnas ja piisava hooldusega suudab puit säilitada oma omadused väga pikka aega. Puit on bioloogiline materjal ja seega keskkonnas häviv materjal ning seda fakti peetakse puidu kõige suuremaks puuduseks võrreldes teiste ehitusmaterjalidega.

Käesolevas magistritöös hinnati välikatses hariliku kuuse vastupidavust väliskeskkonnas. Välikatse ajal hinnatakse puidu lagunemist, värvimuutusi, hallituse ja seente teket, kruvide korrosiooni, pragude teket ja puidu niiskus omadusi ning koos ilmastiku andmetega on võimalik teha järeldusi ilmastiku tingimuste ja katse tulemuste vahel. Harilikust kuusest katsekehad on immutatud puidukaitsevahendiga Impralit KDS, millega immuatamisel on kasutatud kolme erinevat immutamise tsüklit. Hinnatakse kuidas erinevad immutamise tsüklid mõjutavad puidu vastupidavust vananemisele.

Töö tulemused näitasid, et kõik kolm immutamise tsüklit tagasid piisavalt madala puiduniiskuse, et mitte võimaldada puitu lagundavate seente ja hallituse tekkimist. Töödeldud puidu kaitsemehhanism tugineb peamiselt vett hülgava omaduse tekitamisele ning puidu niiskus omaduste parandamisele. Puidu võime imada niiskust on üks vastupidavuse omadustest, mille vähendamine parandab puidu vastupanuvõimet vananemisele ja on oluline puidu säilitamise seisukohast. Pikem immutamise tsükkel annab puidule paremad niiskus omadused, kuna rohkem puidukaitsevahendit on tunginud puidu sisse, mis omakorda tagab väiksema paisumise tangentsiaal pindadel ning vähendab seega pragude tekkimst puidu pinnale. Välikatse ajal olid puidu- ja õhuniiskuse tingimused soodsad kruvide korrrodeerumisele. Galvaniseeritud kruvid korrodeerusid juba kahe nädalaga, mis toetab fakti, et immutatud puit on umbes kaks korda suuremate korrodeerivate omadustega kui töötlemata puit.

Pikima immutamise tsükliga ning madalaima immutamisele eelneva niiskuse sisaldusega harilikust kuusest katsekehadel olid käesolevas töös kõige paremad tulemused, mida võis ka eeldada, kuna nendes oli ka kõige suure puidukaitsevahendi kogus. Puidu kuivatamine enne immutamist puidukaitsevahendiga ning immutamise tsükli pikkus on olulise tähtsusega, et tagada puidus piisav kogus puidukaitsevahendit ning seeläbi parem vastupidavus vananemisele väliskeskkonnas.

73