Ministry of Environment and Gender Equality
Environmental Protection Agency

Wood modification as a path to phasing out biocides
Træmodifikation som vej til udfasning af biocider
MUDP-project

MUDP Report

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Projektet, som er beskrevet i denne rapport, er støttet af Miljøteknologisk Udviklings- og Demonstrationsprogram (MUDP) under Miljøministeriet, der støtter udvikling, test og demonstration af miljøteknologi.

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1. Introduction

Biocides are used in wood preservation to protect against various microorganisms such as fungi. Biocides kill or inhibit the growth of microorganisms and can become harmful to the environment, animals, and humans when they are released into the environment, for example, through leaching.

1.1 Background

Biocides are chemical components designed to kill or inhibit organisms by blocking vital metabolic processes within the organism. Biocides are used to protect e.g. surfaces and materials against pests, bacteria, fungi, and other unwanted organisms. Biocides have many beneficial properties which makes them crucial in our everyday lives, and they can be found in e.g. disinfectants, wood preservatives, and pest control.

In outdoor wood products biocides protects the wood against several kinds of microorganisms, mainly wood destroying fungi also referred to as rot or decay and wood colouring fungi also referred to as blue stain and moulds. By inhibiting the growth of these microorganisms the strength of the wood and the aesthetics are preserved leading to a longer lifetime of the wood products. However, the biocides can leach into the surrounding environment and groundwater. In the environment, biocides can affect biodiversity and impact humans with, for example, allergenic and reproductive toxic effects. [1]

1.2 Challenges and Sustainability Concerns in Wood Preservation

Today, a large part of the outdoor wood used in construction consists of pressure-treated wood with biocides, while only a few biocide-free treatments of wood are available. These methods include thermal treatment, acetylation, and furfuryl alcohol treatment. However, each of these methods has significant disadvantages, such as reduced wood strength, high energy consumption during production, and limited applicability to specific wood species. Therefore, biocides are still used in wood preservatives.

In the construction industry an increased focus on sustainability has emerged due to new legislation. From 2023, a life cycle assessment (LCA) must be made for all new buildings to determine the carbon footprint pr. m^2. Here a limit value for e.g. 1000 m^2 building is set to 12 kg CO_2 eq. pr. m^2 and this value is expected to be decreased through the years. [2] This legislation effects the use of wood products in buildings since they have a beneficial impact on the carbon footprint of a building e.g. for each ton of Portland cement substituted with wood products an average of 2.5 tons CO_2 eq. emissions is avoided [3]. With tightened legislation within the construction industry regarding the carbon footprint of a building it is expected that the use of wood products will increase resulting in increased use of biocides.

The use of biocides has gained major political, industrial, and societal focus due to their undesirable effects on humans, animals, and the environment. In 2022 the Biocidal Agreement was adopted resulting in stricter regulation and approval requirements for use of biocides and a focus on phasing out problematic substances. [4] However, phasing out problematic substances can become a problem if no alternative substances can be found. An example of this is the use of the biocide propiconazole which is used in e.g. wood preservatives. Besides its ability to protect

wood against fungi, propiconazole has potential endocrine-disrupting, carcinogenic, mutagenic, and reproductive-toxic properties. Due to this propiconazole is one of the problematic substances which is being evaluated for safety and environmental impact with the Biocidal Agreement. However, in 2023 the use of propiconazole was prolonged to 2030 due its protection properties for construction wood and wood production in the construction industry. This was decided due to no viable alternative for propiconazole being available and to give the industry time to develop a viable alternative. [5]

1.3 Project goal

The purpose of the project is to develop a biocide-free solution that demonstrates the potential to eliminate biocides in outdoor wood products within a framework that is both industrially and commercially relevant. This is achieved using the supercritical CO_2 ($scCO_2$) process, which is a state-of-the-art wood impregnation technique

The project builds on knowledge obtained in the previous project 'Impregnation of wood without fungicide' (Imprægnering af træ uden fungicid) founded by The Danish Environmental Protection Agency [6]. The project consortium consists of the Danish Technological Institute, Superwood A/S and Velux A/S.

2. Selection of substitution materials

To determine which polymers/chemicals would be suitable for substitution of biocide in wood impregnation requirement specification are made to select which polymers/chemicals would be interesting for further testing to impregnate wood using $scCO_2$. Based on the requirement specification seven polymers have been selected for further testing together with a reference biocide mix.

2.1 Requirement specification

To align expectations regarding the goal and ensure the direction of development throughout the project, requirements for, among other things, technical performance, process conditions, cost price, and environmental and health impact have been set. During the preparation of the requirement specifications, each parameter is assessed based on risk and severity, i.e., what is the risk that the specification will not be met and what is the severity if this happens. This focuses the work on achieving the most critical parameters. The process requirement for $scCO_2$ impregnation have been crucial to set up equipment (3.1) and selection of relevant chemistry (polymers) with respect to e.g. melting and boiling point. In TABLE 1 the process requirements can be seen.

TABLE 1. Requirement specification for $scCO_2$ impregnation process which selected polymers must meet. The requirements are ranked from 1 to 5 based on how important it is for the chemicals to meet the requirement, where 1 is low importance and 5 is high importance.

Area	Requirement	Importance of requirement [1 to 5]
Process time	Less than 5 hours	3
Temperature	50 to 70 °C	3
Pressure	Less than 150 atm.	3
Vapour pressure	Reagents must have a boiling point > 250 °C	3
Liquid at dosing temperature	Reagents must have a melting point < 20 °C	4
High solubility in $scCO_2$	Reagent must be soluble in $scCO_2$	3
Environmentally safe	Must not pose a hazard during process or to the environment	3
Catalyst addition	Catalyst should not give rise to unwanted side reactions	3
Easy dosing of chemicals	High pressure dosing with vaporising should be possible	2
Co-solvent	Addition of co-solvent should be < 0.5 g/(L CO_2)	3
Simple process	As few process steps as possible (1-3 steps)	3
Type of wood/substrate	Must work on scots pine (sapwood/heartwood) and spruce wood at normal humidity level of 12-18 % (non-dried wood)	5

Besides process requirements the selected polymers must also meet some product requirements for the wood product. The product requirements can be seen in TABLE 2.

TABLE 2. Requirement specification for end wood product which selected polymers must meet. The requirements are ranked from 1 to 5 based on how important it is for the chemicals to meet the requirement, where 1 is low importance and 5 is high importance.

Area	Requirement	Importance of requirement [1 to 5]
Adhesion	Glue or paint should have unaltered properties and curing on the modified wood surface.	2
Stability	Should be stable during further refinement of wood product e.g. no corrosion, decay or discolouring.	5
Water content and uptake	Wood should be treated at normal humidity level of 12-18%	3
UV protection	Modification should protect against long-term UV exposure.	5
Mechanical properties	Strength and flexibility should be retained or enhanced.	5
Dimensional stability	Dimensional stability should be increased relative to un-modified wood and have increased water repellent properties.	3
Thermal insulation	Important to retain or improve in window applications.	3
Blue stain	Important not to observe visual appearance	5
Mould growth	Mold growth should be avoided	1
VOC	Outgassing should be avoided	5

Lastly the processing of wood products in $scCO_2$ with the selected polymers must be economically viable. The requirements for the costing of the processing are shown in TABLE 3.

TABLE 3. Requirement specification for costing which selected polymers must meet. The requirements are ranked from 1 to 5 based on how important it is for the chemicals to meet the requirement, where 1 is low importance and 5 is high importance.

Area	Requirement	Importance of requirement [1 to 5]
Price of chemicals	Should be reasonable when comparing to current solution (Maximum of 20 % more cost then current solution)	4
Price of final product	Maximum of 20% should originate from modification process	4

Based on these requirements the polymers are selected which are then used for wood impregnation and further testing.

2.2 Selection of polymers

The selection of polymers has been done based on the requirement specification set in section 2.1 and through dialog with chemicals suppliers to identify polymers which makes chemical and environmental sense to test in an impregnation process with $scCO_2$ as the solvent. The selected polymers are subsequently used for wood impregnation and tested to determine if they can act as alternatives to biocides.

In dialogue with various chemical suppliers, it has been made clear that the identified polymers should have the desired chemical properties described in the requirement specifications (section 2.1). Especially a qualitative assessment of whether the chemicals are soluble in $scCO_2$ was critical in the selection of the polymers.

TABLE 4. Investigated polymers for wood impregnation in $scCO_2$ and reference chemical.

Polymer	Type
A1	Silicone oil, for aq. Microemulsion
A2	"Silicone fluid", for aq. Microemulsion
A3	Paraffins, zirconium acetate, acetic acid
B1	Non-ionic silicone polyether
B2	Alkyl-functionalized, low viscosity silicone resin
C1	For aq. microemulsion, acetic acid
C2	Silanes, siloxanes
C3	Silanes, siloxanes, Sn catalyst
D1	EO-PO-EO diol
D2	EO-PO-EO diol
Reference	Biocide

The polymers are intended for chemical modification of wood where a model substrate can be used if necessary. The model substrate is starch, which contains hydroxyl group like hemicellulose in wood and it has been shown in literature reports that it can be modified with e.g. acetic acid anhydride in $scCO_2$. Chemicals meant for covalent modification of hydroxyl groups should be screened on the model substrate for quickly determining whether the chemistry is feasible. The selected polymers do not form covalent bonds which form well defined structures that are easily identified or quantified by e.g. IR-spectroscopy. Instead, they can crosslink or form hydrogen bonds to the cellulosic structure. Therefore, the polymers are tested directly on wood samples to see if they have hydroscopic and anti-fungal effects.

In TABLE 4 10 polymers and one reference chemical are shown, and these polymers has been selected based on the requirement specification listed in section 2.1. However, polymers A1 and A2 were never received from the supplier which is why no further testing has been done using these two polymers. Polymer A3 was received but it was solid at room temperature meaning that it is not suitable for wood impregnation using $scCO_2$ since a requirement is that the substance must be liquid at dosing temperature < 20 °C, as stated in TABLE 1. Therefore the 10 polymers were reduced to 7 which is listed in TABLE 5.

TABLE 5. Selected polymers and reference chemical for further testing for wood impregnation using $scCO_2$.

Polymer	Type
B1	Non-ionic silicone polyether
B2	Alkyl-functionalized, low viscosity silicone resin
C1	For aq. microemulsion, acetic acid
C2	Silanes, siloxanes
C3	Silanes, siloxanes, Sn catalyst
D1	EO-PO-EO diol
D2	EO-PO-EO diol

Polymer	Type
Reference	Biocide

3. Development of modification chemistry in $scCO_2$

To investigate if the selected 7 polymers can be used for impregnation a lab-scale $scCO_2$ impregnation reactor are constructed. This is done to create a screening process to determine which polymers should be used for wood impregnation at the pilot plant. Due to safety reasons the first lab-scale setup (flow setup) was discarded, and a batch setup was constructed instead. However, it turned out that the screening of the polymers could be conducted at the same pace and on larger wood samples in the pilot reactor. Therefore, the impregnations were moved to the pilot plant setup and afterwards the impregnated wood blocks were sent for analysis.

3.1 Super critical CO_2

To control the degree of modification of wood with different chemicals, it is necessary to control key reaction parameters in the $scCO_2$ setup, e.g., pressure and temperature. The critical point for carbon dioxide is 73.8 bar and 31 ˚C. The setup was designed to maintain an operating pressure of 150 bar and 70˚C to mimic those use in the industrial impregnation plant. In FIGURE 1 a phase diagram for CO_2 is shown, where the super critical area is shown. When CO_2 reaches the supercritical stage, it behaves both as a gas and a liquid, allowing for high penetration of wood by solvents that are soluble in CO_2.

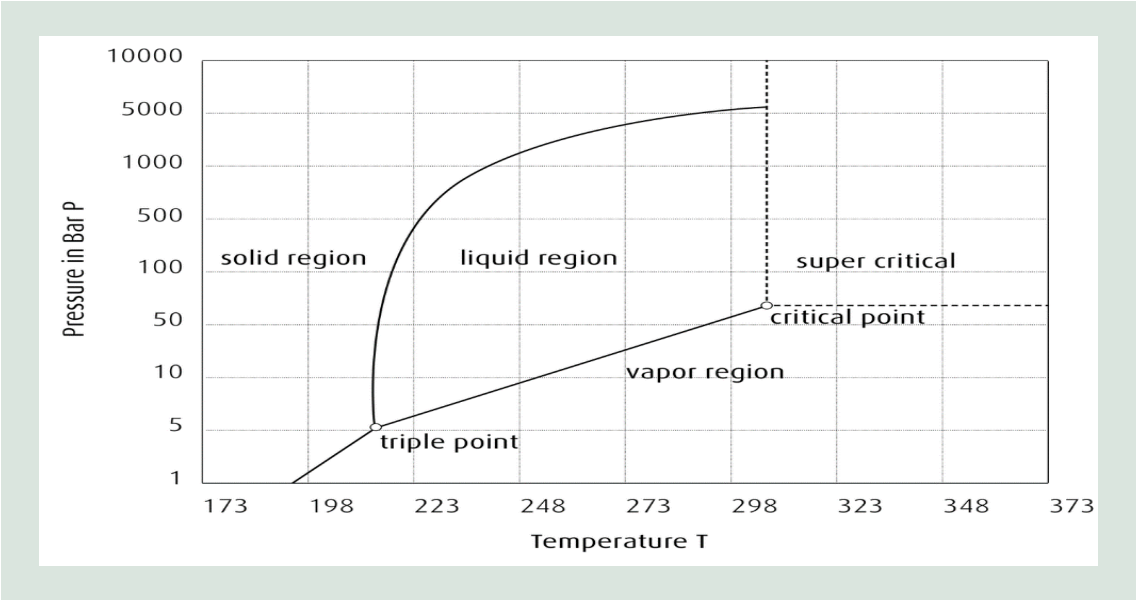

FIGURE 1. Phase diagram of CO_2. Temperature in K.

3.2 Process setup for modification chemistry in $scCO_2$

To evaluate the selected polymers a test setup is constructed. This configuration necessitates a series of unit operations, comprising a CO_2 source, cooling system, pump mechanism, preheating unit, reactor vessel, reactor heating element, pressure release mechanism, and associated safety systems (e.g., pressure relief valves and rupture discs). It is of paramount importance to maintain the CO_2 in the appropriate phase throughout the respective unit operations. For instance, the CO_2 should be cooled to 5 °C at the bottle pressure of 55 bar to ensure its liquid state prior to entering the liquid pump system.

Additionally, installing CO_2 sensors near the equipment is essential for detecting potential leaks, thereby enhancing the overall safety of the experimental setup.

In the project, two different equipment setups have been used. For simplicity, one setup will be referred to as a flow setup and the subsequent setup as a batch setup. The impregnations and experiments are designed to quickly screen chemicals and other impregnation agents to determine which should be used on the larger $scCO_2$ pilot scale reactor. In FIGURE 2 the two equipment setups can be seen.

FIGURE 2. The two equipment setup. The one on the left shows the flow setup and the one on the right shows the batch setup.

One goal has been to identify a model substrate where any covalent modifications could quickly and simply be identified via characterization methods (e.g. infrared spectroscopy or nuclear magnetic resonance spectroscopy) and the model substrate simultaneously reflects the chemistry in wood as closely as possible. Ultimately, starch was chosen as model substrate as it is chemically similar and easily can be characterized by infra-red spectroscopy. While starch can serve as a model substrate, it does not fully represent the complex structure and heterogeneity of wood. Wood also contains lignin, extractives, and other components that can influence the reactivity and accessibility of targeted hemicellulose structures.

Initially, modification chemistry from the literature was reproduced in $scCO_2$. However, the results were not reproducible in the applied flow setup. Furthermore, different chemicals for starch modification in $scCO_2$ were tested, but without success.
During the execution of the experiments, it became clear that the applied flow setup had several disadvantages, including safety concerns, time-consuming setup, and limited possibilities for stirring and temperature control (inside the reaction chamber). Based on these drawbacks, a

new experimental equipment for a batch setup was designed. With the installation of the new batch setup, several of the disadvantages were resolved.

For the batch setup the selected polymers are to be tested on small wood samples. In FIGURE 3 the storage of wood blocks before and after impregnation is shown Wood absorbs or releases moisture depending on the surrounding environment. By storing the wood samples at a fixed relative humidity allows for more accurate and reproducible measurements of their mass.

FIGURE 3. Storage of wood blocks before and after impregnation with selected polymers. The storage conditions are fixed at 59 % relative humidity using a saturated sodium bromide (NaBr) solution.

When using the batch setup, the reactor is loaded with wood samples. The loading of the reactor is shown in FIGURE 4.

FIGURE 4. Loading process of the reactor before impregnation of wood in $scCO_2$.

After loading the batch setup, the impregnation process can begin, where the reactor is placed in a heating element as shown in FIGURE 5.

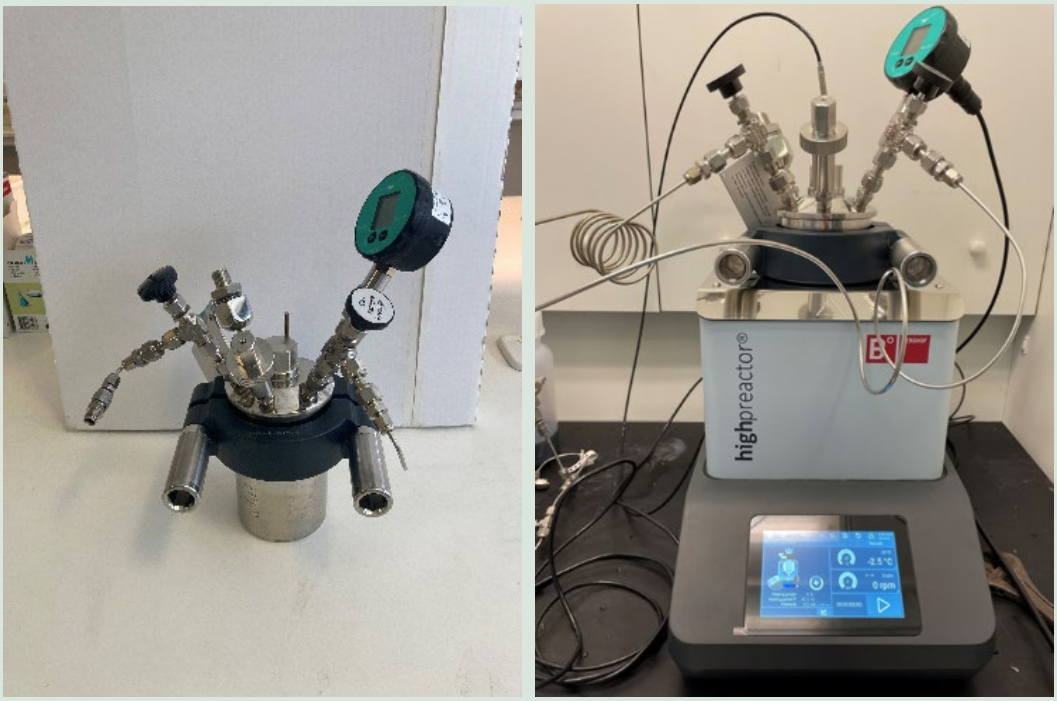

FIGURE 5. The test setup for the batch reactor.

Due to time constraints in the project, it was prioritized to test on wood with polymers identified in 2.2. In the batch reactor several $scCO_2$ impregnations were conducted A conventional biocide mix was used as described above providing similar content to that obtained in the pilot reactor. However, the buildup of pressure was irregular in the batch reactor and since the screening of the polymers could be conducted at the similar pace and on larger wood samples in the pilot reactor the impregnations were moved to the pilot plant setup and afterwards the impregnated wood blocks were sent for analysis.

4. Impregnation of wood in $scCO_2$

Seven different polymers have been selected for wood impregnation in scCO2 where different dosages of polymer have been tested, as well as the introduction of additives. To give an indication of whether the penetration of polymer into the wood has succeeded, the weight gain after treatment has been measured before the treated samples are sent for further testing.

4.1 $scCO_2$ impregnation of wood using polymers

The selected polymers seen in TABLE 4 have been tested using $scCO_2$ impregnation. The aim of these experiments was to evaluate the potential of each polymers ability to improve the wood's decay resistance without the use of biocides. To determine whether penetration of polymer has occurred in the wood, weight gain was measured before additional testing was performed. The results of the additional testing are presented in section 5.4. This method has the potential to reduce the need for biocides, especially fungicides, by creating a barrier against water penetration, which is a primary cause of fungal growth in wood.

The polymers have been tested on spruce and pine wood.

4.1.1 $scCO_2$ Impregnation process

The $scCO_2$ impregnation process involves careful control of pressure and temperature, which are crucial for achieving the desired supercritical state. FIGURE 6 details the sequence of pressure and temperature setting and relief, which is an essential part of the process. The impregnation process itself can vary in duration from 3 to 5 hours depending on the dimensions of the wood.

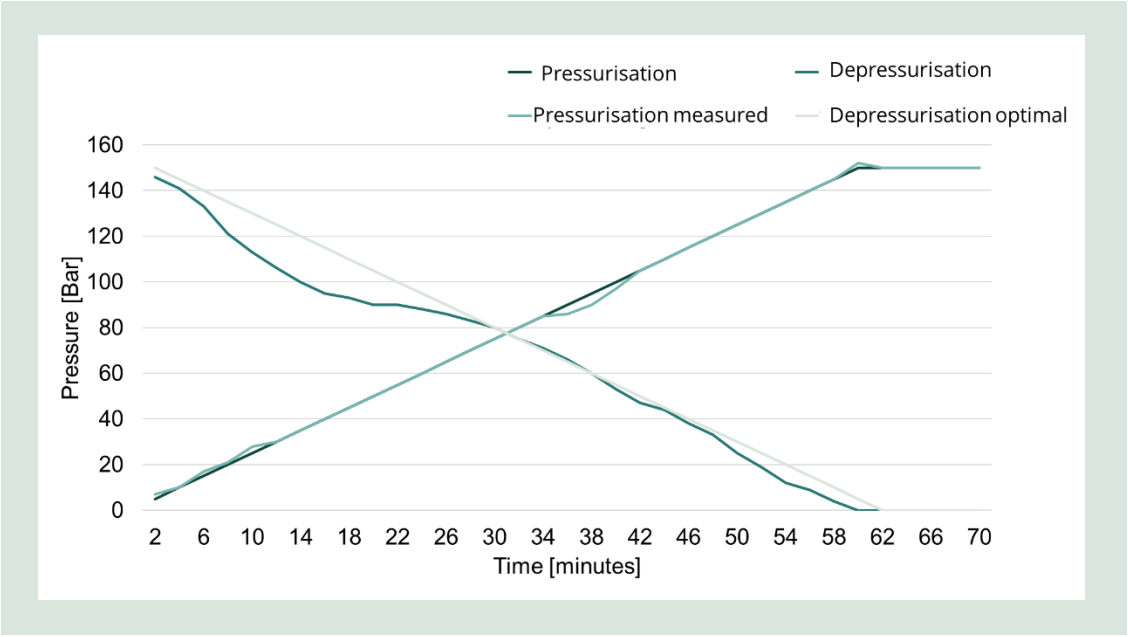

FIGURE 6. Pressure and time for $scCO_2$ impregnation process.

Furthermore, the installation of CO2 sensors near the equipment is essential to detect potential leaks, thereby enhancing the overall safety of the experimental setup. Once this state is achieved, the parameters are kept stable to ensure effective penetration of the polymers into the wood. After impregnation, there is a controlled release of pressure and temperature to conclude the process. This precise control is necessary to ensure that the polymers are correctly and evenly deposited within the wood structure.

4.1.2 Results of wood impregnation using polymers

The results of the impregnation experiments show the effectiveness of various polymers. TABLE 6 summarises the results for weight gain of EN113 wood blocks and spruce after impregnation with different polymers at 10% dosages.

TABLE 6. Treatment series 1: Weight gain results after being impregnated by different polymers. A negative result indicates weight loss, and a positive result indicates weight gain (WPG: representing the ratio of the amount of chemicals added in wood on the mass of wood before impregnation). Some uncertainty is related to these results due to water evaporation during the $scCO_2$ impregnation process.

Polymer	Dosage	EN113 blocks [WPG %]	Spruce wood [WPG%]
B1	10 %	1.82	0.61
B2	10 %	0.20	-0.07
C1	10 %	1.04	0.64
C2	10 %	1.55	0.80
C3	10 %	-0.09	-0.46
D1	10 %	2.01	-0.18
D2	10 %	3.15	-1.22

To make the process more economical feasible the dosage of B1 and D1 is decreased to 2 %. B1 and D1 is selected based on their weight percent gain in EN113 blocks and spruce wood.

Furthermore, two different additives are introduced to investigate if they will affect decay resistance of the treated wood samples. The two additives were selected because of observed synergistic effect when used with other wood preservative products. The results are summarised in TABLE 7.

TABLE 7. Treatment series 2: Weight gain results after being impregnated by different polymers. A negative result indicates weight loss, and a positive result indicates weight gain (WPG: representing the ratio of the amount of chemicals added in wood on the mass of wood before impregnation). Some uncertainty is related to these results due to water evaporation during the $scCO_2$ impregnation process. *) Weight gain measured after 24 hours.

Polymer	Dosage	EN113 blocks [WPG %]	Spruce wood [WPG %]
B1-1 *)	2 %	2.37	-1.42
D1-1	2 %	5.71	-1.87
B1-2	2 % + 0.4 % additive 1	2.31	-1.95
B1-3	2 % + 0.1 % additive 2	2.76	-2.32

The results found in TABLE 6 and TABLE 7 shows that certain polymers, particularly polymeric siloxanes (B1,B2,C1,C2, and C3) and ethylene/propylene oxide (D1 and D2) polymers, have high solubility in $scCO_2$. These properties make them promising candidates for wood impregnation since $scCO_2$ can deeply penetrate the wood and evenly deposit the polymers. It should be noted that there is some uncertainty associated with the weight gain measurements due to water loss from the wood during the impregnation process.

All blocks with a weight gain % higher than 1.5 % has been sent for further testing. This limit has been decided since a lower amount of such chemicals in the wood would most likely barely influence wood properties. The further testing is described in section 5.

4.1.3 Conclusion

Although positive effects of polymer treatment have been observed, based on the observed weight gain for some of the polymers, there is still uncertainty regarding weight gain due to water loss during the impregnation process. Therefore, it is necessary to conduct more trials with larger spruce samples to determine how the polymers penetrate the wood and their long-term effects.

Further research is required to understand the precise mechanisms and conditions affecting polymer effectiveness. This will include both laboratory and field tests to ensure that the method can be widely and effectively applied to different types of wood and under various climatic conditions. These types of experiments are planned to be carried out after the project have ended and will provide important data that can help optimise the impregnation process and document its effectiveness as a biocide-free method for wood protection.

5. Evaluation of impregnated wood

The polymers used for wood impregnation have been investigated for decay resistance, hygroscopicity, and chemical distribution within the treated wood samples. The results show that three out of the seven tested polymers have some decay protection.

5.1 Decay resistance

Biocides are used for wood protection against microorganisms of several kinds, mainly wood destroying fungi or wood colouring fungi.

Wood colouring fungi, mainly referred to as blue stain and moulds, are growing in the wood structure, feeding on organic materials like starch or other potential nutrients stored in wood cells and are not attacking the wood polymers, namely cellulose, hemicelluloses and lignin. Therefore, wood colouring fungi represent mainly an aesthetical disorder since their hyphae contain melanin which is visible as dark blue/grey stains in wood. Wood colouring fungi also lead to an increase in wood impregnability, since they tend to open pathways otherwise blocked by stored nutrients in the wood tracheids. However, they do not represent a short-term threat regarding wood strength.

Wood destroying fungi, mainly referred to as rot or decay, on the other hand, feed on the wood polymers which leads to significant loss in strength of the wood material attacked. For this reason, it has been decided to assess wood resistance to wood destroying fungi after treatment with the selected polymers.

Due to the long-term exposure of wood to fungi inherent to this evaluation (16 weeks according to EN113), it was decided to follow a test method developed by Bravery [7], which involves using smaller wood specimens while maintaining the volume/surface ratio, thereby reducing the exposure duration to 6 weeks. This deviation from standard will be kept in mind for results interpretation, since the specimens' size might impact the treatment distribution, and time is an important factor in biological resistance evaluation.

5.2 Hygroscopicity

The necessary conditions for wood-destroying basidiomycetes to attack and grow on wood material include sufficiently high moisture content in the wood, appropriate temperature, and the presence of nutrients. The optimal moisture content for decay fungi development is between 35 % and 50 %, but it is possible from 18-20 %. The optimal temperature range for decay fungi development is between 25 and 35 °C, but it is possible from 20°C.

One possible approach to avoid wood destroying basidiomycetes is to modify the equilibrium moisture content of wood by chemical treatments. To do this the moisture uptake must be reduced enough to keep the equilibrium moisture content below 18-20 % at all times. The evaluation of the wood hygroscopic properties after treatment is therefore very informative and will be used as an evaluation parameter of the selected polymers for wood impregnation.

5.3 Materials and methods

In this section the methods for evaluation of impregnated wood samples with the selected polymers seen in TABLE 5 are described. The impregnated wood samples are evaluated based on decay resistance and hygroscopicity. Furthermore, the chemical distribution of polymer is investigated to evaluate the given polymer's ability to penetrate the wood samples.

5.3.1 Materials

Wood material

The wood material is Scots pine sapwood sourced from Danish Technological Institute.

Chemicals

The chemicals are listed in section 2.2.

5.3.2 Methods

Decay resistance evaluation

The assessment of decay resistance has been carried out following the guidelines of the EN113-2:2020 [8], however, several adaptations has been implemented.

The wood samples dimensions and duration of exposure to fungi have been adapted to L30xT15xR5 mm (screening blocks) instead of L50xT25xR15 mm and 6 weeks instead of 16 weeks, following Bravery's guidelines [7]. No pre-ageing of the wood treated samples has been carried out before exposure.

The decay procedure is as follows:

1. Per treatment variant, 10 samples have been used for assessment. 6 for the assessment of decay resistance, 2 for the calculation of correction factor and 2 for the gravimetric measurement of moisture content.
2. All samples were conditioned at 20 °C and 65 % relative humidity (RH) until constant mass.
3. Samples for exposure to decay and for the calculation of the correction factor are sterilized by gamma radiation.
4. Samples for exposure to decay are aseptically placed on grown mycelium of *Coniophora puteana* in petri dishes casted with malt-agar culture medium. Samples for calculation of correction factor are aseptically placed on malt-agar culture medium, non-inoculated with fungi.
5. Petri dishes are place in climate chambers set at 23 °C and 75 % RH for incubation over 6 weeks.
6. At the end of the 6 weeks exposure, wood samples are taken out of Petri dishes, brushed to eliminate mycelium materials adhering on the surface, and weighed. The wood samples are weighed again after 48 hours drying at 103°C in the oven.

The mass loss due to decay is calculated from the final wood mass after exposure. The correction factor is subtracted from the calculated dry wood mass (see TABLE 6 and TABLE 7) before exposure. The correction factor represents the mass variation of wood samples that are not exposed to fungi but stored on the non-inoculation culture medium in the same conditions for the same amount of time. This is done to take in account any potential mass change due to other factors that fungi decay. Pictures are taken all along the procedure to document the test.

Hygroscopicity

The assessment of hygroscopicity was carried out by exposing 10 to 15 wood samples per variant, treated and not treated, to high RH until constant mass.

The procedure is as follows:

1. Treated and untreated samples are conditioned to 20 °C and 65 % RH until constant mass.
2. Mass and dimensions of samples are recorded.

3. Samples are exposed to a high humidity environment (ca. 95-98 % RH) at stable temperature until constant mass. Sample mass is recorded over time.
4. Mass and dimensions of samples are recorded.

The hygroscopicity test allows to determine the equilibrium moisture content (EMC) of the wood samples. Because mass is recorded over time, it is possible to compare the kinetics of water vapor absorption of samples of all kinds.

Chemicals distribution observation

To evaluate the diffusion of treatment polymers in the wood structures, spruce samples of 20x50x150 mm were cut to reveal a cross-section (transverse cut) for observation at SEM-EDX (scanning electron microscopy with energy dispersive X-rat spectroscopy), which allowed element identification. Silicon-containing chemicals allowed for observation of the silicon (Si) in the wood samples. The same observation has been carried out on Scots pine specimens of 5x15x30 mm after decay exposure. Only qualitative analysis was conducted in the frame of this project by observing the Si distribution in the wood structure.

5.4 Results and discussion

The polymer treated wood samples has been divided into two treatment series. Treatment series 1 contains treated wood samples with the polymers shown in TABLE 6 whereas treatment series 2 contains the treated wood samples with polymers shown in TABLE 7.

5.4.1 Selection of polymers for decay resistance and hygroscopicity evaluations

Weight percent gain

The weight percent gain results shown in TABLE 6 for treatment series 1 and in TABLE 7 for treatment series 2 the polymer treated wood samples are approximate values where the moisture content has been undefined. For the selection of polymers for further evaluation the weight percent gain has been adjusted where the wood moisture content has been considered. This has been done for the EN113 blocks and for the screening blocks described in section 5.3.2. It has not been done for the spruce wood samples since the chemical impregnation is very low, which can be explained by the combined influence of the species anatomical features and the specimens' dimensions. The adjusted weight percent gain can be seen in TABLE 8.

TABLE 8. Treatment series 1: Weight percent gain for where wood moisture content has been considered. WPG: representing the ratio of the amount of chemicals added in wood on the mass of wood before impregnation. Here the results are shown for the EN113 and for screening blocks which are also pine wood but with smaller volume.

Polymer	EN113 blocks [WPG %]	Screening blocks [WPG %]
B1	3.3	2.0
B2	-0.4	-0.9
C1	0.3	-0.4
C2	3.2	0.6
C3	0.1	-0.2
D1	4.1	3.6
D2	N/D	N/D

In the wood samples different uptake of the polymers can be seen. A higher uptake is noticed in smaller specimens. Wood samples which have a weight percent gain higher than 1.5 % has been selected for further evaluation since a lower amount of polymer present in the wood most likely not influence wood properties.

For decay resistance testing the weight percent gain for EN113 blocks has been used to determine which wood sample should be exposed to decay. Here B1, C2, and D1 have a weight percent gain higher than 1.5 %. It was not possible to obtain adjusted values for D2 but the results in TABLE 6 shows a weight percent gain of 3.15 %. Therefore, D2 is also included for decay resistance testing. The decay resistance testing is however performed on the screening blocks due to reasons described in section 5.3.2.

For Hygroscopicity all seven polymer treated wood samples have been exposed to various relative humidities to identify if an effect could have been overlooked.

The same adjustments for weight percent gain have been made for treatment series 2. The adjusted weight percent gain for treatment series 2 can be seen in TABLE 9.

TABLE 9. Treatment series 2: Weight percent gain for where wood moisture content has been considered. WPG: representing the ratio of the amount of chemicals added in wood on the mass of wood before impregnation. Here the results are shown for the EN113 and for screening blocks which are also pine wood but with smaller volume.

Polymer	EN113 blocks [WPG %]	Screening blocks [WPG %]
B1-1	1.92	4.10
D1-1	2.22	1.43
B1-2	1.93	3.32
B1-3	1.92	4.10

As seen in TABLE 9 all four polymer treated samples have a weight percent gain higher than 1.5 % and therefore all samples are exposed to decay. The EMC of all samples is also evaluated.

Chemicals distribution in wood

To investigate if the polymers have been able to diffusion into wood or if it is simply present on the surface of the sample the chemical distribution has been evaluated. This has been done as a qualitative analysis investigating of Si could be observed in the samples. This has been done for the EN113 and the spruce wood blocks. The results can be seen in TABLE 10.

TABLE 10. Identification of Si in different wood samples treated with different polymers. *Significant amount of Si is found in samples showing low mass loss due to decay, while samples showing high mass loss also display a lower amount of Si in the wood structure after decay.

Polymer	Chemistry	EN113 blocks	Spruce wood
B1	Si-containing	Traces	Traces at the specimen core. Significant down to a depth at least 1 cm.
B2	Si-containing	N/D	Traces at the specimen sore.
C1	Si-containing	N/D	Traces at the specimen core. Low down to a depth at least 1 cm.
C2	Si-containing	Significant *	Significant down to core.
C3	Si-containing	N/D	Traces at the specimen core. Medium down to a depth at least 1 cm.
D1	CHO	Traces	Traces

Polymer	Chemistry	EN113 blocks	Spruce wood
D2	CHO	Traces	Traces

As reported in TABLE 10 polymer C2 has shown a good diffusion into wood, even in Spruce specimens. B1 does not seem to impregnate the whole specimen homogeneously. C1,C2, and C3 show medium and low amount, and only traces of Si in the wood structure, respectively. D1 and D2 showed traces of Si as well even though the polymers are CHO-based, which could result from some pollution during the treatment process. These observations are in accordance with weight percent gain values measured.

5.4.2 Decay resistance evaluation

Treatment series 1

The exposure of treatment series 1 was conducted between the 23/01-2024 and the 05/03-2024. In FIGURE 7 the progress of the decay resistance test for D2 can be seen during the 6 weeks.

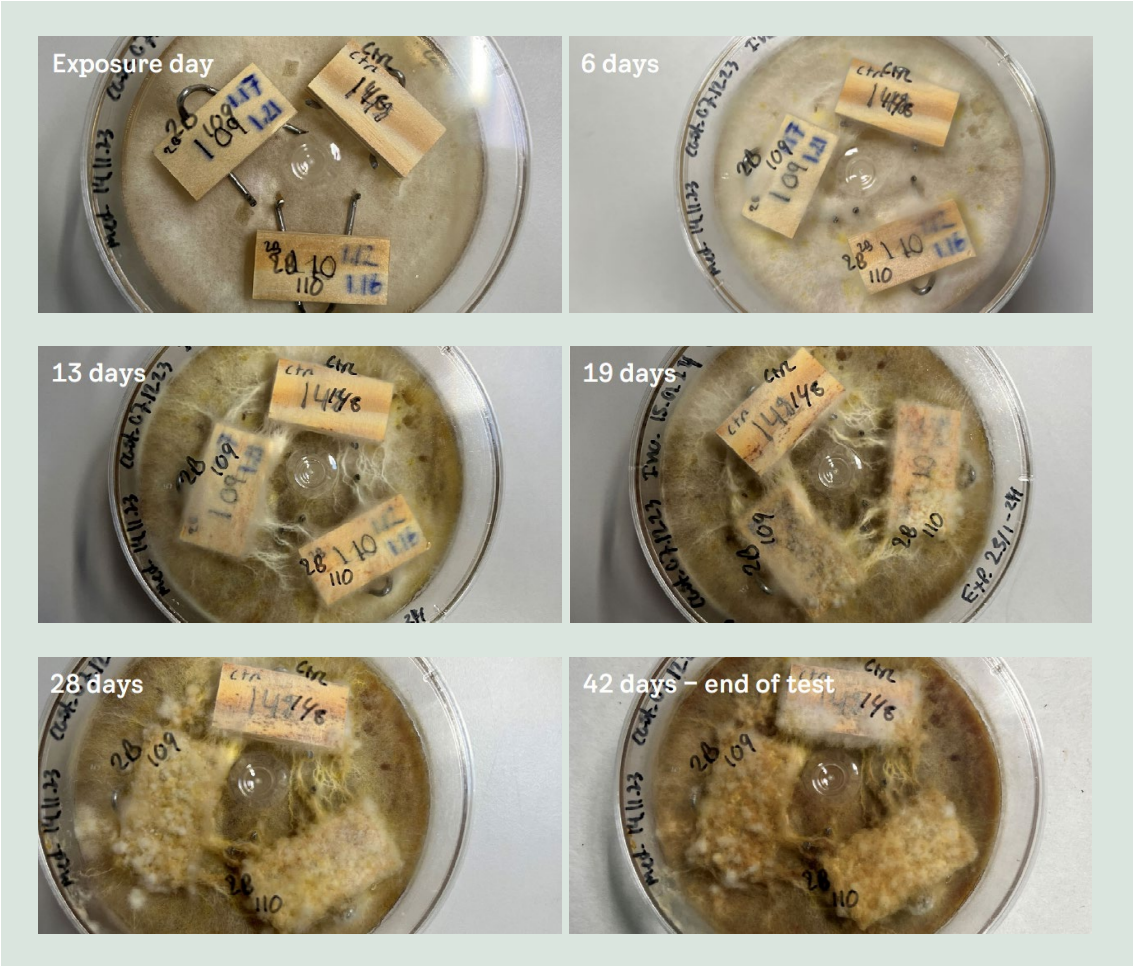

FIGURE 7. Progress of decay resistance test for D2 treated wood samples and a control sample during the 6 weeks. The controls sample are untreated samples that were exposed together with treated specimens in the same petri dish. There are 3 specimens per Petri dish (2 treated and 1 untreated).

The mass loss values after 6 weeks decay exposure for each of the four polymers are displayed in TABLE 11.

TABLE 11. Mass loss values after the polymer treated samples has been exposed to decay for 6 weeks. Control samples (untreated samples) were exposed to decay as well control if the polymers had any effect. The treated samples mass loss values are displayed with mention on how many samples were considered representative of the test due to observed waterlogging. The untreated virulence controls are untreated specimens that are exposed to fungi separately to determine the fungi virulence at the time of the test.

Polymer	Mass loss [%]		Waterlogging observed on:
	Treated samples	Control samples	
B1	15.5 (3 samples)	29.3	3/6 samples
C2	9.4 (5 samples)	30.1	5/10 samples
D1	10.9 (1 sample)	32.5	5/6 samples
D2	28.5 (9 samples)	25.8	1/10 samples
Untreated virulence control	23.7 (9 samples)		3/12 samples

The interpretation of the results is complicated by significant waterlogging phenomenon. Waterlogging means that the wood specimen moisture content increases during the exposure to a level where fungi growth is made impossible due to scarce oxygen availability.

The samples presenting water logging usually show a very low mass loss, that cannot be interpreted as conferred resistance from the treatment with polymer. Some samples were more impacted by water logging than others (i.e. 5 out of 6 specimens treated with treatment D1, whereas only 1 out of 10 specimens treated with treatment D2). The interpretation of mass loss is then sounder for some samples than for others. Because some virulence samples (untreated) were waterlogged as well, it does not seem to be an effect of the chemicals, but rather a combined consequence of the test set-up and the wood samples. In this set-up water logging is observed from time to time, but rarely to such an extent as seen in TABLE 11. For further evaluations, it might be beneficial to expose a significantly higher number of polymer-treated wood samples.

With 23.7 % virulence, the fungi strength is considered acceptable for a 6-week exposure. By comparing the mass loss of treated samples with the mass loss of both control and virulence specimens, it can be concluded that treatment D2 confers no resistance to decay, whereas treatments B1, C2, and D1 seem to show some effect.

Treatment series 2

The exposure of treatment series 2 was conducted between the 03.05.2024 and the 26.06.2024. The mass loss values are displayed in the table below.

TABLE 12. Mass loss values after the polymer treated samples has been exposed to decay for 6 weeks. Control samples (untreated samples) were exposed to decay as well control if the polymers had any effect. The untreated virulence controls are untreated specimens that are exposed to fungi separately to determine the fungi virulence at the time of the test.

Polymer	Mass loss [%]	
	Treated samples	Control samples
B1-1	19.6 (10 samples)	34.2
D1-1	33.6 (10 samples)	33.6
B1-2	19.9 (10 sample)	34.2
B1-3	24.8 (10 samples)	28.0
Untreated virulence control	28.9 (14 samples)	

The treated samples mass loss values are displayed with mention of how many samples were considered representative of the test. There was no water logging effect observed in these trials. The untreated virulence controls are untreated specimens that are exposed to fungi separately to determine the fungi virulence at the time of the test.

With 28.9 % virulence, the fungi strength is considered acceptable for a 6-week exposure. By comparing the mass loss of polymer treated samples with the mass loss of both control and virulence specimens, it can be concluded that treatment B1-3 and D1-1 do not confer any resistance to decay, whereas treatments B1-1 and B1-2 only have a very limited effect.

5.4.3 Hygroscopicity evaluation

Treatment series 1

The EMC of polymer-treated wood samples for all seven selected polymers, after stabilization at high relative humidity (close to 100 % RH) and after soaking in liquid water at room temperature, is displayed in TABLE 13.

TABLE 13. Equilibrium moisture content (EMC) for polymer-treated wood samples and mass uptake of water after water soaking. Control samples was used for comparison to investigate the polymer treatment effect on these parameters.

Polymer	EMC at 100 % RH	WU water soaking
B1	27.5 % ± 0.41 %	98.7 % ± 6.71 %
B2	28.2 % ± 0.82 %	105.9 % ± 7.36 %
C1	27.6 % ± 0.65 %	94.3 % ± 4.89 %
C2	26.2 % ± 0.55 %	97.8 % ± 5.91 %
C3	28.5 % ± 0.39	93.6 & ± 6.54 %
D1	26.7 % ± 0.41 %	98.7 % ± 6.71 %
D2	27.0 % ± 0.41 %	100.6 % ± 6.22 %
Control samples	28.0 % ± 0.77 %	104.2 % ± 7.44 %

There is no EMC reduction conferred by polymer B2 and C3, in comparison to untreated control samples when exposed to high relative humidity. Polymer B1, C1, C2, D1 and D2 shows a slight decrease in EMC compared to the control samples.

Regarding the mass uptake at saturation, treatments C1 and C3 seem to show the biggest effect, while treatments B1, B2, and D2 are not showing any effect on water uptake.

Treatment series 2

The EMC of polymer-treated wood samples of all seven selected polymers after stabilization at high relative humidity (close to 100 % RH) can be seen in TABLE 14.

TABLE 14. Equilibrium moisture content (EMC) for polymer-treated wood samples. Control samples was used for comparison to investigate the polymer treatment effect on these parameters.

Polymer	EMC at 100 % RH
B1-1	28.3 % ± 0.65 %
D1-1	28.9 % ± 1.04 %
B1-2	28.2 % ± 0.73 %
B1-3	28.6 % ± 0.64 %

Chemicals evaluated in treatment series 2 did not lead to any reduction in EMC when wood is exposed to high relative humidity. For the time of reporting, the soaking of polymer-treated wood samples in water was still in progress and therefore these results have not been included in the reporting.

5.5 Treatment series 1 – result summary

In treatment series 1, four of the seven different polymer types shown in TABLE 6 were selected for decay resistance evaluation based on the chemical weight uptake. Out of the 4 selected polymers, one polymer (D2) does not confer any decay protection, whereas three polymers (B1, C2, and D1) lead to some decay protection (mass loss values from 9 % to 16 %).

The four selected polymer treated wood samples show a decrease in EMC of ~4 % in average in comparison to untreated specimens' EMC, which leads to $EMC_{\sim 100\ \%\ RH}$ of 27 % in average for the four selected samples. The selected polymers do not seem to provide a high enough decrease in EMC to ensure a durable and significant impact on fungal growth if EMC decrease is the only mode of action, as shown by mass loss due to decay.

From the fungi growth observation during decay exposure, and from the mass loss due to decay, it can be concluded that the four selected polymers do not seem to have fungicidal effect.

Polymers B1, C2, and D1 form treatment series 1 seem to be the more promising treatments to pursue with.

5.6 Treatment series 2 – result summary

For treatment series 2 all four polymers shown in TABLE 7 were selected (B1-1, D1-1, B1-2, B1-3) for decay resistance evaluation and EMC. There is no significant reduction in EMC conferred to wood by any of treatment series 2 polymers, nor significant conferred decay resistance.

6. Environmental and health assessment

For the selected polymers an initial environmental and health assessment has been conducted. This has been done based on the safety data sheets for the selected polymers to evaluate the impact on the environment and human health compared to biocides.

6.1 Environmental Assessment

The initial environmental assessment of the various chemicals was conducted by evaluating the safety data sheets (SDS) for the identified polymers and chemicals used for wood impregnation. The identified polymers and chemicals can be seen in TABLE 4.

TABLE 15 provides an overview of the environmental impacts of various substances used in the wood modification process. These substances include silicone resins, surfactants, antioxidants, and other additives.

TABLE 15. Environmental impact based on hazard statements found in SDS for polymer and reference biocide in concentrated form.

Polymer	Environmental Impact
B1	Harmful to aquatic life
B2	Potential aquatic toxicity May form formaldehyde vapours when heated
C1	Potential aquatic toxicity Limited biodegradability
C2	Potential aquatic toxicity Limited biodegradability
C3	Potential aquatic toxicity Releases methanol and ethanol upon hydrolysis
D1	Limited biodegradability
D2	Limited biodegradability
Additive 1 in B1-2	No significant impacts identified
Additive 2 in B1-3	Aquatic toxicity Bioaccumulative potential
A3	Potential aquatic toxicity
Reference	Toxic to aquatic life with long lasting effects

Many of the substances, such as C1 and C2 pose potential aquatic toxicity risks. Some, like the additive in B1-3 have bioaccumulative potential. Several products, particularly the silicone-based ones, show limited biodegradability.

When comparing the hazard statements of the polymers and additives investigated as biocide substitutes to the reference biocide, the investigated polymers may be harmful or potentially

toxic to aquatic life and have limited biodegradability. However, it does not seem that the investigated polymers are a harmful to the environment in the same degree as the reference biocide.

This preliminary assessment highlights the importance of careful handling and appropriate safety measures when working with these substances, as well as the need for proper environmental considerations to mitigate potential ecological impacts.

6.2 Health assessment

The initial health assessment of the various chemicals was conducted by evaluating the safety data sheets (SDS) for the identified polymers and chemicals used for wood impregnation. The identified polymers and chemicals can be seen in TABLE 4.

TABLE 16 provides an overview of the health impacts of various substances used in the wood modification process. These substances include silicone resins, surfactants, antioxidants, and other additives.

TABLE 16. Health impact based on hazard statements found in SDS, when a given polymer is used in concentrated form.

Polymer	Health Impact
B1	Harmful if inhaled - Serious eye damage potential
B2	Low toxicity if swallowed Essentially non-irritating to skin and eyes May cause discomfort if inhaled
C1	Serious eye damage potential Contains volatile organic compounds
C2	Flammable liquid and vapor Releases methanol upon hydrolysis (toxic if inhaled/ingested)
C3	Flammable Suspected reproductive toxicant May cause organ damage (immune system) through prolonged exposure
D1	Low acute toxicity
D2	Low acute toxicity
Additive 1 in B1-2	Low toxicity concern based on available data
Additive 2 in B1-3	Suspected endocrine disruptor
Reference	May cause an allergic skin reaction Causes serious eye damage Suspected of damaging fertility or the unborn child May cause damage to organs through prolonged or repeated exposure

The health impacts of the substances vary widely. Several, including C2 and C3, are flammable and can release potentially harmful substances like methanol upon hydrolysis. Eye irritation or damage is a common concern, with products like C1 potentially causing serious eye damage.

Some substances present more severe health risks. For instance, C3 is suspected of being a reproductive toxicant and may cause organ damage through prolonged exposure. Additive in B1-3 is suspected of being an endocrine disruptor. It is noteworthy that some substances, such as the additive in B1-2 and D1 and D2, show relatively low toxicity concerns based on available data.

When comparing the different hazard statements of the polymers and additives investigated to substitute biocide with the reference biocide C3 and Additive in B1-3 have similar effects. The investigated polymers may be harmful when handled in the impregnation process and a focus the work environment if handling any of the investigated polymers should be taken.

This preliminary assessment highlights the importance of careful handling and appropriate safety measures when working with these substances, as well as the need for proper environmental considerations to mitigate potential ecological impacts.

7. Evaluation and future work

7.1 Evaluation of potential for tested polymers to substitute biocides

Some of the investigated chemicals had a minor effect on the hygroscopicity of the polymer-treated wood samples. However, the durability testing shows that this was not enough to provide an efficient protection of the wood against decay fungi.

In TABLE 17 an overview of the test results and the evaluation if the given polymer could be suitable for substituting biocides for wood impregnation can be found in TABLE 17.

TABLE 17. Overview of which polymers has been successfully used for impregnation of wood samples, if decay resistance occurred and if a decrease in equilibrium moisture content (EMC) occurred in the polymer-treated wood samples.

Poly-mer	Impregnation	Decay resistance	Hygroscopicity	Suitable to substitute biocide for wood impregnation [✓-✓✓✓✓✓]
A1	Not tested	Not tested	Not tested	N/D
A2	Not tested	Not tested	Not tested	N/D
A3	Not liquid at dosing temper-ature	Not tested	Not tested	N/D
B1	Yes	Lead to some decay protection	Does not provide a high enough decrease in EMC	✓✓✓
B2	Yes	Not tested due to low weight percent gain	Does not provide a high enough decrease in EMC	✓
C1	Yes	Not tested due to low weight percent gain	Does not provide a high enough decrease in EMC	✓
C2	Yes	Lead to some decay protection	Does not provide a high enough decrease in EMC	✓✓✓
C3	Yes	Not tested due to low weight percent gain	Does not provide a high enough decrease in EMC	✓
D1	Yes	Lead to some decay protection	Does not provide a high enough decrease in EMC	✓✓✓
D2	Yes	Does not confer any decay protection	Does not provide a high enough decrease in EMC	✓✓
B1-1	Yes	No significant con-ferred decay re-sistance	No significant reduc-tion in EMC	✓✓
D1-1	Yes	No significant con-ferred decay re-sistance	No significant reduc-tion in EMC	✓✓

Poly-mer	Impregnation	Decay resistance	Hygroscopicity	Suitable to substitute biocide for wood impregnation [✓-✓✓✓✓✓]
B1-2	Yes	No significant conferred decay resistance	No significant reduction in EMC	✓✓
B1-3	Yes	No significant conferred decay resistance	No significant reduction in EMC	✓✓

Based on the results polymer B1, C2, and D1 showed the most potential for substitution of biocides in wood impregnation since all three leads to some decay protection whereas the rest of the polymers exposed to decay showed no significant decay resistance. Polymer B1 and C2 are chemically more similar to each other than to D1. However, it cannot be concluded which polymer type would be a better alternative for biocides.

It is uncertain whether process improvements and/or higher amounts of chemicals would yield better results. It is also uncertain if a decrease of hygroscopicity alone will be sufficient to protect the wood from fungi attack – especially in connection with coatings or application situations where there is a risk of water trapping in the wood. More experiments with the polymers need to be conducted to obtain more information do determine if any of the investigated polymers is truly suitable for substituting biocides in wood impregnation. Furthermore, a more thorough investigation into the environmental and health impacts of the investigated polymers is necessary to determine if they are better for the environment, human health, and biodiversity compared to biocides.

7.2 Future work

In the project some decay resistance in the wood samples treated with polymer B1, C2, and D1 was obtained. These results are promising but polymer B1, C2, and D1 cannot substitute biocides without further work and investigation. With the established pilot plant for $scCO_2$ as a method for testing wood treatment future research and development for substitution of biocides should consider:

1. **Continued cross-industry exploration:** The polymers investigated in this project is used in other industries but the wood impregnation industry. Therefore, investigation of chemicals and treatments used in other industries that may serve as viable substitutes for biocides should be continued. This includes studying advanced polymers, coatings, and sealants that have demonstrated efficacy in protecting materials against microbial degradation which also will be suitable for impregnation by $scCO_2$.

2. **Continued Screening of Chemicals:** Engage in ongoing dialogue with chemical suppliers to identify and test additional polymers and chemicals based on the findings in this project. As an example, D1 and D2 are similar but with different molecular weights where D1 lead to some decay protection and D2 did not. Therefore, polymers with lower molecular weights should be tested to investigate if decay resistance could be obtained. Furthermore, testing different polymers/chemicals such as paraffin oil/wax (like A3) and other innovative compounds could provide new insights into effective biocide alternatives. In the dialogue with chemical suppliers a deeper understanding of the polymers which showed potential for substituting biocides should also be obtained in order to make more qualified selection of additional polymers and chemicals for further testing.

3. **Water uptake:** Water uptake of the polymer impregnated wood differs depending on the method of water exposure, where soaking for shorter periods of time resulted in

less water uptake compared to prolonged exposure to high relative humidity. This could indicate that the polymer could have a partial water repellent effect. Therefore, it is interesting to test water uptake under 'real conditions' (outdoor with wind, rain, etc.) to see if the polymers have an impact on the speed of the water uptake and therefore could provide decay resistance.

4. **Optimising chemical formulation:** For effective substitution, it is crucial to refine the chemical formulations used in wood treatments. This includes optimising the concentration, combination, and application methods of alternative chemicals to ensure they provide comparable or superior protection to traditional biocides. For known modification chemistries, ensure that the wood is properly dried before treatment. Wet wood requires extensive foundational research.

5. **Process Optimisation:** Continue to refine the $scCO_2$ impregnation process with the most promising candidates identified in this study (B1, C2, and D1). This includes refining process conditions and equipment to maximize the penetration and efficacy of the alternative treatments.

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Wood modification as a path to phasing out biocides

The report explores the potential to replace biocides in wood impregnation with alternative polymer-based treatments using supercritical CO2 (scCO2) as the impregnation method. Biocides are currently used to protect wood from degradation but pose environmental and health risks. Seven polymers were evaluated based on their technical performance, environmental impact, and health risks.

The results indicate that three polymers (B1, C2, and D1) have potential to enhance wood's resistance to fungal decay, but the effect is limited. None of the polymers sufficiently reduced wood's moisture uptake to prevent fungal growth on their own. Challenges were also observed regarding the polymers' diffusion into the wood and their long-term durability.
The environmental and health assessment revealed that the tested polymers generally have lower toxicity and environmental impact compared to traditional biocides, though some polymers still pose risks, such as potential aquatic toxicity and bioaccumulation.
The conclusion is that none of the tested polymers can substitute biocides in wood impregnation. Polymer B1, C2, and D1 show the most promising properties, but further research and process optimization are required to develop an effective and sustainable biocide-free solution. Future work should focus on improving chemical formulations, testing under real-world conditions, and conducting additional environmental and health assessments.

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