Ine Tønnessen Straumbotn

### Climate aging of miscellaneous wood surfaces and their characterization by Fourier transform infrared radiation spectroscopy

Master's thesis in Civil and Environmental Engineering Supervisor: Bjørn Petter Jelle Co-supervisor: Silje Kathrin Asphaug June 2023

NTNU Norwegian University of Science and Technology Faculty of Engineering Department of Civil and Environmental Engineering

Master's thesis



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### Abstract

According to the climate models worked out by the Intergovernmental panel on climate change (IPCC), building materials requirement to resistance will increase. Simultaneously, the UN sustainable development goals set requirements to choices of building materials, where resistance and durability must be balanced against sustainability. Therefore, during such assessments, it is necessary to predict the durability of the materials, to be able to set the correct maintenance interval for a longer service life.

In collaboration with SINTEF and manufacturing enterprise AG-Tre, the durability of miscellaneous claddings has been examined: charred wood, charred wood with ultraviolet (UV) resistant stain, primed and painted wood, and royal-impregnated wood. By exposing the claddings to natural and artificial climate aging, it has been investigated which degradation mechanisms are taking place by using Fourier transform infrared (FTIR) spectroscopy. The claddings were also photographed with a specialized camera setup to examine visual changes.

The results show that visually, the artificial weathering left the samples of charred wood faded. However, a clear trend of higher color stability was visible in royal-impregnated and primed and painted wood. Chemically, from the FTIR analysis, indicators of lignin degradations appear at the front surface of the claddings, except for primed and painted wood. However, no clear indications of chemical changes were shown at the inside relatively close to the front surface, indicating relatively weather resistant wooden claddings. Nevertheless, due to several possible uncertainties observed in the analyze process, further investigations with e.g. longer aging time will be necessary to examine the developments of the observed results.

The results from the tests does not clearly indicate that charred wood is considerably less durable than the other examined claddings. This is positive results for charred wood as an eco-friendly alternative to exterior cladding and provide useful information that will become increasingly important to protect our buildings from the impacts of forthcoming climate changes and reduce greenhouse gases. Nevertheless, further work is necessary in order to possibly determine the expected service life and maintenance interval for charred wood beyond the findings that have arisen in this thesis. Examples of further work could be extended aging time, for the same weathering test carried out in this study, and life cycle assessment to quantify the emissions of greenhouse gases.

### Sammendrag

I følge klimamodellene til FNs klimapanel vil behovet for motstandsdyktige byggematerialer øke i tide fremover. Samtidig setter bærekraftsmålene krav til materialvalg, hvor motstandsdyktighet og holdbarhet må balanseres opp mot bærekraft. Under slike vurderinger er det derfor nødvendig å kjenne til holdbarheten til materialene, og dermed kunne sette riktig vedlikeholdsintervall for en lenger levetid.

I samarbeid med SINTEF og kledningsleverandør AG-Tre er holbarheten til diverse utvendige kledninger undersøkt: brent kledning, brent kledning med ultraviolet (UV) beis, grunnet og malt kledning og royalimpregnert kledning. Gjennom å påkjenne kledningsprøvene for naturlig og kunstig klimaaldring er det undersøkt hvilke nedbrytningsprosesser som finner sted ved hjelp av Fourier transform infrarød (FTIR) spektroskopi. Prøvene er i tillegg fotografert med et spesialisert kameraoppsett for å undersøke visuelle endringer.

Visuelt viste den akselererte klimaaldringen at brent kledning med og uten UV beis grånet. De resterende kledningene, royalimpregnert og grunnet og malt kledning, viste derimot en mye høyere fargestabilitet. Kjemisk, fra FTIR-analysen, var det små indikatorer på nedbrytning av lignin på fremsiden av alle kledningene, bortsett fra for grunnet og malt kledning. Derimot var det ingen klare indikasjoner på kjemiske endringer på innsiden relativt nær framsiden for noen av kledningene, noe som indikerer relativt motstandsdyktige trekledninger. Likevel, på grunn av flere mulige observerte usikkerheter knyttet til analyseprosessen, er det nødvendig med for eksempel lenger aldringstid for å undersøke utviklingen til de observerte resultatene.

Resultater fra prøvingen indikerer dermed at brent kledning ikke har noe betydelig dårligere kjemisk holdbarhet enn de andre undersøkte kledningene. Dette er positive resultater for brent kledning som et miljøvennlig alternativ til utvendig kledning, og gir nyttig informasjon som vil bli viktigere for å beskytte bygningene våre mot kommende klimaendringer og redusere klimautslipp. Det bør likevel nevnes at videre studier er nødvendig for å kunne si noe om den forventede levetiden og vedlikeholdsbehovet til denne typen kledning utover funnene som er gjort i denne oppgaven. Eksempler på videre studier kan være forlenget aldringstid, for de samme aldringstestene gjort i denne studien, og livssyklusanalyse for å kunne kvantifisere klimagassutslippene.

### Preface

This thesis marks the end of my two-year MSc. studies at the Norwegian University of Science and Technology. During spring 2023 this thesis was carried out in association with the Department of Civil and Environmental Engineering.

The thesis is comprised of one scientific article exploring missing fields within the research of charred wood. The article is submitted for publication in the journal of *Wood Material Science and Engineering*.

First and foremost, I want to thank my supervisor professor Bjørn Petter Jelle. Early, you pointed out to me that FTIR spectroscopy of wood is challenging, but just recently I realized what you actually meant. Thank you for all the knowledge sharing within the field, and your patience in the analyzing process. This has been of great help when working on the thesis.

I would also like to thank my co-supervisor Silje Kathrin Asphaug at SINTEF Community. I remember well the first meeting where you introduced me to charred wood. Since then, it has been very interesting to explore this with you. Thank you for all your support with the laboratory work and for being available when I needed it.

Special thanks are also extended to Lasse Rindal and Jan Are Haugen at AG-Tre for the opportunity to examine your products and the very kind visit where I got to see the charring process.

Ine T. Straumbotn

Ine Tønnessen Straumbotn Trondheim, Norway June 2023

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## Introduction to thesis

This introduction covers the work on the article "Natural and artificial climate aging of charred and other wooden claddings characterized by Fourier transform infrared radiation spectroscopy", written during spring 2023. The aim is to present the background for the thesis, the process behind, and the challenges.

Other work carried out in connection to this master's thesis, beyond the article, is attached in the appendix. This involves visual evaluation of the test wall placed at the collaboration manufacturing enterprise and estimations and calculations of acceleration factors for the aging apparatus.

### Background

This master's thesis is a collaboration between NTNU, SINTEF, and AG-Tre Brent, where the client is AG-Tre Brent. AG-Tre Brent is a subsidiary of AG-Tre, which is a manufacturing enterprise in Orkland municipality that produces interior wood paneling and exterior cladding in spruce and pine. They are specialists in surface treatments of wood, and has now, in collaboration with AG-Tre Brent, developed cladding and paneling with charred surface.

Wood charring is an old technique used to increase the durability of wood (Kymäläinen et al. 2017), which originated from Japan (called yakisugi). Today, the knowledge is based on experience and tradition, with insufficient scientific research within the field, which makes it difficult to decide the durability. Thus, AG-Tre Brent wants to analyze their claddings, in collaboration with SINTEF, to prove and investigate their durability. SINTEF has access to laboratories where materials and products can be tested for different properties. The laboratories are also managed by NTNU, which has made it possible to develop this master's thesis based on the project to AG-Tre Brent. In addition to carrying out the test commissioned by AG-Tre Brent, this master's thesis has made it possible to carry out several durability tests on charred wood, and with this further increase the area of knowledge.

### Work progress

AG-Tre had prepared samples for miscellaneous types of claddings which were delivered to SINTEF on two frames. The first frame was to be exposed to artificial climate aging for three months, and the second frame was to be exposed to natural weathering for minimum one year, standing there today, and until October 2023. SINTEF assembled and managed the weathering tests, and I was responsible for sampling and analyzing the results in collaboration with the supervisors.

The work started in autumn 2022 in connection to the specialization project. Much of the work was done during the autumn. For example, starting up the first weathering tests, taking the first sampling, as well as inspection at AG-Tre to see the charring process. During the weathering, I analyzed the samples both visually and with Fourier transform infrared (FTIR) spectroscopy to continuously assess the durability and the need for longer aging time or other weathering tests. During spring 2023 the ongoing artificial weathering experiment was extended due to of small changes in the claddings. Also, a new artificial

weathering experiment with a higher acceleration factor was initiated, in addition to excluding an exposure factor. This new weathering test ran for two weeks.

Due to the costs of accelerated climate ageing, such tests are expensive to conduct. The first artificial weathering experiment was meant to run for 12 weeks but got extended by 8 weeks so that it remained for 20 weeks. Results and previous research indicate that an even longer aging time would have been useful to better evaluate the durability.

### Aim of study and research questions

The aim of this work is to analyze the durability of charred wood compared with other traditional treatments for wooden claddings. The claddings used for comparison are charred wood with UV resistant wood stain, primed and painted wood, and royal-impregnated wood. The wooden claddings are exposed to natural and artificial climate aging, where the apparatus and climate factors used for artificial climate aging are vertical climate simulator (UV radiation, rain, frost) and Atlas climate chamber (UV radiation). Before, during, and after climate aging, the claddings was analyzed both visually with a specialized camera setup (DigiEye) and chemically with FTIR spectroscopy. In the background of this, the following research questions will attempt to be answered:

- (1) How will the claddings look visually?
- (2) What chemical changes occur during aging?
- (3) Based on (1) and (2), what can be said about the durability of the claddings?

### Article

Natural and artificial climate aging of charred wood and other wooden claddings characterized by Fourier transform infrared radiation spectroscopy

# Natural and artificial climate aging of charred wood and other wooden claddings characterized by Fourier transform infrared radiation spectroscopy

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ARTICLE INFO	ABSTRACT		
Keywords:	According to the climate models worked out by the Intergovernmental panel on climate change		
Aging	(IPCC), building materials requirement to resistance will increase. Simultaneously, the UN		
Weathering	sustainable development goals set requirements to choices of building materials, where res		
Wood	and durability must be balanced against sustainability. Therefore, during such assessments, it		
Charred wood	necessary to predict the durability of the materials, to be able to set the correct maintenance inter		
Cladding	for a longer service life.		
Fourier transform infrared	In this study, charred wood is compared with various treatments for wooden claddings: royal-		
FTIR	impregnated wood, primed and painted wood, and charred wood with ultraviolet (UV) resistant		
	wood stain. An estimate of durability is discussed by applying miscellaneous climate aging		
	exposures, including both natural and artificial setups. The characterization methods used are		
	material characterizations with Fourier transform infrared (FTIR) radiation spectroscopy and visual		
	evaluation with a specialized camera setup.		
	The results show that visually, the artificial weathering left the samples of charred wood faded,		
	whereas royal-impregnated and primed and painted wood in overall demonstrated higher color		
	stability. Chemically, from the FTIR analysis, indicators of lignin degradations appeared at the front		
	surface of the claddings, except for primed and painted wood. However, no clear indications of		
	chemical changes were shown at the inside of the wood samples, relatively close to the front surface,		
	indicating weather resistant wooden claddings. Nevertheless, further studies with longer aging time		
	are necessary to examine the development of observed results.		

#### Scientific one-liner

This study is investigating the durability of charred wood and other wooden claddings, by applying Fourier transform infrared (FTIR) radiation spectroscopy for material characterization and a specialized camera setup for visual observations, for the purpose of detecting any chemical and visual changes during exposure to artificial and natural climate aging, demonstrating that charred wood has a clear trend of lower color stability, however with no clear evidence of chemical changes.

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#### 1 Introduction

Wood is a low-cost and ecological choice for outside claddings of buildings (Kymäläinen et al., 2018). It is a better choice of building material, considering global carbon emissions, than more energy-intensive materials such as bricks, aluminum, steel, and concrete (Tettey & Gustavsson, 2019; Werner & Richter, 2007). Nevertheless, the service life of brick and aluminum may be considerably longer, which leads to variations in maintenance between wood and these kind of building materials. The primary energy for maintenance and repairs can constitute a large share of the life cycle energy use, in addition to the exposure conditions. The need for maintenance can be reduced, but exposure conditions are more difficult to handle and properly take into account.

Wood placed outside is exposed to weathering. Wood degrades due to humidity conditions, solar radiation, temperature changes, rain and moisture (Jelle, 2012). As a hygroscopic material, the cyclic humidity conditions cause dimensional changes such as swelling and shrinkage which creates substantial challenges for coatings (Goodell et al., 2020). Furthermore, solar radiation degrades the lignin component which acts as an adhesive in the wood structure (Kymäläinen et al., 2020). To protect the wood from weathering, applying coatings, waxes, oils or stains is the most common treatment for wooden claddings (Sandak et al., 2019).

The main function of external wooden claddings is to protect the wall core from climatic and mechanical stresses (SINTEF Byggforsk, 2022), which sets high requirements for durability and types of treatment used. With maintenance, painted wooden claddings are long-lasting and easy to recycle and dispose of at the end of their service life (Kymäläinen et al., 2018). Maintenance is required every 2-15 years, depending on the opacity of the finish. However, the exposure site and orientation of the wall have higher impacts than the coating. This makes it difficult to determine the interval of maintenance. The paint has a relatively high environmental impact, about 70% of the environmental load (Strand & Hovde, 1999). In fact, longer painting intervals that are resulting in shorter service lives may lower the overall environmental impacts. Also, the dimensional changes represent a challenge for coatings as only a few polymeric coatings can withstand the recurring cycles of humidity changes (Goodell et al., 2020). Based on these challenges, it seems that a possible key solution is to have a treatment that gives the wood a natural state.

One-sided surface charring is a fully organic and natural modification method, as well as a treatment that leaves most of the wood in its natural state (Kymäläinen et al., 2022a). According to Zicherman and Williamson (1981, as cited in Kymäläinen et al., 2022a), the cell structure of the char layer is similar to unmodified wood. This means that pores and cells retain their shape and size. Another characteristic of charred wood is when charring, the water sorption is thought to diminish because charcoal is more hydrophobic than untreated wood (Kymäläinen et al., 2017). Kymäläinen et al. (2017) argue that hygroscopicity reduces when undergoing pyrolysis and that this will increase both the biological and mechanical durability, as well as decrease cracking, cupping, and fungal activity in the absence of water. However, since charcoal is a porous substance, capillary absorption of water will increase.

Previous studies have shown improved wettability characteristics and sorption properties (Kymäläinen et al., 2017, 2018; Šeda et al., 2021) and changes in chemical composition and functional groups toward a more stable material (Kymäläinen et al., 2020). In the latest study by Kymäläinen et al. (2022b), it is concluded that the hydrophobicity of materials seems to efficiently limit fungal activity which may increase service life. Another research finding is changes in the modified lignin component that indicates photodegradation (Kampe & Pfriem, 2018; Kymäläinen et al., 2020, 2022a). Except this, these studies also reported good weatherability when measured with various methods.

Common for the studies on charred wood is the use of artificial and natural climate aging to degrade the wood samples. The service life of modified wood is best evaluated at natural weathering experiments but is very time-consuming (Kymäläinen et al., 2022c). To obtain results sufficiently fast and within economical limits, accelerated climate aging tests are recommended (Jelle, 2012). An artificial weathering setup overlooks factors such as air pollutants, microbes, chemicals, and biological agents (Cui & Matsumura, 2019), but also eliminates differences between exposure sites (Kymäläinen et al., 2022c).

By taking small specimens before, during, and after climate aging, material characterization by Fourier transform infrared (FTIR) radiation spectroscopy, can be used to determine which degradation mechanisms are taking place and the extent of the aging (Jelle, 2012). It is through FTIR, Kymäläinen et al. (2020) investigated the degradation of the modified lignin. There is also measured a reduction of polar components and OH groups, as well as an increase in aromatic structure which created a surface with reduced hygroscopicity (Kymäläinen et al., 2018).

In addition to material characterization by FTIR several research scientists have investigated various characteristics of charred wood such as fire performance (Buksans et al., 2021; Hasburgh et al., 2021; Machová et al., 2021), decay resistance (Hasburgh et al., 2021; Kymäläinen et al. 2022a, 2022b; Machová et al., 2021), cupping (Ebner et al., 2021; Kymäläinen et al., 2018), color changes (Kymäläinen, et al., 2022a, 2022c; Machová et al., 2021), sorption (Kymäläinen et al., 2017, 2018), modification parameters (Kymäläinen et al., 2022a, 2022c) and chemically changes (Kymäläinen et al., 2018, 2020). In general, the results show that the char layer improved the investigated characteristics compared to untreated wood. However, none of these studies show whether these characteristics are improved compared to traditional claddings.

The aim of this study is to analyze the weathering resistance of charred wood compared with other traditional treatments for wooden claddings by applying miscellaneous climate aging exposures, including both natural and artificial setups. Using material characterization methods such as FTIR and a specialized camera setup, this study will discuss chemical and visual changes in the claddings for the purpose of detecting and comparing the durability of the different claddings. No known studies have investigated whether degradation appears at the inside of charred wooden claddings. By sampling both the front surface and the inside relatively close to the front surface of the wood samples, this study attempts to investigate if chemical changes may possibly appear in these areas.

#### 2 Materials and methods

#### 2.1 Material preparation and samples

The materials and samples used in this study were purchased from a commercial manufacturer of exterior claddings. Norway spruce (Picea abies (L.)) and pine (Pinus sylvestris (L.)) were sourced from central and western Norway. The samples have a tongue and groove profile which is one of the most common exterior cladding profiles in Norway. The dimensions were 210 mm x 297 mm x 20 mm (tangential x longitudinal x radial) for the spruce samples and 279 mm x 297 mm x 18 mm for pine samples.

Three different coatings were used as a reference to charred wood of spruce: primed and painted wood of spruce, charred wood of spruce with UV resistant wood stain, and royal-impregnated wood of pine. The coating application was performed in industrial environments by the collaborating company. The first one, primed and painted wood was treated by transparent alkyd/acrylic primer (Teknol 1888, Teknos), and water- and acrylate-based topcoat (Nordica Eko 3330, Teknos). The second one, UV resistant wood stain (Woodex aqua classic, Teknos) was used on charred wood to protect the wooden surface from moisture, soiling, and solar radiation. The third and last one, royal-impregnated wood is a pressure-impregnated cladding produced by Talgø MøreTre AS (Rectangular Møreroyal-impregnated, Talgø, Surnadal, Norway). After pressure-impregnating, the pine gets boiled in oil under a vacuum. The wood is planed pine with an unplaned surface. The samples were stored in industrial environments after coating application and then placed and sent on a frame to the laboratory for further analysis, as shown in Fig. 1. Four samples per treatment were used, where within the treatment of the claddings, the samples are from the same planed plank, but between the various claddings they are not from same planed plank.





Fig. 1. The experimental frame with codes used for sample identification and labelling. Charred wood (C), charred wood with UV resistant wood stain (UV), primed and painted wood (PP), and royal-impregnated wood (R).

#### 2.2 Charring process

The panel surfaces were charred with a purpose-built machine at the commercial and collaborating manufacturer in central Norway. Flame charring was implemented on longer boards, which were then cut to the dimensions of the samples, 210 mm x 297 mm x 20 mm. The starting moisture content (MC) was approximately 14-16%.

The charring took place in a furnace of steel, see Fig. 2, with the dimensions 5000 mm x 400 mm x 10 mm (length x diameter x thickness). The device has a roller on the bottom of the furnace to facilitate continuous board feed, and two propane torches on top. The torches have an output of 55 kW each and are placed at the end of the steel pipe. It is estimated 40 mm between the top surface of the panels and the torches. The processing speed was set at 6 meters per minute, which roughly corresponds to a processing time in the furnace right under one minute per panel. After heat modification the panels got brushed and water sprayed by a high-pressure washer. The last operation was to remove surface water with a leaf blower before storing the samples.





#### 2.3 Weathering tests

Two experimental frames, equally the one shown in Fig. 1, have been used in the weathering tests. One for natural weathering and one for artificial weathering in a vertical climate simulator. For both frames, the bottom row has been removed, with one of the rows used for artificial weathering in a solar simulation chamber.

Natural weathering tests were performed at the Voll test station in Trondheim, Norway ( $63^{\circ}24'38''$  N,  $10^{\circ}27'15''$  E). Outside the test station, there are two roads placed 26 and 73 m from the experimental frame, that may have added particulate matter to the samples. The experimental frame was orientated towards southwest, with an elevation angle of 30 degrees to increase the exposure to rain and sun. The weathering experiment runs for a year, where the sample collection was carried out for five months starting in October 2022. A collection of samples exposed to 2, 4, 10, 16, and 20 weeks was gathered.

The first artificial weathering experiment was made with a vertical climate simulator using a procedure according to Nordtest Method NT Build 495 (Nordtest Method NT Build 495, 2000). The experimental frame with three samples per treatment was placed inside the chamber. The weathering procedure consists of an exposure cycle of four hours that is repeated six times per day for 20 weeks (a total of 3360 h). The four-hour cycle is made of an indoor laboratory climate zone, followed by UV and IR irradiation zone (black panel temperature of 63 °C), water spray (15 dm<sup>3</sup>/(m<sup>2</sup>h)), and in the end, a freezing zone (-20 °C). All the zones last for one hour. A collection of samples exposed to  $\frac{1}{2}$ , 2, 4, 6, 10, 16, and 20 weeks was gathered.

The second artificial weathering experiment was made with a solar simulation chamber, Atlas SC600 MHG. The bottom row of the experimental frame was used in the chamber, with one sample per treatment. The weathering experiment consists of an exposure of two weeks (336 h). The parameters were set to 60 °C, and relative humidity 50 %. The solar radiation intensity was 1200 W/m<sup>2</sup>, where the metal halide global (MHG) lamps obtain an accelerated simulation of indoor and outdoor weathering. Sample collection exposed to 1, 2, 5, 7, 9, and 14 days was gathered. A summary of the values used in the different weathering tests are shown in Tab. 1.

#### Tab. 1. Experimental setup parameters.

	Voll test station,	Vertical climate simulator	Atlas climate chamber
	Trondheim, Norway	Nordtest Method NT Build 495	SC600 MHG
Weathering	Natural	Artificial	Artificial
Date	21.10.2022 - 10.03.2023	07.10.2022 - 24.02.2023	01.02.2022 - 15.02.2023
Weeks	20	20	2
Temperature	$T_{av} = 0.5 \ ^{\circ}C \ (-4 \ to \ +6 \ ^{\circ}C)$	-20 to +63 °C	60 °C
<b>Relative humidity (RH)</b>	$RH_{av} = 78\%$ (71 to 83 %)	No data	50%
Water	~54 mm (20 to 103 mm)	$15 \text{ dm}^{3}/(\text{m}^{2}\text{h})$	None
Sun	Natural solar radiation	Ca. 45 W/m <sup>2</sup> (UVA+UVB)	1200 W/m <sup>2</sup> (Solar radiation)

#### 2.4 Visual characterization

VeriVide's DigiEye system 700 mm Cube was used for visual characterization. The specialized camera setup was used to obtain digital pictures of the wood samples. The digital color imaging system takes pictures with the same light conditions. This makes it possible to compare the color changes of the samples and depict the visual changes of the claddings.

#### 2.5 FTIR material characterization

Material characterization by FTIR radiation spectroscopy may give information about which degradation mechanisms are taking place and the extent of the aging (Jelle, 2012). The FTIR analysis require small specimens of the wooden claddings taken from the samples before, during, and after accelerated climate aging. Sample collections were taken with a gouge chisel down to approximately three mm depth. To protect the sampling area, fast-setting two component adhesive (Casco Strong Epoxy Rapid) was used. All specimens were stored in zip-lock bags before they got subjected to FTIR analysis.

A Thermo Scientific Nicolet iS50 FTIR Spectrometer was used to obtain attenuated total reflectance (ATR) FTIR spectra of the wood specimens. Each measurement consisted of 32 scans at a resolution of 4 cm<sup>-1</sup> with a diamond crystal in the wave number range of 4000-400 cm<sup>-1</sup>. Irregularities in FTIR spectra between 2200 and 1900 cm<sup>-1</sup> appear due to very large absorption between these wave numbers, which represents the weak point in the ATR diamond crystal. Fortunately, only limited numbers of chemical bonds have spectral absorption bands in this region, which makes the diamond crystal an excellent choice for ATR applications. The data was processed with the OMNIC software. To ensure satisfactory contact between the specimens and the diamond crystal, spectra were collected at various locations on the specimen surface. A minimum of three spectra each were recorded at the front surface and inside wood material relatively close to the front surface. The spectra with the largest peaks normally represent the most correct measurements (i.e. the smallest amount of air voids between the specimens and the ATR crystal), and hence these spectra were chosen. Unfortunately, wood samples have a complex structure, which complicates accurate quantitative measurements (height of absorbance peaks). Air between the diamond crystal and specimens results in a weaker absorbance signal unless other conditions such as impurities etc. may indicate otherwise. Qualitative measurements (location of absorbance peaks at wave numbers) do not represent a problem if the contact area is large enough to ensure a sufficiently strong measurement signal.

When analyzing spectra, changes in material structure appear if the absorbance spectra change in line with the aging time (from measurement to measurement). The change is due to chemical bonds forming or disappearing, where an increasing FTIR absorbance peak indicates an increase in a specific chemical bond, and a decreasing absorbance peak indicates a decrease in a specific chemical bond. If the spectra do not change with the increased aging time, or if the changes are small, the changes may be due to variation in the measurement uncertainty, e.g. variations of the amounts of air voids between the specimens and the ATR diamond crystal as mentioned above. In such cases results may be difficult to evaluate.

For easier comparison between various spectra, they should be plotted with the same scale for the absorbance axis, although this implies fewer spectra distinctions for the cases with fewer accelerated aging changes. Absorbance spectra are chosen because the absorption of electromagnetic radiation, e.g. IR radiation, follows the Beer-Lambert law (Jelle & Nilsen, 2011). This means that the radiation is decreasing exponentially with the penetration depth in the actual material. By plotting the spectra on a logarithmic absorbance scale versus wave number, it is possible to carry out quantitative studies. Physically and mathematically, it follows that a doubling of the logarithmic absorbance is interpreted as a doubling of the concentration of absorption active agents.

#### **3** Results and discussion

#### 3.1 Natural weathering details

Rainfall, the relative humidity, and average monthly temperature logged by Norwegian Meteorological Institute (MET Norway), at Voll weather station are shown in Fig. 3. Specimens for the FTIR analysis were taken in the period from 21st of October 2022 to 10th of March 2023, which corresponds to a total time of 20 weeks. In October and March, Fig. 3 only contains measurements from the actual period the claddings were weathered outside, and specimens taken. Average temperatures ranged from -4 to +6 °C. The precipitation was mostly rain but fall as snow in the winter months (December to February). During these months the samples were occasionally blocked with snow and subjected to relatively small amounts of solar radiation.



Fig. 3. Rainfall (mm), relative humidity (%), and averages of temperature (°C) registered at Voll test station. October and March only contain measurements from 21st of October to 31st of October and 1st of March to 10th of March, i.e. the actual period the samples were natural weathered.

#### 3.2 Visual evaluation

No clear visible differences in color are observed for the claddings naturally aged at the Voll test station. Furthermore, photos of the samples aged in the vertical climate simulator, exposed to UV and IR radiation, rain, and frost, are taken with the specialized camera setup, DigiEye, and shown in Fig. 4. Charred wood and charred wood with UV resistant stain have a clear change in color during the aging period as expected according to recent studies on color changes (Kymäläinen, et al., 2022a, 2022c). Also, there is a change already after 6 weeks of aging. An interesting finding is that charred wood with UV resistant stain may have more change in color despite the UV treatment. A possible explanation for this might be a washing off the char layer due to rain. Unfortunately, color changes are observed for the samples aged in Atlas, see Fig. 5, with exposure of solar radiation and no rain, indicating that rain itself does not cause the discoloration. These findings suggest that it should be investigated whether color stability is a property of the UV stain or not. A note to these findings is that the samples in Atlas climate chamber were aged for two weeks with solar radiation, while the samples in the vertical climate simulator were aged for four and a half weeks with UV radiation (1/4 of 18 weeks, before last DigiEye photo). According to these data, we can infer that rain itself does not cause most of the observed discoloration, but further research is needed in order to investigate these phenomena more closely.



Fig. 4. Samples before, during and after weathering in vertical climate simulator taken with DigiEye camera of charred wood (C), charred wood with UV resistant wood stain (UV), primed and painted wood (PP), and royal-impregnated wood (R). The leftmost samples are the non-aged references.

As mentioned in the above, samples weathered in the Atlas climate chamber depict similar color changes as samples weathered in the vertical climate simulator, with higher color stability for primed and painted and royal-impregnated wood. Even though the vertical climate simulator include exposure to UV radiation for four and a half weeks, and the Atlas climate chamber only two weeks with solar radiation lamp, there is a clear color change for the samples weathered in the climate chamber as well. A possible explanation for this might be due to the stronger solar radiation in the Atlas chamber. Furthermore, Fig. 5 shows the sampling of the small specimens of the wooden claddings that were analyzed with FTIR spectroscopy. The color change in the sampling area is due to degradation of the fast-setting two component adhesive (Casco Strong Epoxy Rapid) which were used to protect the sampling area.

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**Fig. 5.** Samples before and after weathering in Atlas climate chamber taken with phone camera of charred wood (C), charred wood with UV resistant wood stain (UV), primed and painted wood (PP), and royal-impregnated wood (R). The upper samples are the non-aged references. The lower samples show marks after sampling of the specimens used in the FTIR analysis.

#### 3.3 FTIR evaluation

Various measured FTIR spectra with absorbance (optical density =  $log_{10}(1/T)$ , T = transmittance) versus wave number (cm<sup>-1</sup>) in the range 4000 cm<sup>-1</sup> (2.5 µm) to 400 cm<sup>-1</sup> (25 µm) are presented in Figs. 6-8. Fig. 6 shows spectra from artificial climate aging in the vertical climate simulator. Fig. 7 depicts spectra from artificial climate aging in the Atlas climate chamber. Fig. 8 gives spectra from natural outdoor aging at Voll test station. Furthermore, spectra of surface treatments compared to non-aged samples are given in Fig. 9, and a comparison between the different aging methods is discussed in chapter 3.3.5 FTIR comparison of aging methods and aging time. Lastly, a summary of the FTIR analysis is given in chapter 3.3.6 FTIR summary.

#### 3.3.1 Artificial weathering experiments – Vertical climate simulator

FTIR absorbance versus wave number for the four different samples subjected to accelerated climate aging in vertical climate simulator are shown in Fig. 6. When looking at the inside of the specimens, see spectra rightmost in Fig. 6, no spectra show rising absorbance intensities with increased time of aging. There are several possible explanations for this result. Firstly, as mentioned in chapter *2.5 FTIR material characterization*, wood have a complex structure that complicates accurate quantitative measurements (height of absorbance peaks). Air between the ATR diamond crystal and the sample results in a weaker absorbance signal, unless other conditions such as impurities etc. may indicate otherwise. This may explain the lack of correlation between increased absorbance peaks and increased time of aging. Secondly, the sampling was taken with a gouge chisel, making it difficult to get the exact depth for all the specimens. Because the degradation varies with differences in depths, this inaccuracy may influence the height of absorbance peaks. Lastly, the lack of correlation in addition to small changes in the height of absorbance peaks and increased cladding, the spectra representing 2 and 16 weeks of weathering show a curved peak while the other ones show a peak similar to the spectra of the surface treatments in Fig. 9. This may indicate a deeper cut in these samples, where the impregnation is not present to the same extent as at the front surface. According to this observation, the second explanation may be suitable for the results showing no correlation, indicating uncertainty in sampling. However, according to small changes in height of absorbance peak, it may be interpreted that no major chemical changes have taken place at the inside of the samples relatively close to the surface.

When analyzing the front surface of the specimens, several peaks are of interest. For charred wood a high peak at 1059 cm<sup>-1</sup>, representing C–O-stretching of cellulose (Pandey, 1999), is shown for all aging times including the non-aged ones. In contrast, Kampe and Pfriem (2018) observed in their study before weathering no similar peak for charred wood, indicating a chemical change of cellulose due to pyrolysis. A possible explanation for this might be differences in the charring process. If we look at spectra in Fig. 9, a smaller peak at 1059 cm<sup>-1</sup> is shown for the spectra representing the measurements of scraped charred wood before the wood panel gets brushed. This may indicate that parts of the carbonization layer, and its chemical properties, disappear when brushing. Unfortunately, Kampe and Pfriem (2018) concluded in their study that the thickness of the carbonization layer is a critical factor for the weathering protection of wood. Also, they reported that a peak appears after weathering which indicates that a part of the surface layer has been removed due to the weathering, and the spectra get similar peaks as weathered untreated wood. Similar changes are observed in this study, where the absorbance intensities increase with increased time of aging, but with lack of correlation due to previous mentioned possible uncertainties (weak absorbance signal, short aging times etc.). For the other claddings an absorbance peak exists for all spectra where a decreasing absorbance peak appears for charred wood with UV stain and primed and painted, and an increasing absorbance peak is observed for royal-impregnated wood. It should be mentioned that the observed changes do not show a correlation between increased absorbance peak and increased aging time, and therefore must be taken with caution.

A peak at wave number 1508 cm<sup>-1</sup> represents aromatic C=C bands (Srinivas & Pandey, 2012). When looking at charred wood, this absorbance peak reduces with increased time of aging, and almost disappears after 16 weeks. The decrease in the intensity of peak 1508 cm<sup>-1</sup> indicates degradation of the aromatic C=C band. Srinivas and Pandey (2012) reported a rapid decrease in the intensity of lignin associated absorption band during photodegradation, which almost disappeared. Kymäläinen et al. (2020) also reported degradation of the aromatic C=C band, as well as formation of oxidized lignin structures and new carbonyl groups at around 1729 and 1508 cm<sup>-1</sup> after aging. For the other claddings a peak at 1508 cm<sup>-1</sup> barely exists or does not exist at all and may be due to the treatments covering the wood structure. At wave number 1729 cm<sup>-1</sup> all the claddings show a peak, but no changes with increased time of aging. The peak is curved for charred wood and royal-impregnated wood, and sharp for charred wood with UV stain and primed and painted wood due to the treatment as shown in Fig. 9.

A peak at 1595 cm<sup>-1</sup> represents C=C stretching vibration, aromatic ring of lignin (Garside & Wyeth, 2003). If the peak is not detected, it indicates decomposition of lignin by weathering. The spectra of charred wood show this peak for non-aged, 4 days of aging, and 2 weeks of aging, before the intensity of the absorbance peak decreases with increased aging time. For charred wood with UV resistant stain the peak disappears with increased aging (not continuously), indicating degradation of lignin. For the other claddings, no peak is observed and may indicate that the treatments cover the wood surface where no contact with wood structures appears. This is a positive result for primed and painted wood where the topcoat should cover the wood structure and not penetrate as impregnation.



Fig. 6. FTIR absorbance versus wave number for the four different samples subjected to accelerated climate aging in vertical climate simulator, including both front surface and inside of the samples.

#### 3.3.2 Artificial weathering experiments – Atlas climate chamber

FTIR absorbance versus wave number for the different samples subjected to accelerated climate aging in Atlas climate chamber are shown in Fig. 7. At wave number 1059 cm<sup>-1</sup> (C–O-stretching of cellulose) at the inside of the claddings, decreasing absorbance peaks (as compared to the non-aged spectra) are observed for the charred wooden claddings, while the primed and painted wooden cladding show increasing absorbance peaks. The decrease of absorbance peaks for charred wood is greater than the changes for charred wood with UV stain and primed and painted wood, indicating less durable cladding. However, the results must be taken with caution as there is no continuous absorbance change with increased aging time as described in previous chapter. At wave number 1508 cm<sup>-1</sup>, the changes are small for all the claddings and may indicae no degradation of the aromatic C=C band of the inside of the wood samples. At wave number 1595 and 1729 cm<sup>-1</sup> a curved peak is observed for all the claddings, where no indications of degradation of lignin are shown.

When looking at the front surface at wave number 1059 cm<sup>-1</sup>, all the spectra except charred wood with UV stain show signs of aging, but the spectra vary between the aging times, making it difficult to determine the chemical changes. Both negative and positive changes in the absorbance peaks are observed, indicating degradation with disappearance or formation of chemical bonds, respectively. At 1508 cm<sup>-1</sup> for charred wood, the absorbance peak decreased and disappeared fully after only 5 days. This was also shown for charred wood in the vertical climate simulator, where the peaks disappeared after 16 weeks, indicating degradation of the aromatic C=C band. For charred wood with UV stain and primed and painted wood, no absorbance peak appeared, and may be due to the treatment hiding or blocking peaks at this wave number. This may be supported by the spectra in Fig. 8 which shows no absorbance peak at the same wave number for the UV stain and topcoat. The last one, royal-impregnated wood shows rising absorbance intensities with increased time of aging and might indicate degraded structures of lignin. At wave number 1595 cm<sup>-1</sup> there is also no existing peak for charred wood with UV stain and primed and painted wood, a peak at wave number 1508 cm<sup>-1</sup>, e.g., treatment blocking or hiding the peak. For charred wood, a peak at wave number 1595 cm<sup>-1</sup> exists for all aging times, but the peaks vary between the aging times, hence making it difficult to determine whether degradation of lignin appears or not. For royal-impregnated wood, the same changes as observed at wave number 1508 cm<sup>-1</sup> appear, thus giving greater evidence to the degradation of lignin. At 1729 cm<sup>-1</sup> the spectra vary much between the aging times, making it difficult to determine whether there has been any chemical changes. It should be noted that the results, owing to the lack of correlation between increased absorbance peak changes and increased time of aging should be taken with caution according to explanations given in the previous chapter.



Fig. 7. FITR absorbance versus wave number for the four different samples subjected to accelerated climate aging in Atlas climate chamber, including both front surface and inside of the samples.

#### 3.3.3 Natural weathering experiments - Voll

FTIR absorbance versus wave number for the four different samples subjected to natural climate aging at Voll are shown in Fig. 8. The natural weathering experiments may capture factors that are challenging to induce in artificial climate aging such as particulate matter, salts, combination of various factors, etc. Common for the inside of the claddings is lack of correlation between rising absorbance intensities with increased time of aging, same results as for the vertical climate simulator and Atlas climate chamber.

Analyzes at the front surface at wave number 1059 cm<sup>-1</sup>, C-O stretching of cellulose, depicts an absorbance peak for all the claddings, but there are no clear connections between peak changes with increased aging time. The same is valid for wave number 1508 cm<sup>-1</sup>, C=C aromatic band, lignin, except for primed and painted wood where no peak exists. In contrast to weathering tests in the vertical climate simulator and the Atlas climate chamber, an absorbance peak still appears at all aging times at wave number 1508 cm<sup>-1</sup> for charred wood. This may be due to slower aging when weathering in natural outdoor conditions, as expected. At wave number 1729 cm<sup>-1</sup>, claddings treated with UV stain and primer and paint, portray a sharp peak. However, no obvious correlations to aging times are observed for either these ones or the other cladding types. Analyzes at wave number 1595 cm<sup>-1</sup>, aromatic ring of lignin, do not show a peak for the claddings except for charred wood. This may be due to the treatments hiding this peak for the wood front surfaces.



Fig. 8. FTIR absorbance versus wave number for the four different samples subjected to natural climate aging at Voll, including both front surface and inside of the samples.

#### 3.3.4 FTIR of surface treatments

FTIR absorbance spectra versus wave number for the wooden claddings and treatments before aging are shown in Fig. 9. Note that the figure with the royal-impregnated wood do not have any spectrum of the treatment, as it was not possible to obtain the treatment of royal-impregnation cladding from the supplier or elsewhere within the timeframe of the investigations. Nevertheless, what can be observed from Fig. 9 is a similarity between the absorbance peaks on the front surface and inside of the wood, which is as expected since the impregnation penetrates the wood. Further, there are many common peaks between charcoal (scraped off charred wood before the brushing and washing process) and charred wood. This may be due to content of wood fibres in the charcoal. Wood fibres were found in some specimens when examined with a light microscope. Pure charcoal consists of carbon bonds and thus different from the more complex FTIR spectra which would arise from the multitude of different chemical bonds in wooden samples.

According to Pandey (1999, as cited in Kampe & Pfriem, 2018) a peak at 1059 cm<sup>-1</sup>, representing C-O stretching of cellulose, should not exist when the wood is strongly pyrolyzed. When looking at the spectra in Fig. 8, a peak with higher absorbance intensity depicts at this wavenumber indicating that the charred wood sample is not strongly pyrolyzed, as expected due to the brushing process as described in the methods chapter. Since many of the investigated properties are in connection to the char layer, e.g. improved wettability characteristics and sorption properties as well as increase biological and mechanical durability (Kymäläinen et al., 2017, 2018; Šeda et al., 2021), it is desirable that the char layer remains. However, a recent study carried out by Žigon & Pavlič (2023) found that higher water absorption was observed in the samples treated with charring, while removal of the char layer by brushing contributed to lower water uptake. In the same study, the results and observations after natural weathering showed that the samples treated with charring in combination with brushing are the most resistant among the surface treatment combinations tested. Based on this, the claddings are not necessarily more durable when the wood is strongly pyrolyzed, but it is probably more important that the changes are small.

An interesting finding is that for charcoal the FTIR absorbance peak at 1795 cm<sup>-1</sup> is more than doubled from 0.09 to 0.22 compared to the non-aged reference. This is also shown for multiple wave numbers in same plot. Probably, the uncertainties mentioned in chapter 3.3.1 Artificial weathering experiments – Vertical climate simulator may occur here as well, especially quantitative uncertainties (height of absorbance peak). If this is the case, the difference may be explained by the varying contact between the ATR diamond crystal and the sample, due to differences in the composition of the charcoal (sharpened charcoal fiber) and the charred wooden cladding. On the other side, it may also be due to differences in the amount of chemical bonds.

For cladding treated with primer and paint, many common peaks appear for the treatment itself and cladding with treatment, as expected. However, a greater difference was expected between the front surface and the inside of the wood due to the primer and paint are applied as a topcoat and hence do not penetrate so far into the wood like e.g. the impregnation. Noteworthy, previous results showed that e.g. peaks representing lignin were not visible, indicating that the treatment covers sufficiently the primed and painted wood.



Fig 9. FTIR absorbance versus wave number of different surface treatments. Note that it was not possible to obtain the treatment of royalimpregnated cladding from the supplier.

#### 3.3.5 FTIR comparison of aging methods and aging time

To visualize the material changes in the wooden claddings with the accelerated apparatuses and natural weathering, the FTIR absorbance at a specific wave number is plotted versus aging time in Fig. 10. As the methods have different climate acceleration aging factors, the differences in absorbance intensities may be compared and in principle be correlated to differences in the acceleration aging factors. Moreover, Fig. 10 may with limited information show more clearly the weathering development of the samples for each method as compared to the changes in the more complex full FTIR spectra. From Fig. 10, one may observe a small development for alle three methods and within the uncertainty for charred wood with UV stain and primed and painted wood, as previous results also have indicated. For charred wood and royal-impregnated wood, a greater change appears for the vertical climate simulator and Atlas climate chamber, especially at aging times of 14 and 28 days where the absorbance intensity is greater than for the rest of the aging times. This may indicate that the uncertainty is high for the measurements in this study. Therefore, a longer aging time would be of interest and could possibly determine if any chemical changes occur for the miscellaneous treatments or not.



Fig 10. Example of infrared absorbance at a specific wave number of 1059 cm<sup>-1</sup> versus time for three different aging methods.

#### 3.3.6 FTIR summary

For the FTIR absorbance plots it is evident that there is a lack of correlation between changes in absorbance peaks and increased aging time. Almost none of the wave numbers shows correlation between changes in absorbance intensities and increased time of aging. This may indicate a quantitative uncertainty, where air between the ATR diamond crystal and sample has resulted in a weaker absorbance signal, unless other conditions such as impurities etc. may indicate otherwise. Another uncertainty which may have a significant impact is the practical difficulties with obtaining the exact same sampling depth in the wood for all the specimens applying the gouge chisel. Lastly, the small changes in the height of absorbance peaks may also be explained by small or no changes in the chemical structure.

Comparison with other studies on charred wood shows that the aging time for the natural climate aging at Voll is rather short. Kymäläinen et al. (2020; 2022a) have conducted similar studies with natural weathering for both one and two years and reported changes in functional groups. It is important to bear in mind the possible weakness of her findings since the measurements are only taken before and after weathering. If measurements are not taken during weathering, quantitative uncertainties may not easily be shown. The same weakness appears in the Kampe and Pfriem (2018) study, with an artificial weathering of UV radiation right under 6 weeks. However, if the results of that study is correct, it may indicate that the aging time in the weathering experiments performed in this study is too short. The samples in the Atlas climate chamber were weathered with solar radiation for two weeks, and in the vertical climate simulator with UV radiation for five weeks, compared to Kampe and Pfriem (2018) with six weeks of UV radiation. One should bear in mind that this comparison cannot be taken directly due to the other factors such as e.g. type of radiation, intensity, temperature and humidity which also need to be considered. Therefore, it would be interesting to see if definite chemical changes would occur with prolonged aging times.

#### 4 Conclusions

The durability of charred wood and other wooden claddings has been investigated. The samples were weathered both naturally and artificially and characterized visually and chemically with a specialized camera setup and Fourier transform infrared (FTIR) radiation spectroscopy. The results showed that the accelerated artificial weathering left the samples of charred wood faded (both with and without UV resistant wood stain), whereas a clear trend of higher color stability in royal-impregnated wood and primed and painted wood was demonstrated. Chemically, at the inside of the wooden claddings, relatively close to the front surface, no clear indicators of chemical changes could be identified. At the front surface, the artificial weathering experiments with the vertical climate simulator showed indications of degradation of lignin at wave number 1508 and 1595 cm<sup>-1</sup>. The peaks for the samples of charred wood became smaller and almost completely disappeared at the end of the aging period. The other claddings did not depict a similar behavior at the same wave number, which for primed and painted wood is a positive result, indicating that the treatment covers the wood sufficiently. Also, at same wave number, indications of degradation of lignin were observed for the charred wooden claddings which were weathered in the Atlas climate chamber. In addition, royal-impregnated claddings showed indications of degradation of lignin at the same wave numbers. As the uncertainties discussed in this study may be significant, further investigations with e.g. longer aging times will be necessary to examine the development of observed results.

#### 5 References

- Buksans, E., Laiveniece, L., & Lubinskis, V. (2021). Solid wood surface modification by charring and its impact on reaction to fire performance. Engineering for rural development jelgava, 26, 899–905. https://doi.org/10.22616/ERDev.2021.20.TF203
- Cui, X., & Matsumura, J. (2019). Wood surface changes of heat-treated cunninghamia lanceolate following natural weathering. *Forests*, 10(9), 791. https://doi.org/10.3390/f10090791
- Ebner, D., Barbu, M.-C., Klaushofer, J., & Cermak, P. (2021). Surface modification of spruce and fir sawn-timber by charring in the traditional japanese method—Yakisugi. *Polymers*, *13*(10), 1662. https://doi.org/10.3390/polym13101662
- Garside, P., & Wyeth, P. (2003). Identification of cellulosic fibres by FTIR spectroscopy—Thread and single fibre analysis by attenuated total reflectance. *Studies in conservation*, 48(4), 269–275. https://doi.org/10.1179/sic.2003.48.4.269
- Goodell, B., Winandy, J. E., & Morrell, J. J. (2020). Fungal degradation of wood: Emerging data, new insights and changing perceptions. *Coatings*, 10(12), 1210. https://doi.org/10.3390/coatings10121210
- Hasburgh, L. E., Zelinka, S. L., Bishell, A. B., & Kirker, G. T. (2021). Durability and fire performance of charred wood siding (Shou sugi ban). *Forests*, 12(9), 1262. https://doi.org/10.3390/f12091262
- Jelle, B. P. (2012). Accelerated climate ageing of building materials, components and structures in the laboratory. *Journal of materials science*, 47(18), 6475–6496. https://doi.org/10.1007/s10853-012-6349-7
- Jelle, B. P., & Nilsen, T.-N. (2011). Comparison of accelerated climate ageing methods of polymer building materials by attenuated total reflectance Fourier transform infrared radiation spectroscopy. *Construction and building materials*, 25(4), 2122–2132. https://doi.org/10.1016/j.conbuildmat.2010.11.020
- Kampe, A., & Pfriem, A. (2018). A note on artificial weathering of spruce (Picea abies) with a carbonised layer. *International wood products journal*, 9(2), 86–89. https://doi.org/10.1080/20426445.2018.1493841
- Kymäläinen, M., Belt, T., Seppäläinen, H., & Rautkari, L. (2022b). Decay resistance of surface carbonized wood. *Materials*, 15(23), 8410. https://doi.org/10.3390/ma15238410
- Kymäläinen, M., Hautamäki, S., Lillqvist, K., Segerholm, K., & Rautkari, L. (2017). Surface modification of solid wood by charring. *Journal of materials science*, 52, 6111–6119. https://doi.org/10.1007/s10853-017-0850-y
- Kymäläinen, M., Lourençon, T. V., & Lillqvist, K. (2022a). Natural weathering of soft- and hardwoods modified by contact and flame charring methods. *European journal of wood and wood products*, 80(6), 1309–1320. https://doi.org/10.1007/s00107-022-01864-w
- Kymäläinen, M., Sjökvist, T., Dömény, J., & Rautkari, L. (2022c). Artificial weathering of contact-charred wood—The effect of modification duration, wood species and material density. *Materials*, 15(11), 3951. https://doi.org/10.3390/ma15113951
- Kymäläinen, M., Turunen, H., Čermák, P., Hautamäki, S., & Rautkari, L. (2018). Sorption-related characteristics of surface charred spruce wood. *Materials*, 11(11), 2083. https://doi.org/10.3390/ma11112083
- Kymäläinen, M., Turunen, H., & Rautkari, L. (2020). Effect of weathering on surface functional groups of charred norway spruce cladding panels. *Forests*, *11*(12), 1373. https://doi.org/10.3390/f11121373
- Machová, D., Oberle, A., Zárybnická, L., Dohnal, J., Šeda, V., Dömény, J., Vacenovská, V., Kloiber, M., Pěnčík, J., Tippner, J., & Čermák, P. (2021). Surface characteristics of one-sided charred beech wood. *Polymers*, 13(10), 1551. https://doi.org/10.3390/polym13101551
- Nordtest Method NT Build 495. (2000). Building materials and components in the vertical position: Exposure to accelerated climatic strains.

- Pandey, K. K. (1999). A study of chemical structure of soft and hardwood and wood polymers by FTIR spectroscopy. *Journal of applied polymer science*, 71(12), 1969–1975. https://doi.org/10.1002/(SICI)1097-4628(19990321)71:12<1969::AID-APP6>3.0.CO;2-D
- Sandak, A., Sandak, J., Brzezicki, M., & Kutnar, A. (2019). *Bio-based building skin*. Springer Open. https://doi.org/10.1007/978-981-13-3747-5
- Šeda, V., Machová, D., Dohnal, J., Dömény, J., Zárybnická, L., Oberle, A., Vacenovská, V., & Čermák, P. (2021). Effect of weathering on surface functional groups of charred norway spruce cladding panels. *Applied sciences*, 11(9), 4086. https://doi.org/10.3390/app11094086
- SINTEF Byggforsk. (2022). 542.101 Liggende og stående trekledning. https://www.byggforsk.no/dokument/472/liggende og staaende trekledning
- Srinivas, K., & Pandey, K. K. (2012). Photodegradation of thermally modified wood. *Journal of photochemistry and photobiology B: biology*, 117(5), 140–145. https://doi.org/10.1016/j.jphotobiol.2012.09.013
- Strand, S. M., & Hovde, P. J. (1999). Use of service life data in LCA of building materials. 8th International conference on durability of building materials and components, 8dbmc, 1948–1958.
- Tettey, U. Y. A., & Gustavsson, L. (2019). Primary energy and CO2 emissions implications of different insulation, cladding and frame materials for residential buildings. *IOP conference series: earth and environmental science*, 297(1), 012020. https://doi.org/10.1088/1755-1315/297/1/012020
- Werner, F., & Richter, K. (2007). Wooden building products in comparative LCA. The international journal of life cycle assessment, 12(7), 470– 479. https://doi.org/10.1065/lca2007.04.317
- Zicherman, J. B., & Williamson, R. B. (1981). Microstructure of wood char. Wood science and technology, 15(4), 237-249. https://doi.org/10.1007/BF00350942
- Žigon, J., & Pavlič, M. (2023). Assessment of the combined charring and coating treatments as a wood surface protection technique. *Forests*, *14*(3), 440. https://doi.org/10.3390/f14030440

## Further work

There are several research opportunities in the extension of this work. First, and foremost the duration of the project should be extended with additional aging time. The experimental frame at Voll will be weathered for at least one, and perhaps two years, to gain more knowledge of degradation of charred wooden claddings in relations to other cladding types. Furthermore, continuing the artificial climate aging of the samples in the vertical climate simulator would be of interest.

Climate aging in Atlas climate chamber could be explored greater by aging the claddings with different climate factors. In this thesis, the claddings were exposed to only UV radiation. By results from the article and the test wall at AG-Tre (see appendix B), it would be interesting to expose the claddings to both rain and UV radiation, and only rain. By this, we can more closely investigate the phenomena that arose in the article, to what extend rain itself causes the observed discoloration.

Visual and material characterization by DigiEye and FTIR can advantageously be used further. Especially, the experimental frame at Voll should be photographed with DigiEye due to various light conditions outdoors. It should be mentioned that this is planned to be done when the claddings have been standing for one year. Sampling used for FTIR measurements can be done for all the apparatus, in line with extended aging time, since none of the samples showed clear signs of aging. Other characterization methods such as Raman spectroscopy should be considered due to higher uncertainties in measurements of wood with FTIR. Kymäläinen et al. (2022a) have reported better results by using this method, especially when analyzing wood with high degree of carbonization.

Furthermore, a life cycle assessment (LCA) of the claddings should be conducted. If the degradation of charred wood is higher than for the other claddings, the environmental footprint may be higher due to increased requirements of maintenance. Therefore, in connection to extended aging, a LCA should be worked out to determine whether charred wood has a lower environmental impact than the other claddings.

### Conclusions

This master's thesis has analyzed the weathering resistance of miscellaneous wood surfaces. Both natural and artificial climate aging was used to degrade the samples, and the specimens were evaluated by using material characterization methods such as Fourier transform infrared (FTIR) spectroscopy and a specialized camera setup. The specialized camera setup depicts a clear color change in the charred wooden claddings, also the charred wood treated with UV resistant wood stain. The color change appeared after only six weeks in the artificial weathering test with the vertical climate simulator. For the other claddings, primed and painted wood, and royal-impregnated wood, no clear color changes were observed. Chemically, from the FTIR analysis, indicators of lignin degradations appear at the front surface of the claddings, except for primed and painted wood. However, no clear indications of chemical changes were shown at the inside relatively close to the front surface, indicating relatively weather resistant wooden claddings. As the uncertainties discussed in this study may be significant, further investigation with e.g. longer aging times will be necessary to examine the development of the observed results.

Based on the findings mentioned in the above, it can be concluded that charred wood with and without UV resistant wood stain fade to grey relative quickly compared to the other tested claddings that did not show any clear visible difference. However, none of the claddings stand out as considerably more or less durable.

### References

Kymäläinen, M., Hautamäki, S., Lillqvist, K., Segerholm, K., & Rautkari, L. (2017). Surface modification of solid wood by charring. *Journal of materials science*, *52*, 6111–6119. https://doi.org/10.1007/s10853-017-0850-y

Kymäläinen, M., Lourençon, T. V., & Lillqvist, K. (2022a). Natural weathering of soft- and hardwoods modified by contact and flame charring methods. *European journal of wood and wood products, 80*(6), 1309–1320. https://doi.org/10.1007/s00107-022-01864-w

## Appendix

Appendix A: Acceleration factor

Appendix B: Test wall at AG-Tre

### Appendix A: Acceleration factor

To estimate the required aging time in the climate aging apparatus, the acceleration factors need to be calculated [A1]. By doing this, the service life of the product may be determined. In a simplified model one may assume that the total acceleration factor  $AF_{tot}$  equals the product of the acceleration factor caused by UV radiation  $AF_{uv}$  and temperature  $AF_{temp}$ .

The acceleration factor caused by UV radiation may be calculated as directly proportional to the ratio between the total UV energy in the laboratory aging apparatus  $Ø_{lab}$  and the natural outdoor aging  $Ø_{nat}$  for a given period:

$$AF_{uv} = \phi_{lab} / \phi_{nat}$$

The higher UV intensity  $(W/m^2)$  and total energy  $(kWh/m^2)$  in the aging apparatus, the higher acceleration factor. The equation above is valid if one may assume that all the UV radiation contributes to initiating degradation reactions, in addition to an equal spectral distribution for natural and artificial UV radiation, which naturally is never completely fulfilled.

The acceleration factor caused by temperature can be determined with the Arrhenius equation, where the chemical degradation process increases exponentially with increasing temperature. Temperature acceleration factor  $AF_{uv}$  is calculated as the ratio between the reaction rate in the laboratory aging apparatus  $k_{lab}$  and the natural outdoor aging  $k_{nat}$ :

$$AF_{temp} = k_{lab}/k_{nat} = \frac{C_{lab} \cdot e^{-E_{lab}/(RT_{lab})}}{C_{nat} \cdot e^{-E_{nat}/(RT_{nat})}} \approx \frac{e^{-E_{lab}/(RT_{lab})}}{e^{-E_{nat}/(RT_{nat})}}$$

Respectively,  $T_{lab}$  and  $T_{nat}$  denote the temperature in the laboratory aging apparatus and the natural outdoor aging, and R = 8.314 J/(Kmol) is the gas constant. It is assumed that the pre-exponential factor C is temperature independent, giving  $C_{lab} \approx C_{nat}$ . It is also assumed that the activation energy E is independent of temperature and set to  $E_{lab} = E_{nat} = 70\ 000\ J/mol$ . The equation of the total acceleration factor caused by UV radiation and temperature is expressed as follows:

$$AF_{tot} = AF_{uv} \cdot AF_{temp} = \frac{\phi_{lab}}{\phi_{nat}} \cdot \frac{e^{-E_{lab}/(RT_{lab})}}{e^{-E_{nat}/(RT_{nat})}}$$

The equation is of course a strong simplification. Other climate factors such as water, physical strains (e.g. snow loads), and microorganisms will influence AF<sub>tot</sub>. It should also be mentioned that the acceleration factor is strongly dependent upon the natural outdoor aging comparison exposure level, e.g.  $T_{nat}$ . To determine the real AF<sub>tot</sub>, one should perform calculations with short time intervals which hence are integrated, and strictly not base the calculation upon annual or daily values for  $Ø_{nat}$  and  $T_{nat}$ . It is important to note that a natural outdoor exposure period at a high temperature and an equally low temperature, will result in a substantially higher reference temperature than the average temperature. This is due to the exponential increase in reaction rate with increasing temperature, as expressed in the Arrhenius equation. Based on this, the natural outdoor aging comparison by accelerated climate aging in a laboratory, the simplified model may be applied. Anyway, one also should compare the laboratory results with outdoor tests in natural climates. Typical values for AF<sub>tot</sub> are often between 5 and 250.

Calculations of the acceleration factor for artificial climate aging in the vertical climate simulator is difficult to determine due to the exposure cycle of four hours with different temperatures and exposure factors such as rain and frost. However, researchers use an acceleration factor of approximately 12. The acceleration factors of artificial climate aging in the Atlas climate chamber are calculated, with the equation above used, and is shown below:

#### Atlas climate chamber

Temperature						
T <sub>lab</sub> = 60 °C + 273 = 333 K	T <sub>nat</sub> = 22 °C + 273 = 295 K					
UV radiation						
$\emptyset_{lab} = \Sigma (\emptyset_{Atlas} \cdot h_{month})$	$Ø_{nat} = \Sigma (Ø_{month} \cdot h_{month})$					
$Ø_{Atlas} = 1 \ 200 \ W/m^2$	$Ø_{month}$ is monthly average values for radiant flux in W/m <sup>2</sup> of a standard					
hmonth is total nour per month, and is:	reference year taken from NS 3031,					
$n_{31days} = 744 \text{ nours}$	Tab. M.2 [A2]. When comparison with Voll					
$H_{30days} = 720 Hours$	(gradient 30°, orientation South),					
	used.					
By applying the equation above	$\phi_{ian} = 21$ $\phi_{may} = 205$ $\phi_{sen} = 132$					
$Q_{lab} = 10\ 512\ kWh/m^2$	$Ø_{feb} = 56$ $Ø_{iun} = 249$ $Ø_{oct} = 66$					
	$Ø_{mar} = 115$ $Ø_{jul} = 222$ $Ø_{nov} = 34$					
	$Ø_{apr} = 176$ $Ø_{aug} = 198$ $Ø_{des} = 19$					
$h_{month}$ is total hour per month, an $h_{31days} = 744$ hours $h_{30days} = 720$ hours $h_{28days} = 672$ hours						
	By applying the equation above					
	Ø <sub>nat</sub> = 1 093 KWN/M <sup>2</sup> .					
Acceleration factor						
$AF_{uv} = \frac{\emptyset_{lab}}{\emptyset_{nat}} = \frac{10512}{1093} = 9.6$						
$AF_{temp} = \frac{e^{-E_{lab}/(RT_{lab})}}{e^{-E_{nat}/(RT_{nat})}} = 26.0$						
$AF_{tot} = AF_{uv} \cdot AF_{temp} = 250$						

### Appendix B: Test wall at AG-Tre

The collaborating manufacturing enterprise AG-Tre has a test wall at their location in Orkland municipality consisting of charred wooden cladding facing southwest. The wall was installed 8th of June 2022 to see which changes occur in natural conditions. 28th of April 2023 a new photo was taken, 46 weeks and 2 days later, showing a color change along the edge of the wall, see Fig. B1. The changes were already first observed 2nd of September 2022, 12 weeks and 3 days after installation.



**Fig. B1.** Left: The test wall when installing 8th of June 2022. The edges were cut right after the picture was taken. Right: Picture of the test wall taken 28th of April 2023 showing a color change along the edge.

The observed difference in color may be due to rain, with a clear color change 55 cm above the cladding. The distance between the ground and the cladding is approximately 25-30 cm, which may exclude the suspect of the spurt of water [B1]. On the other hand, it seems that the color change has a connection to lack of roof gutter, which causes rain from the roof to drips onto the lower part of the wall and gets soaked whenever it rains. In addition, the eaves (overhang of 35 cm) do not cover sufficiently when wind-driven rain appears so the degradation along the edge becomes even higher.

This experiment shows that charred wood may be more susceptible to weathering by rain than the result in the article shows. This indicates that both UV radiation and rain may change the visual appearance of charred wood, in addition to the orientation of the wall together with the climate. However, further studies are needed to gain more knowledge of the performance of charred wooden claddings.



