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Emissions of Volatile Organic Compounds (VOCs) from Interior Wood Panelling

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Sammendrag

Flyktige organiske forbindelser (VOC-er) er en gruppe av organiske kjemikalier som lett fordamper ved omgivelsestemperaturer, bl.a. fra byggematerialer. Dette gjør at de kan påvirke inneluftkvaliteten. Det finnes mange ulike typer VOC-er, i første rekke er det type materiale som bestemmer hvilke VOCer frigjøres til inneluften. Deretter vil materialbehandling og fysiske forhold bestemme konsentrasjonen og sammensetningen av disse forbindelsene. De siste årene har det blitt et økt fokus på VOC-emisjoner, ettersom konsentrasjoner fra disse forbindelsene er ofte høyere innendørs enn utendørs. Dette kan ha en effekt på menneskehelse, spesielt med tanke på at befolkningen i Skandinavia tilbringer over 90% av tiden sin innendørs. Derfor setter stadig flere reguleringer og lovverk krav til VOC-konsentrasjoner fra byggematerialer bl.a. trematerialer, og nivået av VOC-er innendørs.

Et modellsystem, med bakgrunn i kravene for den europeiske standarden for vurdering av frigjøring av farlige stoffer til inneluft (EN 16516) ble utviklet. Dette ble gjort for å sammenligne VOC-emisjoner fra to forskjellige typer av interiørpanel fra *furu (Pinus Sylvestris)*. De mest vanligst forekommende VOC-ene fra furu ble analysert fra disse prøvene, ved bruk av termodesorpsjon-gasskromatografi (ATD GC-MS). Emisjonene fra tre prøver av ubehandlet interiørpanel, og tre prøver av beiset interiørpanel ble analysert i forhold til kravene i den europeiske byggevareforordningen (EU-LCI).
Målene med dette studiet var å utvikle et modellsystem for analyse av VOC-er, og utforske resultater i forhold til tidligere undersøkelser. Deretter var det et mål å utforske hvordan materialbehandling eventuelt påvirker VOC-emisjoner, samt finne ut om disse materialene oppfyller kravene satt i EU-LCI.

Modellsystemet klarte å kvantifisere seks av de mest forekommende forbindelsene i bartrær, *heksanal, α-pinene, β-pinene, β-myrcene, camphene og 3-carene.* Det ble ikke funnet noen statistisk signifikant forskjell mellom de ubehandlede og behandlede materialene, til tross for at det ubehandlede materialet hadde langt høyere konsentrasjoner enn det ubehandlede materialet. Ingen av prøvene klarte å oppfylle EU-LCI kravene etter 28 dager, men dette kan også skyldes modellsystemet. Det er derfor viktig å utvikle dette systemet videre, spesielt med tanke på at målinger av emisjoner fra VOC-er vil bli et stadig større krav i forskjellige reguleringer og lovverk.

Abstract

Volatile organic compounds (VOCs) are a diverse group of organic chemicals that easily evaporate at ambient temperatures, e.g. from building materials. This makes them likely to affect indoor air quality (IAQ). There are many different types of VOCs, and their concentration and composition depend on a number of different factors. Primary, the type of material determines which VOCs will be present in a given environment. Then other factors, such as the material treatment and physical conditions decide the concentration and composition of these compounds in an environment. There has been an increasing focus on VOC-emissions indoors over the last years, as the concentrations of these compounds are higher indoors than outdoors. VOCs can also have an effect on human health. This has led to an increasing number of regulations establishing requirements for VOC emissions from different types of materials, e.g. wood materials, and the level of VOCs in indoors.

A model system was developed to compare VOC emissions from two different types of *Scots Pine* interior panelling (*Pinus Sylvestris*). The model system was based on the requirements of the European standard for the assessment of the release of hazardous substances for indoor air (EN 16516). The most common VOCs in *Scots Pine* were analysed using thermo-desorption gas chromatography (ATD GC-MS). Overall, the emissions from three samples of untreated interior panel, and three samples of stained interior panel were analysed in accordance to the requirements of the Construction Products Regulation (EU-LCI).
The objectives of this study were to develop a model system for the analysis of VOCs, and to compare the analysis from this model system in relation to previous studies. Other objectives were to explore how material treatment might affect VOC emissions, as well as find out if these materials meet the requirements set in EU-LCI.

The model system was able to quantify six of the most abundant compounds in conifers, i.e. *hexanal, α-pinene, β-pinene, β-myrcene, camphene and 3-carene.* No statistically significant difference was found between the untreated and treated materials, even though the untreated material had substantially higher concentrations than the untreated material. None of the samples met the EU-LCI requirements after 28 days, but this could also be due to the model system. It is therefore important to further develop this system, especially as testing of emissions from VOCs will become an increasingly important requirement in various regulations and legislations.

Abbreviations and definitions

AVOC	Anthropogenic volatile organic compounds
BVOC	Biogenic volatile organic compounds
Ch.	Chapter
CLT	Cross-laminated-timber
CPR	Construction Products Regulation
EEA	European Economic Area
ESTDM	External standard method
EU	European Union
IAP	Indoor air pollutants
IAQ	Indoor air quality
ISTDM	Internal standard method
LCI	Lowest concentration of interest
LOD	Limit of detection
LOQ	Limit of quantification
MRM	Multi reaction monitoring
RH	Relative humidity
RT	Retention time
STDAM	Standard addition method
TVOC	Total volatile organic compounds
VOC	Volatile organic compounds
VVOC	Very volatile organic compounds
SD	Standard deviation
SVOC	Semi volatile organic compounds
WHO	World Health Organization

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1. Introduction and objectives

This master thesis consists of three different objectives. The first objective is to develop a model system capable of measuring VOC emissions over time, as close as possible to the European standard for determination of emissions into indoor air; EN16516: Construction products: Assessment of release of dangerous substances – Determination of emissions into indoor air products (European Committee for Standardization, 2017).

The second objective is to use this model system to quantify VOC emissions from wood two differently treated types of *Scots Pine* interior panelling. One type of panelling will be untreated, while the other type will be coated with acrylate paint. The aim is to examine how the model and analytical system perform, while also analysing the difference between these sample types.

The aim of the third objective is to explore how these test results compare to results from similar studies. Their performance in accordance to critical values in EN 16516 will be also explored.

1.1. Background

The emissions of VOCs from wood and wood-based products has become an increasingly more discussed topic, especially as the use of wood as a building material has become more prevalent globally. VOCs have an important part in indoor air quality (IAQ), as they are considered to be one of the most prevalent groups of indoor air pollutants (IAP). VOCs can therefore have a great impact on the IAQ, with several types of health issues connected to VOCs (Tsakas et al, 2011). Wood VOCs are commonly found in indoor environments, i.e. in wood and wood-based building products, materials, furnishings and furniture (Fechter et al., 2006). IAQ is an important topic of research, as bad IAQ may affect health, comfort, well-being and productivity of residents and has therefore become a focus area over the last decades (Englund, 1999; Spengler et al., 2001).

1.1.1. Indoor air quality (IAQ)

IAQ is a term used to describe the indoor climate within and around residential, public and commercial buildings, while IAPs are various types of particles, microbes and gaseous phase compounds which may affect the IAQ (Fechter et al.,2006; Tsakas et al., 2011). In order to achieve good IAQ, it is necessary to maintain parameters such as T, RH and the ventilation rate within a certain comfort range. At the same time, it is also necessary to keep concentrations of IAPs such as respirable dust, carbon dioxide and VOCs below a certain level. Good IAQ is important as studies show that humans, especially in Scandinavia, spend more than 90% of their time indoors and breath in about 15 000 litres of air per day (Klepeis et al., 2001; Schweizer et al., 2007). Studies by Prussin & Marr (2015) further show that the indoor air can contain concentrations of 1-10 ppm/m^3 of different types of particles and microorganisms, depending on the source materials. The different types of materials used in construction products, along with furnishings, and their treatment also affect IAQ and the types and concentrations of particles and microorganisms present in indoor air. The concentrations and effects of these materials on IAQ are not always known. This leads to their possible determinal effects often not being known until humans have been exposed to these materials. It is therefore important to be both aware of possible pollutants in the air, their sources and how their possible determental health effects can be minimized.

Pollutants in the indoor air, such as various types of particles, microbes and gasesos phase compunds are commonly refered to as indoor air pollutants (IAP). IAPs can be divided based on their effects on human health, how often they appear in indoor air, their usual concentrations, their sources or if the polutant is a chemical or biologic substance (Tsakas et al., 2011). Chemical pollutants can further be divided into organic and inorganic compounds, where organic compounds contain carbon and inorganic do not (Plesser, 2012). When it comes to chemical indoor air pollutants, most of these are gases, vapours and particulate matter. The gaseous phase organic compounds released into the indoor air, such as VOCs, can be associated to various health symtoms ranging from mild irritations and unpleasent odors to serious health problems. Furthermore, the chemical compunds in the indoor air can react with chemicals from the products present in a given indoor enviroment and create additional pollutants which also affect the IAQ (Plesser, 2012).

1.1.2. Impact of indoor air pollutants (IAP) on health and productivity

The impact of IAQ on indoor air quality can have a great effect on health and productivity, with links between bad IAQ and various health symptoms. These symptoms include headache, nausea, respiratory infections and symptoms of building-related illnesses which furthermore include irritation of eye, nose and throat, dry cough, dry or itchy skin, dry mucous membrane, dizziness and nausea, difficulty in concentration, mental fatigue and erythema (Tsai, 2019). Exposure to IAPs, especially chemicals, may also affect the nervous system resulting in impaired concentration and attention along with psychosocial stress reactions (Skulberg et al., 2019). These links between health issues and exposure to IAPs are complex, with few studies exploring how chemical emissions from wood affect humans directly. Even when taking past and present studies on indoor air pollutants into account it is difficult to define this link.

Most of the research regarding health effects from wood based emissions is based on questionnaires and how residents experience indoor air environments (Wolkoff, 2012). Indoor air research commonly studies irritation or olfactory responses, i.e. the thresholds at which humans get a symptom or can detect an odour. These levels depend on the compound and the exposed individual, with certain parts of the population being more sensitive to IAPs. Though, even when taking these sensitive groups into account the existing studies addressing health effects from wood emissions there have not been found any consistent associations between exposure and health outcomes (Skulberg et al., 2019). Furthermore, exposure experiments with terpene oxidation products, such as carboxylic acids, show no statistically significant differences in respiratory or cognitive health (Skulberg et al., 2019). These chemical compounds are important, as they are even more reactive than their corresponding aldehydes and may play an important role in the formation of secondary organic aerosols (SOAs) that might further affect human health.

Current VOC regulations, such as the Lowest Concentration of Interest (EU-LCI) concept, the AgBB scheme, the M1 Emission Classification of Building Materials, the French Agency for Food, Environmental and Occupational Health & Safety (ANSES) VOC regulations and other schemes in Denmark and Belgium are based on potential health risks arising from exposure to individual VOCs (Jäckh et al., 2013). The limit levels for VOCs in these regulations are based on toxicity studies, with both humans and animals being exposed to various types of indoor air pollutants. Though, these studies are not uniform and are often based on both humans and different animal species with possible limitations when it comes to generalizing the results to humans. This is due to the fact that many rodents and rabbits have significantly different nasal passages compared to those of humans. This means that these results may not be directly applicable to humans. Furthermore, exposure experiments with animals are difficult to perform and lack standardization (Doty et al., 2004).

When it comes to human exposure experiments, some weak links between exposure to VOCs and negative health effects have been found (Wolkoff, 2020). Though, these links are based on short term experiments and no significant health effects on participants in this type of studies were found (Gminski et al., 2011a; Gminski et al., 2011b). Skullberg et al. (2019) also found no certain links between exposure to VOCs and negative health effects. There is also doubt that possible negative health effects are due to exposure to IAPs. The perception of the chemicals and work performance might be influenced by an unpleasant odour coming from the IAPs (Skulberg et al., 2019). It is also important to consider that these types of human exposure experiments are often based on short-time exposure, lasting for up to two hours. Thus, there is a need for exposure experiments with a longer duration. This will give further knowledge and data on how long-time exposure to VOCs can affect human health. Another issue is the practicality of this type of studies, i.e. how can humans stay in the same environment long enough to draw conclusions regarding possible health effects from IAPs. Until such long-time studies have been performed it is difficult to draw any certain conclusions when it comes to possible health effects of exposure to VOCs.

2. Theory

Volatile organic compounds (VOCs) are a diverse group of organic chemicals that have a high vapour pressure. Due to their volatility, the VOCs present in indoor air have elevated concentrations and can be up to five times higher than the VOC outdoor levels (Jones, 1999). There are many different types of VOCs, and their nature and concentrations present in any given environment will depend on a range of factors. These factors can be endogenous, including genetic and biochemical factors, or exogenous such as growth conditions, wood treatment, physical and chamber conditions like relative humidity (RH), temperature (T), ventilation and the material storage conditions (Kesselmeier & Staudt, 1999; Pohleven et al., 2019). There is also a difference between VOCs produced and emitted by living processes, compared to those produced and emitted through anthropogenic processes. Biogenic VOCs (BVOCs) are of natural origin and are emitted by living organisms, such as trees and other plants. These VOCs are most commonly found in untreated wood, and are a large part of VOC emissions from softwood trees such as *Scots Pine* (Pohleven et al., 2019). Anthropogenic VOCs (AVOCs) are emitted, for example, from synthetics resins, surface treatments or coatings and are more commonly found in wood composites and other types of wood products. In this study, the focus will mainly be on BVOCs, as they are most common in untreated wood and constitute the largest concentrations of VOCs found in softwood.

There has also been an increased interest in the reduction of VOC product emissions, due to undesirable health effects. Several evaluation and classification schemes, along with guidelines and limit values have been developed with the reduction of VOC product emissions in mind. Several voluntary schemes have been used for the testing and evaluation of building products, furniture along with a harmonized European standard for the assessment of release of dangerous substances –and the determination of emissions into indoor air (Fechter et al., 2006; Oppl, 2014). In addition, several regulation and certification schemes such as Building Research Establishment Environment Assessment Method (BREEAM), Leadership in Energy and Environmental Design (LEED) have performance requirements and VOC content limit values for building products and furniture including paints and varnishes, wood panels, timber structures, wood flooring, laminated floor coverings, wall coverings and flooring products (BREEAM-NOR, 2016; Stratev et al., 2016). National standards for the testing and health evaluation of VOCs from construction products, such as AgBB, M1 and ANSES also exist. This chapter will also cover these types of regulations, along with different types of testing methods used to evaluate VOC emissions.

2.1. Volatile organic compounds (VOCs)

The prevalence of VOC in indoor air has led to VOCs being the most studied group of indoor air pollutants (Spengler et al., 2001). There are several sources of VOCs in indoor air, with the most significant sources being emissions from building materials and human activities inside buildings (Tsakas et al., 2011). The types of VOCs present in indoor air will mainly depend on the wood species and the material treatment (Plesser, 2012). Processed wood materials will have different VOC content compared to untreated wood materials, and coated wood materials will also differ from untreated or processed wood materials. Different wood species will also contain different types of VOCs. Additionaly, VOCs are a diverse group of chemicals consisting of terpenes, aldehydes, ketones, alchohols, aromatic hydrocarbons, acids and esters. These VOC groups might affect human health differently, and their concentrartions and profiles will depend on the abovementioned factors.

Due to the abundance of VOCs, it is important to categorize them in order to analyse them correctly and have an approximation of which VOCs can be expected in a given environment. (World Health Organization (WHO), 1989) categorizes VOCs into three groups based on their volatility, i.e. the boiling point; very volatile organic compounds (VVOCs), volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs). These groups are presented in table 1.

Table 1: VOC definitions according to WHO (WHO, 1989)

Description	Abbreviation	Boiling point range (°C)	Example compounds
Very volatile (gaseous) compounds	VVOC	<0 to 50-100	Propane, butane, formaldehyde*
Volatile organic compounds	VOC	50-100 to 240-260	d-Limonene*, hexanal*
Semi volatile organic compounds	SVOC	240-260 to 380-400	Fire retardants (PCBs, PBB)

**VOCs commonly found in wood*

These definitions are important as they can be used to estimate how certain VOCs will occur in an indoor environment, where VVOCs and VOCs will appear as gasses and SVOCs will appear as airborne particles. The boiling ranges and different VOC definitions also make it possible to estimate from which substances and materials these VOCs will be emitted (Plesser, 2012). VOCs can be found in wood and wood products, VVOCs such as formaldehyde can be found in glued products and SVOCs can be found in paints and coatings (Plesser, 2012) As previously mentioned, the VOCs concentrations may also vary depending factors such as T, RH, the ventilation system and the source strength (Wolkoff, 2012). The boiling point of a VOC will also have an influence on how a particular compound can be analysed, as not all the types of VOCs can be analysed by the same methods. Both these points will be further discussed in their own chapters.

2.2. Total volatile organic compounds (TVOC)

Another important term in regard to VOCs and indoor air pollutants is total volatile organic compounds (TVOC). This is a term that has been loosely used to refer to several different parameters in the context of testing chemical emissions from products (European Committee for Standardization, 2017). Usually, the TVOC term has been used to summarize all the compounds collected by an air sampler containing the Tenax TA sorbent and quantified by using thermal desorption and gas chromatography (TD/GC) analysis (Jäckh et al., 2013). Typically, different types of eco labels, certification schemes and programs for sustainable buildings use TVOC both as a measurement of the total level of VOCs in an indoor environment, and also as a measurement of the total VOC emissions from a given product.

Even though the TVOC term is widely used to evaluate emissions from products and IAQ, it is not a reliable indicator of the effects VOCs may have on human health (Jäckh et al., 2013). This is both due to different TVOC definitions being used differently, and different VOCs having different characteristics. Certain VOCs may have more adverse health effects than others. A high TVOC value may be harmless, while a low TVOC value may result in poor IAQ. This may be due to strong odour perception, or the ratio/composition of different VOCs in any given material.

With this in mind, this study will use the sum of all of the VOCs the analytical system is capable of quantifying as the TVOC definition. This information will be used to examine the concentration changes over time and analyse the differences between the samples used in this study.

2.3. VOC testing methods

Testing VOC emissions into indoor air has been the subject of intensive method development for over 20 years. Different organizations have developed regulations and guidelines for testing and regulating IAPs, as a response to public awareness of their health effects and governmental response to said health effects (Woolfenden, 2009). Traditionally there have been different methods for testing VOC emissions from materials, with different organizations using their own methods. Although, these recent developments have led to the need for standard emission testing methods, which are both robust and repeatable for laboratories to carry out. Uniform testing methods, both between different countries and markets are necessary if manufacturers are to avoid having to submit the same products for emission testing by multiple different emissions certification protocols.

Different testing methods have relied on different sampling methods, detection methods and test or certification protocols. The types of chambers, cells and model rooms and the required testing climate has also varied. Active sampling, where a pump draws a specified amount of air through an adsorbent tube is currently the most used sampling method (Plesser, 2012). This is because active sampling is more reliable than other types of sampling, and commercial adsorbent tubes, e.g. Tenax-tubes, have become ubiquitous (Lundgren et al., 1994). Other testing methods have relied on grab sampling, where Summa®-polished steel or aluminium canisters allow sampling over a short time (10-30 seconds) (Lundgren et al., 1994). There have also been passive samplers, which are similar to active samplers. The main difference between these two types of samplers are that passive samplers do not rely on a pump drawing the air, instead the cross section of the adsorbent tube and the distance between the opening of the tube and the adsorbent surface decide the sampling rate of the passive sampler. Gas detector tubes have also been used, but suffer from low sensitivity (Lundgren et al., 1994).

The most used detection methods are gas chromatography (GC-MS) and flame ionization detection (GC-FID) (Oppl, 2014). Additionally, high-performance liquid chromatography (HPLC) is used for more volatile compounds. Both GC-MS and GC-FID are commonly used to test VOC emissions, but GC-MS has become more common over time. This is due to the GC-MS systems having a good ability for identification, the possibility to check for interferences and sensitivity (Woolfenden, 2009). GC-MS is also the analytical system required in EN16516, along with the use of Tenax tubes and a thermal desorber (European Committee for Standardization, 2017). Test or certification protocols are similar to standard test methods but include additional requirements. The emission test method, when it should be carried out and for how long (e.g. a test period of 28 days, with tests at day 3 and 28), a list of target compounds and pass/fail criteria are types of such requirements. Pass/fail criteria may be based on health evaluated values, such as limit levels above which a product would fail the certification process because a VOC emitting above a set level might have adverse health effects (Woolfenden, 2009).

2.3.1. Standardized methods for testing VOC emissions

There are several standardized methods for testing VOC emissions, with the methods becoming more uniform over the last few years. This is with EN16516 especially in mind, as this is a harmonized European standard for determining emissions of dangerous substances into indoor air. The goal of this standard has been to develop a robust, repeatable and reproducible testing method (Oppl, 2014). EN16516 is based on the ISO 16000 standard series, with a few key differences cf. Table 2. The ISO 16000 standard series has previously been the most used standardized test method for emissions from building products, and is a widely adopted test method in Europe (Plesser, 2012). Other standardization agencies, such as Japanese Industrial Standards (JIS), ASTM International and certification agencies, such as LEED, BREEAM and national regulations such as AgBB, M1 and ANSES have all required the use of the ISO 16000 standard series when testing building products.

Table 2: Differences between ISO 16000 and EN16516 (Oppl, R. 2014)

Parameter	ISO 16000	EN16516
Temperature in ˚C	23 ± 2	23 ±1
RH%	50 ± 5	50 ± 5
Air change rate (per hour)	Variable	0,25 to 1
Chamber material	Stainless steel or glass	Stainless steel or glass
Chamber size	Minimum 20L	Not specified
VOC sampling	Tenax TA	Tenax TA
VOC desorption	Thermal desorption	Thermal desorption
VOC analysis	GC-MS or GC-FID	GC-MS

It is expected that EN16516 will become the main testing method in Europe over the next years, as all construction products traded in the EU are going to require a Declaration of Performance (Oppl, 2014). There are a few other widely used standardized test methods, not based on the ISO 16000 standard series, but these are mostly used as a supplement to this standard series. This has to do with volatility, as not all VOCs can be tested in the same way.

VVOCs, such as formaldehyde, require a more sensitive analytical method in order to be quantified. This is because higher volatility equals a lower boiling point, which makes it much more difficult to analyse these compounds by using the ISO 16000 standard series (Salthammer, 2016; United States Environmental Protection Agency (EPA), 2020). The best known VVOC is formaldehyde, due to its extensive use as a resin in the wood industry and adverse health effects. This has led to several methods being used to quantify formaldehyde emissions from different products. The most common method is EN 717, presented in Table 3, which is recommended in EN 16516 when testing formaldehyde emissions (European Committee for Standardization, 2017). This test method is only applicable for formaldehyde, and is used widely adapted in the European Single Market (Oppl, 2014).

Table 3: EN 717 testing requirements (Oppl, R. 2014)

Parameter	EN 717
Temperature in ˚C	23 ± 0.5
RH%	45 ± 3
Air change rate (per hour)	1.0 ± 0.05
Chamber material	Stainless steel, aluminium, glass, PVC, PMMA…
Chamber size	12m^3, 1m^3 or 225L
VOC sampling	Not applicable
VOC desorption	Not applicable
VOC analysis	HPLC

There are also other methods for testing formaldehyde, but most of these are similar or adapted from EN717 (Kim et al., 2006). Notable methods for testing formaldehyde emissions are the Japanese standard method with a desiccator, and the Korean and Swedish methods (Kim et al., 2006). Though, in the interest of finding a uniform test method for quantifying all the types of VOC there has been some research trying to use EN16516 in this manner. EN16516 has not yet been evaluated for stable concentrations of formaldehyde, but a study by (Wilke & Jann, 2018) compared the existing standard for testing formaldehyde and EN16516. This study showed that EN 16156 is able to measure and quantify formaldehyde exactly with a conversion factor (Wilke & Jann, 2018). These results indicate that EN16156 might be the first universal method for testing all types of VOC in the future.

2.4. Common VOCs found in wood and wood materials

The VOC emissions from wood and wood materials are reflected in the properties of the material, and other intrinsic and extrinsic factors. According to (Pohleven et al., 2019) wood is a complex multicomponent biopolymer with inherent variability, which leads to different types of VOC emissions. These emissions can be attributed to various endogenous and exogenous factors, as previously mentioned. In this study, the chosen species is *Pinus Sylvestris (Scots Pine)*, which is considered to be a high VOC emitting wood species. Volatile terpenes contribute most to the high VOC concentrations (70-90%) seen in *Scots Pine*. Other VOCs, mainly aldehydes (10-25%), organic acids and alcohols contribute to the VOC emissions from *Scots Pine* (Pohleven et al., 2019). This chapter will focus on common VOCs found in *Scots Pine*, how these VOCs are formed and their emission patterns.

The main reason that *Scots Pine* and other softwoods are seen as high emitting VOC species is due to the biological function of volatile terpenes in trees (Pohleven et al., 2019). Terpenes are a large and diverse class of organic compounds produced by a variety of plants, including conifers (Plesser, 2012). They are naturally found as extractives in the resin of softwoods such

Scots Pine, where they form a part of a trees chemical defence against damage, insects and pathogenic organisms (Granström, 2005). There are several groups of terpenes, where monoterpenes make up the majority of the terpenes found in *Scots Pine*. The most abundant monoterpenes in *Scots Pine are α-pinene* and *3-carene Scots Pine*, as seen in several studies (Englund, 1999; Hyttinen et al., 2010; Pohleven et al., 2019).

All the monoterpenes consist of two isoprene units and have the same chemical formula, $C_{10}H_{16}$, where they can be linear (acyclic) or contain rings (Granström, 2005). Figure 1 shows both these structures. 3-carene represents the structure which contains rings, and *β-myrcene* represents the linear structure.

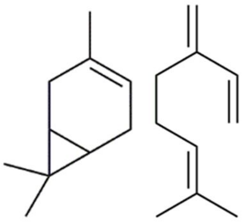

Figure 1: Structural formula of 3-carene (left) and β-myrcene (Aarsbog, L. 2019)

The isoprene units can be derived from two pathways in nature, the mevalonic acid pathway and the methylerythritol phosphate (MEP) pathway (Kesselmeier & Staudt, 1999). The MEP pathway is considered to be the most utilized in nature (Aarsbog, 2019). Two intermediates are produced from both pathways, isopentyl pyrophosphate (IPP) and dimethylallyl pyrophosphate (DMAPP). These give geranyl diphosphate (GPP) through an enzyme-catalysed combination. Through the elimination of phosphate and a combination of other reactions, the GPP can form a range of linear monoterpenes such as the ones analysed in this study.

The resin of the most abundant Norwegian conifers, Norway Spruce and Scots Pine, consists of 25-30% monoterpenes (Granström, 2005). Volatile terpenes, such as monoterpenes and other nonbound VOCs, are characterized by high emission rates (Pohleven et al., 2019). The concentrations of volatile terpenes are high in the beginning but decrease substantially with drying and over time. It is estimated that approximately 50% of the initial concentrations decrease over the first 2 weeks. Volatile terpenes can then generally dominate the emissions from a material for a period of several months, or up to a year.

The other target VOCs in this study are pentanal and hexanal, which are aldehydes. Aldehydes are a group of organic compounds that can be naturally found in wood but are also created during the production of wood-based products (Salem & Böhm, 2013). This group of VOCs is also called secondary VOCs, due to their emission pattern. Secondary VOCs are characterized by initially low VOC concentrations, compared to volatile terpenes, but are emitted over a longer period of time.

Aldehydes are created through a chemical process called autoxidation, which can be used to describe how organic compounds, such as aldehydes are created and degrade in both the atmosphere and indoor air (Frankel, 2005). Degradation of organic compounds in air leads to the formation of many different products, such as free radicals, which are very reactive and contribute to the release of organic compounds over a long period of time. Oxygen has a key role in the autooxidation process, where organic compounds can directly react with organic compounds under ambient conditions (Kesselmeier & Staudt, 1999). Autoxidation is an autocatalytic chain reaction which takes place through a free radical transition state and is a three-step process. This process can be divided into three subsequent stages, autoxidation starts with *chain initiation* which goes through to *chain propagation* which then goes to *chain termination.*

Initiation starts with a direct reaction of the double oxygen bond present VOCs such as hexanal, as seen in figure 2, in simple form with molecular oxygen.

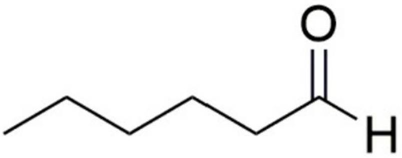

Figure 2: Molecular structure of hexanal (Aarsbog, L. 2019)

During the initation, the formation of auto oxidation reactions occurs. This process comprises of 2 phases. During the first phase free radicals draw hydrogen atoms from methyl groups, then during the second phase oxygen is added to alkyl radicals and results in peroxyl radicals (ROO). Peroxyl radicals can extract hydrogen from methyl groups and create hydroperoxides (ROOH) and new alkyl radicals (R). These later react with oxygen and the process continues.

During *termination* free radicals react with each other and with other non-radical products, or with other molecules (for example antioxidants) so that they create stable radicals which do not contribute to further *propagation.* The elimination of all free radicals is the only way to stop the oxidation reactions. In indoor air this is a process which might take a long time, and studies have shown that VOCs can be emitted for as long as 600 days (Stratev et al., 2016).

2.5. Factors affecting VOC emissions in indoor air

The types of VOCs present in indoor air will mainly depend on the material, but the concentrations of these VOCs will also depend on physical factors in the environment itself (Wolkoff, 2012). The treatment of the material, i.e. the type of paint, coating, glue or if the material has been processed will also affect the VOC concentrations in indoor air. Additionally, wood is not a homogenous material, and there might even be individual differences in VOC emissions within the same tree species, and different individual trees (Granström, 2005). Wood is a natural material and the tree species, the genetic of a specific tree, its age and individual variations within the same tree species will also affect the emissions (Plesser, 2012).

In indoor air the most important factors affecting VOC emissions are physicals factors such temperature, humidity, and ventilation. The effect of temperature has been researched in several studies, with studies showing that VOC emissions elevate faster at high temperatures and that there is an association between VOC concentrations and temperature (Fechter et al., 2006; Wolkoff, 1998). An increase in temperature leads to a proportional increase in VOC concentrations, where a 20˚C increase in temperature can lead to a doubling in total VOC concentrations (Fechter et al., 2006; Lee & Kim, 2012). The effect of humidity on VOC emissions in indoor air has also been thoroughly researched, where increased humidity leads to higher concentrations for some VOCs (Fechter et al., 2006; Wolkoff, 1998). Monoterpenes are more likely to be affected by temperature, specifically an increase in temperature, while humidity has a lesser effect on their concentrations in indoor air. Aldehydes, on the other hand, are more susceptible to increases in humidity, while at the same time being as sensitive to temperature as monoterpenes (Fechter et al., 2006). The general hypothesis is that the emissions from polar compounds, such as aldehydes and alcohols, are more influenced by the humidity in the air (RH) than non-polar compounds such as terpenes (Fechter et al., 2006).

Ventilation is important for diluting indoor air pollutants to acceptable levels and providing fresh air indoor. Airtight buildings might lead to increased VOC emissions, and the occupants might experience discomfort in regard to this. Studies of air velocity in indoor air show that the ventilation rate has not as big of an effect on VOC concentrations as temperature and humidity (Wolkoff, 1998). The most important factor regarding ventilation will be to adequately ventilate spaces with fresh VOC material sources, as the emissions are highest during the first days as the ventilation rate does not increase VOC concentrations such as temperature and humidity.

The coating of a material might have an effect on VOC emissions, where a surface treatment can work as a barrier reducing VOC emissions from a material. Coatings may also have an effect of the moisture buffering of wood and wood materials, limiting the moisture exchange between the material and the air (Hameury, 2007). This will lead an increase in vapour resistance, making the wood materials more resistant to changes in humidity and thus decreasing the effect of humidity on VOC concentrations. Uncoated materials will often have the highest VOC concentrations, while coated materials will have reduced VOC concentrations due to this barrier effect and less moisture buffering (Alpha & Corneau, 2006; Bartekova et al., 2006). Though, it is important to consider the film thickness of the coating as this may affect the barrier effect (Afshari, 2003). This may also affect the VOC content, and the film thickness must therefore be considered in comparison measurements. The coating might also lead to the presence of other VOCs. Some VOCs may come from the production process, such as formaldehyde in plywood (Plesser, 2012). Other VOCs may be created when chemicals in the coating react to chemicals in the environment or the product.

2.6. VOC legislation

The legislation regarding IAP and IAQ can be legally binding or based on recommendations from voluntary certifications schemes and similar programs. The focus can be on either health issues, occupant comfort levels or prevention of irritating odours. As of today, there are no legal frameworks directly connected to VOC emissions from construction products in indoor air in Norway. There is some legislation regarding limiting emissions of VOCs due to the use of organic solvents in certain paints, varnishes and vehicle refreshing products, but this legislation applies to industrial processes and installations (EØS-notatbasen, 2012). Most of the legislation regarding IAQ focuses on temperature, air ventilation and humidity in order to achieve a comfortable and safe indoor air (Direktoratet for byggkvalitet, 2017). The Norwegian Working Environment Act also has some regulations regarding the working environment, where a comfortable and safe indoor air is required (Arbeidsmiljøloven, 2005).

This leads to most of the demands regarding VOC emission testing and critical values in Norway being driven by consumer and market demand, along with foreign companies that have stricter regulations regarding indoor air pollutants (Plesser, 2012). Over the last years certification schemes such as BREEAM have become more and more common, due to increased market demand for buildings with a low environmental footprint. The current BREEAM technical manual has emission limits for different product categories, such as walls, floors and sealants (BREEAM-NOR, 2016). There are no specific emission limits for individual VOCs in the BREEAM system, with the exception of formaldehyde, but rather a total limit for all of the VOCs added together (TVOC). The emission measuring systems are also based on the ISO 16000 standard series. As there are currently no labs specializing in measuring VOCs according to the existing standards in Norway the emissions tests are carried out in laboratories abroad (Plesser, 2012).

2.7. EU-LCI

In 2013 the European Construction Products Regulation (CPR) (EU/305/2011) replaced the former Construction Products Directive (EEC/106/89), with the goal of the CPR being facilitating cross-border trade and overcoming trade barriers in the form of national rules and standards. The aim of the CPR has been to provide a common technical language in harmonised EU product performance standards, for use by both manufacturers and regulators (Jäckh et al., 2013). Regarding VOCs, this regulation requires documentation for construction product emissions of VOCs released into indoor air for several product types. This documentation is necessary for products, in order to receive CE marking. The EU uses CE-marking to ensure that reliable information on product performance is presented in a harmonized manner across Europe, with the main intention of this regulation being to substitute national systems and to facilitate cross-border trade by giving access to this information (Oppl, 2014).

Even when a harmonized standard covers VOC emission, product performance requirements will not be compulsory for the trade of construction products in the whole EU/EEA trade area. Only those countries with relevant national legislation on VOC emissions require a declaration of performance for trade in their markets (Oppl, 2014). As Norway is a part of the European Economic Area (EEA) it is therefore likely that future laws and regulations regarding VOCs from the EU will be adapted into Norwegian law.

The CPR has led to the Lowest Concentration of Interest (LCI) concept having a bigger role when it comes to establishing a VOC framework in Europe. The EU-LCI concept is based on toxicity studies of chemicals being released into indoor air, along with toxicity data and limit values from existing national evaluation systems (Jäckh et al., 2013). EU-LCI values are health-based reference concentrations (µg/m^3) that are meant to evaluate emissions from a single product over 28 days, under the standard for assessing and determining emissions of dangerous substances into indoor air (EN16516). Though, they cannot be used for evaluating indoor air quality (Jäckh et al., 2013). The EU-LCI values represent concentration levels which are considered likely not to cause adverse effects over the longer term, with the goal being to avoid health risks to the public from long time exposure to the listed components. The EU-LCI values are also continuedly updated with new compounds and LCI values for these compounds, and there were already LCI values for over 150 different compounds (Jäckh et al., 2013). Table 4 shows EU-LCI values for some common VOCs found in wood and wood materials.

Table 4:EU-LCI values for VOCs common in wood and wood materials (Jäckh et al., 2013)

Compound	EU LCI (µg/m^3)
1-Butanol	**3000**
Pentanal	**800**
Hexanal	**900**
β-Myrcene	**1400**
β-Pinene	**2500**
Carene	**1500**
Camphene	**1400***
α-Pinene	**1400**
Limonene	**5000**

**Other terpene hydrocarbons*

3. Methods and materials

The methods and materials in this study were based on EN16516, the harmonized European standard for determination of emissions into indoor air. The purpose of following an already established testing method would make it easier to develop representative results. The results from this study could then be compared to results from other emission tests, and earlier research. It turned out that developing this type of model system was very demanding, and the study was not able to completely follow all the requirements in EN16516. This chapter will describe both the development of the model system used in this study, the model system itself, the samples used and the sample preparation method. An overview of the analytical system, including the method validation and quality assurance and quality control will also be described in this chapter.

3.1. Developing a model system for VOC analysis in accordance to EN16516

The model system for quantifying VOC emissions from construction materials would need to meet the requirements of EN16516. These requirements consisted of, among other things, a model system consisting of a climate chamber with a minimum size of 20L, an air change rate of 0,25-1,5 per hour, and a testing climate of 23±1˚C with a RH% of 50 ± 5% (European Committee for Standardization, 2017). This sub-chapter will give an overview of how this study tried to accommodate to the requirements in EN16516.

The first requirement was regarding the VOC chamber size, made from a suitable material. EN16516 proposes the use of stainless steel or glass, as these materials would not contaminate the sample environment. The chamber body, chosen for this study, was therefore made in aluminium, while the lids were made of safety tempered glass. The VOC chambers were provided with a vulcanized 100% silicone gasket, where the gasket had no glue in order to avoid possible VOC interferences and the sealant was silicone grease. In addition to this, the VOC chambers also had to have connectors that made it possible to install an air supply, an air pump and also a valve for releasing the air flowing in from the air supply.

It was also important to find a way to maintain the desired temperature, and we chose to use a climate-controlled cabinet to ensure this. The climate-controlled cabinet was able to keep the required temperature of 23±1˚C, while the humidity was much harder to control. While the climate-controlled cabinet, itself, was able to keep a RH% of 50 ± 5% it was simply not possible to the same for the chambers. This was mostly due to the air supplied having an RH% of 0, which would lower the RH% each time it was supplied to the chambers. This can be seen in Figure B1, Appendix B, where the RH% drops every time new air is supplied to the chambers. To completely control the RH% would therefore require additional equipment, but due to a lack of funds and time we were not able to secure this equipment.

3.2. The model system

The model system consisted of 4x20L vacuum chambers (VC3028AG, VacuumChambers.eu, Ignatki-Osiedle, Poland) placed inside a climate-controlled chamber (Termaks KB8400 F, Termaks AS, Bergen, Norway). The chamber body was made from aluminium, while the lid was made of tempered glass. The chambers were provided with a vulcanized 100% silicone gasket. The chambers also came with two ball valves installed, one connected to the air supply (HiQ Synthetic air 5.0, Linde Gas AS, Oslo, Norway) and another one connected to a sampling pump with an electronic flow controller (SKC Pocket Pump 210-1002, SKC Blandford, Dorset, UK). The air mixture of 70% nitrogen (N_2) and 30% oxygen (O_2) was supplied through a compressed air hose (Tess Miljøtex 10602-06, TESS AS, Lierstranda, Norway), which was then split into four separate air hoses for each of the chambers. The air hoses were then separated by using hose couplings made in brass (TESS 31962-06-06, TESS AS, Lierstranda, Norway). The gas flow was controlled by using a pressure regulator (C-regulator C200/1 AB DiN 10, Linde Gas AS, Oslo Norway). Factors such as T and RH were monitored with a data logger (SenseAnywhere AiroSensor 20-20-24/00, SenseAnywhere, Oud Gastel, the Netherlands), in accordance to EN16516. Figure 3 presents a schematic model system of the used 20L chambers. The main difference between the model system used in this thesis and figure 3 is the number of chambers.

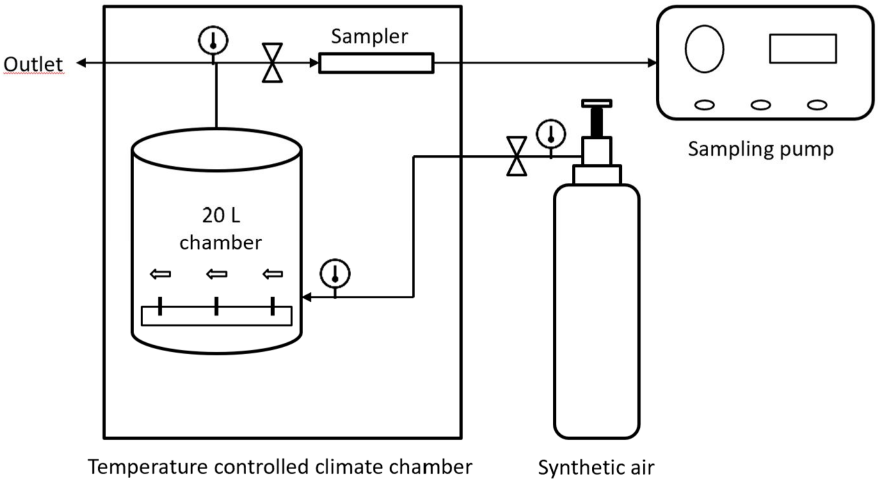

Figure 3: Schematic diagram of the model system. Adapted from Kim et al, 2006.

Prior to sampling, the chambers were sealed and checked for air leaks. The glass lids were secured from movement with a metal weight of 1kg. After this, hexane was released into the chambers for three days. This was done in order to remove possible contaminates, and the hexane was later replaced by an air mixture consisting of 70% N_2 and 30% O_2.

3.3. Materials and sample preparation

3.3.1. Materials

In order to compare the difference in emissions from treated and untreated wood, two different batches of samples were used. The first batch of samples consisted of untreated planed interior panels of Scots Pine of Swedish origin. The samples were directly collected after manufacture, after they had gone through a wood planer. After this, the samples were wrapped in aluminium foil (Toppits aluminium foil, Cofresco Frischhalteprodukte GmbH & Co. KG, Minden, Germany). Then the samples were put in zip-seal bags (Bag LDPE zipper, VWR, Kokstad, Norway) approximately 45-60 minutes after manufacture.

The second batch of samples also consisted of Scots Pine of Swedish origin, the main exemption being that the samples had been treated with wood stain. The samples had been stained with a water-borne acrylate paint (Laqva Top, Sherwin Williams Norway, Skedsmokorset, Norway). These samples had been treated on the interior side while the backside, not exposed to indoor air, remained untreated. This configuration can be seen in figure 4.

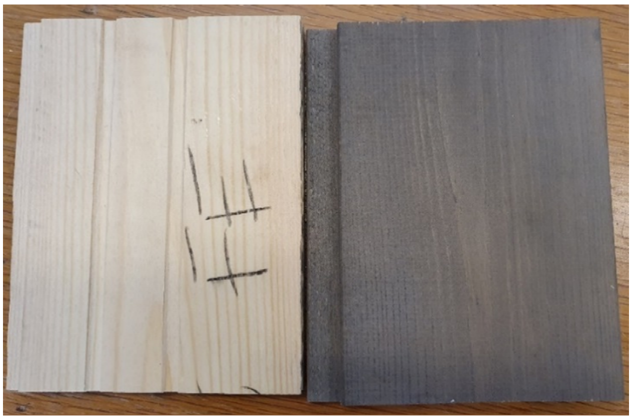

Figure 4: Wood stain samples

The wood stain samples were approximately 30 days old when they were picked up and packed. Table 5 shows the materials and their main characteristics.

Table 5: Sample information

Sample	Tree species	Treatment	Dimensions
U1	Scots pine	None	150x120 mm
U2	Scots pine	None	150x120 mm
U3	Scots pine	None	150x120 mm
WS1	Scots pine	Water-borne acrylate paint	150x120 mm
WS2	Scots pine	Water-borne acrylate paint	150x120 mm
WS3	Scots pine	Water-borne acrylate paint	150x120 mm

3.3.2. Sample preparation

The samples had originally a surface area of 210x120 mm, with a thickness of 13mm. The samples were later cut to 150x120 mm. This was done to make the samples as homogenous as possible and remove possible variations, such as knots and other irregularities. The samples were also weighed, so that information about their basic density and RH could later be calculated.

Before the samples were placed into the chambers, they were sealed. This was done in accordance with NS-EN 16516, with low emitting aluminium tape (tesa® Aluminium tape, tesa SE, Norderstedt, Germany) covering the edges not exposed to indoor air and the backside not exposed to air as shown in Figure 5.

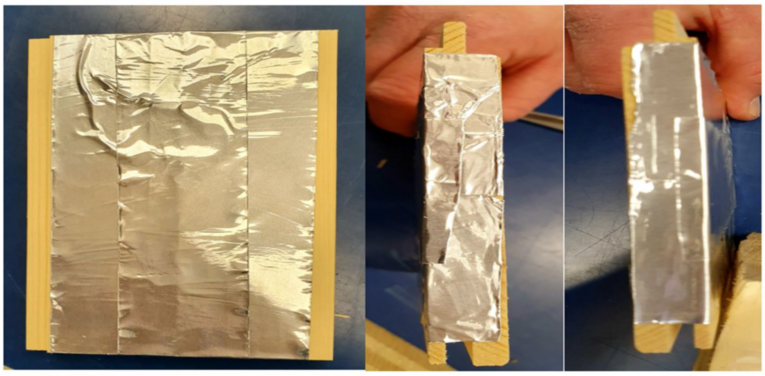

Figure 5: Sample packaging

3.4. Sampling

The VOCs were actively sampled in Perkin Elmer Tenax TA adsorption tubes (Stainless Steel ATD Prepacked Sample Tubes, PerkinElmer Inc, Norwalk, USA) filled with Tenax TA (300 mg) by using an SKC pocket pump with an electronic flow controller. The Tenax tubes were conditioned using thermal desorption at 250°C for 30 minutes before use. The samples were taken through the upper ball valve, which was placed over the tested sample cf. Figure 6. The sampling happened in two stages, where the samples were first collected at a flow of 100 mL/min for 100 minutes. This equals 10L of sampled air. Right after this, duplicates samples were collected at a flow of 80 mL/min for 63 minutes cf. EN16516. This equals 5L of sampled air. The samples were collected at 1, 3, 5, 7, 10, 13, 16, 20, 24 and 28 days. In both testing periods, one chamber was left blank to ensure that there were no contaminants present in the chambers. Background measurements were also collected from the chambers before the emission testing began.

Figure 6: The VOC chambers and an air pump inside the climate cabinet

After each of the samples had been taken, they were packed in aluminium foil before being placed in a zip-seal bag with the samples from the same batch. The samples were then stored in a freezer at -20˚C prior to analysis.

3.5. Sample analysis

The samples were analysed according to EN16516, by using automated thermal desorption gas chromatography mass spectrometry (ATD GC-MS) to identify each of the target compounds. The quantification method used was developed by the Faculty of Chemistry, Biotechnology and Food Science (KBM) at the Norwegian University of Life Sciences (NMBU). The concentrations were expressed in µg/m^3, per EN16516. Quantification was done by using toluene-D8 (Toluene-d$_8$, Chiron AS, Trondheim, Norway) as an internal standard.

3.6. Method validation and quantification

In order to ensure that the results in an analytical process are reliable, consistent and reproducible it is necessary to have a robust method with a good quality assurance and control. Testing blank samples, calibration curves, recovery, calibrating the equipment, determining the limits of detection (LOD) and the limits of quantification (LOQ) are all important parts of this process (Clausen & Kofoed-Sørensen, 2009). It is also important to make sure that the method is valid through a process called method validation. Method validation can be defined as a process used to determine if the analytical process utilized for a specific test is suitable for its intended use, and that it fulfils the necessary quality requirements (Huber, 1998).

This study took several of the aforementioned steps in order to ensure reliable, consistent and reproducible results. Blank samples were used to determine if there were any contaminants present in the analytical process, whether the contaminants came from the samples themselves or from the analytical equipment. Contamination control was done by conditioning the sample tubes and assigning them a number, so it was possible to follow the tubes through sampling and analysis. All necessary laboratory procedures were followed when it came to contamination control, i.e. cleaning of laboratory equipment, fume hoods and other working surfaces were cleaned before and after use.

The external standard method, internal standard method and standard addition method are the most used quantification methods for VOCs (Aarsbog, 2019). EN16516 does not require the use of any specific standard method, but the quantification method has to be appropriate for the target VOCs. That means that at least one suitable standard, containing toluene and at least a set of compounds that is representative of the volatility and polarity range of the relevant target compounds shall be run at the start of each batch of samples (European Committee for Standardization, 2017). In this study, an internal standard method containing toluene D8 diluted in hexane was chosen, in order to accommodate with these requirements. The internal standard was added to both the sample and validation tubes, in accordance with EN16516.

Calibration curves were then calculated based on the chromatographic response to both the analyte and its internal standard. These calibration curves were then used to calculate the VOC concentrations in the samples. This is because calibration curves describe the relationship between the signal response and the concentration of the analyte, and can be used to determine the concentrations of analyte in the samples (Clausen & Kofoed-Sørensen, 2009). The relationship between the signal response and the concentration of the sample is linear in most cases, following equation 3.1. (Aarsbog, 2019).

$$y = ax + b$$

(3.1) $$Where: \quad y = \frac{A_i}{A_{ISTD}} \quad x = \frac{M_i}{M_{ISTD}} \quad b = constant$$

$\frac{A_i}{A_{ISTD}}$ = Area of chromatography peak of native compound (i) or internal standard (ISTD)

$\frac{M_i}{M_{ISTD}}$ = Amount of native compound (i) or internal standard (ISTD)lk.

The quality of the linear model, i.e. how much of the variation in which *y* is explained by the linear relationship is measured by the coefficient of determination (R^2). The closer R^2 is to 1, the better the linear model explains the variations in *y* (Skoog et al., 2013). The calibration parameters used in this study can be seen in Table A2, Appendix A.

Recovery is an important part of the method validation and is directly connected to the calibration curves. The calibration curve is constructed by adding different amounts of the standard into the matrix and process, while the recovery gives information about the extraction method. If the extraction method is poor, either more or less than the amount of the analyte will be recovered (Skoog et al., 2013). For this study, mainly the apparent recovery was used. Apparent recovery is defined as the ratio between the measured analyte and the added analyte, equation 3.2. (Aarsbog, 2019) Due to the use of a calibration curve, apparent recovery is recommended (Burns et al., 2002). This method takes into account the response of an internal standard and corrects the measured amount (Aarsbog, 2019).

(3.2) $$\%\, Apparent\ recovery = \frac{Measured\ amount}{Added\ ammount}$$

Limit of detection (LOD) and limit of quantification are important parts of the method, as results below the LOD are uncertain. LOD is can be defined as the lowest quantity or concentration of a component that can be detected with a given analytical method, but not necessarily quantified (Clausen & Kofoed-Sørensen, 2009). The LOD and LOQ in this study were calculated on the basis of the blank samples, where the LOD was estimated as three times the standard deviation of the analysis of the blank samples. The LOQ was estimated as ten times the standard deviation of the blank samples. The main criteria used for quantification were the retention time (RT), the shape of the chromatographic peaks and the detection limits.

3.7. Sample analysis and analytical system

The Tenax tubes were sealed with PTFE stoppers and directly loaded onto the carousel, with a capacity for 50 tubes, of the automated thermal desorber (ATD 400, Perkin Elmer, Norwalk, CT, USA), checked for leaks, then purged with helium for one minute to remove water vapour and air. The samples were desorbed at 220 °C onto a -15 °C cold trap for 10 min (helium flow 50 ml/min, outsplit flow 47.5 ml/min, this means 5% dilution). The cold trap was then heated rapidly to 220°C and the desorbed samples were flushed through a fused-silica transfer line (0.32 mm I.D., 225°C, helium flow 1 ml/min) to the analytical chromatography column (capillary column, 60 m×0.22mm, 0.25 µm film thickness, Supelco), the helium flow rate is adjusted through adjusting the inlet pressure (MINI PSI) to 25. Column temperatures were ramped as follows: 80 °C for 5 min to 5°C/min to 240°C for 5 min. The total run was for 48 min. Compounds were detected and identified using an Agilent triple quadrupole MS (MS/MS) 7000C system using multiple reaction monitoring (MRM) scan mode.

3.8. Statistical analysis

The statistical analysis was performed using JMP Pro for Windows, version 15 (SAS Institute, Cary, North Carolina, U.S.A.). A repeated measure analysis of variance (ANOVA) was used to test the overall difference between related means (within-subjects effects), in addition to compare the groups of respondents (between-subject effects) (Skulberg et al., 2019). A repeated measures study design is commonly used when examining changes in mean scores over three or more time points, such as in this study. The between subject categories in this study were determined by the subject treatment (untreated vs. wood stain) in addition to the test order. A repeated measures ANOVA also makes it possible to analyse the pattern of response within-subject. The interaction between “time and test” and “VOC concentrations” and between time and “test order” were also evaluated.

4. Results

Background VOC concentrations were quantified in all four chambers, c.f. Table 6. This was done before the main sampling started, with the purpose of finding possible sources of contamination. The samples were quantified using the same method as in chapter 3.4, while the background TVOC concentrations were calculated according to EN16516.

Table 6: Background VOC emissions

Chamber ID	Sample size (L)	Single VOC concentration (µg/m^3)					TVOC (µg/m^3)
		α-pinene	**camphene**	**β-myrcene**	**β-pinene**	**3-carene**	
1	10	5.44	0.31	0.56	0.44	3.92	10.67
2	10	5.88	0.46	0.61	0.72	7.58	15.26
3	10	5.67	0.43	0.60	0.69	6.44	13.85
4	10	6.85	1.34	1.05	1.89	29.81	40.99

A substantial difference between the chambers was observed, with the background VOC concentrations in chamber 2 being higher than for the other chambers. This applied both for the main and the duplicate sample. Though, these concentrations seemed to have only a minor influence on the main results. This can be seen in the main results, where chamber 2 had the lowest VOC concentrations during the sampling period. Furthermore, the background concentrations were not subtracted from the emission test results, due to the chambers being opened and closed when placing the samples. This was done two times after the background emission measurements were taken.

In order to ensure that the chambers functioned optimally, one chamber was selected to be used as a reference chamber for each of the emission test periods. The reference chamber was chosen randomly, and a different chamber was used as the reference for each emission test. The concentrations in the reference chamber followed the same pattern as the chambers with samples inside, indicating that there may have been some contamination. This contamination may have come from the lab itself, or the chambers may have had a problem with the sealing. A possible problem with the chambers not being properly sealed is also indicated by the constantly changing RH (Figure B1, Appendix B).

4.1. Quantification results

4.1.1. Untreated samples

The quantification results showed that the analytical method was not able to quantify all the target VOCs for the untreated samples. Analysing the calibration samples showed some issues with *1-butanol, pentanal and limonene*. These issues were difficult to deal with and resulted in reduced precision in detection/quantification of the target compounds. The latter issue caused the results for *β-myrcene* to be regarded as semi-quantitative, which will be further discussed in chapter 4.2.5.

Looking at the detection of the compounds, the analysis method used in this study is relatively new and still under development at NMBU. This might explain some of the issues with the detection and quantification of *1-butanol, pentanal and limonene.* Emission testing of *1-butanol* was carried out for the first time during this study, and several issues came up. There was a large spread in the RT, making it difficult to identify which top came from *1-butanol*. Changes to the thermal desorption and gas chromatograph were made, but the same issue persisted. The large spread in the RT may also have been caused by high concentrations of *1-butanol*, saturating the GC-MS. An important consequence of this was that the analytical method was not able to analyse this compound. Another possible issue was that the GC-MS had been adjusted for polar compounds, such as monoterpenes, and was therefore not as sensitive for non-polar compounds, such as *1-butanol* and *pentanal*.

Similar issues as for *1-butanol* occurred with *pentanal*, with a too big spread in RT. This made it difficult to discern which top came from pentanal. The latter issues also occurred for *limonene*, even though the method had previously been successful in quantifying this compound. Figure 7 is a chromatograph showing RT for these three compounds expanded over several minutes. The challenges when detecting/quantifying these three compounds were obvious and led to the exclusion of these components from this part of the study.

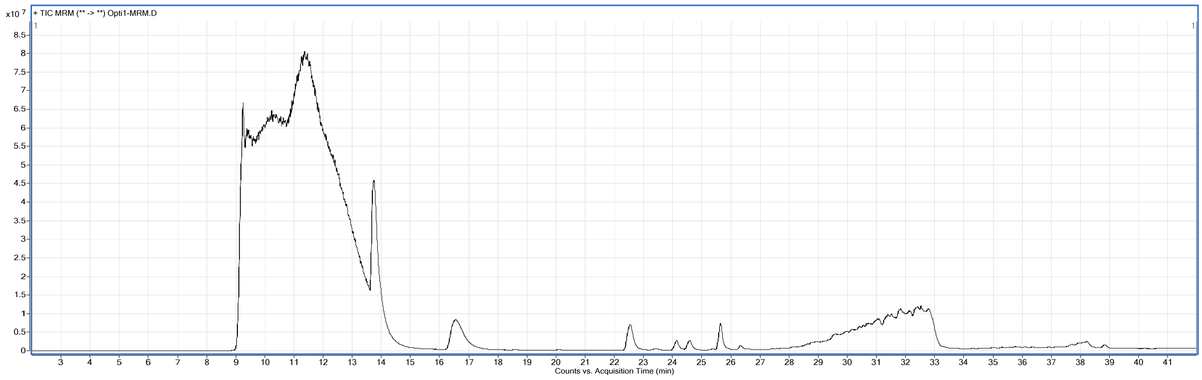

Figure 7: Chromatograph showing a mix of 1-butanol, pentanal and limonene

The calibration samples went later through a 5% dilution, which was successfully used to run further GC-MS analysis. This made it easier to identify the tops, but there were still some issues with the RT. The chambers had been cleaned with *hexane*, which had the same RT as *pentanal*. This led to the analytical system not being able to distinguish *pentanal* from *hexane*.

1-butanol and *limonene* continued to have the same issues as before, and it was not possible to make calibration curves for these compounds. Table 7 shows the limit of detection and instrumental limit of quantification for the target compounds the analytical method was able to quantify.

Table 7: LOD and LOQ for the target compounds

Compound	LOD (µg/m^3)	LOQ (µg/m^3)
1-Butanol	N.A.	N.A.
Pentanal	N.A.	N.A.
Hexanal	0.0001	0.0004
β-Myrcene	0.0208	0.0692
β-Pinene	0.0031	0.0103
3-Carene	0.0070	0.0234
Camphene	0.0104	0.0347
α-Pinene	0.0683	0.2276

4.1.2. Wood stain samples

Due to the issues with both the retention time and the chromatographic peaks for the untreated sample, *cyclohexane* was chosen to be mixed with toluene D8 as an internal standard. Figure 8 shows the differences between the retention times for *hexane* and *cyclohexane*. The three different RTs for *hexane* can be seen in Figure 8, illustrating the importance of choosing a relevant standard when quantifying this type of compounds. Using an inappropriate standard, with similar RT to the target compounds, makes it very difficult to identify which peaks belong to which compounds. Because of this, it was not possible to identify *1-butanol, pentanal and limonene* for the untreated interior wood samples. Cyclohexane, opposed to hexane, made it possible to identify these compounds as seen in chapter 4.3.

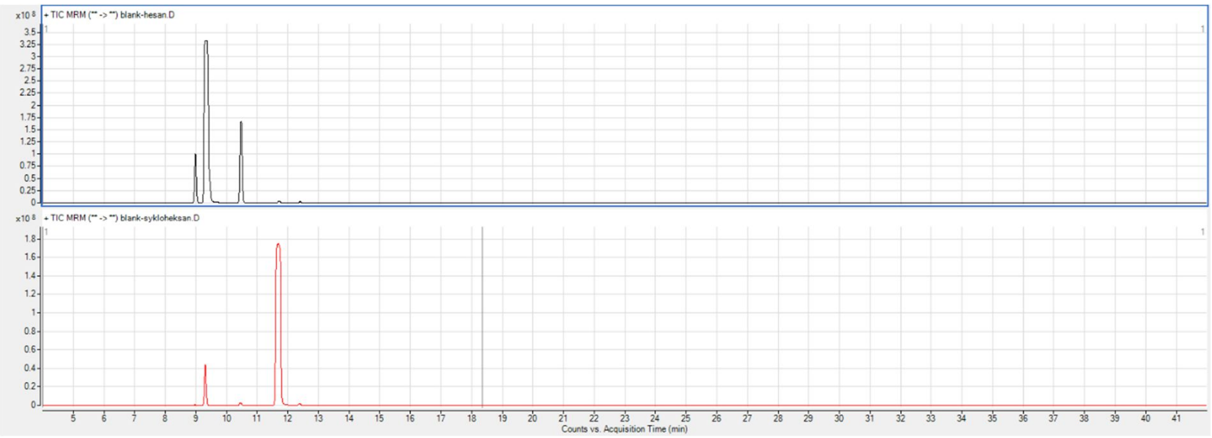

Figure 8: Chromatograph showing the differences between the retention times for hexane (top half of the picture) and cyclohexane (bottom part of the picture)

The LOD and LOQ was the same as for the untreated samples, as these limits were based on the same calibration samples.

4.2. Volatile organic compounds (VOC) in untreated wood panels

4.2.1. TVOC concentrations

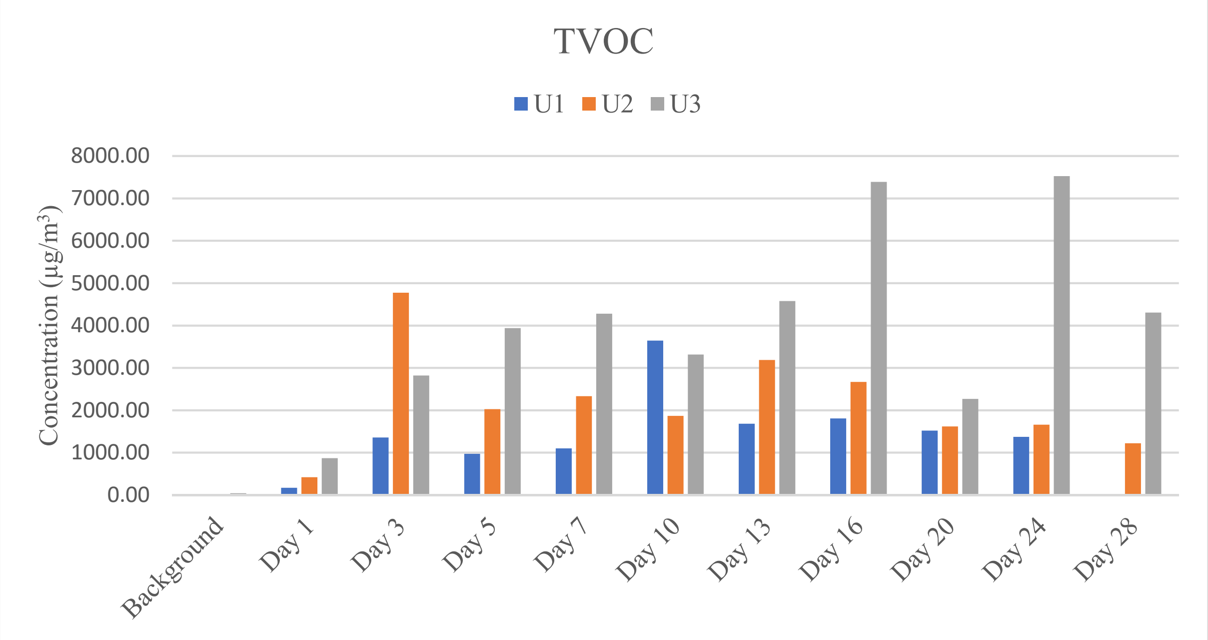

Figure 9: TVOC concentrations from untreated wood panels

Figure 9 shows the TVOC concentrations measured during the sampling period of 28 days, for each of the three untreated samples. Variability in TVOC concentrations during the sampling period was observed between both samples and sampling days. At day 1, the highest observed TVOC concentration was from sample U3. This trend continued throughout the whole sampling period, except for days 3 and 10. U3 had a spike in TVOC concentrations at days 3 and 13, while the TVOC concentration varied between 419-2670 µg/m^3 throughout the sampling period. Sample U1 had a spike in TVOC concentrations at day 10, reaching a concentration of 3642 µg/m^3. During the rest of the sampling period, the TVOC concentrations varied between 171-1806 µg/m^3.

4.2.2. Single VOC concentrations

The single VOC concentrations for the untreated samples are presented in the following section.

4.2.2.1. Hexanal

Figure 10 shows the Hexanal concentrations measured during the sampling period for each of the three untreated samples. Variability in Hexanal concentrations during the sampling period was observed between both samples and sampling days. The sample in chamber 2 had the highest concentrations during the first two sampling days, but sample in chamber 1 had the highest concentrations for the rest of the sampling period. The sample in chamber 3 was substantially different from the other samples, with the lowest concentrations during the sampling period.

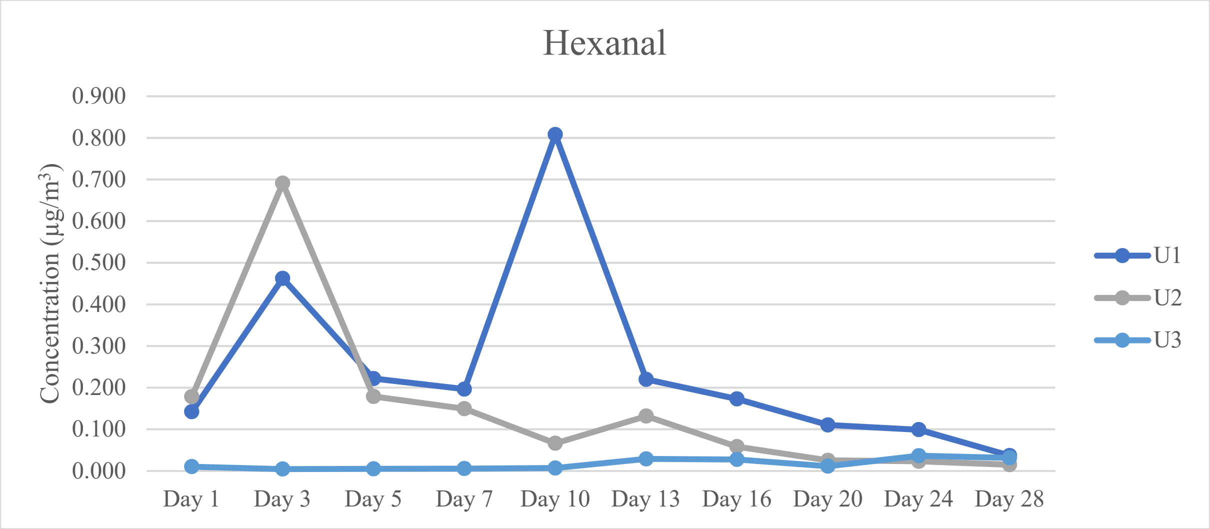

Figure 10: Hexanal concentrations from three untreated samples of Scots Pine wood panels during the testing period

The reference chamber shows no outside concentrations that might have affected the measured concentrations for the samples, cf. table 8. These concentrations were always below 0,05 µg/m^3, which indicates that there was a low possibility for contamination in the chambers with samples. The SD is as expected, with the sample in chamber 3 mainly contributing to the variation between the samples.

Table 8: Hexanal concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[1]	0,002	0,004	0,001	0,002	0,001	0,004	0,003	0,001	0,002	0,001
2 (U1)	0,142	0,462	0,222	0,196	0,808	0,220	0,173	0,110	0,099	0,037
3 (U2)	0,178	0,691	0,179	0,149	0,066	0,132	0,058	0,025	0,023	0,015
4 (U3)	0,010	0,004	0,005	0,006	0,007	0,029	0,027	0,011	0,036	0,031
Avg. con.	0,11	0,39	0,14	0,12	0,29	0,13	0,09	0,05	0,05	0,03
SD	0,09	0,35	0,11	0,10	0,45	0,10	0,08	0,05	0,04	0,01
CV%	80,30	90,59	84,95	84,84	151,94	75,39	88,88	109,11	76,66	41,40

[1] *Reference chamber*

4.2.2.2. α-pinene

The *α-pinene* concentrations measured in this study are presented in Figure 11. The concentrations differ less than the hexanal concentrations, with the sample in chamber 4 emitting more than the other samples for most of the sampling period. Looking at α-pinene generally, this is one of the higher emitted VOCs seen in this study.

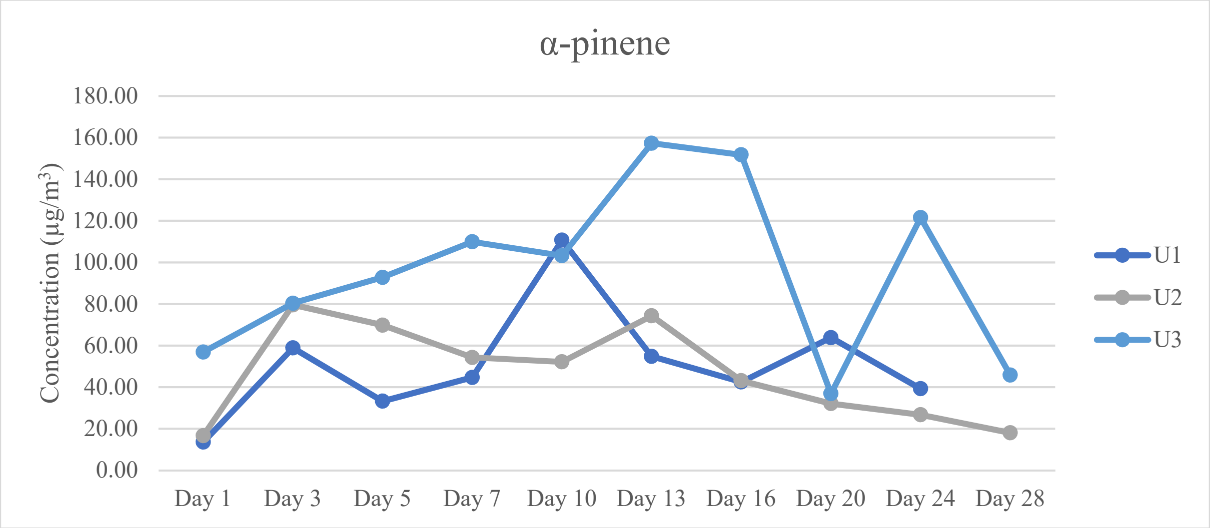

Figure 11: α-pinene concentrations from three untreated samples of Scots Pine wood panels during the testing period

Table 9 presents the *α-pinene* concentrations measured in the reference chamber, where no substantial emission pattern is observed. There is therefore a low probability that the samples might have been contaminated during the test period. The SD is as expected, with the sample in chamber 4 mainly contributing to the variation between the samples.

Table 9: α-pinene concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[I]	0,73	0,4	0,5	0,6	0,7	1,5	1,8	0,8	1,5	1,1
2 (U1)	13,59	58,9	33,3	44,7	110,7	54,8	42,5	63,8	39,3	[II]
3 (U2)	16,70	79,6	69,8	54,3	52,2	74,3	43,1	32,1	26,7	18,1
4 (U3)	56,89	80,3	92,8	109,9	103,2	157,3	151,7	36,9	121,5	45,8
Avg. con.	29,06	72,93	65,31	69,61	88,71	95,47	79,10	44,26	62,51	31,93
SD	24,15	12,20	30,03	35,22	31,85	38,87	62,89	17,10	51,49	19,63
CV%	83,12	16,72	45,98	50,59	35,90	40,72	79,51	38,63	82,37	61,46

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.2.2.3. β-myrcene

The *β-myrcene* concentrations measured in this study are presented in Figure 12. Less variation is observed for this VOC, compared to the other target compounds in this study. Sample U1 reached its highest concentration at day 10, while samples U2 and U3 reached their peak concentrations at days 13 and 16, respectively.

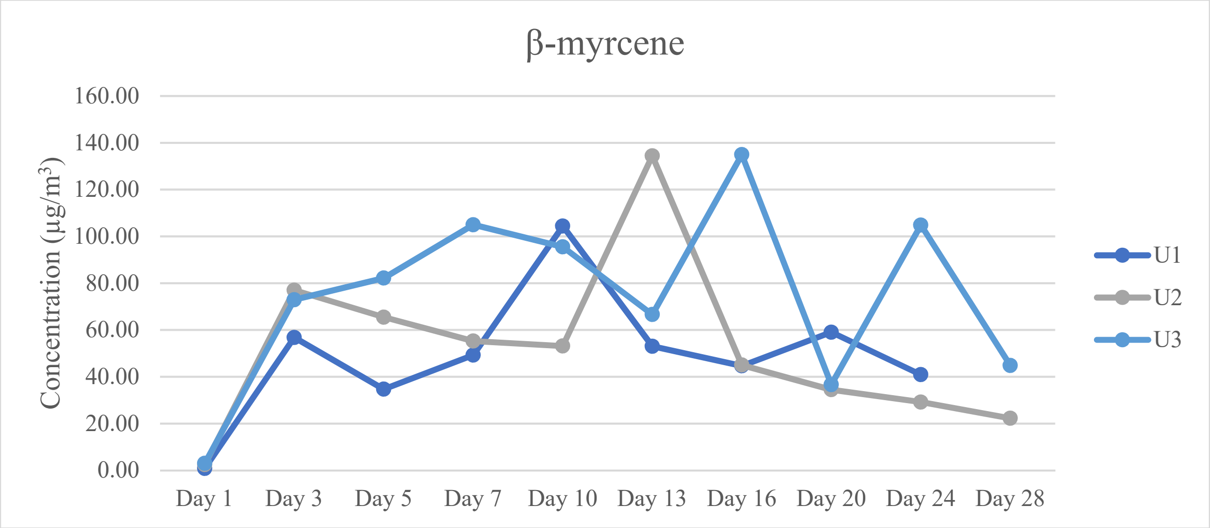

Figure 12: β-myrcene concentrations from three untreated samples of Scots Pine wood panels during the testing period

Table 10 presents the *β-myrcene* concentrations measured in the reference chamber. These concentrations were higher than expected and may indicate that there was a source of contamination. The average concentrations of β-myrcene were also higher than expected, indicating a possible source of contamination contributing to higher concentrations.

Table 10: β-myrcene concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[I]	0,56	7,99	7,99	8,12	8,32	9,06	9,29	8,33	8,96	8,50
2 (U1)	0,82	56,81	34,68	49,29	104,40	53,03	44,67	59,1	40,91	[II]
3 (U2)	2,44	77,02	65,47	55,26	53,14	134,39	45,00	34,5	29,2	22,2
4 (U3)	3,01	72,92	82,19	104,97	95,50	66,59	134,92	36,7	104,8	44,79
Avg. con.	2,09	68,92	60,78	69,84	84,35	84,67	74,86	43,40	58,30	33,51
SD	1,13	10,69	24,10	30,57	27,39	43,59	52,01	13,60	40,71	15,95
CV%	54,22	15,51	39,65	43,77	32,47	51,48	69,48	31,34	69,83	47,58

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.2.2.4. β-pinene

Figure 13 presents the *β-pinene* concentrations measured in the reference chamber. The variability between the samples is not as high as for the other samples. Sample U1 had the lowest measured concentrations, with a peak at day 1. For the rest of the sampling period, samples U2 and U3 had the highest observed concentrations.

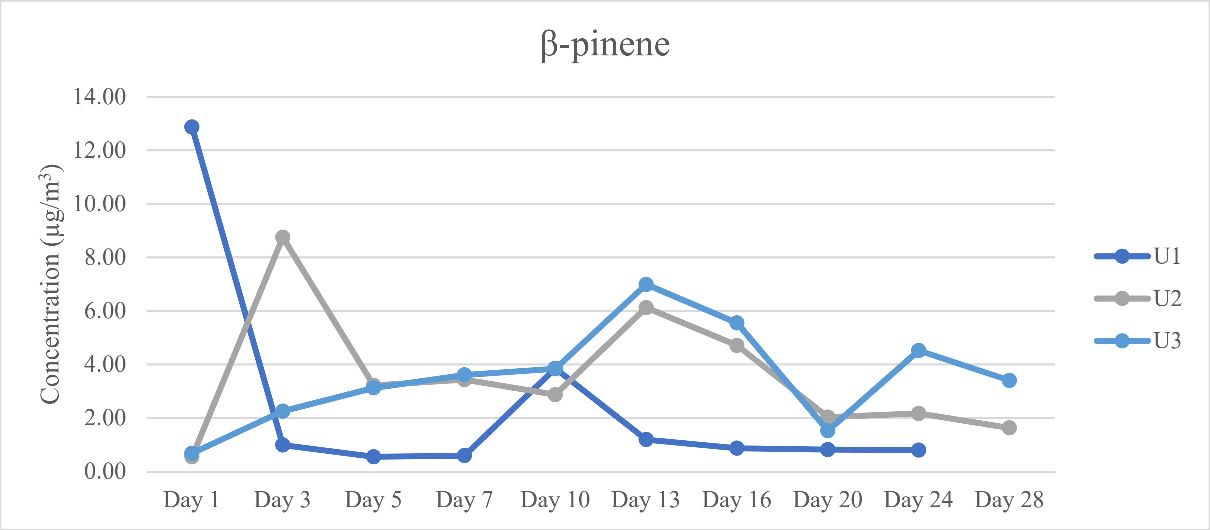

Figure 13: β-pinene concentrations from three untreated samples of Scots Pine wood panels during the testing period

The *β-pinene* concentrations observed in the reference chamber indicate a low probability for outside concentrations that might have affected the measured concentrations for the samples, cf. Table 11. The SD is as expected, with the different samples contributing to a higher SD on different days.

Table 11: β-pinene concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%).

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[1]	0,34	0,06	0,05	0,055	0,06	0,09	0,10	0,0	0,1	0,1
2 (U1)	12,87	0,99	0,55	0,597	3,85	1,19	0,87	0,8	0,8	[III]
3 (U2)	0,55	8,75	3,22	3,433	2,87	6,12	4,71	2,0	2,2	1,6
4 (U3)	0,68	2,26	3,12	3,608	3,84	6,99	5,55	1,5	4,5	3,4
Avg. con.	4,70	4,00	2,30	2,55	3,52	4,77	3,71	1,46	2,50	2,52
SD	7,08	4,16	1,51	1,69	0,56	3,13	2,50	0,61	1,88	1,25
CV%	150,56	104,14	65,88	66,40	15,97	65,58	67,24	41,77	75,34	49,80

[1] Reference chamber

[III] This sample was lost due to a malfunction in the ATD GC-MS system

4.2.2.5. Camphene

The *camphene* concentrations measured in this study are presented in Figure 14. Variability in Hexanal concentrations during the sampling period was observed between both samples and sampling days. Sample U3 had the highest camphene concentrations on seven of the sample days. The exceptions were days 3, 10 and 20. Sample U2 had the highest concentration at day 3, and sample U3 had the highest concentration on days 10 and 20.

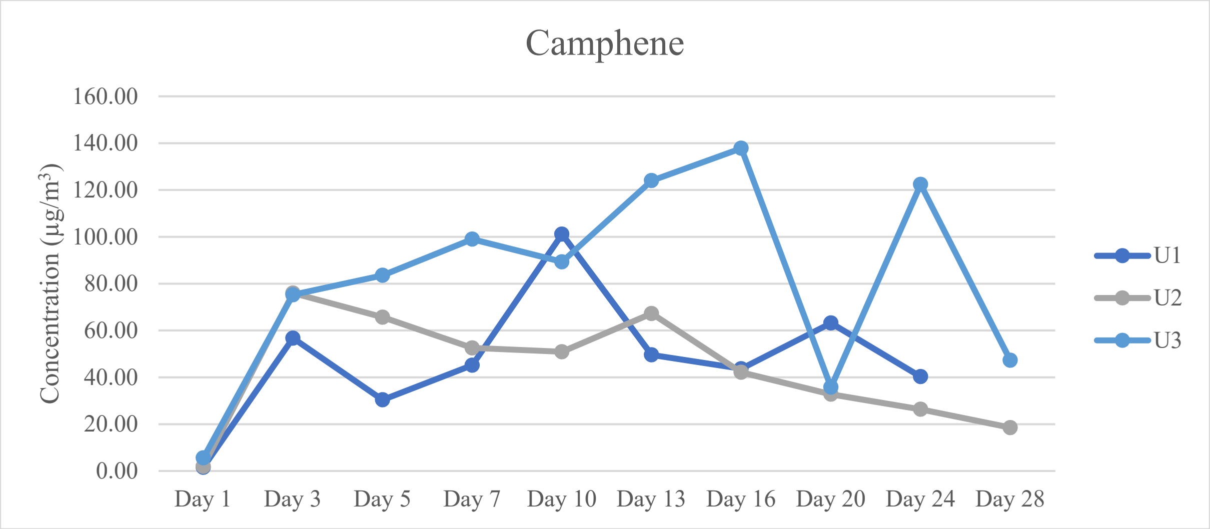

Figure 14: Camphene concentrations from three untreated samples of Scots Pine wood panels during the testing period

Table 12 presents the camphene concentrations measured in the reference chamber. These concentrations were low, with several of the samples having so low concentrations that the ATD GC-MS system was not able to detect them. These indicates a low probability of contamination that might have affected the analysis.

Table 12: Camphene concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[I]	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1,0	1,7	1,2
2 (U1)	1,59	56,7	30,4	45,2	101,1	49,6	43,7	63,2	40,3	[II]
3 (U2)	1,99	76,0	65,7	52,6	50,9	67,3	42,1	32,8	26,3	18,5
4 (U3)	5,62	75,2	83,6	99,0	89,3	124,0	137,8	35,8	122,4	47,3
Avg. con.	3,07	69,30	59,88	65,59	80,44	80,28	74,54	43,92	63,01	32,89
SD	2,22	10,90	27,05	29,17	26,25	38,87	54,78	16,76	51,88	20,31
CV%	72,48	15,73	45,18	44,48	32,64	48,42	73,49	38,17	82,34	61,76

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.2.2.6. 3-carene

The *3-carene* concentrations measured in this study are presented in Figure 15. 3-carene is the highest emitting VOC in this study, with sample U3 mostly contributing to this. Both less variation and lower concentrations was observed between samples U2 and U3.

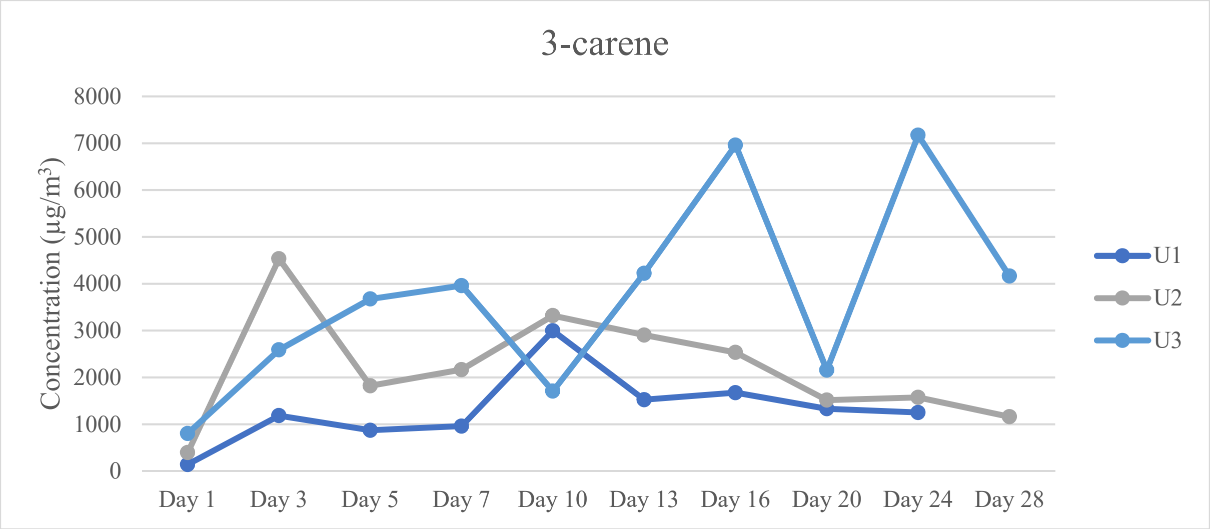

Figure 15: 3-carene concentrations from three untreated samples of Scots Pine wood panels during the testing period

The 3-carene concentrations observed in the reference chamber indicate a low probability for contamination, compared to the concentrations from the chambers with samples inside. This can be seen in Table 13. The SD is as expected, with sample U3 mostly contributing to the variation. This is also why a higher SD was observed.

Table 13: 3-carene concentrations from three different untreated samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1[I]	1,64	6,1	10,8	15,4	20,4	45,8	59,8	33,8	59,1	45,0
2 (U1)	141,5	1184,3	871,6	961,4	2996,8	1523,9	1674,2	1332,0	1251,2	[II]
3 (U2)	397,36	4532,8	1822,2	2167,1	3320,7	2905,4	2534,7	1515,5	1574,4	1159,8
4 (U3)	801,84	2589,4	3675,1	3960,6	1706,5	4223,6	6960,3	2156,8	7170,9	4164,3
Avg. con.	446,90	2768,84	2122,97	2363,03	2674,70	2884,29	3723,05	1668,10	3332,15	2662,05
SD	332,94	1681,44	1425,76	1509,14	853,94	1349,97	2836,37	433,06	3328,36	2124,50
CV%	74,50	60,73	67,16	63,86	31,93	46,80	76,18	25,96	99,89	79,81

[I] Reference chamber

[II] This sample was lost due to a malfunction in the ATD GC-MS system

4.3. Wood stain samples

4.3.1. TVOC concentrations

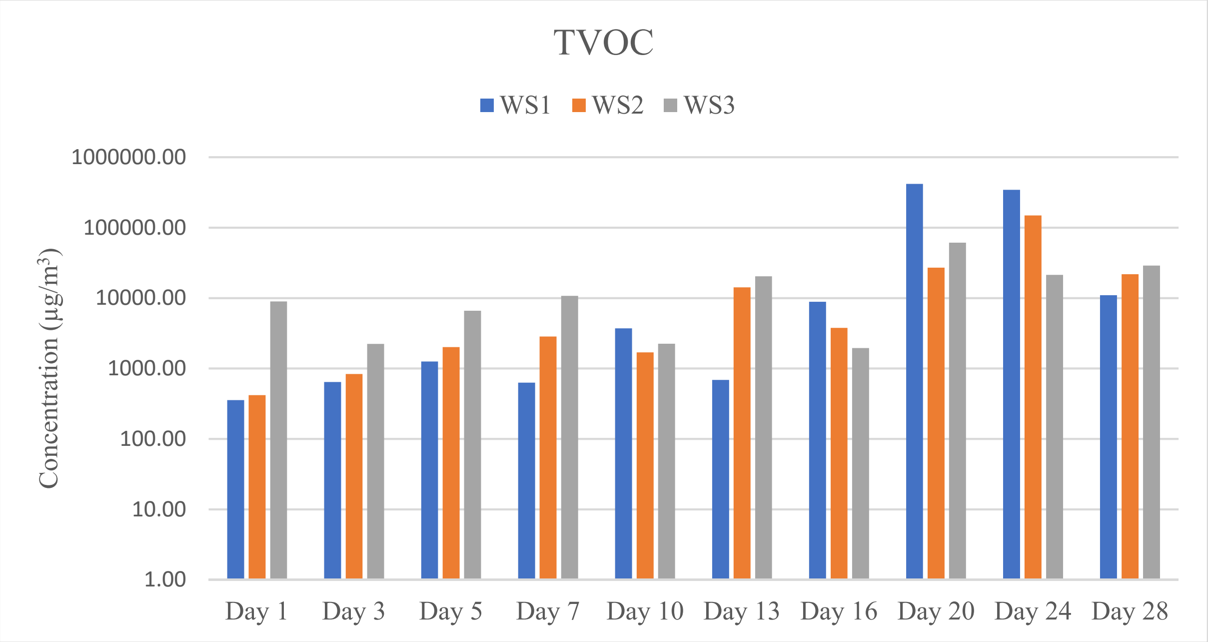

Figure 16: TVOC concentrations from wood stain samples

Figure 16 shows the TVOC concentrations measured during the sampling period of 28 days, for each of the three wood stain samples. The concentrations for these samples were substantially higher than the ones for the untreated wood samples, and the data had therefore to be put on a logarithmic scale with a base of ten. The emission pattern was similar to the one observed for the untreated wood samples, only with much higher concentrations. Variability in TVOC concentrations during the sampling period was observed between both samples and sampling days. At day 1, the highest observed TVOC concentration was from sample WS3. This trend continued for most of the sampling period, with the exception of days 16, 20 and 24. Sample 1 had a spike in TVOC concentrations at these three days. WS2 had a spike on day 13, but this concentration was still lower than the one for WS3. The TVOC concentration ranged from 346,2 µg/m^3 to 328 622 µg/m^3 during the sampling period.

4.3.2. Single VOC concentrations

The single VOC concentrations for the wood stain samples are presented in the following section.

4.3.2.1. Hexanal

Figure 17 shows the hexanal concentrations observed during the sampling period, for each of the three wood stain samples. A variability in concentrations was observed both between samples and sampling days. Sample WS3 had the highest observed concentrations for most of the sampling period, substantially higher than the other two samples. The only exceptions were at day 24, where WS1 had the highest concentrations. WS1 and WS2 had similar concentrations during the sampling period, with the exception of a spike for WS1 on day 20. The concentration was also higher for WS1 on day 24, before these samples had very similar concentrations on day 28. All the samples had peak concentrations between days 20 and 24.

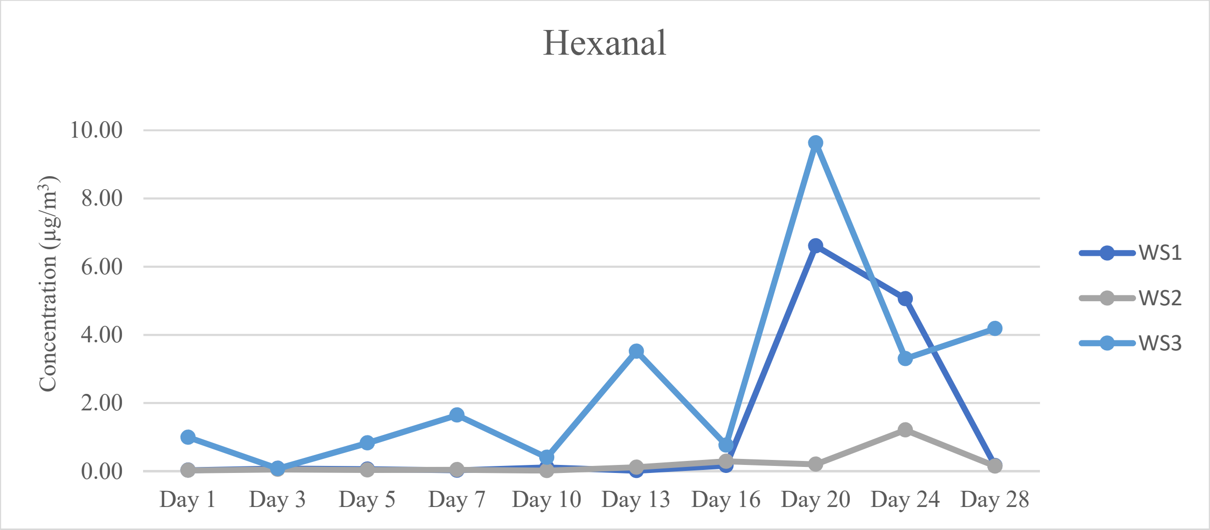

Figure 17: Hexanal concentrations from three wood stain samples of Scots Pine wood panels during the testing period

Table 14 shows that the concentrations in the reference chamber had lower concentrations than the chambers with samples. Though, it is possible to ascertain an emission pattern for the reference chamber similar to the other chambers. This might indicate that there was a possible leak in the chambers, which might have had an influence on the results. The SD is as expected, with sample WS3 mostly contributing to the variation between the samples.

Table 14: Hexanal concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1 (WS1)	0.03	0.08	0.06	0.03	0.11	0.02	0.16	6.6	5.06	0.16
2 (WS2)	0.03	0.05	0.04	0.04	0.02	0.12	0.29	0.20	1.21	0.15
3[I]	0.003	0.004	0.007	0.011	0.008	0.004	0.05	0.07	3.28	0.26
4 (WS3)	1.00	0.08	0.83	1.65	0.40	3.52	0.76	9.63	3.30	4.18
Avg. con.	0.35	0.07	0.31	0.57	0.18	1.22	0.41	5.48	3.19	1.50
SD	0.56	0.02	0.45	0.93	0.20	1.99	0.32	4.81	1.93	2.33
CV%	159.95	22.64	146.23	162.61	113.30	163.88	77.81	87.82	60.43	155.20

[I] *Reference chamber*

4.3.2.2. α-pinene

The α-pinene concentrations observed in this study can be seen in Figure 18. A lot of variability was observed between both the samples and sampling days, and due to the high concentrations observed a logarithmic scale with a base of 10 was used. Sample WS1 had the highest observed concentrations on day 10, 20, and 24. WS2 had the highest observed concentration on day 16. Meanwhile, WS3 had the highest observed concentrations on days 1, 3, 5, 7, 13 and 28. All of the samples had peak concentrations between days 20 and 24.

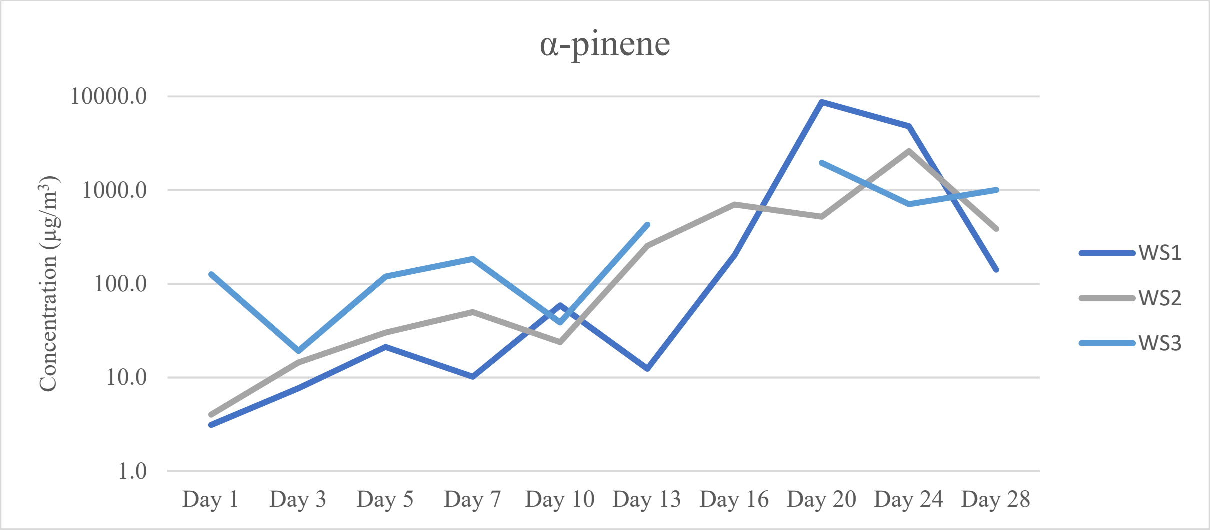

Figure 18: α -pinene concentrations from three wood stain samples of Scots Pine wood panels during the testing period

The concentrations in the reference chamber, seen in Table 15, show a similar emission pattern to the samples. This might indicate a leak in the chambers, and it is difficult to exclude possible contaminations in the chambers. The observed concentrations might therefore have been affected, and the high concentrations observed might be due to this. The SD is as expected, with the different samples contributing to the observed variation on different days.

Table 15: α -pinene concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1 (WS1)	3.12	7.67	21.19	10.17	58.91	12.42	202.18	8704.20	4807.81	141.34
2 (WS2)	4.02	14.46	30.18	49.99	23.82	254.59	702.27	519.74	2604.80	386.49
3[I]	1.91	7.47	16.01	19.98	9.58	6.17	79.54	65.31	2492.95	156.67
4 (WS3)	126.39	19.19	119.39	184.49	38.65	427.70	[II]	1957.08	707.03	1005.59
Avg. con.	44.51	13.77	56.92	81.55	40.46	231.57	452.23	3727.01	2706.55	511.14
SD	70.91	5.79	54.29	91.34	17.62	208.60	353.61	4369.88	2052.28	445.40
CV%	159.33	42.03	95.37	112.01	43.54	90.08	78.19	117.25	75.83	87.14

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.3.2.3. β-myrcene

Figure 19 shows the β-myrcene concentrations observed during the sampling period, for each of the three wood stain samples. Due to the high concentrations observed a logarithmic scale with a base of 10 was used, and a lot of variability between both the samples and sampling days was observed. Sample WS1 had the highest observed concentrations on day 20 and 24, while sample WS2 had the highest observed concentrations on days 16 and 28. On days 1, 3, 5, 7, 10 and 13 WS3 had the highest concentrations. All of the samples had peaks between days 20 and 24.

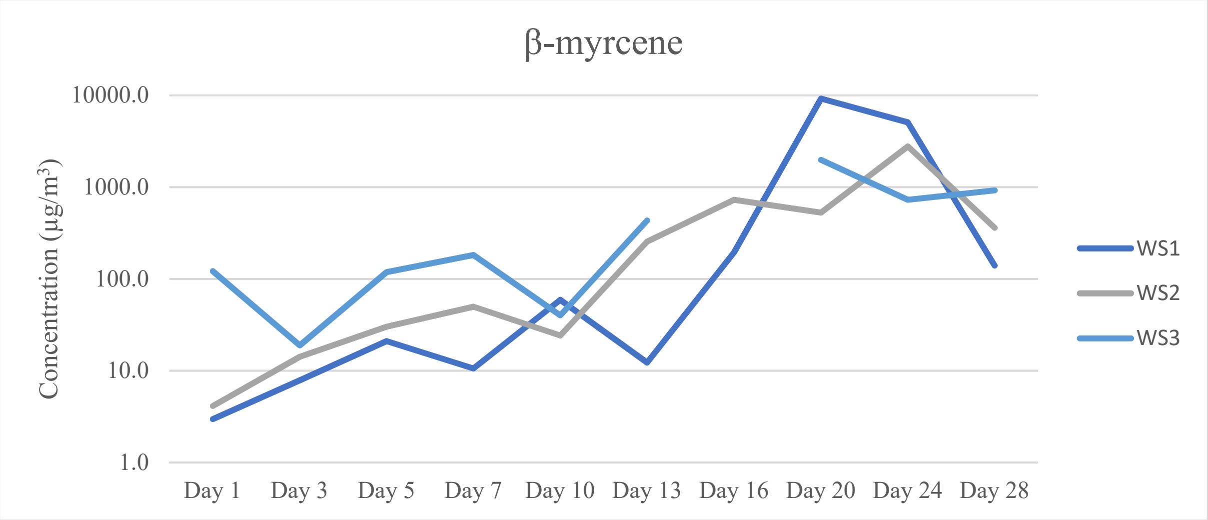

Figure 1910: β -myrcene concentrations from three wood stain samples of Scots Pine wood panels during the testing period

The concentrations in the reference chamber, seen in Table 16, show a similar emission pattern to the samples. This might indicate a leak in the chambers, and it is difficult to exclude possible contaminations in the chambers. The observed concentrations might therefore have been affected, and the high concentrations observed might be due to this. The SD is as expected, with the different samples contributing to the observed variation on different days.

Table 16: β -myrcene concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1 (WS1)	2.97	7.87	21.00	10.56	59.11	12.30	194.49	9217.04	5079.65	140.11
2 (WS2)	4.13	14.17	30.22	49.75	24.23	255.65	729.43	527.28	2782.18	360.03
3[I]	2.10	7.66	15.56	19.28	9.62	6.54	82.88	71.31	2711.73	151.04
4 (WS3)	121.58	18.88	118.67	182.23	40.10	433.99	[III]	1979.86	727.49	924.09
Avg. con.	42.89	13.64	56.63	80.85	41.15	233.98	461.96	3908.06	2863.11	474.74
SD	68.15	5.52	53.93	89.96	17.46	211.68	378.26	4654.72	2177.21	404.38
CV%	158.88	40.51	95.23	111.27	42.44	90.47	81.88	119.11	76.04	85.18

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.3.2.4. β-pinene

The β-pinene concentrations observed in this study can be seen in Figure 20. A substantial amount of variation was observed between both the samples and sampling days. WS1 had the highest observed concentrations on days 20 and 24, while WS2 had the highest observed concentrations on day 16. Sample WS3 had the highest observed concentrations on days 1, 3, 5, 7, 10, 13 and 28.

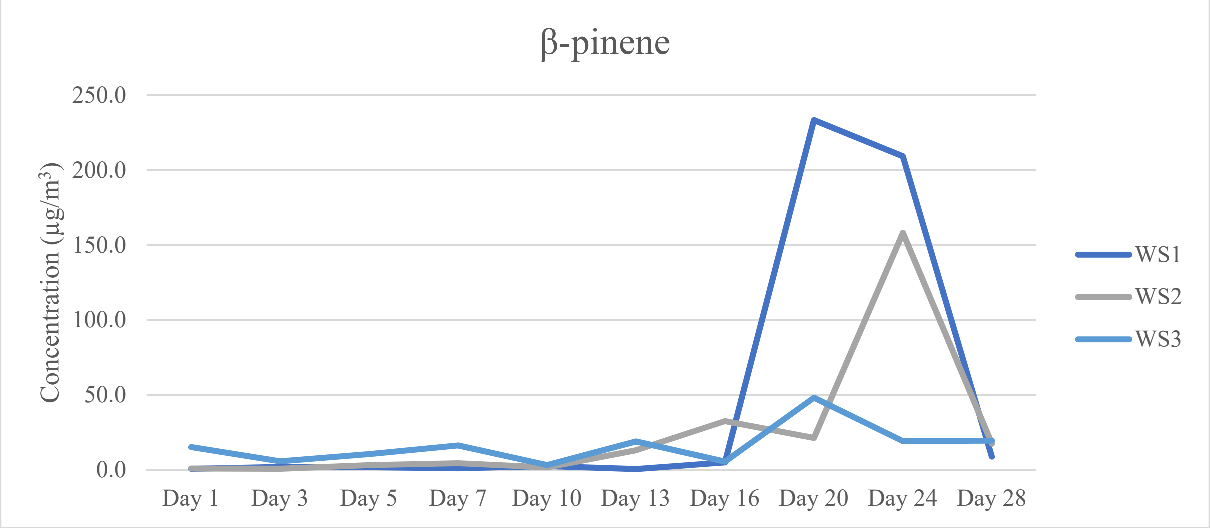

Figure 20: β -pinene concentrations from three wood stain samples of Scots Pine wood panels during the testing period

Table 17 shows that the concentrations in the reference chamber had lower concentrations than the chambers with samples. Though, it is possible to ascertain an emission pattern for the reference chamber similar to the other chambers with samples. This might indicate that there was a possible leak in the chambers, which might have had an influence on the results. The SD is as expected, with different samples contributing to the variation between the samples on different days.

Table 17: β -pinene concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibrations range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1	0.77	2.08	1.83	1.02	2.59	0.61	5.02	233.41	209.28	8.92
2	0.92	0.82	3.22	4.52	1.73	13.15	32.57	21.35	158.11	17.48
3[I]	0.49	0.78	1.32	1.74	0.95	0.34	4.04	3.77	103.85	6.58
4	15.25	5.73	10.62	16.36	3.25	18.98	5.58	48.19	19.19	19.55
Avg. con.	5.65	2.88	5.22	7.30	2.52	10.92	14.19	100.98	128.86	15.32
SD	8.32	2.55	4.72	8.04	0.76	9.39	15.75	115.47	98.36	5.64
CV%	147.26	88.59	90.42	110.11	30.12	85.99	109.46	114.34	76.33	36.82

[I] *Reference chamber*

[II] *These concentrations were above the calibration range, and therefore extrapolated*

4.3.2.5. Camphene

Figure 21 shows the camphene concentrations observed during the sampling period, for each of the three wood stain samples. Due to the high concentrations observed a logarithmic scale with a base of 10 was used, and a lot of variability between both the samples and sampling days was observed. Sample WS1 had the highest observed concentrations on days 10, 20 and 24, while sample WS2 had the highest observed concentrations on day 16. On days 1, 3, 5, 7, 13 and 28 WS3 had the highest concentrations. All of the samples had peaks between days 20 and 24.

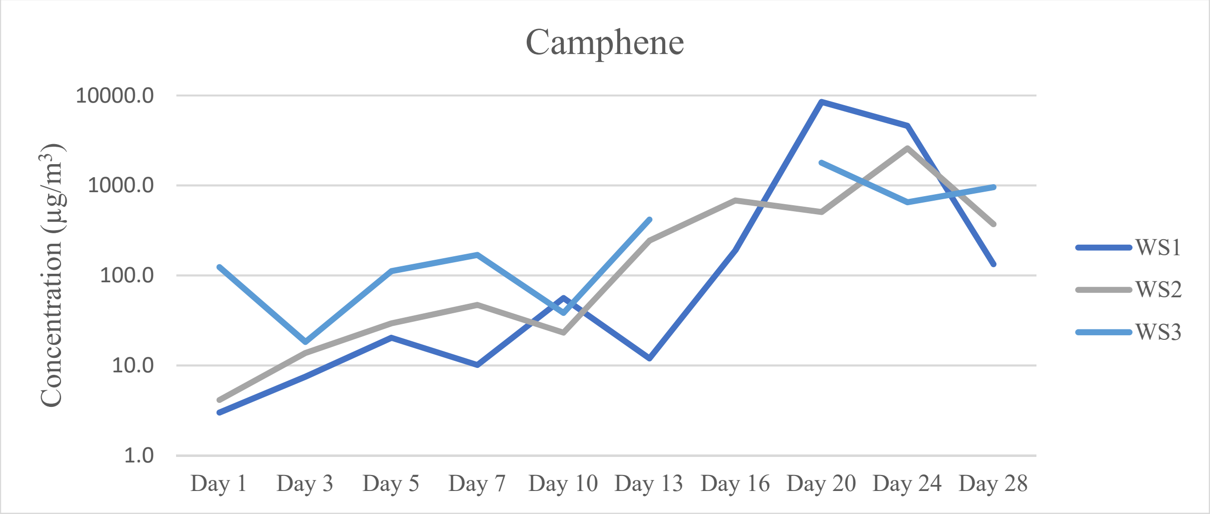

Figure 21: Camphene concentrations from three wood stain samples of Scots Pine wood panels during the testing period

The concentrations in the reference chamber, seen in Table 18, show a similar emission pattern to the samples. This might indicate a leak in the chambers, making it difficult to exclude possible contaminations. The observed concentrations might therefore have been affected, and the high concentrations observed might be due to this. The SD is as expected, with the different samples contributing to the observed variation on different days.

Table 18: Camphene concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibration range, and therefore extrapolated.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1	3.00	7.52	20.36	10.13	56.05	12.00	190.71	8478.99	4600.56	133.22
2	4.13	13.76	29.43	47.02	23.20	243.91	680.52	507.03	2586.16	371.45
3[I]	1.97	7.44	15.27	20.18	8.30	6.25	75.23	64.96	2417.99	150.06
4	124.02	18.26	111.82	169.40	38.28	418.19	[II]	1789.64	650.67	956.64
Avg. con.	43.72	13.18	53.87	75.52	39.18	224.70	435.62	3591.89	2612.46	487.10
SD	69.55	5.39	50.39	83.37	16.44	203.77	346.35	4280.67	1975.07	423.72
CV%	159.08	40.92	93.54	110.40	41.97	90.69	79.51	119.18	75.60	86.99

[I] *Reference chamber*

[II] *This sample was lost due to a malfunction in the ATD GC-MS system*

4.3.2.6. 3-carene

The 3-carene concentrations observed in this study can be seen in Figure 22. A lot of variability was observed between both the samples and sampling days, and due to the high concentrations observed a logarithmic scale with a base of 10 was used. Sample WS1 had the highest observed concentrations on day 10, 20, and 24. WS2 had the highest observed concentration on day 16. Meanwhile, WS3 had the highest observed concentrations on days 1, 3, 5, 7,13 and 28. All of the samples had peak concentrations between days 20 and 24.

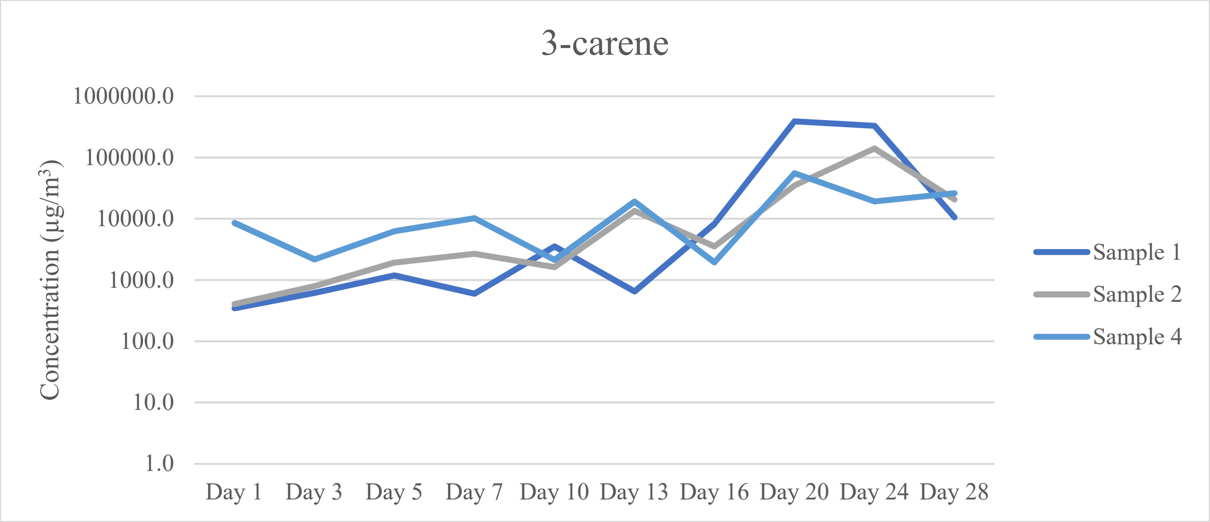

Figure 22: 3-carene concentrations from three wood stain samples of Scots Pine wood panels during the testing period

Table 19 shows that the concentrations in the reference chamber had lower concentrations than the chambers with samples. Though, it is possible to identify an emission pattern for the reference chamber similar to the other chambers with samples. This might indicate that there was a possible leak in the chambers, which might have had an influence on the results. The SD is as expected, with different samples contributing to the variation between the samples on different days.

Table 19: 3-carene concentrations from three wood stain samples of Scots pine wood panels during the testing period, with average concentrations, standard deviation (SD) and standard deviation in percent (CV%). The highlighted values are above the calibration range.

Chamber	Day 1	Day 3	Day 5	Day 7	Day 10	Day 13	Day 16	Day 20	Day 24	Day 28
1	346.22	616.70	1189.62	596.02	3527.33	650.53	8239.02	389909.03	328622.36	10570.69
2	404.22	791.47	1923.37	2684.15	1623.35	13413.12	34821.71	25402.66	140637.16	20615.89
3[I]	241.03	449.52	862.81	1192.21	649.64	309.28	3533.22	3102.98	134346.86	7487.11
4	8553.99	2173.84	6242.85	10204.95	2129.55	19066.46	1940.91	55443.83	19203.00	26033.26
Avg. con.	3101.47	1194.00	3118.61	4495.04	2426.74	11043.37	160058.19	156918.50	162820.84	19073.28
SD	4722.10	853.05	2730.43	5053.95	986.16	9433.89	199323.54	202334.02	155897.96	7845.86
CV%	152.25	71.44	87.55	112.43	40.64	85.43	124.53	128.94	95.75	41.14

[I] *Reference chamber*

[II] *These concentrations were above the calibration range, and therefore extrapolated*

4.4. Comparison of treated versus untreated samples

Even though the relative emission levels differed between the untreated and wood stain samples, ANOVA did not confirm statistically significant differences between these two sample types. The p-level was at 0.1480, as presented in Table 20. The emissions from the untreated samples exhibited less variation than the wood stain samples, cf. Figure 23. Furthermore, ANOVA did not confirm statistically significant differences for any of the other sampling days. Thus, ANOVA could not confirm statistically significant differences between the sample types throughout the sampling period.

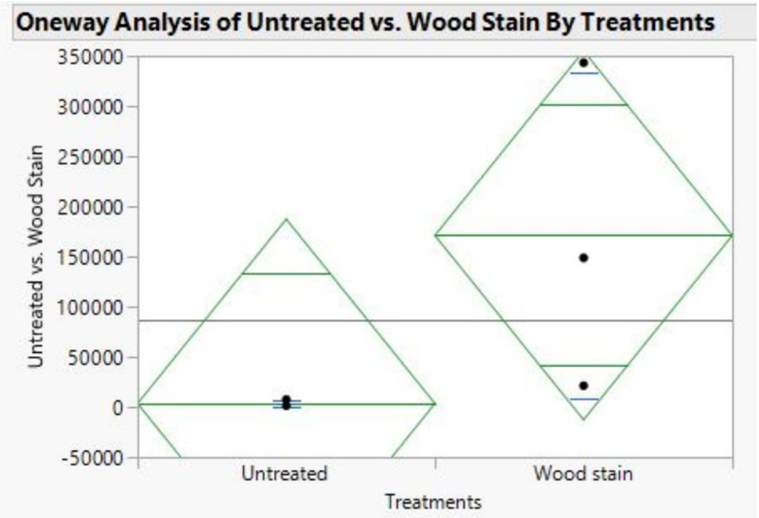

Figure 23: Comparison of treated vs. untreated samples on day 24

Table 20: ANOVA table for day 24

Source	DF	SS	MS	F	p
Treatment	1	4.2143e + 10	4.214e + 10	3.2035	0.1480
Error	4	5.2621e + 10	1.316e + 10		
C. Total	5	9.4764e +10			

ANOVA assumes that the variances are equal, and the data are normally distributed (Dean et al., 2017). This did not hold for the observations in this study. A possible solution for this is to use a nonparametric test, which does not assume normality in the data. An example of a nonparametric test is a Wilcoxon signed rank test, though we were not able to use this test in this case. This was due to the sample size being too small to use this type of test. The conclusion was therefore that there was not enough data to reject the null hypothesis for any of the time points.

5. Discussion

High concentrations of VOCs were observed in both untreated and wood stain samples. This was not unexpected, as *Scots Pine* is considered to be a high emitting wood species. According to Pohleven et al. (2019) intraspecific variations have been shown to affect VOC emissions from trees. The samples used in this study came from different trees, and the observed variations in emission profiles were expected. It is also important to mention that earlier studies found a variability in emissions among the same type of chambers (Afshari, 2003).

Findings from similar studies indicate that this model and analytical system performed well, having in mind that the emissions pattern, VOC content and composition were comparable. The criteria for the analytical system i.e. the method validation and quantification were acceptable and suggested an overall good sensitivity for the target compounds.

5.1. Untreated wood panels

A high variation in the specific VOCs measured in the present study observed for the untreated wood panels. This variation may have been caused by both endogenous factors, including genetic and biochemical factors, and on exogenous factors such as growth conditions, material, and sample treatment along with chamber conditions (Pohleven et al., 2019). It is therefore important to consider these factors when testing VOC emissions from different wood samples.

The sample type and material treatment may have generated higher VOC emissions for several of the compounds in this study. Namely *3-carene, camphene and β-myrcene* while *hexanal*, *β-pinene* and *a-pinene* were within the normal range based on earlier studies as seen in chapter 4.2. The samples chosen for this study were collected right after manufacture, directly after planing. This process has been shown to generate higher VOC emission rates (Risholm-Sundman et al., 1998). Thus, the time period between planing and analysis may have had a direct impact on the VOC concentrations, as especially monoterpenes emit the most at an early stage (Pohleven et al., 2019). The samples were additionally cut down to fit inside the chambers, releasing fresh VOCs which may also have led to increasing emissions.

The large intraindividual variability of volatile emissions from *Scots Pine* can mostly be attributed to genetic factors, according to Englund (1999). Volatile terpenes are not homogenously distributed in softwoods, but rather stored in discrete resin canals which produce terpene rich sections within the material (Pohleven et al., 2019). These sections must be considered when comparing VOC emissions from different samples, as they may lead to a variability in the VOC emissions within the same material. The ratio of heartwood to softwood is another factor which may affect VOC emissions, as heartwood has been shown to generate higher VOC emission rates in Scots Pine (Ingram et al., 2000). Besides genetic factors, the emissions of VOCs from wood panels may also have been influenced by intraspecific variations, such as the date of harvesting, the portion of knots and other defect areas alongside the presence of tree/wood injuries such as different types of impacts.

5.1.1. TVOC and single VOC concentrations

The highest levels of observed VOCs in the study were monoterpenes. A variability in these concentrations was observed between both samples and sampling days, during the study. There were several TVOC spikes which may have been caused by different sources, like issues with sampling, internal standard preparations, or issues in the analytical system.

Similar spikes during the sampling period have also been observed in studies by (Stratev et al., 2016; Hyttinen et al. 2010). Possible explanations for these spikes are high RH and ventilation issues. Such RH variability was also present during this study, as the RH fluctuated from 0,4-72,4% (Table A3, Appendix A), There was a clear pattern in how the RH changed, where it fell during the sampling and then rose again after sampling. Temperature does not seem to be a contributing factor in this study, as the recorded temperature was constant at 23˚C ±1 (Figure B2, appendix B).

Further, the lack of ventilation in the model system may have had an effect on both the VOC and TVOC concentrations. Air exchanges only occurred during sampling periods, as there was no constant airflow. This led to VOCs accumulating in the chambers, thus elevating their concentrations. This differs from other studies, where a constant air flow dilutes and decreases VOC concentrations. Hence, it is possible that the intervals between the sampling days may have influenced the VOC emissions observed at different stages in the sampling period. The TVOC concentrations were generally lower between days 16-28, cf. Figure 9, despite the VOCs having more time to accumulate in the chambers. In contrast, the emissions were higher before this, which may rather be attributed to the VOC emission profile rather than the lack of ventilation.

The VOC emission pattern can be summarized by increasing emissions during the first 16 days, with a noticeable emission increase on day 3. A relative stable emission was then observed from days 5-16, with some variations between samples and sampling days. After this period, a gradual decrease was observed for most of the samples and compounds. This is a similar pattern to a study by Englund (1999), with untreated samples from *Scots Pine.*

3-carene was the most abundant single VOC throughout the sampling periods for untreated samples, as seen in Ch. 4.2.6. *3-carene* was the highest measured VOC in a study by Englund (1999), while *α-pinene is* usually the highest measured VOC in most emissions studies of *Scots Pine.* The *3-carene* concentrations in the present study were more than 10 times higher than the *α-pinene* concentrations.

The quality of the analytical system can be assured by looking at the criteria for the quality control, i.e. apparent recovery, LOD, LOQ and calibration curves (Appendix A, Table A2). These were within an acceptable range and seem not to have affected the *3-carene* concentrations. The apparent recovery was at 68,3 ± 18,1%, indicating that there was low possibility of *3-carene* contamination during the analysis. The results were high and had to be extrapolated, but this should not have influenced the quality of the results. The calibration had an R^2 over 0,95, which is acceptable for the purposes of this type of study (Skoog, 2013).

The high *3-carene* concentrations may also be related to the wood treatment. Englund (1999) and Granström (2005) found that the VOC profiles and concentrations in wood are also affected by seasonal changes, with individual VOCs being more abundant in certain temperate seasons. Drying wood, as in the case with these samples, may lead to chemical reactions and the possible change of VOC profiles and concentrations.

As mentioned above, the *α-pinene* concentrations were lower than expected. This may have been caused by seasonal changes, or possible chemical reactions. However, the analytical parameters were within the expected range and seem not to impact the results.

The concentrations of *β-myrcene* and *camphene* were also observed in higher concentrations than expected, compared to previous findings. Typically, the concenctarions of *β-myrcene* are somewhat higher than the concentration of *camphene*, but still the concentrations of these VOCs are as high as those for *α-pinene.* Possible reasons for these deviations may be caused by genetic factors, or issues during the sampling. *β-myrcene* is formed from the dehydration and isomerization of *geraniol* (Eggersdorfer, 2000). The samples were dried during the manufacturing process, and this may have led to an increase in the concentrations of *β-myrcene.*

The higher levels of *camphene* may be attributed to some of the previously mentioned factors, i.e. endogenous or exogenous factors with acceptable analytical parameters. The *β-pinene* concentrations were as expected, only contributing a little to the overall TVOC concentrations. No issues with the analytical system stood out.

Hexanal was found to be the least emitting VOC in untreated samples. Hexanal, along with several other aldehydes, is emitted in relatively low concentrations in softwood such as *Scots Pine* according to Pohleven et al. (2019). Only a small amount of *hexanal* naturally occurs in softwoods, and is formed from chemically or physically bound compounds that are released by chemical or physical degradation of wood or wood extractives (Granström, 2005; Pohleven et al., 2019). Aldehydes are also emitted more evenly, and with lower concentrations over a longer time in contrast to volatile terpenes (Pohleven et al., 2019).

5.2. Wood stain samples

The wood stain samples had a similar VOC profile to the untreated samples, with the exception that the concentrations were much higher. This variation may have been caused by endogenous and exogenous factors, along with chamber conditions. These factors have also to be considered, as they were in Ch. 1.1. The main difference between the wood stain samples and the untreated wood panels was the coating. Water-borne acrylate paint was applied on the test material and may have affected both the VOC profile and concentration.

5.2.1. TVOC and single VOC concentrations

The VOC emission results from the wood stain samples were also dominated by volatile monoterpenes. Several spikes were also observed for these samples and may have been caused by the same factors as the ones for the untreated samples. Salo (2017) also experienced this type of spikes on wood samples coated with a similar type of paint. This may therefore be a normal occurrence in emission pattern for *Scots Pine.*

In general, the emission pattern was similar to the emission pattern for the untreated samples with a few differences. The TVOC concentrations were far higher during the first days of the test, reaching their peak at days 20-24, and decreased more rapidly thereafter, vs. the untreated samples, cf. the other studies done on coated samples from Scots Pine.

3-carene was the most abundant single VOC for the wood stain samples, even though *α-pinene* has been found to be the most abundant VOC in similar studies. This difference is likely to have been caused by other factors, since monoterpene emissions are less likely affected by coatings (Salo, 2017). The quality control was the same as for the untreated samples, thus satisfactory for *3-carene*. Endogenous and exogenous factors may have influenced the *3-carene* concentrations, cf. Ch. 5.1.1.

The profile of the other single VOCs was similar to the one for the untreated samples, with lower *α-pinene* concentrations than expected. *β-myrcene* and *camphene* had higher concentrations than expected, probably due to the aforementioned factors. *β-pinene* concentrations were as expected, and no particular issues stood out. Finally, hexanal was found to be the least emitting VOC in this portion of the study.

5.3. Comparison of untreated versus wood stain samples

The results for the untreated and wood stain samples can be seen in Table 19 and Table 20. *3-carene* did not pass the EU LCI criteria after 28 days for any of the samples. Some of this can be attributed to the model system, due to the lack of ventilation. The VOCs were not diluted, as they usually are under standard testing conditions in EN 16516. This may have caused higher concentrations. ANOVA did not confirm significant statistical differences between untreated and wood stain samples, cf. Ch. 4.4.

Even though no statistically significant differences were found, there was still a substantial a difference in the emission profile between these two sample types. The wood stain samples started with higher concentrations than the untreated samples, and also reached their peak concentrations later. This may have been caused by the coating, as studies have found that coated wood samples follow the emission pattern of the specific coating (Salo, 2017). Coatings are dominated by secondary VOCs, which reach an equilibrium in their emissions later than primary VOCs such as monoterpenes.

The emission profile of a material is also affected by changes in the boundary layer, as VOCs have to be emitted through this layer (Afshari et al., 2003). This layer should act as a barrier which reduces the emissions, but the film thickness may also have an effect on the emissions. The film thickness was not measured, but should be considered in this type of comparison measurements. The storing conditions of the coatings also may influence the results, as terpenes are volatile at ambient temperatures and may transfer over to the coatings. Terpenes are not commonly found in coatings (Plesser, 2012), but this transmission risk should be taken into account when interpreting this type of results.

Table 21: Untreated wood panel concentrations compared to EN16516

Single VOCs	**Concentration** (µg/m³)					**EU LCI** (µg/m³)
	U2	**U3**	**WS1**	**WS2**	**WS3**	
Hexanal	0.02	0.02	0.16	0.15	4.18	900
α-pinene	18.1	45.8	141.34	386.49	1005.59	1400
β-myrcene	22.2	44.79	140.11	360.03	924.09	1400
β-pinene	1.6	3.4	8.92	17.48	19.55	1400
3-carene	1159.8	4164.3	10570.69	20615.89	26033.26	1500
camphene	18.5	47.3	133.22	371.45	956.64	1400
TVOC	1220	4305	10994	361484	28943	N/A

6. Conclusions

The main objectives of this study were to establish a VOC model system, measure VOC emissions from two different types of *Scots Pine interior panel* and to explore how these results compared to those from other studies.

VOC emissions from both untreated and wood stain samples were measured with good accuracy in this model system, despite some issues with RH and a possible leak in the chambers. An alternative approach is to stabilize RH and the possible leak by fine-tuning the chambers and humidifying the air.

The results demonstrated that VOCs from the untreated and wood stain samples mainly included monoterpenes and traces of aldehydes. High concentrations of α-pinene, *β-myrcene, camphene and 3-carene* were observed in both sample types. The obtained results for *3-carene* demonstrate that the use of *Scots Pine* does not pass the EU-LCI limit for either sample type. The other compounds were within these limits.

Even though there was some difference in profiles of VOCs released from untreated and wood stain samples, ANOVA did not confirm a statistically significant difference between these two sample types. This may be due to the low number of samples, but this could not be evaluated by nonparametric tests.

There is no big discrepancy between this and other studies, but the high concentrations of 3-carene might be an issue. Furthermore, the results for *β-myrcene* were semi quantitative and might not be as high if measured with better accuracy. The results pinpoint a low dominance of aldehydes in indoor applications, which was as expected and desired.

In the future, it may be beneficial to increase the number of VOC chambers in order to perform statistical calculations. It may also be beneficial to increase the number of sampling days, in order to further examine the spikes seen in the present study. There seems to be a lack of research on this topic, and as the spikes may be a pattern of the VOC emission profile they should be addressed.

7. References

Aarsbog, L. (2019). *Solvent-free quantification for selected volatile organics in Artic air: application of thermal desorption-gas chromatography-mass spectrometry*. Master thesis. Ås: Norges miljø- og biovitenskapelige universitet. Available at: https://nmbu.brage.unit.no/nmbu-xmlui/handle/11250/2642505 (accessed: 21.02.2020).

Afshari, A., Lundgren, B. & Ekberg, L. (2003). Comparison of threee small chamber test methods for VOC emission rates from paint. *Indoor Air*, 13 (2): 156-165. doi: 10.1034/j.1600-0668.2003.00146.x.

Alpha, B. & Corneau, D. (2006). *Effectiveness of barriers to minimize VOC emissions including formaldehyde*. Forest Products Journal. Available at: http://agris.fao.org/agris-search/search.do?recordID=US201301104188 (accessed: 21.02.2020).

Arbeidsmiljøloven. (2005). *Lov om arbeidsmiljø, arbeidstid og stillingsvern mv.* . Available at: https://lovdata.no/dokument/NL/lov/2005-06-17-62 (accessed: 18.02.2020).

Bartekova, A., Lungu, C., Shmulsky, R., Huelman, P. & Park, J. (2006). Laboratory evaluation of volatile organic compounds emissions from coated and uncoated oriented strandboard. *Forest Products Journal*, 56 (2): 85-90. Available at: https://www.academia.edu/18731951/Laboratory_evaluation_of_volatile_organic_compounds_emissions_from_coated_and_uncoated_oriented_strandboard (accessed: 20.02.2020).

BREEAM-NOR. (2016). *BREEAM-NOR 2016 Technical Manual v.1.2*. TECHNICAL MANUAL SD5075NOR. 05.02.2020. Available at: https://byggalliansen.no/wp-content/uploads/2019/06/SD-5075NOR-BREEAM-NOR-2016-New-Construction-v.1.2.pdf.

Burns, D., Danzer, K. & Townshend, A. (2002). Use of the term "recovery" and "apparent recovery" in analytical procedures (IUPAC Recommendations 2002). *Pure and Applied Chemistry - PURE APPL CHEM*, 74 (11): 2201-2205. doi: 10.1351/pac200274112201.

Clausen, P. A. & Kofoed-Sørensen, V. (2009). Sampling and Analysis of SVOCs and POMs in Indoor Air. In Salthammer, T. & Uhde, E. (eds) *Organic indoor air pollutants 2nd edition*, pp. 19-47. Weinheim: WILEY-VCH Verlag GmbH & Co.

Dean, A., Voss, D. & Dragulic, D. (2017). *Design and Analysis of Experiments Second Edition*. New York: Springer International Publishing AG.

Direktoratet for byggkvalitet. (2017). B*yggteknisk forskrift (TEK17) med veiledning*. Available at: https://dibk.no/byggereglene/byggteknisk-forskrift-tek17/ (accessed: 18.02.2020).

Doty, R. L., Cometto-Muñiz, J. E., Jalowayski, A. A., Dalton, P., Kendal-Reed, M. & Hodgson, M. (2004). Assessment of Upper Respiratory Tract and Ocular Irritative Effects of Volatile Chemicals in Humans. *Critical Reviews in Toxicology*, 34 (2): 85-142. doi: 10.1080/10408440490269586.

Eggersdorfer, M. (2000). Terpenes. In Bellussi, G., Bohnet, M., Bus, J., Drauz, K., Greim, H., Jackel, K.-P., Karst, U., Kleemann, A., Kreysa, G., Laird, T., et al. (eds) vol. 36 *Ullmann's Encyclopedia of Industrial Chemistry*, pp. 29-45: Wiley-VCH Verlag GmbH & Co. doi: 10.1002/14356007.a26_205

Englund, F. (1999). *Emissions of volatile organic compounds (VOC) from wood*. Report I 9901001. Stockholm: Tratek, Swedish Institute for Wood Technology Research.

European Committee for Standardization. (2017). *EN 16516 Construction products: Assessment of release of dangerous substances - Determination of emissions into indoor air*. Brussels: European Committee for Standardization,.

EØS-notatbasen. (2012). *Tekniske tilpasninger 2004/42/EC - VOC*. Available at: https://www.regjeringen.no/no/sub/eos-notatbasen/notatene/2011/mai/tekniske-tilpasninger-200442ec---voc/id2432568/ (accessed: 18.02.2020).

Fechter, J.-O., Englund, F. & Lundin, A. (2006). Association between temperature, relative humidity and concentration of volatile organic compounds from wooden furniture in a model room. *Wood Material Science & Engineering*, 1 (2): 69-75. doi: 10.1080/17480270600900551.

Frankel, E. N. (2005). *Lipid Oxidation*. Davis: Bridgewater: Oily Press.

Gminski, R., Marutzky, R., Kevekordes, S., Fuhrmann, F., Bürger, W., Hauschke, D., Ebner, W. & Mersch-Sundermann, V. (2011a). Chemosensory irritations and pulmonary effects of acute exposure to emissions from oriented strand board. *Human & Experimental Toxicology*, 30 (9): 1204-1221. doi: 10.1177/0960327110388537.

Gminski, R., Marutzky, R., Kevekordes, S., Fuhrmann, F., Bürger, W., Hauschke, D., Ebner, W. & Mersch-Sundermann, V. (2011b). Sensory irritations and pulmonary effects in human volunteers following short-term exposure to pinewood emissions. *Journal of Wood Science*, 57: 436–445. doi: 10.1007/s10086-011-1182-1.

Granström, K. (2005). *Emissions of volatile organic compounds from wood*. Doctoral dissertation. Karlstad: Karlstad University Studies. Available at: https://www.diva-portal.org/smash/get/diva2:24672/FULLTEXT01.pdf (accessed: 30.01.2020).

Hameury, S. (2007). Influence of coating system on the moisture buffering capacity of panels of Pinus sylvestris L. *Wood Material Science & Engineering*, 2 (3-4): 97-105. doi: 10.1080/17480270801906167.

Harb, P., Locoge, N. & Thevenet, F. (2018). Emissions and treatment of VOCs emitted from wood-based construction materials: Impact on indoor air quality. *Chemical Engineering Journal*, 354: 641-652. doi: 10.1016/j.cej.2018.08.085.

Huber, L. (1998). *Validation and Qualification in Analytical Laboratories*. 1 ed. Boca Raton: CRC Press.

Hyttinen, M., Masalin-Weijo, M., Kalliokoski, P. & Pasanen, P. (2010). Comparison of VOC emissions between air-dried and heat-treated Norway spruce (Picea abies), Scots pine (Pinus sylvesteris) and European aspen (Populus tremula) wood. *Atmospheric Environment*, 44 (38): 5028-5033. doi: 10.1016/j.atmosenv.2010.07.018.

Ingram, L. L. J., Templeton, M. C., McGraw, G. W. & Hemingway, R. W. (2000). Knot, Heartwood, and Sapwood Extractives Related to VOCs from Drying Southern Pine Lumber. *Journal of Wood Chemistry and Technology*, 20 (4): 415-439. doi: 10.1080/02773810009351892.

Jäckh, R., Annys, E., Heinzow, B., Witterseh, T., Glöckner, M., Kephalopoulos, S., Coutalides, R., Lathauwer, D., Uhl, M., Brouwere, K., et al. (2013). *Harmonisation framework for health based evaluation of indoor emissions from construction products in the European Union using the EU-LCI concept*. Environment and Quality of Life Report No 29. Available at: https://op.europa.eu/en/publication-detail/-/publication/d3d78842-bc95-4984-a2fe-2317731324bd (accessed: 25.01.2020).

Jones, A. P. (1999). Indoor air quality and health. *Atmospheric Environment*, 33 (28): 4535-4564. doi: https://doi.org/10.1016/S1352-2310(99)00272-1.

Kesselmeier, J. & Staudt, M. (1999). Biogenic Volatile Organic Compounds (VOC): An Overview on Emission, Physiology and Ecology. *Journal of Atmospheric Chemistry*, 33 (1): 23-88. doi: 10.1023/A:1006127516791.

Kim, S., Kim, H.-J. & Moon, S.-J. (2006). Evaluation of VOC Emissions from Building Finishing Materials Using a Small Chamber and VOC Analyser. *Indoor and Built Environment*, 15 (6): 511-523. doi: 10.1177/1420326X06072040.

Klepeis, N. E., Nelson, W. C., Ott, W. R., Robinson, J. P., Tsang, A. M., Switzer, P., Behar, J. V., Hern, S. C. & Engelmann, W. H. (2001). The National Human Activity Pattern Survey (NHAPS): a resource for assessing exposure to environmental pollutants. *Journal of Exposure Science & Environmental Epidemiology*, 11 (3): 231-252. doi: 10.1038/sj.jea.7500165.

Lee, Y.-K. & Kim, H.-J. (2012). The effect of temperature on VOCs and carbonyl compounds emission from wooden flooring by thermal extractor test method. *Building and Environment*, 53: 95–99. doi: 10.1016/j.buildenv.2011.10.016.

Lundgren, B., Crump, D., Knöppel, H., Laurent, A.-M., Lebret, E., Rothweiler, H., Seifert, B., Wolkoff, P. & Cavallo, D. (1994). *Sampling strategies for volatile organic compounds (VOCs) in indoor air* Environment and Quality of Life Report No 14. Available at: http://www.inive.org/medias/eca/eca_report14.pdf (accessed: 13.02.2020).

Manninen, A.-M., Pasanen, P. & Holopainen, J. (2002). Comparing the VOC emissions between air-dried and heat-treated Scots pine wood. *Atmospheric Environment*, 36 (11): 1763-1768. doi: 10.1016/S1352-2310(02)00152-8.

Oppl, R. (2014). New European VOC emissions testing method CEN/TS 16516 and CE marking of construction products. *Gefahrstoffe - Reinhaltung der Luft*, 74 (3): 62-68. Available at: https://www.eurofins.com/media/1721/gst-2014-x738-oppl-cen-ts-16516.pdf (accessed: 25.01.2020).

Plesser, T. (2012). *Emisjoner fra byggevarer: Måling i laboratorium og resultater*. Byggforskserien. Oslo: SINTEF Byggforsk.

Pohleven, J., Burnard, M. & Kutnar, A. (2019). Volatile organic compounds emitted from untreated and thermally modified wood - a review. *Wood and Fiber Science*, 51 (3): 231-254. doi: 10.22382/wfs-2019-023.

Prussin, A. J. & Marr, L. C. (2015). Sources of airborne microorganisms in the built environment. *Microbiome*, 3 (1): 78-88. doi: 10.1186/s40168-015-0144-z.

Risholm-Sundman, M., Lundgren, M., Vestin, E. & Herder, P. (1998). Emissions of acetic acid and other volatile organic compounds from different species of solid wood. *Holz als Roh- und Werkstoff*, 56 (2): 125-129. doi: 10.1007/s001070050282.

Salem, M. Z. M. & Böhm, M. (2013). Understanding of Formaldehyde Emissions from Solid Wood: An Overview. *BioResources*, 8 (3): 4775-4790. doi: 10.15376/biores.8.3.4775-4790.

Salo, L. *The Effects of Coatings on the Indoor Air Emissions of Wood Board. Master Thesis. Helsinki: Aalto University*. Avaliable at:

https://aaltodoc.aalto.fi/bitstream/handle/123456789/29368/master_Salo_Laura_2017.pdf?sequence=1&isAllowed=y (accessed at 30.01.2020).

Salthammer, T. (2016). Very volatile organic compounds: an understudied class of indoor air pollutants. *Indoor Air*, 26 (1): 25-38. doi: 10.1111/ina.12173.

Schweizer, C., Edwards, R. D., Bayer-Oglesby, L., Gauderman, W. J., Ilacqua, V., Juhani Jantunen, M., Lai, H. K., Nieuwenhuijsen, M. & Künzli, N. (2007). Indoor time–microenvironment–activity patterns in seven regions of Europe. *Journal of Exposure Science & Environmental Epidemiology*, 17 (2): 170-181. doi: 10.1038/sj.jes.7500490.

Skoog, D. A., West, D. M., Holler, J. F. & Crouch, S. R. (2013). *Fundamentals of Analytical Chemistry: Ninth Edition*. Belmont: Brooks/Cole, Cengage Learning.

Skulberg, K. R., Nyrud, A. Q., Goffeng, L. O. & Wisthaler , A. (2019). Health and Exposure to VOCs From Pinewood in Indoor Environments. *Frontiers in Built Environment*, 5 (107): 1-11. doi: 10.3389/fbuil.2019.00107.

Spengler, J. D., McCarthy, J. F. & Samet, J. M. (2001). *Indoor Air Quality Handbook*. New York: McGraw-Hill Education.

Stratev, D., Fürhapper, C., Niedermayer, S., Habla, E., Nohava, M. & Weigl, M. (2016). From model rooms towards a modelled indoor environment. *International Wood Products Journal*, 7 (4): 195-201. doi: 10.1080/20426445.2016.1222254.

Tsai, W.-T. (2019). An overview of health hazards of volatile organic compounds regulated as indoor air pollutants. *Reviews on Environmental Health*, 34 (1): 81-89. doi: 10.1515/reveh-2018-0046.

Tsakas, P. M., Siskos, P. A. & Siskos, P. (2011). Indoor Air Pollutants and the Impact on Human Health. In Mazzeo, N. (ed.) *Chemistry, Emission Control*. Athens: IntechOpen.

United States Environmental Protection Agency (EPA). (2020). *Technical Overview of Volatile Organic Compounds*. Available at: https://www.epa.gov/indoor-air-quality-iaq/technical-overview-volatile-organic-compounds (accessed: 27.02.2020).

Wilke, O. & Jann, O. (eds). (2018). *Comparison of Formaldehyde Concentrations in Emission Test Chambers Using EN 717-1 and EN 16516*. Philadelphia: Indoor Air.

Wolkoff, P. (1998). Impact of air velocity, temperature, humidity, and air on long-term VOC emissions from building products. *Atmospheric Environment*, 32 (14): 2659–2668. doi: 10.1016/S1352-2310(97)00402-0.

Wolkoff, P. (2020). Indoor air chemistry: Terpene reaction products and airway effects. *International journal of hygiene and environmental health*, 225: 1-8. doi: 10.1016/j.ijheh.2019.113439.

Wolkoff, P. (2012). Indoor air pollutants in office environments: Assessment of comfort, health, and performance. *International Journal of Hygiene and Environmental Health*, 216 (4): 371-394. doi: 10.1016/j.ijheh.2012.08.001.

Wolpert, B. (2012). *Emission and abundance of biogenic volatile organic compounds in wind-throw areas of upland spruce forests in Bavaria*. Doctoral dissertation. Munich: Technische Universität München. Available at: https://pdfs.semanticscholar.org/cec3/284b4e5cee9c25050091a037cb7403f38ef0.pdf (accessed: 24.04.2020).

Woolfenden, E. (2009). Standarized methods for testing emissions of organic vapours from building products to indoor air. In Salthammer, T. & Uhde, E. (eds) *Organic indoor air pollutants 2nd edition*, pp. 119-144. Weinheim: WILEY-VCH Verlag GmbH & Co.

World Health Organization (WHO). (1989). Indoor air quality: Organic pollutants. *Environmental Technology Letters*, 10 (9): 855-858. doi: 10.1080/09593338909384805.

Zhou, S., Liu, H., Ding, Y. & Wu, Y. (2019). The effects of temperature and humidity on the VOC emission rate from dry building materials. *IOP Conference Series: Materials Science and Engineering*, 609: 1-6. doi: 10.1088/1757-899x/609/4/042001.

Appendix

Appendix A. Background emissions

Table A1: Apparent recovery and RSD [%] for 50pg, 500pg and 5 ng

Analyte	Recovery ± RSD [%] 50 pg	Recovery ± RSD [%] 500 pg	Recovery ± RSD [%] 5 ng
Hexanal	66,1±22,98	103,5 ± 12,7	
α-pinene		98,9 ± 25,9	96,6 ± 20,0
β-myrcene		971,7 ± 17,7	
β-pinene		141,8 ± 21,2	75,5 ± 12,0
3-carene			68,3 ± 18,1
camphene		82,9 ± 25,0	

Table A2: Calibration parameters for untreated wood samples

Analyte	Linear range [pg]	R^2	Origin treatment	Weight	a	b
Hexanal	0,01-3	0.992	force	none	0.005622	0
α-pinene	0,5-1000	0.984	force	none	1.31×10^{-4}	0
β-myrcene	0,5-1000	0.994	ignore	none	3.53×10^{-7}	0.001662
β-pinene	0,2-200	0.99	force	none	7.81×10^{-5}	0
3-carene	0,5-1000	0.979	force	none	1.97×10^{-6}	0
camphene	1-1000	0.992	force	none	2.98×10^{-5}	0

Table A3: Calibration parameters for wood stain samples

Analyte	Linear range [pg]	R^2	Origin treatment	Weight	a	b
Hexanal	0,01-3	0.992	force	none	0.005622	0
α-pinene	0,5-1000	0.984	force	none	1.31×10^{-4}	0
β-myrcene	0,5-1000	0.994	ignore	none	2.07×10^{-6}	- 0.1579
β-pinene	0,2-200	0.99	force	none	7.81×10^{-5}	0
3-carene	0,5-1000	0.979	force	none	1.97×10^{-6}	0
camphene	1-1000	0.992	force	none	2.98×10^{-5}	0

Appendix B. Temperature and humidity parameters for the samples

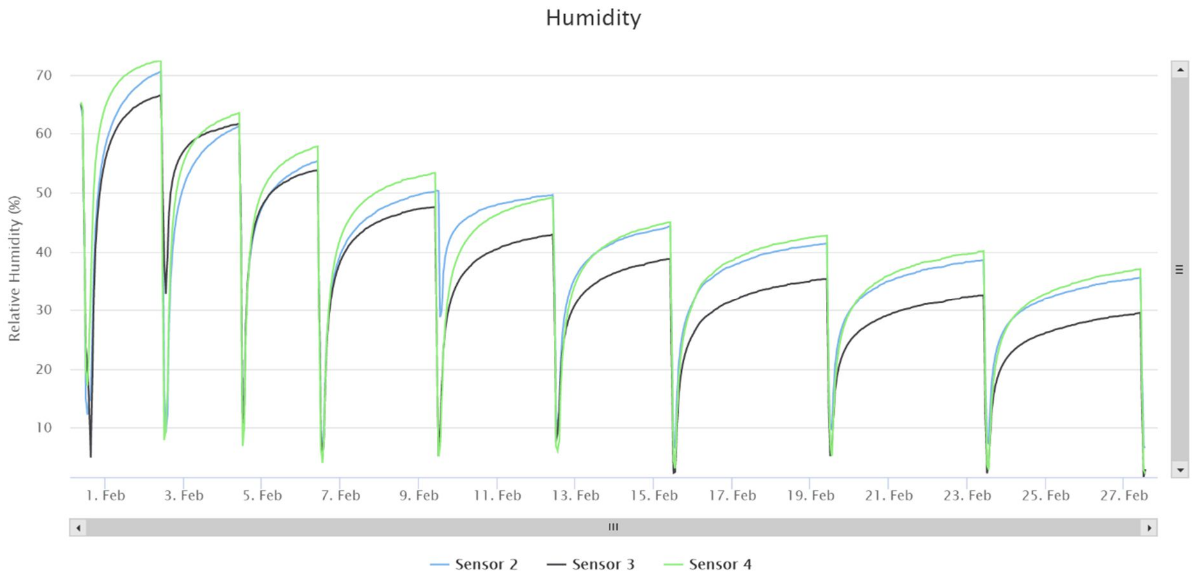

Figure B1: RH during the sampling period for untreated interior wood panels. Each sensor number corresponds to the sample number.

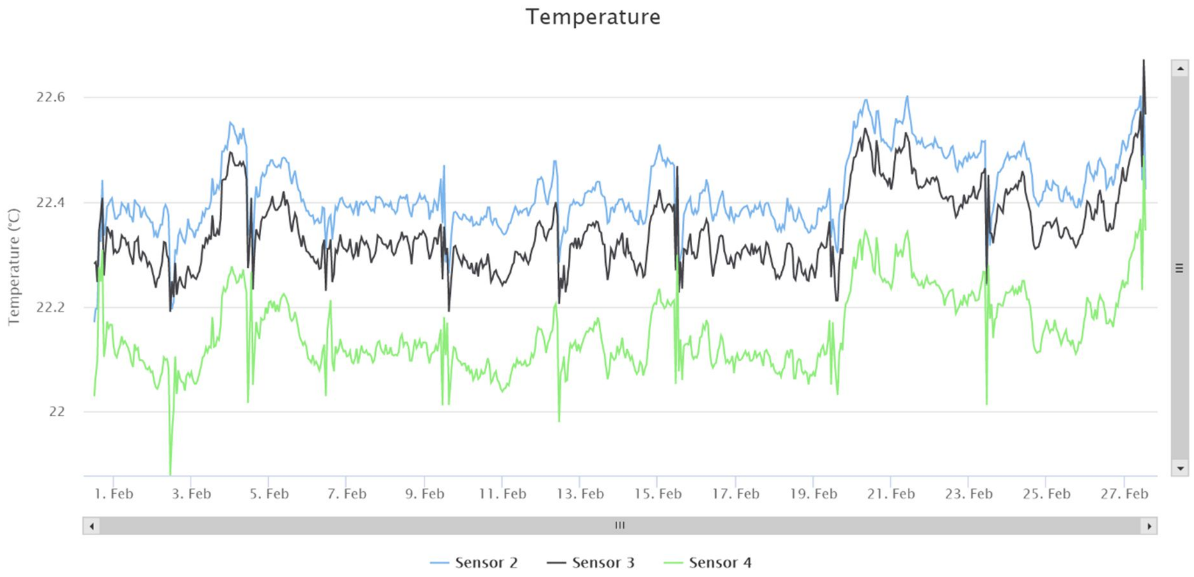

Figure B2: Temperature (˚C) during the sampling period for untreated wood panels. Each sensor number corresponds to the sample number.

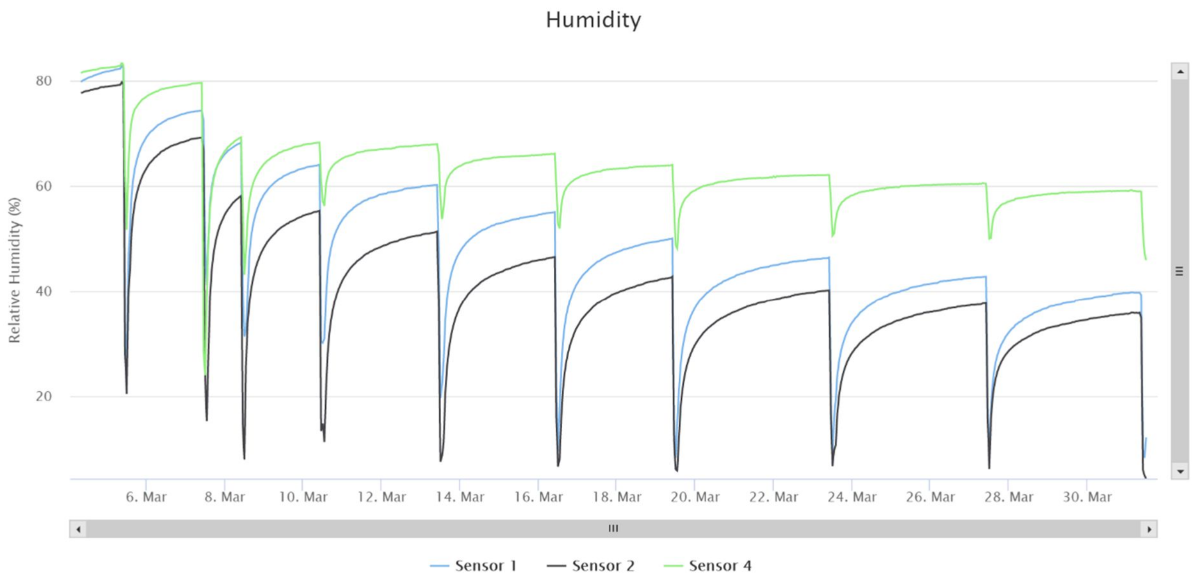

Figure B3: RH during the sampling period for the wood stain samples. Sensor 1 corresponds to sample WS1, sensor 2 corresponds to sample WS2 and sensor 4 corresponds to sample WS3.

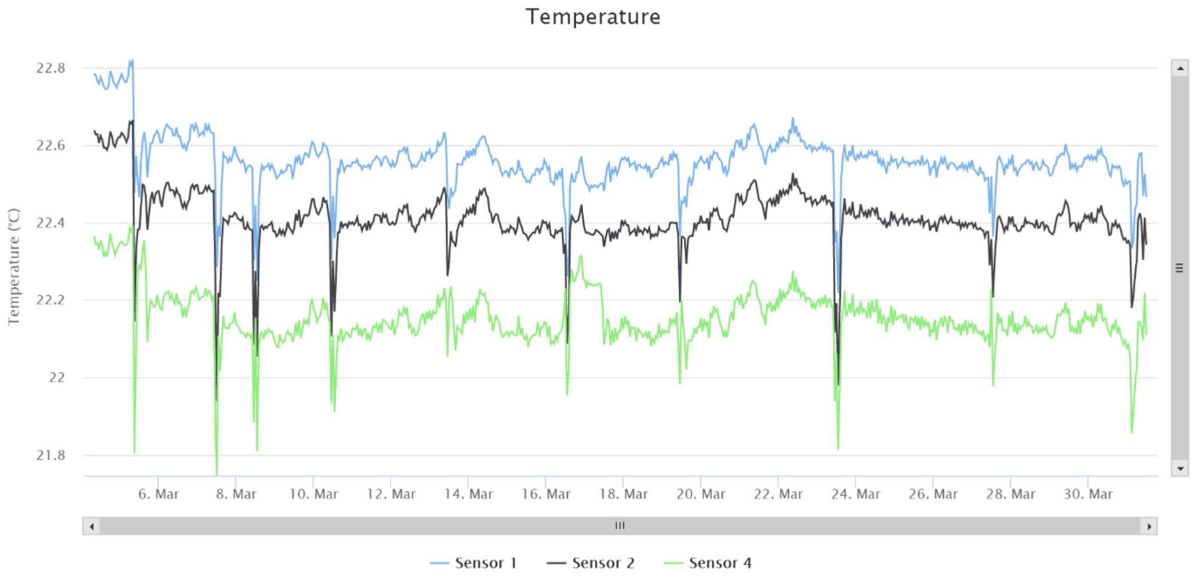

Figure B4: Temperature (˚C) during the sampling period for the wood stain samples. Sensor 1 corresponds to sample WS1, sensor 2 corresponds to sample WS2 and sensor 4 corresponds to sample WS3.

Appendix C: Experimental information

Table C1: Sampling information for the untreated interior wood panel samples

Date	Day	Time	Sampling volume
31.01.2020	1	10:30-12:10	10 000 mL
02.02.2020	3	10:30-12:10	10 000 mL
04.02.2020	5	10:30-12:10	10 000 mL
06.02.2020	7	10:15-11:55	10 000 mL
09.02.2020	10	10:15-11:55	10 000 mL
12.02.2020	13	10:15-11:55	10 000 mL
15.02.2020	16	10:05-11:45	10 000 mL
19.02.2020	20	10:05-11:45	10 000 mL
23.02.2020	24	10:00-11:40	10 000 mL
27.02.2020	28	10:00-11:40	10 000 mL

Table C2: Sampling information for the wood stain samples

Date	Day	Time	Sampling volume
04.03.2020	1	10:30-12:10	10 000 mL
06.03.2020	3	10:30-12:10	10 000 mL
08.03.2020	5	10:30-12:10	10 000 mL
10.03.2020	7	10:15-11:55	10 000 mL
13.03.2020	10	10:15-11:55	10 000 mL
16.03.2020	13	10:15-11:55	10 000 mL
19.03.2020	16	10:05-11:45	10 000 mL
23.03.2020	20	10:05-11:45	10 000 mL
27.03.2020	24	10:00-11:40	10 000 mL
31.03.2020	28	10:00-11:40	10 000 mL

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