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Volatile organic compounds emitted from Scots pine and Norway spruce wood

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Abstract

Volatile organic compound (VOC) emissions from Scots pine and Norway spruce wood were evaluated in green and kiln-dried conditions using Micro-Chamber (μ-CTE™) air-sampling, thermal desorption, and GC–MS. A total of 411 specimens were tested to evaluate sampling distributions, effect of kiln drying, and the chemical composition of VOC emissions in detail. There was no significant difference in emissions between spruce heartwood and sapwood. The median total VOC emission rate from spruce was 93% lower than that from pine heartwood in green condition, whereas in the kiln-dried state, the emission was 98% lower. Compared with pine sapwood, the median emissions from spruce were 85% and 94% lower in green and kiln-dried conditions, respectively. Pine sapwood emitted 55% and 79% less VOC than heartwood in green and kiln-dried conditions. Except for pine sapwood, all groups exhibited positively skewed distributions. The skewness was particularly noticeable in the green pine heartwood sample, where the top 10% of VOC scores increased the mean VOC emission by 67%. Kiln drying had a substantial impact on VOC emissions. The median emission reduction due to drying amounted to 93%, 79%, and 80% for spruce, pine heartwood, and pine sapwood, respectively. A strong positive linear relationship was observed between the emissions in the green condition and the emission reduction caused by drying. Except for 3-carene, the emission profiles were similar among the tested groups.

1 Introduction

Volatile organic compounds (VOC) comprise a large group of several hundred diverse compounds present in atmospheric air, primarily in gaseous form (Król et al. 2010). The World Health Organization (WHO) differentiates between very volatile organic compounds (VVOC), volatile organic compounds (VOC), and semi-volatile organic compounds (SVOC), based on rather vaguely defined boiling point boundaries (Salthammer and Bahadir 2009; Roffael et al. 2015; Salthammer 2016).

In the past few years, exposure to VOC has gained more attention in the building sector because of a higher health consciousness of residents and administrative authorities. Therefore, threshold values and enforceable standards for indoor VOC emissions have been recommended or set in different countries. The number of VOC-related scientific publications has increased in the past few decades, also indicating the growing concern in science and in practical building regulations (Zhang et al. 2010; Höllbacher 2016; Birmili et al. 2018; Cheng et al. 2019).

Human activities (Höllbacher et al. 2017) and outdoor air entering the building (Jia et al. 2005; Salthammer and Bahadir 2009; Massolo et al. 2010; Villanueva et al. 2018) may contaminate indoor air in a rather discontinuous manner. However, internal sources such as building products, furniture, and textiles are major long-lasting sources of VOC emissions indoors (Salthammer and Bahadir 2009; Brown et al. 2013; Que et al. 2013; Chin et al. 2014; Campagnolo et al. 2017).

Wood is a material widely used for the manufacturing of building products. Softwoods, especially wood species with high resin content, generally emit considerably more VOC than hardwoods (Risholm-Sundman et al. 1998; Englund

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1999; Peters et al. 2008). Monoterpenes, which represent a volatile component of canal resin, are the major VOC found in softwoods. Monoterpenes have a vital function in the defense system of a living tree. Besides being toxic for some insects and microbial agents (Fäldt et al. 2006; Eberhardt et al. 2009), their primary function is to provide a suitable gum flow of the canal resin by lowering its viscosity and to eventually evaporate, leaving the wound protected with an oleophilic material (Back 2000). Alongside terpenes, a wide range of volatile terpene derivatives—terpenoids—and secondary emission products such as aldehydes and carboxylic acids are found in softwoods. However, these substances are present in much lower amounts than terpenes (Back 2000). Uhde and Salthammer (2007) explained and schematized the formation of primary and secondary emissions, which corresponds well to those of wood and wood-based products.

In a recent review, Pohleven et al. (2019) provided a broad overview of the most critical aspects of VOC emissions from untreated and thermally modified wood. Moreover, their study indicated the scarcity of publications dealing with VOC emissions from untreated wood. In addition, as several factors influence VOC emissions from wood, it is difficult to grasp the representative values and compare the results between different studies. Different testing equipment and processing parameters yield different emission scores. The emission rate and chemical composition of VOC vary significantly among different wood species and different (wood-based) products. Moreover, Back and Ekman (2000) pointed out the same issue with high variability in combination with small sample sizes. This issue pervades most of the recently published studies regarding VOC emissions from wood as well. The number of samples per tested group has ranged in most studies from 1 to approximately 10. With such a low sample size, only general patterns can be identified. However, to be able to reduce the VOC emissions from wood products, much more detailed information is needed.

The present study was conducted to address the above-mentioned issues. Two softwood species of major industrial importance in Europe—Scots pine (*Pinus sylvestris* L.) and Norway spruce (*Picea abies* (L.) Karst.)—were evaluated in terms of VOC emissions. The emission rate distributions—their shape, spread, and central tendency—were investigated and compared in detail for the first time. The aim was to quantify differences between tested groups more accurately by considering the variation in VOC-emission rates within each tested group and to provide additional insights by closely describing their VOC-emission rate distributions. The effect of kiln drying on VOC emissions was also evaluated. The findings from this analysis should help evaluate the potential of wood drying to further reduce the VOC emissions of wood products. Eventually, the chemical composition of the collected VOC samples, which is of crucial importance for a toxicological evaluation, was

investigated in detail and compared between the tested groups. A micro-chamber air-sampling method (μ-CTE™, Markes Int.; Schripp et al. 2007; Salthammer and Bahadir 2009) enabled considerably faster VOC sampling than the traditional emission chambers. However, when investigating wood samples, micro-chamber sampling generally returns higher emission values than the standard emission test chamber method described in EN ISO 16000-9 (2008) and EN 16516 (2018). This is due to different sampling conditions such as the loading factor, air exchange rate, and specimen shape (Stratev 2018). Consequently, a direct comparison with the standard test chamber method on an absolute scale (i.e., area-specific emission rate) is limited. Nevertheless, comparisons on a relative scale are possible and should yield valuable information.

2 Materials and methods

2.1 Materials

The test material, boards of Norway spruce (*P. abies*) and Scots pine (*P. sylvestris*), was collected from various partner sawmills. The round timber was harvested from different forest sites in Austria, Slovenia, and Bavaria in Germany. Additional Scots pine boards were supplied from North Karelia in Finland and Boksitogorsky district in Russia to assess whether there are significant differences in VOC emissions between the growing sites in Central, Eastern, and Northern Europe. After log-processing, boards of 4–5 m in length were separated into central boards and side boards, each corresponding to a different log. Boards for further preparation were selected randomly from each group. Two 0.5 m long pieces were cut from each selected board, the first one in green condition, immediately after the selection was done, and the second one after kiln drying. Drying was performed using conventional drying schedules with temperatures of up to 70 °C. Immediately after cutting, the boards were sealed in aluminum bags to prevent the loss of volatiles and sent successively within 6 months to the testing laboratory. Boards from packages damaged during transportation were not included in the analysis.

At the testing laboratory, the boards were stored at room temperature according to EN ISO 16000-11 (2006). Four test cubes with dimensions of $20 \times 20 \times 20$ mm^3 were cut out from each board. One part of spruce specimens in green condition was separated into sapwood and heartwood groups to test the difference in VOC emissions from spruce heartwood and sapwood in the green condition, which was part of this study. The differentiation between heartwood and sapwood was also done for all pine specimens. Not all specimens could be evaluated due to technical reasons during VOC sampling or the subsequent thermal desorption and analysis.

On average 3.57 cubes per board (median = 4) were tested. A total of 237 spruce specimens and 174 pine specimens were tested.

2.2 VOC sampling, extraction, and analysis

The Micro-Chamber/Thermal Extractor™ (Markes Int., Schripp et al. 2007) is a dynamic headspace unit used to collect VOC and SVOC emitted from products and materials. It is a multiple chamber system. Six such systems, each having six chambers, were used in this study. This allowed to perform large-sample size experiments and more reliable estimation of central tendency and spread within each group tested.

The test cubes were weighed and placed into the microchambers immediately after preparation. VOC sampling was conducted after 1 h of equilibration period. During these two steps, purified air was passed through each chamber at a flow rate of 50 ± 2 ml min^{-1} and a temperature of 23 °C. VOC were collected on Tenax® TA sorbent packed in stainless steel tubes (EN ISO 16000-6 2012, Markes Int.). The tubes were directly attached to the exhaust point of each chamber. One tube was attached to an empty chamber of each extractor unit during a sampling sequence to take a blank measurement. Therefore, a fully loaded sampling sequence comprised 30 VOC samples and six blank measurements. Prior to VOC sampling, 0.37 µg toluene-d8 dissolved in methanol was added to each sampling tube as an internal standard.

An automated thermal desorption system (TD-100, Markes Int.) was used for sample extraction, focusing on analytes and their introduction into a gas chromatography (GC) column. A cold trap packed with Tenax® TA (U-T9TNX-2S, Markes Int.) was used for analyte concentration inside the TD unit. The thermal desorption parameters used in this study were set according to EN ISO 16000-6 2012. Primary and secondary splits were set according to the expected concentration of the most abundant compound.

A gas chromatography-mass spectrometry (GC–MS) system was used for the identification and quantitative analysis of the emitted substances. The analytes were introduced in a helium stream from the thermal desorption unit into a GC column. A 100% dimethylpolysiloxane column (HP-PONA, 50 m × 0.2 mm × 0.5 µm; Agilent Technologies) was used for analyte separation. The MS detector was set to acquire data over an m/z range of 12-400. VOC were identified by comparing the collected spectrum with data from commercial mass-spectral libraries (John Wiley & Sons). Prior to VOC sampling, an internal standard was added to compensate for any variability due to the GC injection (Slack et al. 2003).

Quantification of α-pinene, β-pinene, 3-carene, and limonene was performed by including a response factor obtained from calibration standards. Other substances were quantified as toluene-d8 equivalents (TE). The total volatile compound (TVOC) value included all substances occurring within the GC retention time window ranging from n-hexane to n-hexadecane (ECA 1997; Massold et al. 2005; Salthammer and Bahadir 2009). Short-chain aldehydes such as formaldehyde and acetaldehyde were, therefore, not included in the analysis.

2.3 Data analysis

Data analysis was conducted in R programing language using available packages for inferential statistics. Data visualization was performed in Python programing language using the library Matplotlib.

A Welch two-sample t-test was implemented to evaluate the difference in TVOC emission rates from spruce heartwood and sapwood in the green condition (see Sect. 3.1). A Welch correction was used to account for the violated assumption of homogeneity of variances.

Several statistical methods were applied to evaluate the TVOC-emission rates from Norway spruce, Scots pine heartwood, and sapwood in green and kiln-dried conditions (see Sect. 3.1). The Welch-ANOVA and Games-Howell post hoc tests were used to evaluate the between-group differences. The tests were conducted on log-transformed data because the original data were highly skewed and approximately log-normal. A Student's t-test was applied to the log-transformed data to estimate the 95% confidence intervals (CIs) for the mean values on the log scale. The *mean* and its CI on the log scale correspond to the *median* and its CI after back-transforming the data to its original scale (Bland and Altman 1996a; Olsson 2005). Accordingly, the *difference of means* between the two groups yielded by the Games-Howell test on the log scale becomes the *ratio of medians* after back-transformation (Bland and Altman 1996b; Lee 2020). This applies only if the original data are log-normal, as it was in this study. Bootstrapping was used to estimate the CIs for the *means* on the original scale. More precisely, the bootstrap t CI method explained in detail by Chihara and Hesterberg (2018) was used. This method yields more accurate confidence intervals for strongly skewed data than symmetric t intervals or bootstrap percentile CIs (Chihara and Hesterberg 2018). The number of resamples was 50,000.

Mixed ANOVA and pairwise t-tests were applied to estimate the reduction in TVOC emissions due to kiln drying (see Sect. 3.2). Pairwise t-tests were used to estimate the CIs as well. Again, the tests were conducted on the log-transformed data and then back-transformed to the original scale. In addition, a simple linear regression model was fit to estimate the effect of the initial emission rate (in the green condition) on the reduction in TVOC-emission rates due to drying.

The same bootstrapping method used for TVOC-emission was applied to estimate CIs for the *means* of the individual compounds (see Sect. 3.3).

3 Results and discussion

3.1 TVOC emitted from spruce and pine heartwood and sapwood

One subgoal of this study was to evaluate the difference in TVOC emission rates from spruce heartwood and sapwood in the green condition. A strong overlap was observed in distributions between the two groups, which is shown in Fig. 1. Consequently, the Welch two-sample t-test revealed no significant difference between the groups, $t(68.83) = 1.2$, $p = 0.23$. As the original data were right-skewed, the t-test was conducted on log-transformed data.

This result was expected because spruce heartwood and sapwood contain similar amounts of nonvolatiles in their canal resin (Ekman and Holmbom 2000). Moreover, little or no additional resin is formed in the heartwood transition zone of spruce (Back 2000). Nisula (2018) reported that the resin acid contents in Norway spruce sapwood and heartwood ranged from 0.05 to 0.2% and from 0.06 to 0.2%, respectively, on a dry wood basis. In contrast, Czajka et al. (2020) reported 2.5 times higher TVOC emissions from spruce sapwood. However, no post-hoc results were presented to show whether the difference is statistically significant even though highly overlapping CIs were reported and

a small sample size ($n = 3$) was used in the study. Steckel et al. (2010) also observed higher emissions from a single spruce sapwood sample. Nevertheless, the micrographs of the tested specimens confirmed that the higher emission was caused by the presence of stress-induced resin ducts in the sapwood specimen.

The TVOC emission rates from Norway spruce, and Scots pine heartwood and sapwood, in both the green and kiln-dried conditions, were also evaluated. For easier comparison, Fig. 2 shows the TVOC emission rate distributions and individual measurements on a $\log_{10}$ scale. The Welch-ANOVA and Games-Howell post hoc tests revealed highly significant differences between all groups ($p < 0.001$), in both the green and kiln-dried conditions (Table 1). Pairwise group comparisons, represented as ratios of the group *medians* (see Sect. 2.3), including their CIs, are listed in Table 1. The ratios of the group *means* are also presented. However, these values should serve only as a descriptive metric as they were not part of the statistical test (see Sect. 2.3). The 95% CIs for the *mean* and *median* of each observed group are listed in Table 2.

In the green condition, the median TVOC emission rate of spruce amounted to 7% (95% CI [4.6%, 10.4%]) and 15% (95% CI [11.9%, 19.5%]) of that from pine heartwood and pine sapwood, respectively. The difference was even more pronounced in the kiln-dried condition. Spruce emitted 1.7% (95% CI [1.0%, 2.8%]) and 5.7% (95% CI [3.9%, 8.1%]) of that from pine heartwood and sapwood. (Table 1). Large differences were anticipated between pine and spruce TVOC emission rates, as their resin canal systems and resin amounts differ substantially. In general, pine wood has the most well-developed resin canal system (Back 2000). Their epithelial cells, which are responsible for the resin production, are thin-walled—they easily expand, and the resin is rapidly pressed out. In contrast, the thick-walled and lignified epithelial cells in spruce wood scarcely expand, and their resin canals are rarely entirely filled with resin (Back 2000). Nisula (2018) reported up to 37 times higher nonvolatile resin acid content in pine heartwood than in spruce wood.

Pine sapwood emitted 45% (95% CI [30.8%, 67.0%]) and 30% (95% CI [17.9%, 51.0%]) of that from pine heartwood in the green and kiln-dried conditions, respectively (Table 1). In pine sapwood, canal resin can be found solely in the resin canals, which is not the case in heartwood (Back 2000). Before lignification, the epithelial cells in the sapwood-heartwood transition zone produce additional resin. The resin fills the canals and is pressed out in the surrounding tissue (Back 2000). Nisula (2018) observed 0.2% resin acids in Scots pine sapwood on a dry weight basis, compared to 1.6–2.2% in the heartwood.

Emission patterns similar to those found in this study were reported in various publications. However, in most
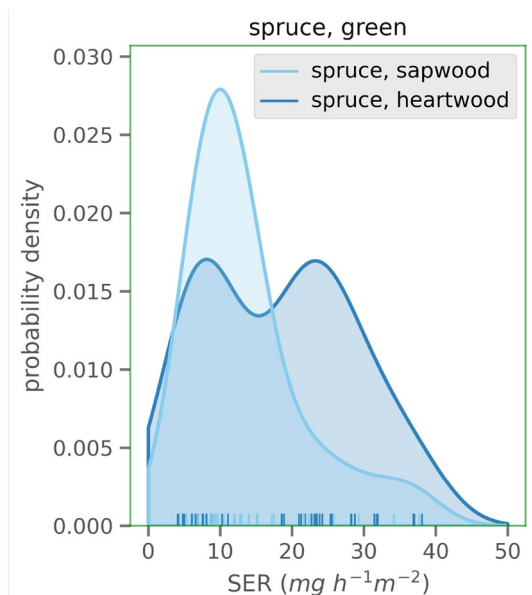
Fig. 1 TVOC-emission rate distributions for spruce heartwood and sapwood in the green condition; SER—Area-specific emission rate
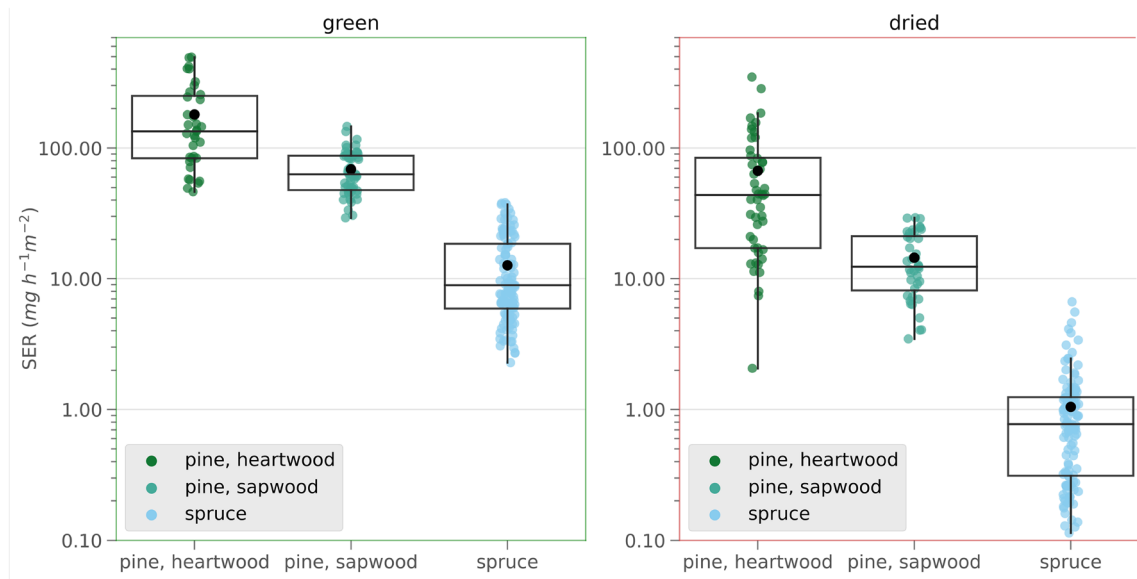
Fig. 2 TVOC-emission rates for pine heartwood, pine sapwood and spruce. (Y-axis $\log_{10}$); black dots represent the mean values; other dots represent individual measurements

Table 1 Between-group comparisons: Norway spruce, Scots pine heartwood, and sapwood; Welch-ANOVA and Games-Howell post hoc test

Welch-ANOVA	F	df	denom df	p-value
	489.59	5	139.81	<0.001

	Ratio (mean)	Games-Howell post-hoc					
		Ratio (median)	CI.lo	CI.hi	t	df	p
Green							
Pine sapwood: pine heartwood	0.382	0.454	0.308	0.670	6.0	47.8	<0.001
Spruce: pine heartwood	0.070	0.069	0.046	0.104	19.5	56.5	<0.001
Spruce: pine sapwood	0.183	0.153	0.119	0.195	22.1	159.7	<0.001
Kiln-dried							
Pine sapwood: pine heartwood	0.215	0.302	0.179	0.510	6.7	75.8	<0.001
Spruce: pine heartwood	0.016	0.017	0.010	0.028	23.4	76.6	<0.001
Spruce: pine sapwood	0.072	0.057	0.039	0.081	22.9	104.7	<0.001

previous studies, the sample sizes were too small, considering the very high *within-group* differences to quantify the *between-group* differences in a reliable manner. Czajka et al. (2020) recorded much higher VOC emissions from pine heartwood than from spruce heartwood in the green condition, whereas Risholm-Sundman et al. (1998), Englund (1999) and Larsen et al. (2000) found considerably higher emissions from pine than from spruce in dried samples. Higher emissions from pine heartwood in contrast to pine sapwood were observed in several studies as well. Czajka et al. (2020) reported higher emissions from pine heartwood in the green condition, and Dix et al. (2004), Roffael (2006) and Roffael et al. (2015) recorded a similar pattern from pine heartwood strands. A similar

tendency was reported by Steckel (2010) and Larsen et al. (2000) for dried samples.

A strong disagreement compared to the present findings was reported by Czajka et al. (2020), who observed slightly higher emissions from spruce sapwood than from pine sapwood in the green condition. The present analysis indicated that although there was a slight overlap between these two groups (Fig. 2), the probability for such observations to occur is relatively low, even with small sample sizes.

A large sample size allowed us to evaluate the shape and spread of the emission rate distribution within each group. All groups, especially pine heartwood, manifested strongly right-skewed distributions (Fig. 3). This is critical for data analysis and interpretation and, to the best of the authors'

Table 2 TVOC-emission rates for pine heartwood, pine sapwood and spruce, including 95% confidence intervals; % confidence interval deviation from the mean and median in %; n – sample size

		95%-Confidence intervals for the means								95%-Confidence intervals for the medians						
		SER (mg m^{-2} h^{-1})			%					SER (mg m^{-2} h^{-1})			%		
	n	mean	CI.lo	CI.hi	mean	CI.lo	CI.hi		n	median	CI.lo	CI.hi	Median	CI.lo	CI.hi
Green								**Green**							
Pine heartwood	35	180.3	138.2	238.2	100	76.7	132.1	Pine heart-wood	35	134.2	110.8	180.1	100	82.6	134.2
Pine sapwood	51	68.9	61.7	77.2	100	89.6	112.0	Pine sapwood	51	63.2	57.6	71.4	100	91.1	113.0
Spruce	118	12.6	10.9	14.7	100	86.5	116.7	Spruce	118	8.9	8.6	11.2	100	96.6	125.8
Kiln-dried								**Kiln-dried**							
Pine heartwood	48	67.1	48.7	105.2	100	72.6	156.8	Pine heart-wood	48	43.7	30	55.6	100	68.6	127.2
Pine sapwood	40	14.4	12	18	100	83.3	125.0	Pine sapwood	40	12.4	10.2	14.9	100	82.3	120.2
Spruce	119	1	0.9	1.3	100	90.0	130.0	Spruce	119	0.8	0.6	0.8	100	75.0	100.0
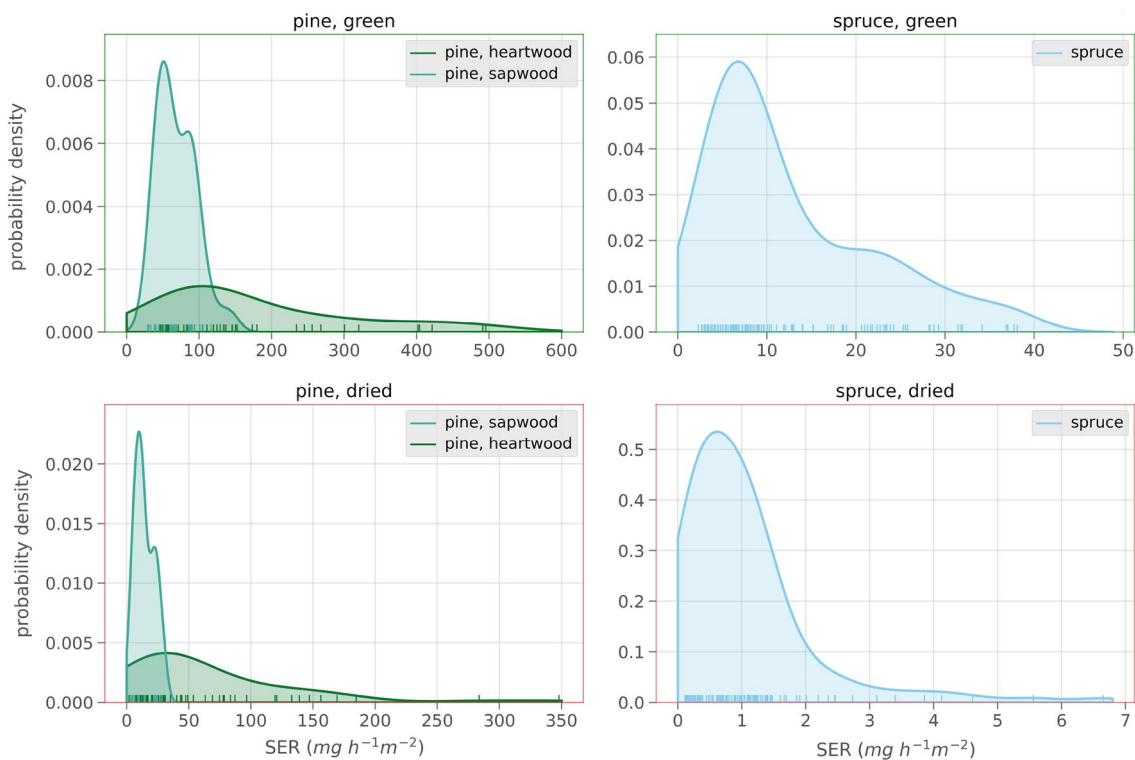
Fig. 3 TVOC-emission rate distributions for Norway spruce and Scots pine sapwood and heartwood

knowledge, was not considered in previous studies on a similar topic. Positively skewed data resulted in a considerable shift in mean values to the right. Compared to the median, the shift ranged from 9% for the kiln-dried pine sapwood to even 53% for the kiln-dried pine heartwood. This implies that only the highest 10% of the pine heartwood sample increased the mean emission by almost 40%. In practice, sorting out such high resinous boards could

yield considerable reductions in VOC emissions of the final product. A very high *within-group* variability was observed as well, primarily in high-emitting groups (Fig. 3), indicating that small sample sizes could easily lead to biased estimations.

No statistically significant differences in TVOC emission rates were detected between the growing sites in Central, Eastern, and Northern Europe. However, the sample size

was probably too low after adding this additional factor in the analysis.

3.2 Effect of kiln drying on VOC emissions

The effect of kiln drying on VOC emissions was also evaluated. Figure 4 shows the emission rates from pine heartwood, pine sapwood, and spruce. Each line connects specimens (wood cubes) extracted from a single board—their mean emission rates—in the green condition and after kiln drying. The boxplots in Fig. 4 (right) depict the distributions of the absolute reductions in emission rates after kiln drying for each of the three observed groups. Although the relative emission reduction was higher for spruce wood (Fig. 4, left, Table 3), the absolute emission reduction was, as anticipated, considerably greater for the two pine groups.

As shown in Fig. 4 (left, middle), the emission reduction due to drying depends highly on the "starting" emissions from a specimen (emission in the green condition), which probably reflects its resin content in the green condition. These two variables exhibited a strong, positive, linear relationship in all the three observed groups (Fig. 5). A simple linear regression model yielded a good approximation of the emission reduction. Interestingly, although the intercepts differed, the three groups shared a very similar slope—the increase rate in emission reduction.

The median emission reduction due to kiln drying amounted to approximately 80% (95% CI [62.7%, 88%]) for Scots pine sapwood and heartwood, and approximately 93% (95% CI [89.5%, 94.9%]) for Norway spruce. The mixed ANOVA post hoc results are listed in Table 3.

Englund and Nussbaum (2000) observed the loss of terpene content in Scots pine and Norway spruce after drying at various temperatures. They found that pine heartwood and sapwood lost 25–50% and 20–40% of their original terpene content, respectively. However, spruce lost 10–50% of its terpene content. Thompson and Ingram (2006) reported somewhat closer results to the ones of this study here. They recorded terpene content losses of 40% and 47% after drying loblolly pine heartwood and sapwood, respectively.

The abovementioned two studies observed the absolute terpene amount driven off by drying (content loss). However, in the present study, the emission *rates* in the green and kiln-dried conditions were measured. Although they should be correlated, these two metrics do not necessarily need to be directly comparable. For instance, an increase in resin viscosity caused by terpene loss during drying could hinder the resin flow and lead to disproportionally lower emission rates from dried wood.
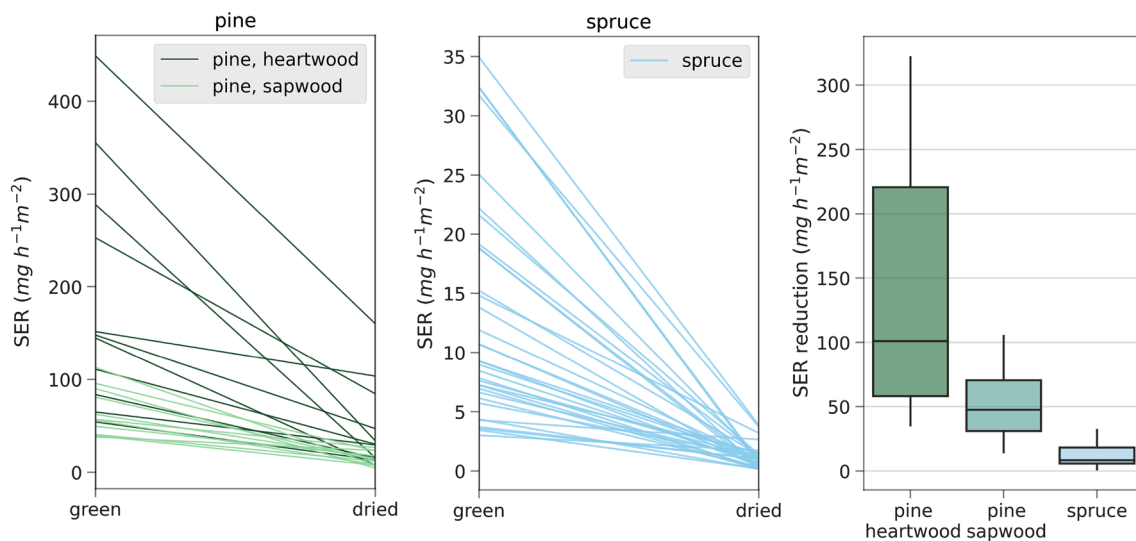
Fig. 4 (left, middle) Pairwise comparison of TVOC emission rates (SER) before and after kiln drying; (right) TVOC emission rate reduction due to drying

Table 3 Mixed ANOVA post-hoc results for medians; green vs. kiln-dried condition

	Ratio green:dried	CI.lo	CI.hi	n1	n2	p.adj
Spruce	13.70	9.54	19.67	34	34	<0.001
Pine heartwood	4.71	2.68	8.31	11	11	<0.001
Pine sapwood	4.89	2.94	8.14	11	11	<0.001
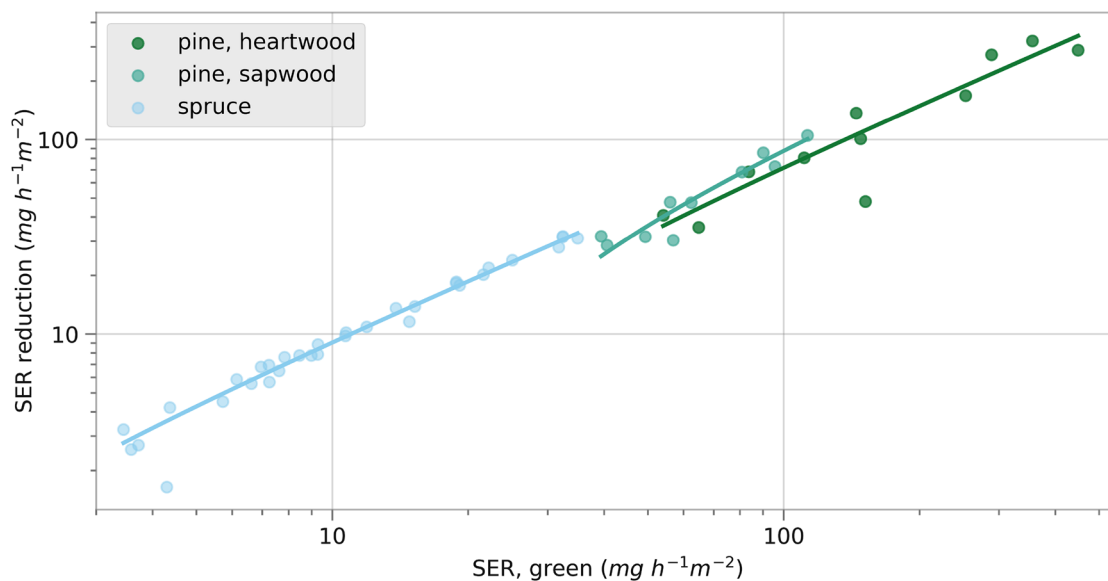
Fig. 5 Absolute TVOC-emission rate reduction as a function of TVOC-emission rate in the green condition; x- and y-axis $\log_{10}$

3.3 VOC emission profiles

Terpenes dominated the VOC emissions in pine samples, in both the green and kiln-dried conditions. The *mean* share of terpenes in all the identified VOC ranged from 93.7 to 97.1% in the green pine samples and from 83.8 to 96.3% in the kiln-dried pine sapwood. On the absolute scale, the terpene emission reflected the TVOC emission rates discussed in Sect. 3.1 (Table 4). Pine heartwood emitted much higher amounts of terpenes than pine softwood in both the green and kiln-dried conditions. Similar to the TVOC emission rate, there was a substantial reduction in the terpene emission rate due to drying. Compared with terpenes, terpenoid and aldehyde emissions were drastically lower. On average, they ranged from 1.7 to 4% and from 0.1 to 0.9%, respectively. The only exception was the kiln-dried pine sapwood, in which an increase in aldehyde emissions and a significant drop in terpene emissions increased the aldehyde share to 11%. However, even with this increase, the absolute aldehyde emissions were low compared with terpene emissions from other pine groups. Short-chain aldehydes such as formaldehyde and acetaldehyde were not included in the analysis. Table 4 shows detailed results, including the mean, median, confidence intervals, and quartiles for the *compound groups* and the most abundant *substances*.

Although there were large differences in emissions between pine categories on the absolute scale, the mean *emission profiles*, shown as dashed lines with 95% CIs in Fig. 6, were similar. The lines represent profiles with categorical variables on the x-axis as in parallel coordinate plots. They do not represent a process as in traditional line plots. The mean shares of the most abundant VOC with CIs are

presented in Fig. 7. The most abundant compound in each pine category was α-pinene, followed by β-pinene, 3-carene, and limonene and sabinene (Figs. 6 and 7). The unexpectedly low mean content of 3-carene in the kiln-dried pine heartwood was most probably due to the small sample size, considering the specific 3-carene distribution (discussed later), rather than a representative 3-carene mean value. Due to difficulties in separating limonene from sabinene during the GC analysis, individual quantification was not possible. Therefore, their emissions were merged and represented as a single value. In general, hexanal was the most abundant aldehyde. However, its concentrations were low compared to those of the most abundant terpenes. Although the emissions of individual terpenes decreased considerably after drying, the mean hexanal emissions increased, especially in pine sapwood. Englund (1999) also observed higher emissions of aldehydes in pine sapwood than in pine heartwood.

Although the relationships between compounds shown in Fig. 6 mostly reflected those shown in Fig. 7, this was not always the case. For instance, the mean 3-carene emissions were higher than β-pinene emissions for pine samples in the green condition, as shown in Fig. 6, whereas the opposite result can be observed in Fig. 7. This is because each observation was normalized (0–100%) before calculating the shares presented in Fig. 7. Hence, all samples were equally weighted. In contrast, Fig. 6 represents the mean *absolute* emissions of individual compounds (no normalization), which implies a more substantial impact of highly concentrated samples. Compared with Fig. 7, Fig. 6 allows for a more robust comparison of emission profiles between different groups as it is less sensitive to changes in the emissions of individual compounds. For example, Fig. 7 depicts quite

different terpene profiles for pine heartwood and sapwood in the dried condition. However, Fig. 6 reveals that these changes are primarily due to an unexpectedly low 3-carene emission in the heartwood group (discussed later) and somewhat higher hexanal emission in the softwood group.

The VOC emission profiles of other terpenes are, in fact, extremely similar between the two groups.

The distributions of the most important terpenes in the case of pine samples were right-skewed, which is indicated by the higher mean values than the medium values and the

Table 4 Absolute emission rates and shares for the compound groups and the most abundant substances including the mean and median values, confidence intervalsand quartiles

Pine (green)

heartwood

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**180.3**	140.1	233.5	**134.2**	83.8	250.5	**100.0**			**100.0**		
terpenes	**171.7**	133.7	224.3	**131.9**	78.7	231.1	**97.1**	95.5	97.9	**98.2**	95.9	99.0
terpenoids	**4.0**	2.0	10.7	**1.2**	0.6	3.0	**2.1**	1.4	3.5	**1.0**	0.5	2.9
aldehydes	**0.2**	0.1	0.3	**0.1**	0.0	0.2	**0.1**	0.1	0.2	**0.1**	0.0	0.2
α-pinene^d	102.9	80.4	137.3	75.6	45.6	128.1	59.6	55.3	64.4	56.3	50.3	70.1
β-pinene^d	19.8	12.9	28.6	6.2	2.7	35.3	12.6	8.6	17.2	4.6	1.7	21.6
3-carene^d	25.6	14.1	43.9	0.1	0.0	42.0	10.5	6.5	15.1	0.1	0.0	24.9
limonene+sabinene^d	14.6	11.1	18.6	11.3	4.7	21.9	8.5	7.2	10.4	8.1	5.6	9.8
hexanal	0.03	0.00	0.85	0.00	0.00	0.00	0.0	0.0	0.7	0.0	0.0	0.0
camphene	3.93	2.71	5.62	1.89	1.10	4.81	1.9	1.7	2.3	1.7	1.4	2.0
α-terpineol	1.22			0.54	0.00	1.07	0.8			0.5	0.0	1.2
α-terpinolene	1.98			0.73	0.28	3.34	1.0			0.7	0.2	1.3
cymene	1.08			0.14	0.10	1.06	0.5			0.2	0.1	0.9

sapwood

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**68.9**	61.9	77.1	**63.2**	47.7	87.6	**100.0**			**100.0**		
terpenes	**63.4**	56.1	71.7	**58.9**	40.2	83.8	**93.7**	90.0	95.8	**97.2**	93.9	99.0
terpenoids	**2.0**	1.3	3.0	**0.8**	0.2	2.3	**4.0**	2.5	6.4	**0.9**	0.3	4.2
aldehydes	**0.2**	0.1	0.3	**0.0**	0.0	0.2	**0.3**	0.2	0.6	**0.1**	0.0	0.3
α-pinene^d	37.4	32.8	42.2	35.4	22.5	50.3	53.9	50.1	57.6	55.6	45.5	60.1
β-pinene^d	8.6	6.1	12.1	2.9	0.9	13.2	13.2	9.5	17.2	5.0	1.4	28.4
3-carene^d	10.4	6.9	14.8	1.5	0.0	21.0	12.2	8.4	16.4	3.7	0.0	22.6
limonene+sabinene^d	3.5	3.0	4.1	3.1	2.0	4.8	5.7	4.8	6.8	4.5	3.2	8.2
hexanal	0.04	0.01	0.12	0.00	0.00	0.00	0.1	0.0	0.1	0.0	0.0	0.0
camphene	1.27	1.03	1.56	1.07	0.53	1.95	2.3	1.8	2.9	1.5	0.9	3.4
α-terpineol	0.67			0.00	0.00	0.82	1.3			0.0	0.0	1.5
α-terpinolene	0.90			0.44	0.27	1.30	1.3			0.6	0.3	2.2
cymene	0.37			0.23	0.07	0.60	0.6			0.3	0.1	0.9

Pine (kiln-dried)

heartwood

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**67.1**	49.8	95.2	**43.7**	17.2	84.4	**100.0**			**100.0**		
terpenes	**65.1**	48.0	93.0	**41.8**	16.4	84.3	**96.3**	95.3	97.2	**97.2**	93.8	98.9
terpenoids	**0.5**	0.4	0.7	**0.4**	0.1	0.7	**1.7**	1.1	2.7	**0.9**	0.2	1.6
aldehydes	**0.2**	0.1	0.4	**0.0**	0.0	0.1	**0.9**	0.4	1.9	**0.0**	0.0	0.3
α-pinene^d	47.3	34.7	65.2	26.6	11.3	67.4	67.3	64.2	70.8	64.9	57.0	76.9
β-pinene^d	8.5	4.8	19.9	2.6	1.2	8.9	13.2	10.1	16.3	14.2	1.9	22.5
3-carene^d	0.9	0.3	2.2	0.0	0.0	0.0	2.6	1.0	5.6	0.0	0.0	0.0
limonene+sabinene^d	5.8	3.9	8.8	2.3	1.0	6.3	7.3	6.1	8.9	5.7	4.5	9.0
hexanal	0.10	0.05	0.25	0.00	0.00	0.01	0.5	0.2	1.2	0.0	0.0	0.1
camphene	1.37	0.90	2.14	0.54	0.23	1.46	1.9	1.6	2.3	1.5	1.1	2.5
α-terpineol	0.11			0.00	0.00	0.00	0.6			0.0	0.0	0.0
α-terpinolene	0.53			0.32	0.10	0.62	0.7			0.8	0.4	1.1
cymene	0.16			0.04	0.00	0.15	0.3			0.1	0.0	0.4

sapwood

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**14.4**	12.1	17.0	**12.4**	8.1	21.2	**100.0**			**100.0**		
terpenes	**11.9**	9.8	14.4	**10.7**	6.0	17.4	**83.8**	78.1	87.8	**90.8**	82.4	93.2
terpenoids	**0.4**	0.3	0.6	**0.2**	0.1	0.4	**2.4**	1.7	3.3	**2.0**	0.9	2.5
aldehydes	**1.0**	0.7	1.3	**0.8**	0.2	1.4	**11.0**	7.5	15.8	**6.2**	2.1	13.7
α-pinene^d	8.2	6.7	9.8	7.7	3.8	12.4	54.7	51.0	58.3	53.2	46.7	65.5
β-pinene^d	1.2	0.7	2.0	0.2	0.1	1.8	8.0	5.1	12.1	2.5	0.7	15.3
3-carene^d	1.3	0.8	2.1	0.2	0.0	2.3	8.5	5.5	12.4	2.6	0.2	14.5
limonene+sabinene^d	0.63	0.47	0.82	0.43	0.15	1.10	4.1	3.3	5.1	3.8	1.6	5.8
hexanal	0.72	0.52	0.95	0.56	0.16	1.11	7.4	5.0	10.5	4.4	1.4	9.7
camphene	0.24	0.18	0.32	0.22	0.09	0.32	1.5	1.2	1.9	1.3	0.9	2.4
α-terpineol	0.18			0.00	0.00	0.14	1.0			0.0	0.0	1.0
α-terpinolene	0.11			0.03	0.00	0.18	0.7			0.3	0.0	1.2
cymene	0.07			0.05	0.00	0.10	0.6			0.3	0.0	1.2

Spruce

green

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**12.63**	11.06	14.46	**8.91**	5.92	18.54	**100.00**			**100.00**		
terpenes	**11.27**	9.88	12.86	**7.93**	5.31	16.53	**92.6**	91.4	93.6	**93.2**	89.4	97.5
terpenoids	**0.55**	0.40	0.76	**0.18**	0.08	0.53	**3.7**	3.1	4.4	**2.7**	0.7	5.6
aldehydes	**0.05**	0.04	0.09	**0.00**	0.00	0.00	**0.9**	0.6	1.5	**0.0**	0.0	0.4
α-pinene^d	6.14	5.39	7.04	4.47	2.74	8.43	50.6	48.3	52.9	50.7	41.4	60.0
β-pinene^d	2.02	1.65	2.49	1.20	0.40	2.88	17.1	14.8	19.4	14.2	5.3	29.2
3-carene^d	0.24	0.17	0.34	0.07	0.03	0.21	1.5	1.3	1.9	0.9	0.4	2.2
limonene+sabinene^d	2.15	1.74	2.72	1.13	0.75	2.39	15.2	13.7	16.9	13.6	9.6	18.3
hexanal	0.04	0.02	0.08	0.00	0.00	0.04	0.4	0.3	0.6	0.0	0.0	0.4
camphene	0.34	0.26	0.46	0.17	0.11	0.27	2.5	2.2	2.8	1.9	1.5	3.4
α-terpineol	0.25			0.08	0.00	0.30	1.5			0.9	0.0	2.7
α-terpinolene	0.20			0.04	0.00	0.09	1.0			0.5	0.0	1.2
cymene	0.21			0.12	0.07	0.19	1.5			1.3	0.8	2.0

kiln-dried

compounds	SER^a (mg m^{-2} h^{-1})						%^{b}					
	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e	mean	CI lo^e	CI hi^e	median	Q1^e	Q3^e
TVOC^c	**1.05**	0.88	1.29	**0.77**	0.31	1.24	**100.00**			**100.00**		
terpenes	**0.90**	0.73	1.14	**0.65**	0.25	1.09	**87.1**	84.0	89.8	**94.2**	79.6	98.4
terpenoids	**0.01**	0.01	0.01	**0.00**	0.00	0.00	**0.8**	0.5	1.1	**0.0**	0.0	0.1
aldehydes	**0.05**	0.03	0.07	**0.02**	0.00	0.00	**8.0**	6.2	10.5	**2.4**	0.0	11.7
α-pinene^d	0.58	0.48	0.73	0.42	0.18	0.77	54.0	51.1	56.8	57.1	42.6	64.8
β-pinene^d	0.17	0.13	0.23	0.05	0.02	0.24	11.9	10.4	13.6	9.8	5.0	19.1
3-carene^d	0.02	0.02	0.03	0.01	0.00	0.03	1.8	1.5	2.3	1.4	0.0	2.6
limonene+sabinene^d	0.08	0.07	0.11	0.04	0.02	0.10	7.1	6.3	8.0	6.4	3.7	10.0
hexanal	0.03	0.02	0.05	0.00	0.00	0.03	3.4	2.3	5.0	0.0	0.0	3.2
camphene	0.02	0.02	0.03	0.02	0.01	0.03	2.5	2.1	2.9	2.2	1.1	3.3
α-terpineol	0.00			0.00	0.00	0.00	0.3			0.0	0.0	0.0
α-terpinolene	0.00			0.00	0.00	0.00	0.2			0.0	0.0	0.0
cymene	0.01			0.00	0.00	0.01	0.7			0.0	0.0	0.6

[a] SER—area specific emission rate

[b] Terpenes, terpenoids and aldehydes in % of all idetified compounds; individual compounds in % of TVOC (identified + not identified); each observation normalized (0–100%)

[c] TVOC—total volatile organic compounds (acc. to EN 16516)

[d] Compound specific; other compounds quantified as toluene equivalents

[e] CI lo (lower bound), and CI hi (upper bound) of the 95%-confidence interval for the mean; Q1—the first quartile; Q3—the third quartile

high range between the median and the third quartile (Q3) in Table 4. The skewness of α-pinene in pine sapwood was much lower than that in heartwood, which was reflected in the TVOC distribution as well (Fig. 3). However, β-pinene exhibited a strong right skew in each pine group. The distribution of 3-carene was different from that of other major terpenes. In most pine samples, no 3-carene was detected, or its emission was negligible (the medians shown in Table 4). However, a significant number of specimens showed very high 3-carene concentrations, pushing the mean 3-carene emissions above those of β-pinene (Fig. 6). This finding is somewhat in contrast with several studies, which reported considerably higher amounts of 3-carene than β-pinene (Englund 1999; Larsen et al. 2000; Wajs et al. 2007; Hyttinen et al. 2010). However, Scots pine samples with no or low amounts of 3-carene were also reported (Lange and Weißmann 1988; Weissmann and Lange 1990; Manninen et al. 2002). Interestingly, the present study revealed a negative relationship between 3-carene and β-pinene. Specimens with high 3-carene concentrations tended to emit lower amounts of β-pinene, and vice versa, specimens without 3-carene exhibited the highest β-pinene emissions. Although this was not always the case, a clear pattern could be observed (Fig. 8). In contrast to 3-carene, β-pinene was present in all pine samples. The spread of distributions of individual VOC was highly related to their absolute emission rates. Higher emission rates implied higher variability and vice versa. Such a pattern was detected in all groups and can also be observed in Fig. 3.

The emission rates of terpenes from spruce were drastically lower than those from pine heartwood and sapwood (Table 4). This is also reflected in the TVOC-emission rates. However, the mean share of terpenes in all the identified VOC emitted from spruce was comparable to that from pine (Table 4). It amounted to 92.6% and 87.1% for the green and kiln-dried spruce, respectively. Low emission rates of terpenoids and aldehydes were also recorded. An even more significant difference in emission rates between the green and kiln-dried wood was observed compared with pine. The emission rates of terpenes and terpenoids decreased significantly after drying, which resulted in an increase in aldehyde share in dried samples to 8.1%.

Besides emitting much lower amounts of VOC than pine, the emission profiles of spruce in the green and kiln-dried conditions were similar to those of pine (Figs. 6 and 7). A major difference was detected in 3-carene emission, which played only a minor role in spruce specimens, in both the green and dried conditions. Another difference was a relatively high mean emission rate of limonene and sabinene in spruce specimens in the green condition, which was comparable to that of β-pinene. However, this was not the case for the kiln-dried group. Further investigations are required to explain this discrepancy.

In contrast to pine, 3-carene was present in most spruce samples. The pattern of its distribution was comparable to that of other terpenes. The distribution of other significant terpenes emitted from spruce showed a similar pattern to that from pine samples. They exhibited a significant right skew, and their spread highly correlated with the absolute emission rates.

The literature generally agrees on the major conclusions concerning the VOC emission profiles. Terpenes represent by far the most abundant group of compounds released from both Scots pine (Risholm-Sundman et al. 1998; Englund 1999; Larsen et al. 2000; Manninen et al. 2002; Wajs et al. 2007; Hyttinen et al. 2010; Czajka et al. 2020) and Norway spruce (Wajs et al. 2006, 2007; Peters et al. 2008; Hyttinen et al. 2010; Steckel et al. 2010; Czajka et al. 2020). The major compounds emitted were α-pinene, β-pinene, 3-carene, limonene, and hexanal. However, the reported amounts of the individual compounds and their shares in the total VOC emissions vary considerably among studies. Such inconsistency is expected, given the substantial *within-species* variation documented in this study and the very low sample sizes generally used in earlier studies. The contributions of individual compounds reported by Hyttinen et al. (2010) and Manninen et al. (2002) for the air-dried pine are primarily in the range between the first (Q1) and third quartile (Q3) observed in the present study (Table 4), or at least within the entire range. Czajka et al. (2020) recorded a low share (55%) of terpenes in the total VOC emissions for pine sapwood and an unexpectedly high share of α-pinene (88%) for pine heartwood. The former result is due to a very high share of aliphatic and aromatic hydrocarbons. In contrast, Wajs et al. (2007) documented an α-pinene share of as low as 28% and 3-carene as the most abundant compound. These values do not fit in the range observed in the present study. For Norway spruce, the relative amounts of individual compounds recorded by Wajs et al. (2006, 2007) and Czajka et al. (2020) primarily are in the range between Q1 and Q3 observed in this study. Hyttinen et al. (2010) found a very low relative amount (28%) of α-pinene, emitted from air-dried spruce, which strongly disagrees with the current results.

4 Conclusion

This study has attempted to overcome the recurring problem of small sample sizes when quantifying and comparing VOC emissions from wood and indicated the drawbacks when reporting the mean values without providing more information about the underlying sampling distribution.

The VOC emission distributions of Scots pine heartwood and sapwood partially overlap. Around 50% of the
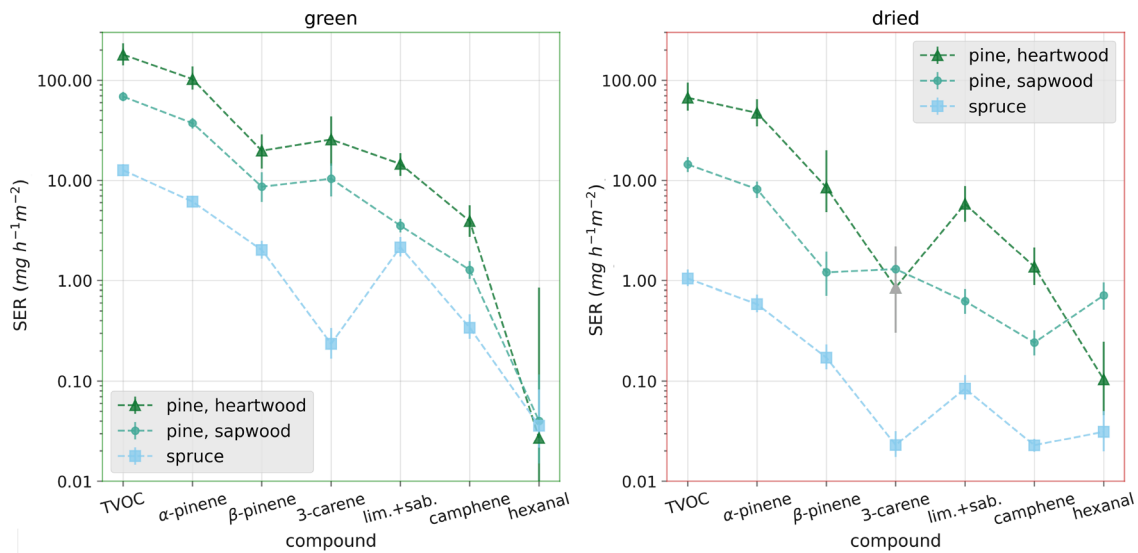
Fig. 6 VOC-emission profiles of Scots pine and Norway spruce in green and dried conditions, depicted as SER—Area specific emission rate; error bars represent 95% confidence intervals; 3-carene in dried pine heartwood not representative (see below)
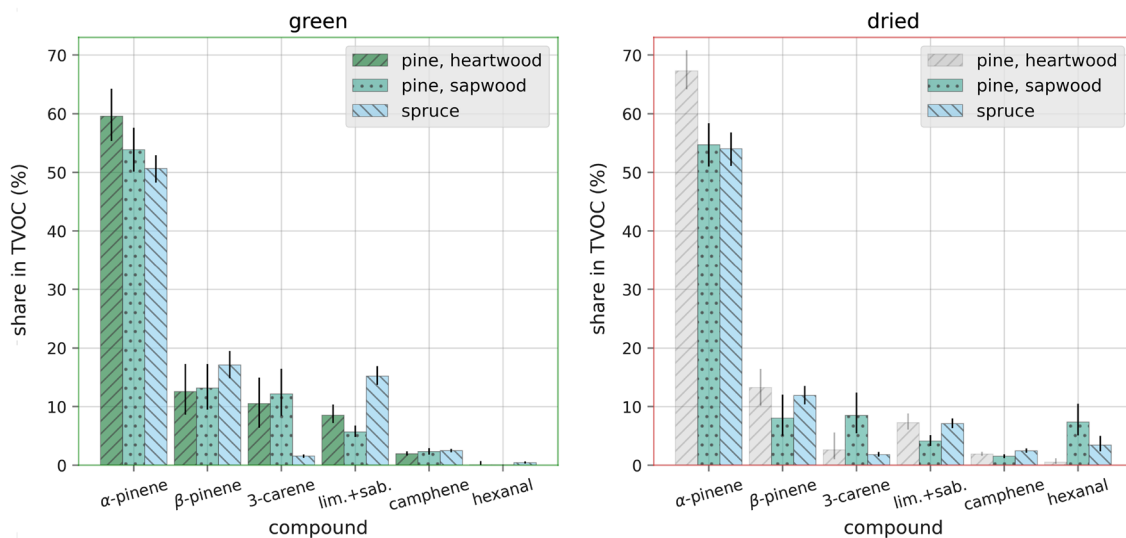

Fig. 7 VOC-emission profiles of Scots pine and Norway spruce in green and dried conditions, depicted as the substances' mean shares in TVOC; error bars represent 95% confidence intervals; dried pine heartwood not representative (see below)

heartwood measurements fall within the range of the sapwood emissions. However, a significant portion of the heartwood observations exceeded the sapwood emissions substantially. Consequently, the median sapwood emission was 55% and 75% lower than that from pine heartwood in the green and kiln-dried conditions. The discrepancy in the mean values was even more pronounced and amounted to 62% and 79%. The VOC emissions from Norway spruce wood amounted only to a small fraction of those emitted from Scots pine wood, 1.7–15%, especially in the kiln-dried group. The emissions from spruce heartwood and

sapwood strongly overlapped, and no statistically significant difference was observed.

The mean values considerably deviated from the median values in the pine heartwood and spruce groups due to the positively skewed distributions, indicating a disproportionally strong impact of the high-emitting specimens on the mean value. Pine sapwood samples were considerably less skewed.

Kiln drying substantially reduces VOC emissions, and the extent of the reduction depends very much on the emission of the drying material in the green condition. A strong
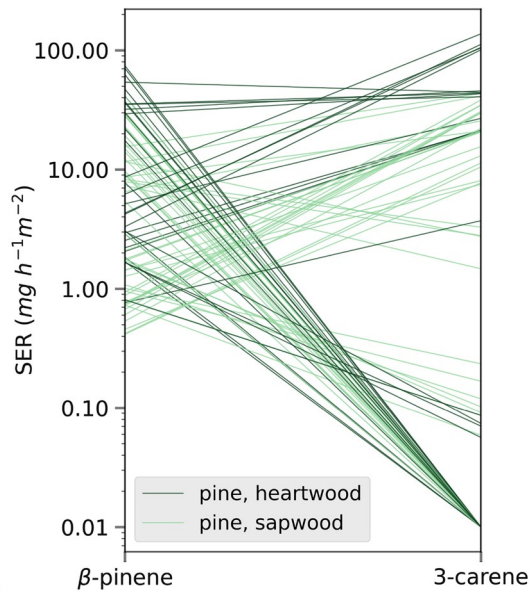
Fig. 8 Negative relationship between β-pinene and 3-carene in pine samples; each line represents one VOC measurement

positive linear relationship was observed between these two variables. It would be valuable to determine those drying parameters that influence this reduction the most and whether modifying the drying schedules could result in further emission reduction. The strong effect of the initial emission on the VOC-reduction should be considered when designing and evaluating such experiments.

Monoterpenes dominated the VOC emissions of both species, with α-pinene being by far the most abundant compound. The distributions of the most important terpenes in pine samples were right-skewed. The skewness of α-pinene in pine sapwood was considerably lower than that in heartwood, which was reflected in the TVOC distribution. However, β-pinene exhibited a strong right skew in each pine category. The distribution of 3-carene differed significantly compared with other compounds in pine samples. Although no or only very low emissions of 3-carene were detected in most pine specimens, many specimens emitted substantial amounts of 3-carene. Interestingly, a negative correlation was detected between 3-carene and β-pinene emissions in pine samples. However, the 3-carene content in spruce samples was less significant. The most significant terpenes emitted from spruce were right-skewed.

The high variability in VOC emissions within each tested group indicates that small sample sizes can easily lead to inflated or deflated central tendency estimates—the mean and median. Moreover, highly skewed data, as shown in the present study, strongly influence the mean. The mean captures only a part of the whole picture, and when reported as

the only statistic, it may even result in a biased interpretation of the data.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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