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Emission rates of bio-based building materials, a method description for qualifying and quantifying VOC emissions

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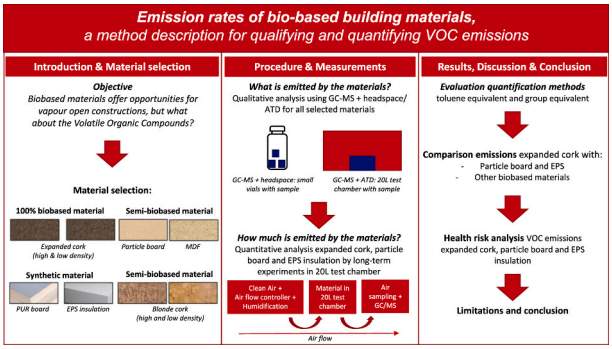
HIGHLIGHTS

- VOC emissions from biobased insulation materials are hardly reported yet.
- Expanded cork emits lower amounts of VOCs than particle board and EPS insulation.
- Group compared to toluene equivalent provides more accurate quantitative results.

GRAPHICAL ABSTRACT



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ABSTRACT

Biobased insulation materials offer opportunities to use vapor-open building constructions. Such constructions allow direct interaction between the biobased material and the indoor environment. This interaction raises questions about indoor air quality concerning volatile organic compounds (VOCs). This study presents results for the VOC emissions from biobased materials. It consists of two parts: 1) qualification of VOC emissions (compounds) from several biobased and non-biobased building materials, and 2) quantification of VOC emissions (emission rate) from expanded cork (biobased), particle board (semi-biobased), and EPS insulation. By quantifying the emission rate, the exposure to the released VOC emissions at room temperature in a standardized room can be compared to health limit requirements. Gas chromatography and mass spectroscopy (GC–MS) is used to derive the individual VOC emissions and the Total Volatile Organic Compounds (TVOC) from these materials. For qualification, two different sampling techniques are used in which temperature is introduced as a variable to investigate its effect on the type of compounds emitted. For quantification, the toluene equivalent approach is compared to the group equivalent approach. From the analyses it is concluded that temperature has an effect on the type of VOC compounds emitted from (biobased) materials. Results from the quantification indicate that expanded cork and particle board emit no harmful substances at a level that can affect human health. For EPS insulation, elevated levels of benzene were found to exceed healthy limits. The toluene equivalent approach for quantifying the emission, generally, underestimates the rate as compared to the more accurate group equivalent approach.

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1. Introduction

The construction industry is currently experiencing a transition regarding circularity and energy conservation. One possible way to decrease the amount of CO_2 emissions and energy demand, and promote circularity is by using biobased insulation materials. Biobased materials are materials that come from (1) living nature, including livestock, horticulture, agriculture and forestry, and (2) can regrow during the life of a building (Kennisbank Biobased Bouwen, n.d.). Especially the latter part of this description shows the potential of biobased materials compared to synthetic materials regarding energy and circularity. However, the use of such materials may have unintended consequences concerning indoor air quality.

Biobased building materials have the potential to react with the indoor environment. This reflects to the vapor open construction biobased materials are applied to. When a construction is damp open, this means that the damp-resistant layers are eliminated in the construction, allowing indoor moisture directly to interact with the biobased (insulation) material. In this way, a so-called 'breathing' situation is created in which indoor moisture can be controlled in a natural manner. When applied, vapor-open structures will have many square meters of wall surfaces/insulation materials that can directly interact with the indoor environment. This does not only raise questions about humidity but also for indoor air quality parameters, including volatile organic compounds (VOC). As of today, there is a lack of knowledge about the effect of VOC emissions emitted by biobased materials on indoor air quality, while at the same time unsubstantiated claims are being made that biobased materials can automatically be considered healthy.

VOC emissions are gaseous emissions with a specific individual chemical structure that, among other things, evaporate from the surface of a material into the air. This type of emissions is an understudied class within the field of indoor air quality. According to the International Energy Agency, database Pandora and the World Health Organisation (International Energy Agency, 2020; La Rochelle Universite, n.d.; World Health Organisation European Centre for Environment and Health, 2010) VOC emissions become more important. Economic growth and development in lifestyle changes increase the use of various building materials, including biobased building materials. This, combined with the increased air tightness of buildings and the large amount of time people spend indoors, poses a new risk for humans (World Health Organisation, 2010).

Elevated amounts of VOC emissions have the potential to cause (1) irritant effects including perception of unpleasant odours, mucous membrane irritation and exacerbation of asthma; (2) systemic effects such as fatigue and difficulty concentrating; and (3) toxic, chronic effects such as carcinogenicity (Girman, 1989). Therefore, it is important to investigate the impact of newly developed biobased insulation materials directly interfering with the indoor air on its potential to emit VOC compounds and determine the rate of emission of these compounds, to analyse the potential risk it may pose to human health.

Studies on VOC emissions of building products are not new. It was already found that finishing layers and wood (board) products with synthetic resins have the tendency to emit elevated levels of VOC (da Silva, 2020; Van Dam and Van Den Oever, 2019). However, what was found in the study of He et al. (He et al., 2012a) is that, when looking more deeply at the impact of VOC emissions related to single elements inside of wood boards, the Total Volatile Organic Compounds (TVOC) from raw wood chips are proportionally higher than when these wood chips are processed into application-ready wood boards. TVOC is defined as the sum of all single VOC emission with a carbon range between C6 and C16. This outcome raises the question whether biobased materials, that largely consist of raw plant materials, especially when no finishing layer is applied to these materials, emit VOCs at a higher rate as compared to processed building products.

An investigative literature study was performed on TVOC and single VOC emissions from biobased building materials to identify the status of research in this area and the research methods applied for analysis. Since the last major literature review in 1996 (Haghighat and Bellis, 1998), new studies have been performed on wood-based boards and on some biobased materials. Wood-based boards have been analysed much more as compared to biobased materials. Particle board and MDF were analysed most. Between 1997 and 2022, 12 studies have been reported on particle board regarding TVOC emissions and 14 regarding single VOC emissions. For MDF 7 studies have been performed on TVOC emissions and 13 on single VOC emissions. Although wood based materials are the most investigated, in the end only a limited amount of research has been performed on these materials looking at the time that has passed.

Biobased materials other than wood boards have not been studied in literature extensively: TVOC – 2 studies for hempfibre (Maskell et al., 2015; Koivula et al., 2005), 1 study for cork (Silva and Fernandes, 1997) and hemplime (Maskell et al., 2015). Single VOC - 2 studies on hempfibre (Maskell et al., 2015; Adamová et al., 2019), 2 studies on cork (Silva and Fernandes, 1997; Horn, 1998) and 1 study on hemplime and mycelium (Tirillini et al., 2000). This creates a gap in knowledge concerning the VOC emissions of these types of materials. This accounts both for single target VOCs as well as for total VOC emissions.

Looking at studies that have made initial steps toward investigating biobased (insulation) materials, it can be stated that evaluated emissions of harmful TVOC compounds from biobased materials did not exceed the set limits (da Silva, 2020; Maskell et al., 2015; Durai Prabhakaran et al., 2015; Adamová et al., 2020; De Visser et al., 2015; Richter and Horn, 2015; Richter et al., 2021; Romano et al., 2019). Nevertheless, the conclusion may not be drawn that this automatically will account for other contemporary and future biobased insulation materials and finishing layers, as only a small selection of biobased materials is yet investigated.

More background information on the topic can be found in the studies of Maskell et al. (Maskell et al., 2015) and Richter et al. (Richter and Horn, 2015). Both studies investigated VOC emissions from the surface of biobased building materials. The results from these studies conclude that no high amounts of TVOC, TSVOC or formaldehyde emissions were found in the tested biobased materials. However, for some biobased products elevated levels were found of single components. This creates a major concern.

The increased TVOC emissions as found from single plant-based products used for wood boards, the lack of research in the field of biobased materials and the potential of elevated levels of single VOC emissions regarding biobased materials identifies the need to increase our knowledge on the emission of VOCs from biobased building materials. Biobased materials that serve as both insulation and wall finishes are of main interest as these porous materials are in direct contact with the indoor environment and may affect the indoor air quality.

Therefore, the main goal of the research presented was to determine the (T)VOC emission from selected biobased materials. In this way the knowledge regarding VOC emissions on these newly developed materials and on VOC emissions in general can be increased. In addition, we critically evaluated the research method to highlight the challenges that come with experimentally evaluating material emissions.

2. Method

2.1. Material selection

Three types of materials have been selected for the study, based on their potential application:

(1) Fully biobased materials made of 100 % natural products – Expanded cork (cork particle board were, due to the production process, cork particles stick together by the naturally released suberin) with a high and low material density [kg/m^3];
(2) Semi-synthetic materials made of natural products with a synthetic resin – MDF, particle board and blonde cork;

(3) Fully synthetic material made of 100 % non-biobased materials – EPS insulation and PUR insulation plates.

In the process we included semi- and non-biobased products for comparison of the outcomes.

2.2. Procedure

A two staged approach was taken for the measurements. First a qualitative assessment of the VOC compounds was determined. Here the most important VOC compounds emitted were identified. Next, a quantitative assessment was performed, in order to determine the emission rate of selected VOC compounds, as determined from the qualitative study.

Gas chromatography and mass spectroscopy (GC–MS) (Hübschmann, 2015) was applied in this study for the analysis of the selected materials. Two methods were applied: (1) GC–MS with adsorption/thermal desorption (ATD), and (2) GC–MS with headspace. The qualitative study allowed to identify what emissions evaporate from the materials surface under static circumstances, and at various temperatures. This presents the starting point for the quantified emission rates.

The quantitative study focused at retrieving the amount of VOC emission (rate) that is evaporated from the investigated material. In the process of that analysis, the quality of the standard quantification method, which applies the toluene method was evaluated as well. A new type of quantifying strategy was introduced, which is based on the evaluation of group equivalents. The intention of this updated quantification strategy is to arrive at a more precise emission rate outcomes as compared to the toluene method.

2.3. Qualification measurements

The first step for investigating VOC emissions is to qualify the emission under static conditions. This step must be taken before quantification of the emission can take place. From the qualification the settings of the GC–MS device can be tuned and identify potentially harmful emissions. For this step all materials, as shown in Fig. 1, were tested. As part of the analysis, two set-ups of the GC–MS technique have been applied: (1) GC–MS with adsorption/thermal desorption (ATD), and (2) GC–MS with headspace. The two set-ups of the GC–MS allowed for analysing the effect of temperature on the compounds emitted from the materials investigated.

The materials tested were as fresh as possible and stored under sealed conditions. For all tests the same batch of samples was used.

2.4. GC–MS with ATD

The first setup is Gas Chromatography with Mass Spectroscopy and Thermal adsorption/desorption (GC–MS + ATD). For this a test environment has been designed and built. The test environment is made of a stainless steel cylinder (20*L*) with a supply tube of clean air on the one end and an exhaust tube on the other end to extract emissions from the environment in the test chamber (Fig. 2). The shape and connections of the test chamber are based on references found in literature (da Silva, 2020; Maskell et al., 2015; Son et al., 2013), combined with requirements as stated in ISO 16000:9–2006 (NEN-EN ISO 16000-9:2006, n.d.).

With GC–MS + ATD samples are taken from a larger test environment compared to the headspace technique and samples are taken by sorption tubes. These tubes are heated, which causes the compounds to be released from the sorption tubes and being transferred to the GC–MS device. During the measurements, the temperature and relative humidity are based on the conditions in the room (RH 30 %, temperature ±22 °C). No air flow is introduced in this setup, i.e. there was no ventilation taking place. This allowed the concentration of emitted material to increase to higher levels and thus increased the chance of identifying VOC emissions that have low emission rates. The device being used for the measurement is Gas Chromatograph Clarus 680 (Perkin Elmer) with an Elite-5MS column (60 m; 0.25 mm DF1) coupled to a turbomatrix Air Monitoring Trap 350 ATD system (Perkin Elmer) and a Mass spectrometer Clarus SQ 8 T (Perkin Elmer). Samples are taken on AIRTOXIC desorption tubes and sampled with a GSP-300FT-2 air sampler with a flow rate of 100 mL/min and a volume of 3 L.

All selected test materials were tested under the indicated conditions. For each material, three samples are taken from the test environment, i.e., a cylinder with the individual material: one after 24 h, one

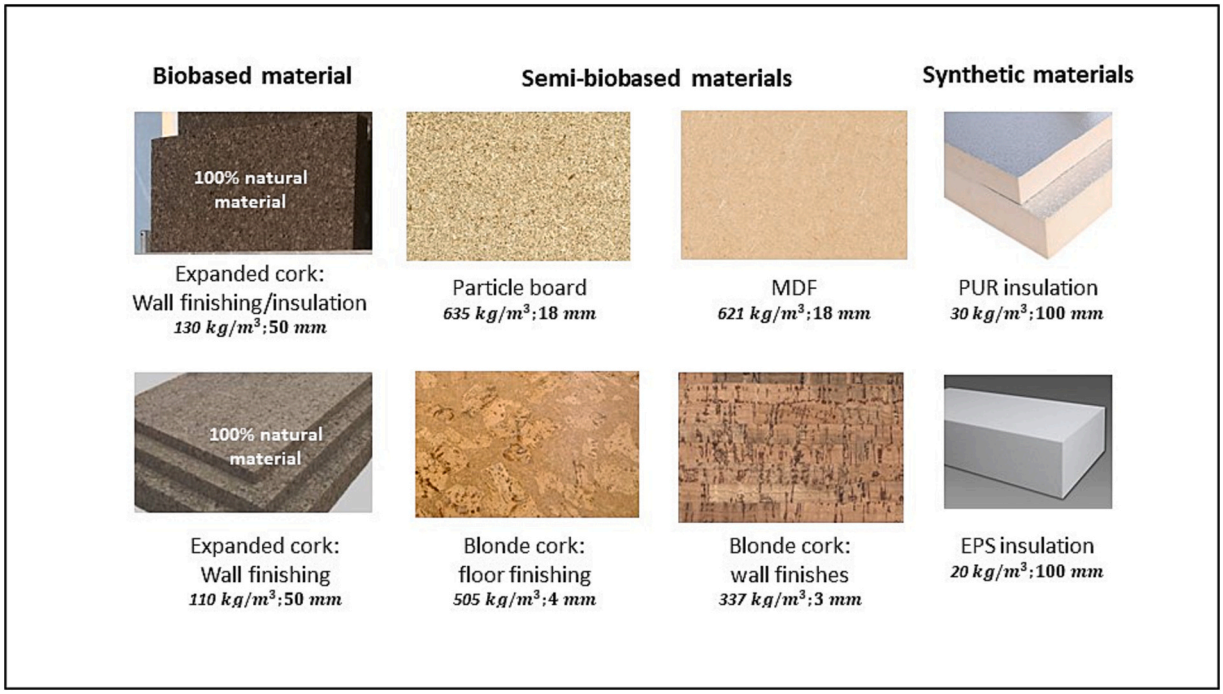

Fig. 1. Investigated materials, including biobased, semi-biobased and synthetic materials.

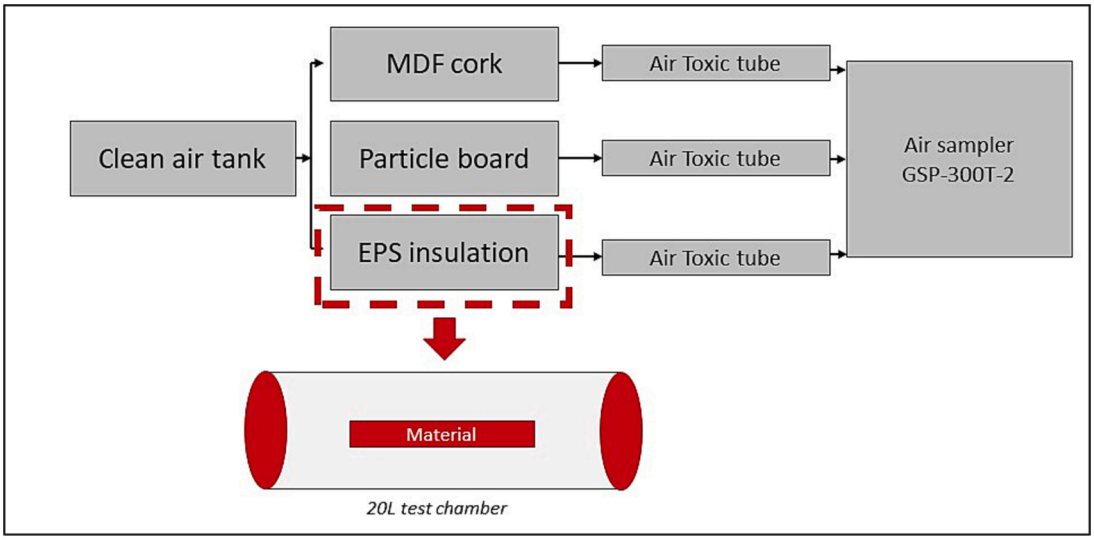

Fig. 2. Setup for the measurements with GC–MS and ATD.

after 48 h and one after 72 h. After taking each sample, a vacuum was created in the cylinder. To return to a normal atmospheric pressure, the volume that was taken from the cylinders by the air sampler was added again using an air flow controlled tube with clean air attached to the cylinder. The material loading ratio was set as high as possible to enforce high amounts of emissions to be evaporated in the air (Table 1).

After sampling emissions from the test environment onto AIRtoxic tubes, the desorption tubes were analysed by the GC–MS. Analysis was done applying the GC–MS settings as shown in Table 2.

2.5. GC–MS with headspace

This second analysis set-up combined GC–MS with a head space (HS). With this technique small crushed samples are placed in small vials and are being analysed under various thermal conditions. In this case, no external transfer of tubes is taking place. The vials are being heated at pre-set temperature conditions to investigate the influence of temperature on the emissions release. The materials are being exposed for a certain amount of time to this temperature, forcing the test materials to emit compounds. Once this step is done, the emissions are captured directly from the vials by means of a thin injection needle, transferring the emissions directly into the GC–MS system. The qualitative analysis is therefore based on the temperature of the vials, the sampling from the vials by a needle and the GC–MS settings.

For the experiments small samples were prepared with a maximum fill of the vials by the material up to halfway the bottles (+/− 37.5 mm; see Fig. 3). To fill the vials, six material samples were extracted from two or three places from each material to analyse the effect of homogeneity of the material considering VOC emissions. Per sample vial a combination is made of three separate samples from each location to limit the sample error of the material.

The sample location was divided into inside and outside samples. Inside samples include samples of 20–25 cm inside the full-sized material. Outside samples are taken 5 cm from the border of the material to determine the homogeneity of the material. For non-fresh MDF and particle board, also samples were taken at the border of the material that was exposed to the indoor environment, in order to analyse the influence on the emissions at the sides of the material after being exposed to the indoor environment for a certain amount of time. In Fig. 4 a selection of the material samples is shown. The samples had a 1–10 % deviation in weight.

The GC–MS + headspace applied uses a headspace carousel device (*Shimadzu GC–MS-QP2020 with a non-polar column 30 m, 0.25* µm*, 0.25 mm coupled to HS-20*) with small, closed sample tubes of 20 ml that were heated at 45 °C for 12 h. Due to pressure differences, emissions will be released from the material into the headspace. When the gasses in the headspace and in the material reach an equilibrium, samples are taken from the headspace and analysed by GC–MS. Table 3 summarizes the applied GC–MS settings for investigating the materials.

2.6. Quantification measurements

In order to determine the amount of VOC emitted from the surface of the materials, GC–MS with ATD technique was used. The same settings have been used as for the GC–MS with ATD analysis in the qualification studies (see Table 2). In these measurements, due practical limitations, a selection was made of the materials tested qualitatively. MDF cork, particle board and EPS have been investigated. During the experiments, the materials were covered with aluminium foil at the sides and the bottom of the material, to assume a representative emission situation. In this case only the top of the material is in direct contact with the indoor air in the cylinders. The loading factor therefore is smaller compared to the qualitative measurements (see Table 4). The materials were freshly unpacked just before the start of the measurements.

The goal of this experiment was to quantify the concentration of VOC in the air of the cylinder from 3 days, up to 28 days exposure in an indoor environment with continuous ventilation. These measurements align as close as possible to the standard NEN-EN ISO 16000-9:2006. This standard provides guidelines to determine the area specific emission rate of VOCs from building products.

Table 1
Materials area, loading factor and density of the tested materials used for ATD measurements for qualification.

	Particle board fresh	Particle board non-fresh	MDF fresh	MDF cork	IDF cork	Cork floor	Cork wall	EPS	PIR
Sample area [m^2]	0.104	0.207	0.103	0.243	0.237	0.191	0.177	0.325	0.288
Loading factor [m^{-1}]	5.21	10.37	5.15	12.13	11.85	9.54	8.85	16.25	14.40
Weight [g]	521.90	959.7	759.7	602.41	408.10	192.8	71.7	140.80	360.9

Table 2
ATD and GC–MS settings of qualitative analysis of sample materials.

Sample	Semi-synthetic and cork materials	Synthetic materials
Sampling method	3 l samples 100 ml/min	1.5 l samples 100 ml/min
Settings GC	GCMS3 Initial: 50 °C for 2 min Ramp 1: 1 °C/min to 60 °C, hold for 1 min Ramp 2: 10°/min to 250°, hold for 2 min	GCMS5 Initial: 50 °C for 2 min Ramp 1: 5 °C/min to 150 °C, hold for 0 min Ramp 2: 10°/min to 250°, hold for 2 min
Split ratio	100 %	
Settings MS	*m/z* 40–700	
Settings ATD	Toluene slow (Based on previous research performed in the lab (Verbunt, 2016))	

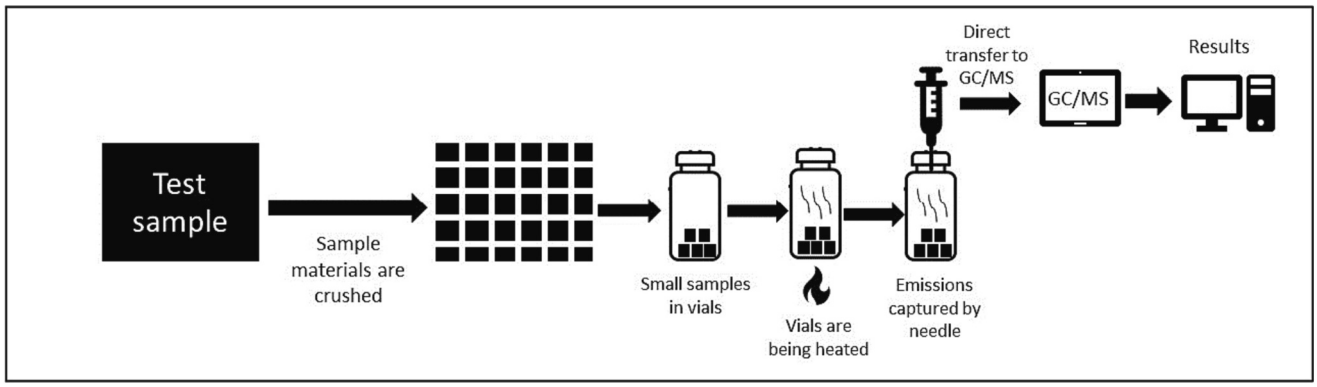

Fig. 3. Air sampling process GC–MS + headspace.

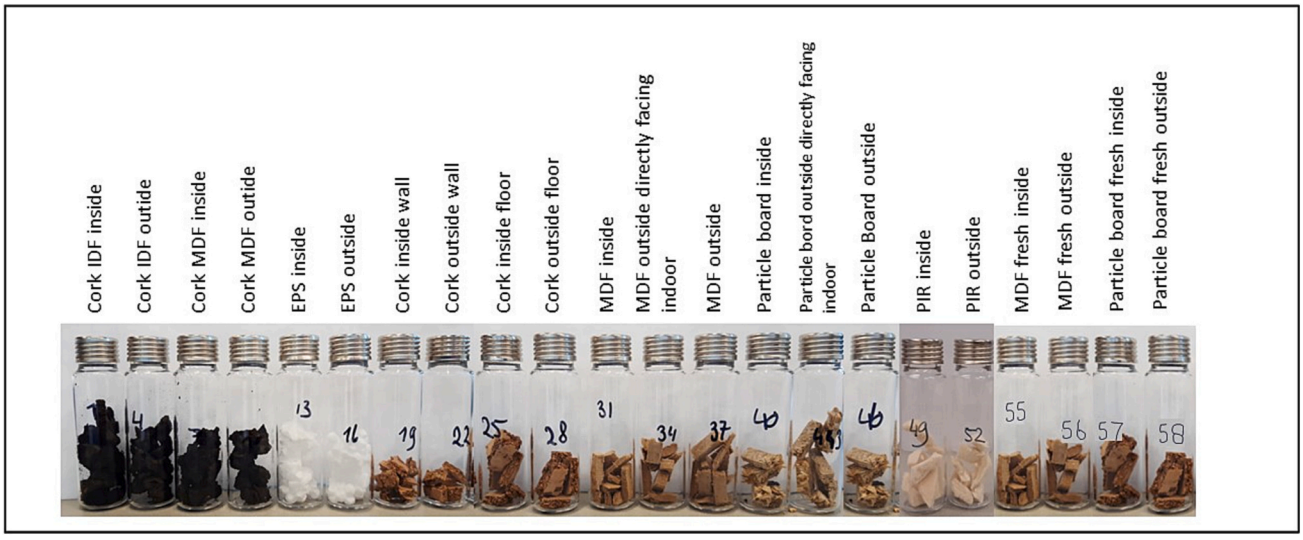

Fig. 4. Material samples in Headspace vials.

The cylindrical test chambers are similar to the 20 L test chambers applied in the qualification study. The boxes are air-tight and connected to a clean air supply. The air flow is controlled by a mass flow controller (Bronckhorst). The air flow is monitored and logged continuously, as are temperature and relative humidity (Fig. 5).

A constant air change rate of 0.5 air changes per hour was assumed. This results in an air flow rate of 0.1667 L/min for each test chamber. The air is conditioned in terms of relative humidity, which has been set to a 50 % ±5 %. The temperature could not be controlled within the test chamber and follows the controlled room temperature which was set to 21 °C +/− 0.5 °C.

Air sampling on AIRTOXIC desorption tubes has been done using an air sampling device (model GSP-300FT-2 of GASTEC). The exhaust air at the emission chamber outlet is used for sampling. The air sampling system is directly coupled to the exhaust air. In between AIRTOXIC tubes are located.

Indoor concentrations have been measured over a month. The test samples were taken after 3 days (72 +/− 2 h) and 28 days (which are the minimum amount of samples that have to be taken according to NEN-EN ISO 16000-9:2006), with additional samples taken at day 1, 7, 14 and 21 to study the decay of the emission rate. All details of the quantification measurements are summarized in Table 5.

The emissions measured are recalculated using the area specific emission rate (SER). SER is the emission rate, TVOC or single emitted VOCs from the non-covered material surface in µg per time unit under standardized environmental conditions, including a constant tempera-

Table 3
Headspace and GC–MS settings of qualitative analysis of sample materials.

Sample preparation	0.08–1.75 g sample in test vials
Sampling method	Sample time: 10 min Heating vial: 12 h
Column	30 m, 0.25 um, 0.25 mm; Non-polar Transfer gas: helium
Settings GC	Initial: 50 °C for 2 min; Ramp 1: 1 °C/min to 60 °C, hold for 1 min; Ramp 2: 10°/min to 250°, hold for 2 min
Settings MS	m/z: 40–700

Table 4
Overview of materials characteristics for quantification measurements.

	Particle board fresh	MDF cork	EPS
Sample area [m^2]	0.106	0.106	0.089
Loading factor [m^2/m^3]	5.31	5.31	4.43
Weight [g]	521.90	602.41	140.80
Measured density [kg/m^3]	657.4	137.7	15.9

ture, relative humidity and area specific air flow rate, with a certain loading factor. It is calculated from:

$$SER_a = \frac{C_t * ACH_t}{L_{At}} \text{ At time t} \quad (1)$$

With

$$C_t = \frac{Ma}{V_a} \quad (2)$$

Where:

- Ser_a: area specific emission rate test material $\left[\frac{\mu g}{m^2 h}\right]$
- C_t: concentration substance in test chamber $\left[\frac{\mu g}{m^3}\right]$
- M_a: mass component in an air sample [μg]
- V_a: volume air sample [m^3]
- ACH: Air change rate [h^{-1}]
- L_{At}: loading factor test room [m^2/m^3]

To determine the substance concentration (Crr) in a reference room of 30 m^3 the loading factor (L) of a standard reference room should be included as well. The loading factor [m^2/m^3] is dependent on the area size of the surface of the material directly facing the indoor air in m^2 related to the volume of the chamber in m^3. During the measurements a relatively large loading factor was chosen compared to most of the reference studies found. This has to do with the expectation that cork will emit low emissions and would therefore produce low peaks. In order to align the loading factor, a relatively high loading factor was applied for EPS and particle board as well.

To calculate the actual substance emission for a standard reference room, the loading factor for such a room must be included. It is assumed that the materials are only used in the walls. Therefore, a loading factor of 1.0 is applied. Crr was calculated, according to Eq. 3 for the days sampled, up till 28 days after the start of the experiment.

$$Crr = \frac{SER_a * L_{Ar}}{AC_r} \text{ At time t} \quad (3)$$

Where:

- C_{rr}: Actual substance emission in a standard room $\left[\frac{\mu g}{m^3}\right]$
- L_{ar}: loading ratio reference room [1 m^2/m^3 for wall materials]
- Ac_r: air change rate in reference room [0.5 h^{-1}]

The authors are aware that the material behaviour can deviate in a large standard test chamber compared to the small test chamber used in this experiment, including the material state and environmental factors. Following the procedure of NEN-EN ISO 16000-9:2006 an environment is created that is as close as possible to a real environment as found in larger rooms. With this we assume that deviations in outcomes are as limited as possible. However, in a real situation there is a probability that the air layer nearby the material is more stagnant, meaning that the material emits a different amount of VOC emissions then obtained from the small test chamber. To address this problem to some extent, use was made of an aluminium device placed on the inside at the entrance of the test tube, which allowed clean air to be blown in varying directions. This prevented the air from being blown directly over the material at a high velocity, which is assumed more similar to a real environment. More

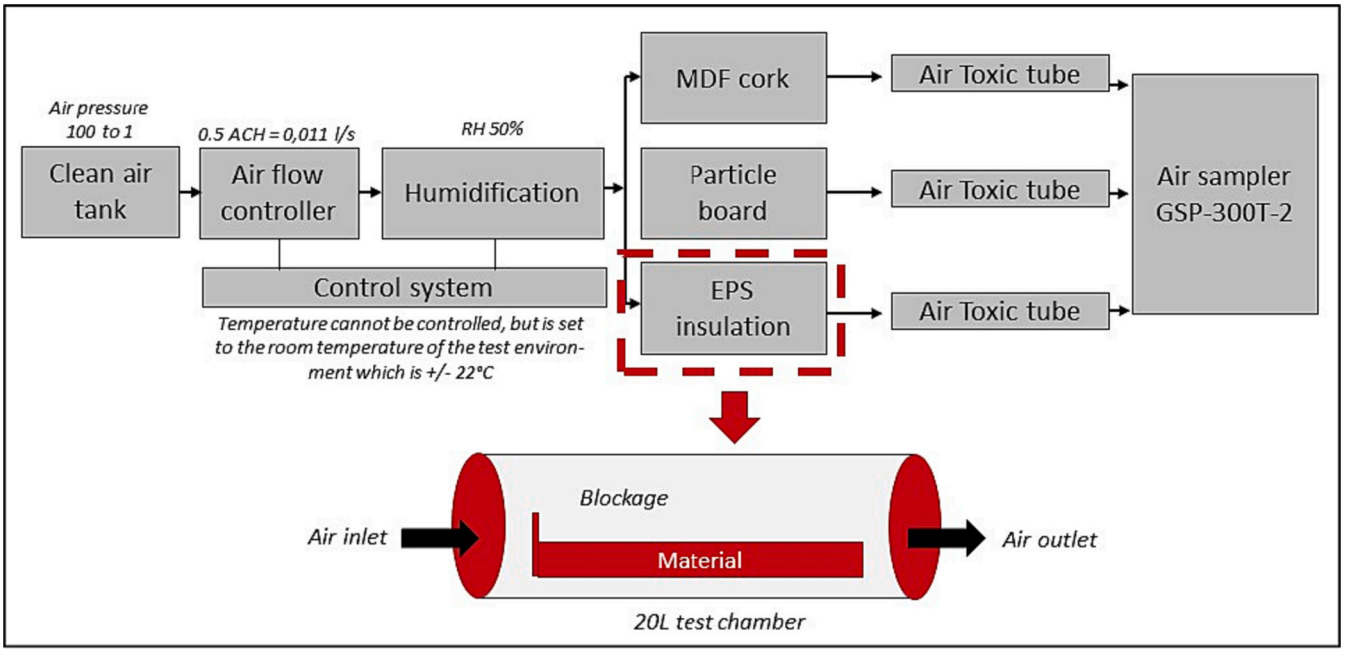

Fig. 5. Overview test setup dynamic measurements.

Table 5
Overview of details quantification measurements.

Test condition	Dynamic chamber
Sample area [m^2]	Particle board: 0.106, Cork: 0.106 m2, EPS: 0.0885
Volume [l]	20
Loading factor [m^2/m^3]	Particle board:5.31, Cork: 5.31, EPS 4.43
Air change rate [h^{-1}]	0.5
Air supply (l/min)	0.1667 L/min per box
Equilibration time [day]	1, 3, 7, 14, 21, 28
Temperature, RH	21 °C +/− 0.5 °C; 50 % +/− 5 %
Compounds, sample flow and total sampling	VOC: 50–200 ml/min for 10–30 min
Inlet air	Clean air
Analysis method	VOC: GC–MS + ATD

research is needed to investigate the differences in outcomes between a real-sized test chamber and small test chambers. This is out of the scope of this research.

2.6.1. Total VOC (TVOC) calculations

For the total VOC emissions in a sample, different methods can be adhered to. According to EN-16516:2017 TVOC is the sum of all VOC components between C6-C16 with a minimum concentration of 5 μg/m^3. As the AIRTOXIC tubes have a range up to about C12-C14/C15 there is a small deviation from this definition. In addition, due to the assumed low emission values for cork, the total chromatogram area between C6-C12-C14/C15 is included in the results, with values of <5 μg/m^3 included as well. Background emissions are subtracted from the total sum (Haerinck, 2019).

2.6.2. Toluene versus group equivalents

The most common method for quantifying VOC emissions uses an external calibration curve (ASHRAE, 2017). Here a standard sample is used containing a known concentration of substances. These are injected with different amounts on the sorption tubes. Based on the response of the GC–MS and ATD a linear relationship can be obtained between the amount of TIC counts on the tubes and the amount of a component in ng.

In general, the toluene equivalent is often used (NEN/EN 16516:2017+A1:2020, n.d.). The TIC responses of individual substances are converted to the response of toluene in the GC–MS. The advantage of this technique is that only one calibration curve is needed to compare with all components. The disadvantage is that it assumes that all emissions have a similar response on the GS-MS + ATD device as toluene. This assumption may not hold true in any case and may lead to over- or underestimated outcomes.

Therefore, in addition, an alternative approach was included in the analysis as well (Perkin Elmer and de Kort, n.d.). In order to determine emissions more accurately, the method of measuring VOC emissions based on group equivalents was applied. This method has been applied previously by Cariou (Cariou et al., 2016). Conclusions from that study were that the impact was small for aromatics and aliphatic hydrocarbons, but differences were noticeable for oxygenated molecules such as alcohols and ketones. Since cork products were expected to emit low amount of emissions, accuracy was deemed very important in this study, therefore the additional analysis via the group equivalent approach.

The qualitative results were used to detect the most common VOC emissions from the investigated samples for expanded cork, particle board and EPS insulation. Besides aromatic hydrocarbons such as toluene, also other groups were found in high quantities. Based on this information, a selection was made of VOC emission types that were used in addition to toluene for the quantification. This can be seen in Table 6. Due to research constraints, remaining groups, such as acids, were only investigated through the toluene equivalent.

A multi-standard (Sigma Aldrich 40,353-U) containing 48 emission components, including the emissions from Table 6, was used for developing the calibration curves. Five different amounts of the multi-standard were added to the GC–MS, with a weight of 20, 50, 100, 200 and 400 ng. The samples were diluted with methanol. Per volume six sample tubes were made, resulting in a total of 30 datapoints for the calibration curves of the various compounds extracted from the multi-standard. Based on the response factor, a calibration curve was developed. For additional verification, pure components of each of the equivalents were also tested with the GC–MS system to assure that the found peaks correspond to the emissions in the multi-standard being used for calibration.

The volumes for the calibration curve are injected onto the AIR-TOXIC tubes by using a CSLR device of Markes International. This device is coupled to a nitrogen air flow. The substances are injected into the device, mixed with the nitrogen and flushed through the AIRTOXIC tubes. The air flow of the nitrogen is set to 100 ml/min. In this way the sample extraction is similar to the sample extraction of the test materials and can therefore be compared. The results are obtained using GC–MS + ATD with GCMS3 settings as shown in Table 2.The calibration curve of toluene is shown in Fig. 6. The calibration curves of the group equivalents are shown in the annex.

3. Results and discussion

3.1. Qualitative analyses

For the quantitative analysis expanded cork, EPS insulation, PUR insulation, particle board, MDF and blonde cork were investigated. All materials, except for the biobased expanded cork, were used in this study as reference materials to verify the results of biobased expanded cork, as relatively more literature can be found on these materials. The emissions found in the tested reference materials are in line with the emissions found in literature (He et al., 2012a; Kim et al., 2006; Wi et al., 2021; Kusch, 2017; Pajaro-Castro et al., 2014; Cabanes et al., 2020; Scheirs and Priddy, 2003; Baumann et al., 1999a; Henneuse-Boxus and Pacary, 2003; He et al., 2012b; Svedberg et al., 2004; Baumann et al., 1999b; Ministry of food and environment Denmark, 2020; Laopaiboon et al., 2006; Shakeel, 2018).

Fig. 7 summarizes the results from the qualitative analysis for all the investigated materials. The results are presented as so-called *chemical fingerprints*, which presents the chemical groups found per material. An overview is shown of the emissions measured with the headspace method (45 °C) and the ATD method (21 °C). IDF cork is expanded cork with a density of 110 kg/m^3 and MDF cork is expanded cork with a density of 130 kg/m^3.

Table 6
Overview of additional VOC-emission groups applied for the quantification of the emissions.

Equivalent	Group	CAS nr.	Used for quantification of:
Limonene	Terpenes	138–86-3	Terpenes
Heptane	Aliphatic hydrocarbons	142–82-5	Aliphatic hydrocarbons
Butanol	Alcohols	71–36-3	Alcohols
2-methyl-4-pentanone	Ketone	18–10-1	Ketone
Decanal	Aldehydes	112–31-2	Aldehydes
Toluene	Aromatic hydrocarbons	108–88-3	Aromatic hydrocarbons and remaining groups

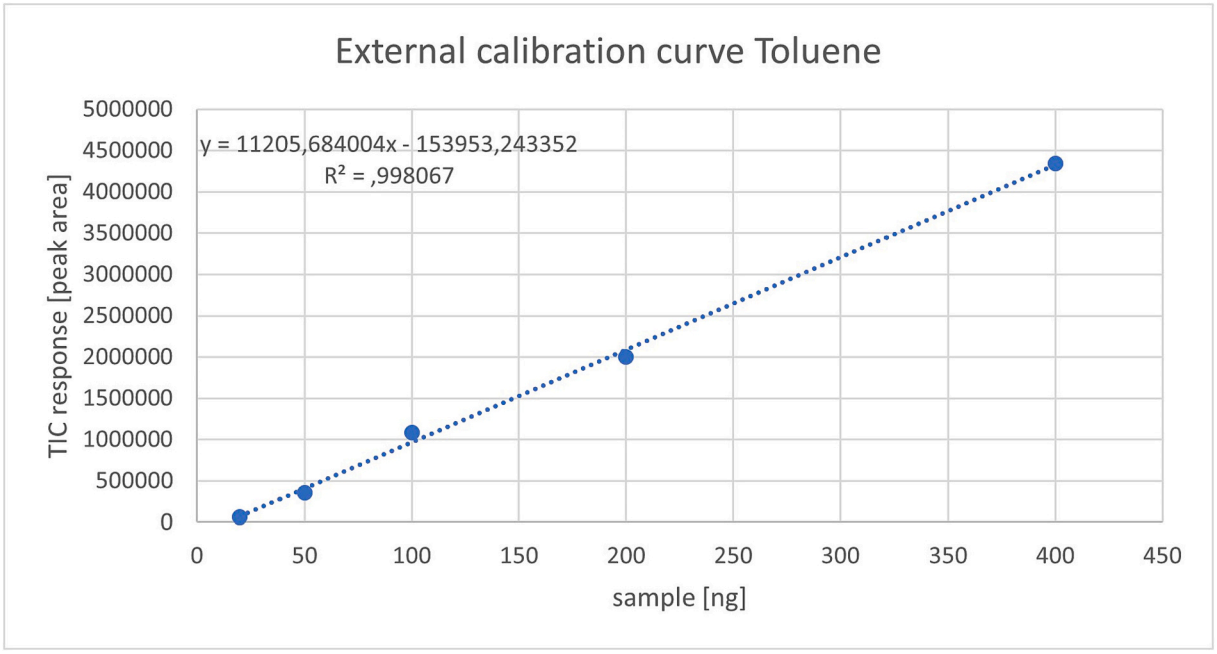

Fig. 6. External calibration curve Toluene.

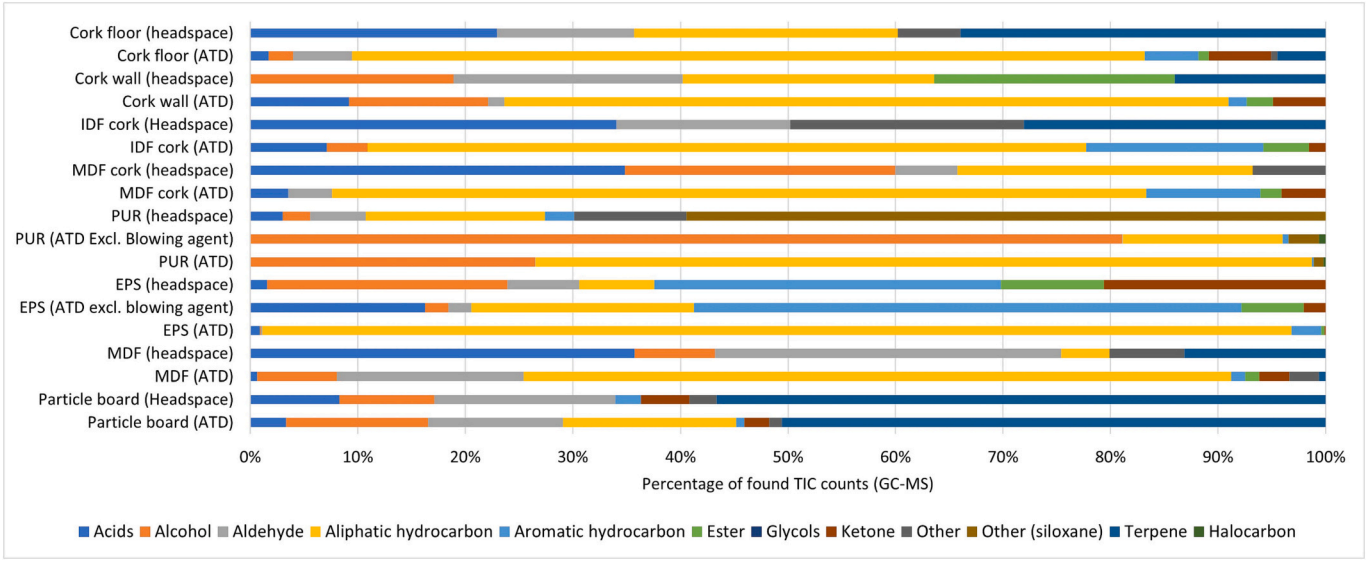

Fig. 7. Chemical fingerprint particle board, EPS insulation and expanded cork comparing results GC–MS with ATD and GC–MS with headspace method.

Table 7
percentage difference between group equivalent and toluene equivalent for different TIC count values.

Compound	TIC counts			
	1.00E+09	*1.00E+08*	*1.00E+07*	*1.00E+06*
Toluene eq.	0 [%]	0 [%]	0 [%]	0 [%]
Aliphatic hydrocarbon eq.	74 [%]	74 [%]	73 [%]	64 [%]
Alcohol eq.	914 [%]	911 [%]	883 [%]	639 [%]
Terpene eq.	−29 [%]	−29 [%]	−28 [%]	−23 [%]
Ketone eq.	107 [%]	106 [%]	105 [%]	90 [%]
Aldehyde eq.	12 [%]	12 [%]	12 [%]	15 [%]

Difference > 0 %: underestimation by toluene equivalent.
Difference < 0 %: overestimation by toluene equivalent.
Difference = 0 %: perfect match.

Table 8
TVOC values: toluene eq. versus group eq. for tested materials.

Day	TVOC emissions [µg/m³]					
	Particle board		Cork		EPS	
	Group eq.	Toluene eq.	Group eq.	Toluene eq.	Group eq.	Toluene eq.
Day 1	367	272	320	234	687	523
Day 3	201	141	111	107	356	310
Day 7	ND		77	63	263	231
Day 14	152	113	84	77	257	233
Day 21	71	50	58	49	159	130
Day 28	60	38	18	15	38	26
Healthy value TVOC according to WHO (World Health Organisation, 2010) after 28 days	<200					

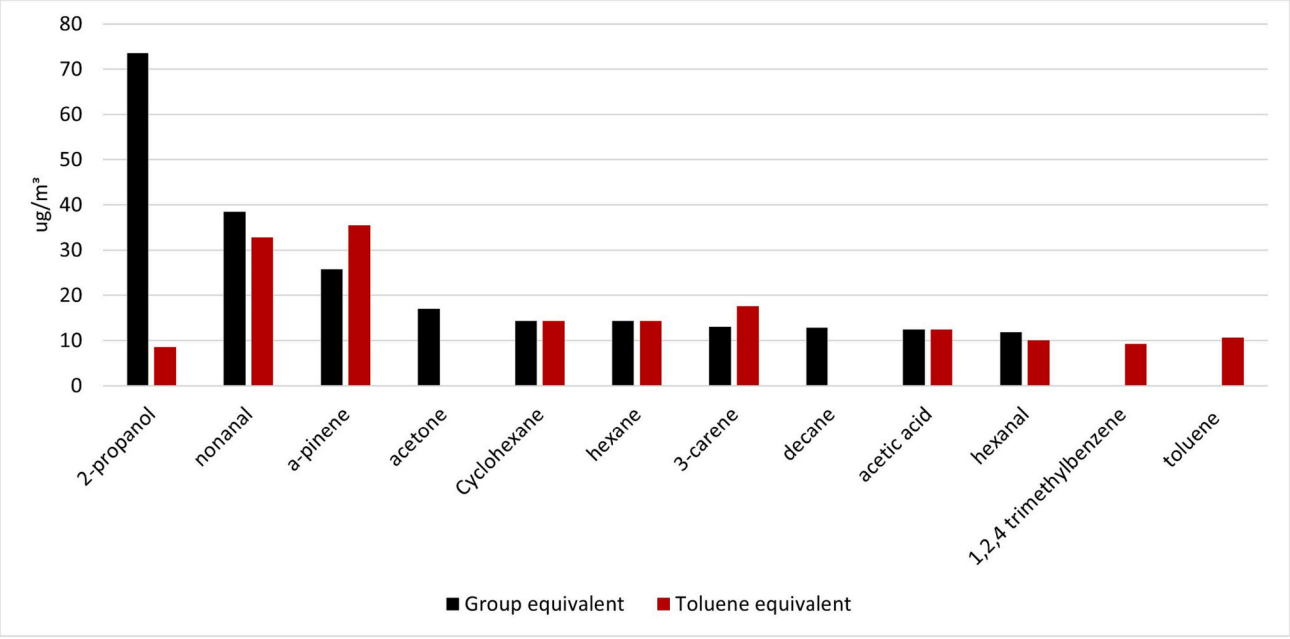

Fig. 8. Top ten compounds with highest emittance for particle board: group equivalent versus toluene equivalent.

Fig. 7 shows that the different emissions found for expanded cork (IDF and MDF) can be separated in groups. Aliphatic hydrocarbons were found in both materials at room temperature and acids mainly at an elevated temperature. The main concerns for single emissions emitted from the materials include furfural, toluene, acetic acid, phenol, acetone and various terpene emissions. These emissions have been reported in literature for blonde cork, and are most harmful for the health of humans (Salthammer, 2000). At room temperature toluene and acetic acid were found for expanded cork, while at an elevated temperature also phenol was found.

Therefore, from the results, it can be concluded that temperature affects the type of emissions emitted from the tested (bio-based) materials. This is explained by the fact that at an elevated temperature, chemical reactions take place, while also emissions with a higher boiling point can be found.

3.2. Quantitative analyses

3.2.1. Toluene versus group equivalents

By comparing the toluene and group equivalent method, the differences are shown using each method, indicating the variance in results. Table 7 presents the comparison in TIC counts [−] based on the concentration of the three test materials for TVOC (C6 to ~C12-C14/15) for the group equivalent approach versus the toluene equivalent approach. Table 8 presents the TVOC emission rates for the different materials investigated. Outcomes for both type of quantification techniques are presented. Finally, in Fig. 8 the comparison is shown for individual components as found in particle board.

Table 7 indicates that every compound, except terpenes, is underestimated when the toluene equivalent approach (aromatic group) is applied. As can be seen, there is a particularly large difference for alcohol. Due to the fact that alcohols were hard to measure with the GC/MS + ATD setup used in this experiment, this may have influenced the results. As it was not expected that many alcohols would be found in expanded cork, the accuracy of the GC/MS device for alcohols possible deviation in outcomes is not taken into account for further analysis.

Also ketones and aliphatic hydrocarbons present relatively large differences. At smaller TIC counts the relative differences are somewhat smaller, until the point where the calibration curve is not sufficiently accurate anymore for quantifying emissions. Of the selected compounds, only terpene emissions appear to be overestimated when applying the toluene equivalent approach.

In line with the results presented in Table 7, Table 8 shows that the TVOC emissions rate is affected by the approach used for the quantification. This is in line with the studie of Cariou et al. (Cariou et al., 2016). The group equivalent outcomes present higher emission rates for all materials investigated. As literature, generally, only presents toluene equivalent outcomes (Maskell et al., 2015), this would mean that emission rates may be underestimated. This of course depends on the material investigated.

Table 9
TVOC area specific emission rate - measured results versus references for biobased materials.

Material	TVOC Area specific emission rate [μg/m^{2}h^{-1}]	
	Day 3	Day 28
Expanded cork (group eq./toluene eq.)[a]	52/50	19/16
EPS insulation (group eq./toluene eq.)[a]	177/155	38/26
Particle board (group eq./toluene eq.)[a]	100/70	30/19
Cellulose flakes[b]	11	ND
Wool[b]	4	ND
Hemp fibre[b]	33	8
wood fibre/wool[b]	911	160
Hemp lime mix (330 kg/m³)[b]	34	ND
Hemp lime mix (275 kg/m³)[b]	28	ND
Rigid wood fibre[b]	14	9

[a] TVOC emissions based on C6-C14/15 instead of C6-C16 due to limitations desorption tubes.
[b] Maskell (Maskell et al., 2015).

As Fig. 8 shows, the difference can be linked to single target components. For particle board, the group equivalent, presents 2-propanol with a high indoor concentration rate, while with the toluene equivalent outcome is in the order of 90 % lower. This is explained by the fact that alcohol is significantly underestimated by the toluene equivalent as shown in Table 7. On the other hand, in line with the outcomes presented in Table 7, terpenes are overestimated with the toluene equivalent.

3.2.2. Indoor concentration emissions rates expanded cork

Table 9 presents the outcomes (in μg/m^{2}h^{-1}) for expanded cork, particle board and EPS, from the 28 days experiments. In Table 9, emission rates from other biobased materials from the research of Maskell (Maskell et al., 2015) are included for comparison.

As shown in Table 9, expanded cork has elevated emission rates compared to other biobased materials at day 3 and day 28, except for wood fibre/wood wool. The results of expanded cork, however, are lower compared to the conventional materials EPS and particle board.

Looking more closely to the indoor concentration in a standard reference room, the emission rates for expanded cork, from day 3 to day 14, are almost two to three times smaller than particle board and EPS insulation board, the reference materials, as is shown in Table 10. The concentration of expanded cork in a standard reference room decreased to 18 μg/m^3 after 28 days, which is well below the recommended reference value of 200–500 μg/m^3 of Molhave et al. (Mølhave, 1997).

The AgBB scheme, including the assessment of single VOC emissions based on LCI, has been used as a guideline for assessing the acceptability of the emission rates for TVOC and single VOC components in practice (eco-INSTITUT Germany GmbH, n.d.). A number of conditions are assumed in the analysis. The carcinogenic substances are only evaluated after 3 days and after 28 days. Other non-carcinogens only are evaluated after 28 days. The emissions of Semi VOC emission is not evaluated due to the limited VOC range of the airtoxic tubes.

Following these conditions, emissions from expanded cork (and particle board) meet the AgBB requirements. The TVOC and all single VOC emissions remained below the limits as stated by AgBB and LCI. After 28 days, most of the measured VOC emissions were below 1 μg/m^3. EPS does not meet these requirements. After 28 days, emission rates of benzene still exceeded carcinogenic limits.

In Table 11 an overview is shown of the measured VOC emissions from expanded cork. In bold emissions are shown that are potentially harmful at certain emission rates according to Agency for Toxic Substances and Disease Registry (ATSDR), European commission LCI and/or the World Health Organisation (WHO).

3.3. Limitations of the research

The analysis of the VOC emission from materials are encounter several uncertainties. An extensive review on these uncertainties is described in (de Kort, 2022).

Although material selection was performed carefully, material uncertainties can be expected, including (1) the production process influencing the type and amount of VOC emissions per product, (2) the age of the material and (3) limited information on what emissions can be expected from these materials.

Furthermore, the authors are aware that the measurement process can also influence the final results, including errors such as (1) measurement equipment (limited errors due to fine calibrations and verification of the system), (2) the assumption that the chosen loading factor does not have an influence on the final results (Que et al., 2013), (3) the sampling speed and time determining what emissions will be captured and (4) used sorption tubes, that were AIRtoxic tubes instead of generally used Tenax Tubes. This latter aspect may affect the final TVOC emission rates which can be higher than presented in this research. However, from literature study (de Kort, 2022) it could be concluded that expanded cork or any the reference materials applied would emit high amounts of emissions with carbon compounds above the maximum level of Air Toxic Tubes. The impact on the TVOC emissions is therefore considered as limited. However, on the impact of single VOC emissions, more research should be performed.

Table 10
TVOC indoor concentration rates expressed in toluene and group equivalents for the tested materials in a standard test room.

Day	TVOC emissions [μg/m³]					
	Particle board		Cork		EPS	
	Group eq.	Toluene eq.	Group eq.	Toluene eq.	Group eq.	Toluene eq.
Day 1	367	272	320	234	687	523
Day 3	201	141	111	107	356	310
Day 7	ND		77	63	263	231
Day 14	152	113	84	77	257	233
Day 21	71	50	58	49	159	130
Day 28	60	38	18	15	38	26
Healthy value TVOC according to WHO (World Health Organisation, 2010) after 28 days	<200					

Table 11
Overview of compounds as measured for Expanded cork and its toxicity.[a]

Expanded cork							
	3 days		28 days				Check/fail
Compound	Experiment [μg/m³]	C1 (<10 μg/m³)	Experiment [μg/m³]	C1 (<1 μg/m³)	LCI value [μg/m³]	$R < 1$	
1,2,3-trimethyl benzene	0.9	Not carcinogenic	1.0	Not carcinogenic	450	0	Approved
1,2,4 trimethyl benzene	0.9				450	0	Approved
1,3,5 trimethyl benzene	0.9		<1		450	0	Approved
2,4-dimethylpentane	3.1						Approved
2-Ethyl toluene	0.9		<1		550	0	Approved
3-Ethyl-2-methyl heptane	2.6						Approved
3-Methyl butanal	3.2						Approved
4-Ethyl toluene	0.9		<1		550	0	Approved
a-Pinene	3.8				2500	0	Approved
Benzene	0.9	approved	0	approved			Approved
b-Pinene	1.3	Not carcinogenic		Not carcinogenic	1400	0	Approved
Cumene	1.5				1700	0	Approved
Cyclohexane	22.0						Approved
Decanal	3.1		1.4		900	0	Approved
Decane	17.8		1.5				Approved
Dodecane	2.6						Approved
Ethylbenzene	0.9		1.1		850	0	Approved
Heptane	1.7						Approved
Hexane	3.1						Approved
Limonene	1.7				5000	0	Approved
Methyl benzene	0.9				450	0	Approved
Methylcyclopentane	3.9						Approved
Nonanal	4.8		1.9		900	0	Approved
Nonane	2.2						Approved
Nonene	2.0						Approved
Pentadecane	1.5		1.1				Approved
Propylbenzene	0.9		4.3		950	0	Approved
Styrene	0.9				250	0	Approved
Tetradecane	1.8		1.2				Approved
Toluene	11.5		1.0		2900	0	Approved
Tridecane	2.0		1.2				Approved
Undecane	2.4						Approved
Xylene	2.0				500	0	Approved
Sum of VOC with unknown LCI-value			1.1	Sum of VOC with unknown LCI < 100 μg/m³			APPROVED
							Approved

[a] In bold emissions are shown that are potentially harmful at certain emission rates according to Agency for Toxic Substances and Disease Registry (ATSDR), European commission LCI and/ or the World Health Organisation (WHO).

Though the calibration curves were developed carefully, potential uncertainties may arise due to (1) manual application of the multi-standard on desorption tubes, (2) a small deviation in the multi-standard itself, (3) the low detectability of alcohols with GC–MS and ATD, and (4) a deviation in the relative standard deviation of repeatability (RSD). This latter aspect needs more explanation. According to Yrieix (Yrieix et al., 2010) it is important to observe this value to express the closeness of agreement between sampling results. Preferably, the RSD should be <15 %. In Table 12 the values obtained in this research can be found. The RSD values are calculated using Eq. 4.

$$RSD\ [\%] = 100\ S/\bar{x} \quad (4)$$

With:
S: Standard deviation samples taken for calibration curve.
$\bar{x}$: average samples taken for calibration curve.
What can be seen is that the higher the emission value, the better the repeatability of the TIC response. This was also found in the study of Yrieix (Yrieix et al., 2010). Aldehyde emissions cause a relatively high RSD value. This has to do with the fact that the response of aldehydes using GC–MS with sorption tubes is less accurate than using DNPH coated cartridges according to NEN-EN ISO 16000-3:2011 (Yrieix et al., 2010; Salthammer and Mentese, 2008). This may result in deviations around 45 %. What also can be seen is that for most investigated compounds the RSD is above the preferred 15 % deviation. This has mainly to do with the application of the multi-standard on the sorption tubes. Due to the fact that this was done manually, low RSD values were hard to obtain, especially for low emission rates. The RSD values presented in Table 12 are therefore the best values that could be obtained in this study. The calibration curves developed for quantification in this study are based on these RSD values.

According to Yrieix (Yrieix et al., 2010) the advice is to set the uncertainty for single compounds emitted at high levels (>10 μg/m³) to 20

Table 12
Relative standard deviation of repeatability of group equivalents used in this study.

	Relative standard deviation of repeatability (RSD) [%]					
ng multi-standard	Heptane	Butanol	4-methyl-2-pentanone	Toluene	Limonene	Decanal
20	51	–	43	36.5	12.2	23.2
50	37.1	–	32.4	26.6	27	50.7
100	11.6	17.7	21.3	19.2	20.7	41.8
200	9.5	19.9	14.3	17.3	22.9	41.3
400	7.6	14.5	9.8	14.6	21.6	39.7

% and for single compounds emitted at low levels (<10 μg/m^3) to 40 %. Generally, this is in line with the RSD values found in this study for heptane, butanol, 4-methyl-2-pentanone, toluene and limonene (10 μg/m^3 = $^+/_-$ 150 ng). For aldehyde emissions (decanal) this is not representative.

Finally, finding low amounts of alcohol in the GC–MS was very difficult. Peaks with low amounts could be found in the samples of the materials and in the samples of the multi-standard. However, no clear calibration line could be made with low quantities of the multi-standard. Therefore, for alcohol the calibration curve was based on slightly higher carbon components to ensure a better calibration line. Therefore, errors may be found among the alcohols that may affect the results.

4. Conclusion

This study addressed the qualification and quantification of VOC emissions from expanded cork, a biobased building material, and various non-biobased building materials. Two different sampling methods were used for qualitatively identifying the VOC emissions and one sampling method for retrieving quantified emissions values, in order to assess the effect of VOC emissions from biobased and non-biobased materials on the indoor air quality.

Following the qualitative analysis, for the investigated biobased materials, the harmful substances furfural and phenol were not found at room temperature, but acetic acid, toluene and acetone were. At 45 °C also phenol was found. An elevated temperature therefore has an influence on the emitted VOC emissions from expanded cork.

Quantified VOC emissions rates from expanded cork, particle board and EPS showed that expanded cork and particle board did stay within health limit requirements. EPS insulation emitted elevated levels of benzene and therefore did not adhere to these requirements. Compared to other biobased materials studied in the research of Maskell et al. (Maskell et al., 2015), expanded cork emits higher values of TVOC after 3 and 28 days.

When comparing the toluene equivalent versus the group equivalent approach, in case of quantifying the emission rates, it can be concluded that VOC emissions generally are underestimated when applying the toluene equivalent approach. This is explained by the specific response of the GC–MS device to the various chemical groups. When using the group equivalent approach, more realistic values can be derived for aliphatic hydrocarbons, alcohols, terpenes, aldehydes and ketones.

In practice, this research adds to the knowledge base on VOC emissions from various biobased and non-biobased materials. The results can be used to create more awareness of the emission of building materials and its effect on the indoor air quality and therefore human health. Because knowledge of emitted components and emission rates from building materials is still limited continued analysis of building materials is advised, including biobased building materials. A next step in this is to investigate the materials in a full-sized test chamber and to include other parameters as well in the analysis, including relative humidity. In that analysis, the group equivalent approach is preferred over the toluene equivalent approach to quantify those emission rates. Furthermore, in future research more tests should be performed to increase the repeatability of outcomes.

CRediT authorship contribution statement

J.M.A. de Kort: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation. **F. Gauvin:** Writing – review & editing, Supervision, Methodology, Conceptualization. **M.G.L.C. Loomans:** Writing – review & editing, Supervision, Methodology, Conceptualization. **H.J.H. Brouwers:** Writing – review & editing, Supervision, Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

I have refered to my thesis which is an open acces. There all data can be found.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.167158.

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