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Manuscript title: Influence of Eco-materials on Indoor Air Quality

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Abstract

A growing strategy to reduce the energy consumption of buildings involves a combination of increased air tightness and high levels of insulation. However, an undesirable consequence of this approach is a deterioration of the Indoor Air Quality and accumulation of airborne pollutants, resulting from the reduction in ventilation. The chemical nature and concentration of indoor air pollutants is dependent on the building materials and activities of the occupiers. Recent studies have raised awareness of the effect of Indoor Air Quality on the perceived comfort levels, health and well-being of humans. This paper investigates the role of commercially available natural building materials including lime mortars, natural fibres and wood panels on the Indoor Air Quality. Initially the emissions of Volatile Organic Compounds (VOCs) from building materials were identified and measured. Subsequent tests then considered the adsorption and re-emission behaviour of four VOCs; toluene, limonene, dodecane and formaldehyde. The significance of this paper lies in its demonstration that emissions are dependent on the chemical composition of building materials and the production process, whereas the adsorption/desorption characteristics are related to material microstructure and polarity of the VOCs. The results allow the performance of a construction material, in terms of its influence on indoor air quality, to be deduced from a knowledge of chemical composition and microstructure. This paper provides a new approach for assessing the influence of different building materials on indoor air quality when exposed to gaseous pollutants.

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1. Introduction

Concerns about poor Indoor Air Quality (IAQ) have been rising due to its effect on health and well-being of building occupants that typically spend more than 80 % of their life indoors.[1-3] Poor IAQ has been linked to *Sick Building Syndrome* (SBS) and *Building Related Illness* (BRI) which has been defined as a range of symptoms associated with spending time in a building.[3-5] The symptoms associated with SBS and BRI include headaches, eye and respiratory irritation, dizziness and nausea, fatigue, difficulty in concentration, chest tightness, muscle aches, and in cases of prolonged exposure have the potential to cause chronic health effects. IAQ is strongly influenced by ventilation, temperature and humidity, lighting, airborne particles (*e.g.* dust) and airborne chemical pollutants (*e.g.* Volatile Organic Compounds, VOCs). In order to reduce the problems caused by poor IAQ, the ECO-SEE FP7 project aims to develop new eco-materials and components for the purpose of creating both healthier and more energy efficient buildings through hygrothermal (heat and moisture) regulation and the removal of airborne chemical pollutants through both passive capture and photocatalysis.[6,7] The adoption of natural materials in this project is driven by reducing the embodied energy and carbon impact of construction. This paper is focused on studying the role of natural building materials in improving the IAQ by studying emissions and adsorption/desorption of VOCs.

The World Health Organisation (WHO) classifies VOCs as organic compounds with boiling points from 50 °C to 260 °C; below 50 °C they are classed as very-VOCs (VVOCs). After a large number of studies on the effects of formaldehyde and VOCs on human health, the WHO guidelines[8] recommended a limit for formaldehyde concentration of 100 μg/m^3, and across

Europe guidelines for Total VOC (TVOC) concentration range from 200 to 500 μg/m^3 within indoor environments. Mølhave provided an approximate guideline on the potential impacts on health associated with levels of TVOCs as given in Table 1.[9]

VOCs found in an indoor environment can originate from a wide range of sources such as outdoor (traffic, industries), human activities (cleaning, cooking), furniture and building materials.[4, 10-12] Emissions from building materials are classified as primary or secondary. Primary emissions include the physical release of non-bound VOCs, which are present in a new product (e.g. additives, antioxidants, solvent reactants). Secondary emissions represent the release of chemically or physically bound VOCs, which can be produced by chemical reactions in the product or in the indoor environment such as oxidation, chemical or physical degradation or sorption processes.[13] The nature of VOCs emitted from products varies according to the chemical composition and production processes of the materials.[14] Terpenes (natural VOCs, such as α- and β-pinene), and aldehydes (such as hexanal and pentanal) are commonly found in wood based materials due to hydrolysis and oxidation processes of the hemicellulose.[15,16] Emissions of formaldehyde are observed in the case of wood fibre materials such as medium density fibreboard (MDF). This is related to the use of resins such as urea-formaldehyde to bind the wood fibres.[17,18] Aliphatic hydrocarbons (such as dodecane) and aromatic hydrocarbons (such as toluene, styrene, ethylbenzene) found in the indoor air are typically solvent residues from the manufacturing processes, paint solvents, silicone sealants, adhesives etc.[4,18]

When exposed to a polluted environment, building materials can act as a sink by adsorbing the airborne pollutants and re-emitting them later.[19,20] This phenomenon is known as the sink

effect and can significantly affect the concentrations of VOCs in indoor environments. Several mathematical models are being developed and studied to estimate the impact of the sink effect on the VOCs concentrations in indoor environments; however, there has been no study of the relationship between physical and chemical properties of the building materials and their sorption characteristics.[19-24] The sorption phenomena can occur through physical interactions – physisorption, or through chemical phenomenon– chemisorption.[25,26] Gunschera studied the physical adsorption behaviour of commercial zeolites when exposed to chlorobenzene, α-pinene, 2-ethoxyethylacetate and pentanal.[27] The authors correlated the VOCs' molecular 3D dimensions and structure with the dimensions of the zeolite pores and observed that zeolites with larger pores adsorbed high quantities of VOCs independently of the size and structure of VOC molecule, while zeolites with small pores showed low capacity to adsorb large VOC molecules. Elongated VOC molecules can also be adsorbed by smaller pores, but in less quantity when compared with zeolites with larger pores. At the end of their studies, the authors also observed that this behaviour was not observed in some materials and they suggest that other factors, such as polarities and specific surface area, can play an important role in the adsorption capacity of the zeolites. An example of chemisorption is the reaction between the proteins in sheep wool and formaldehyde.[28-30] Some other studies have been undertaken addressing sorption of VOCs by building materials, however there is no discussion regarding physical and chemical properties.[31-32] This paper reports the influence of lime mortar, natural fibre and wood based building materials on Indoor Air Quality. The study specifically quantifies VOC and formaldehyde emissions whilst

also investigating the sink behaviour of toluene, limonene, dodecane and formaldehyde using a purpose built environmental chamber.

2. Materials and experimental methodologies

2.1 Materials

Figure 1 shows the 15 natural building materials studied in this paper. These materials are commercially available and classified as either insulation, coatings or wood panels accordingly to their application. With the exception of sheep wool, all fibrous insulation materials were cellulose based and divided into two categories: flexible and rigid. Flexible insulation materials are essentially loose fibres and include the sheep wool, hemp fibres and wood fibres. Rigid insulation materials have a higher density compared to the loose fibre products and also incorporate a binder. In the case of hemp-lime, the binder used was a formulated air lime containing some hydraulic addition (Portland cement CEM I). The hemp-lime mix was prepared under laboratory conditions according to the manufacturer's instructions. In order to reduce any variation in VOC emissions which may be attributed to storage conditions and time since manufacture, the insulation materials tested were supplied directly from the production line.

The coatings tested were specified for internal use as either base or finishing coats. Coatings containing lime A and clay A are marketed for use as top/finishing coats whereas lime B and clay B are used as base coats. Lime A is a plaster based on air lime containing some hydraulic addition (Portland cement CEM I) and aggregate (sand) and was supplied pre-mixed as a proprietary product where only water additions were required prior to use. Lime B is a binder consisted of air lime containing some hydraulic addition (Portland cement CEM I) which was mixed with

either sand (Lime B + Sand) or hemp shives (Lime B + Hemp) as the aggregate. The binder used to formulate hemp-lime insulation material, described before, is different, with respect to composition and microstructure from both binders used in Lime A plaster and Lime B renders. All cast specimens were produced following the manufactures guidance for water additions, mixing times and where applicable sand or hemp additions.

Two types of commonly used wood panel were studied: Medium Density Fibreboard (MDF) and chipboard. The majority of MDF products available, such as panels and furniture, are covered in a resin coated paper layer for both moisture resistance and aesthetic appearance. A comparison between uncoated and coated wood panels was made allowing the effect of the coating to be evaluated. As was the case for the fiber board, wood panels were supplied directly from the production line by the manufacturer.

2.2 Indoor air quality assessment

Emissions and adsorption/desorption cycles were carried out in 2 L environmental chambers in accordance with methodology based on BS EN ISO 16000-9.[33] The custom-made rig is composed essentially of three main components: 1) pure air generator, 2) VOCs and formaldehyde chambers (used only for adsorption/desorption studies) and 2 L cylindrical chambers in which the materials are placed, Figure 2 a). The rig has capacity to test several materials simultaneously and to run one reference chamber (no material) for comparison. Two main lines are used in this assembly: 1) pure air line, where pure air flows directly from the generator to the chambers and 2) dopant air line, where pure air passes through the VOCs and formaldehyde (HCHO) sources and then to the

chambers. One valve was placed at the end of each line to control which whether pure air, or air containing the dopant, flowed through the chambers.

The temperature was maintained at 23 °C ± 2 °C and 50 % ± 5 % relative humidity. Mass flow controllers were placed after the pure air generator to adjust the air flow rate going to the VOCs and formaldehyde chambers (dopant air) and the air following directly to the 2 L chambers. The inlet air flow rate in the environmental chambers was kept at 200 mL/min (equivalent to an air change rate of 6 h^{-1}). All tubes, valves and joints used in the rig assembly were made of emission-free and non-adsorbing materials to avoid influences in the experiments. Volatile organic compounds were sampled on to Tenax TA® tubes and formaldehyde by using 2,4-Dinitrophenylhydrazine (DNPH) cartridges.

2.2.1 Emissions testing

VOCs and formaldehyde emissions were carried out using methodology based on the standard BS EN ISO 16000-9.[33] Specimens with nominal dimensions of 20 cm × 6 cm × 2-5 cm were enclosed by an emission free aluminium metal boat, so that emissions could occur only from the exposed surface into the pure air, as shown in Figure 2 b). Sampling points were carried out after 3 and 28 days of pure air passing through the chambers with an air flow rate of 50 mL/min for VOCs and 120 mL/min for formaldehyde.

2.2.2 Adsorption and re-emission behaviour

One insulation material, one coating and two wood panel samples were chosen to study the adsorption/desorption behaviour of VOCs and formaldehyde: Sheep wool, Lime A, uncoated

MDF and coated MDF. Three VOCs and formaldehyde were selected for this research with boiling points from – 19 °C to 216 °C as shown in Table 2.

The four VOCs tested represented the range of molar masses and chemical characteristics typically found within indoor environments. Formaldehyde is considered a very-VOC due to its very low boiling point. It is often found in indoor environments and can cause severe health effects.[8] Toluene is an aromatic hydrocarbon and its emissions sources can be solvents, tobacco smoke, cooking activities, fuels *etc*. Limonene is classified as terpene because it is produced by a variety of plants and some insects. This chemical compound was chosen because it is often found in indoor environments together with other terpenes (such as α-pinene) and furthermore it is widely used in cleaning products and indoor fresheners when a citrus fragrance is desired. Dodecane is a straight chain alkane composed only by carbon and hydrogen atoms. It was selected to represent the majority of alkanes emitted from building materials, fuels and solvents.

For the adsorption/desorption experiments, sources of VOCs and formaldehyde were used and placed in separated chambers, as shown in Figure 2 a), in order to produce a polluted air (called dopant air). A permeation tube of formaldehyde (classified as HRT – High Rate Disposable Permeation Tube) was acquired from *KIN-TEK*™ to be used as a source. Custom home-made diffusion tubes were used as VOCs sources. Prior to starting the adsorption/desorption experiment the mass loss of each source was calculated in a daily basis. Once the emission rate was demonstrated to be stable, signified by a constant mass loss, the source was used.

The adsorption phase initiated when dopant air started passing through the chambers. The chambers outlet air was then sampled at 1, 24, 48 and 72 hours with an air flow rate of 150

mL/min. The desorption phase was deemed to have started when the dopant air was turned off and pure air channelled to flow through the chambers. At this stage, some of the VOCs and formaldehyde adsorbed within the materials surface could start to be re-emitted. Samples were then drawn at 1, 24 and 48 h. Figure 3 shows the schematic of the 2 L chambers adsorption/desorption methodology.

To compare the adsorption/desorption behaviour of different materials a reference (empty) chamber was analysed at the same time.

When the dopant air flows through the chamber (**A**), some of the VOCs carried by the air will be adsorbed onto the material surface (**C**) and the rest will flow to the outlet of the chamber (**D**). The VOCs concentration in the outlet will be equal to the concentration inside the chamber (**B**). The sink effect is represented by the concentration **C**, which will be re-emitted later by the materials. As stated previously, for comparison purposes, one of the chambers was kept without any material (reference chamber). This chamber showed the maximum VOCs concentration (as emitted by the VOCs sources), and no sink effect was observed.

2.2.3 Chemical analysis

VOCs and formaldehyde analyses were carried out by using procedures accredited to BS EN ISO/IEC 17025.[34] The VOC analysis was undertaken by thermal desorption (TurboMatrix 350, Perkin Elmer) and gas chromatography (AutoSystem XL, Perkin Elmer) coupled with a mass spectrometer (TurboMass, Perkin Elmer) in accordance with BS ISO 16000-6.[35] This analysis allows the identification and quantification of the concentration of each individual VOC and also the TVOC trapped in the Tenax TA® tube. To ensure high quality of the results, prior to the VOC

analysis one standard quality sample made with known concentrations of n-hexane, toluene, limonene and hexadecane was analysed on the gas chromatograph, and another on the mass spectrometer. The formaldehyde sampled on to 2,4-DNPH cartridges was analysed by the High Performance Liquid Chromatography (Gilson Inc.) following BS ISO 16000-3.[36] Like the VOCs analysis, prior to the analysis of formaldehyde, standard solutions with known concentrations of formaldehyde were analysed. The formaldehyde desorption from the 2,4-DNPH cartridges was carried out by solvent extraction using acetonitrile (HiPerSolv CHROMANORM® ACS super gradient grade for HPLC, VWR chemicals). The overall accuracy of the emissions testing, adsorption/desorption experiment and VOCs and formaldehyde analysis is higher than 90%.

3. Results and discussion

3.1 Emissions testing

The specific emissions rates of formaldehyde ($\mu g/m^2/h$) of the six insulation materials, five coatings and four wood panels after 3 and 28 days are presented in Figure 4. Formaldehyde emissions rates decreased with time for all materials tested. A similar observation was made by Yu & Crump.[18] Wood fibre showed the lowest decrease of the formaldehyde emissions rate (~14 %) and both hemp limes showed the highest decrease, about 50 %. The highest emissions of formaldehyde were observed on the wood based panels, emitting up to 300 $\mu g/m^2/h$.

The higher formaldehyde emissions rate from the wood based materials may be related to the urea-formaldehyde resins used to bind the wood fibres. Sheep wool presented the lowest formaldehyde emission rate. By comparing coated wood panels with uncoated ones (such as MDF and chipboard) it is observed that uncoated wood panels have higher formaldehyde emissions

compared with those that are coated. This indicates that the paper coating on the surface of the wood panels acts as a barrier to the diffusion of formaldehyde through the material.

In Figure 5 the area specific emissions rate of VOCs from the 15 materials are presented. Apart from Wood fibre, materials showed low VOCs emissions rates. The main VOCs emitted were as follows:

- Wood based materials (insulation and wood panels): acetic acid, furfural, pentanal, hexanal, α-pinene, 3-carene, limonene, octanal, nonanal and decanal;
- Coatings: 1-butanol.

Acetic acid and furfural emitted by wood based materials such as hemp fibres, wood fibre and wood fibre board are associated with the degradation of the hemicellulose.[18] The origin of acetic acid is expected to be from the elimination of acetyl-groups and furfural is formed from pentoses and hexoses by elimination of water.[16] High emissions of the both of these VOCs are also associated to the heat-treated wood or kiln-dried products. An example of this is the wood fibre material, which emitted 911 $\mu g/m^2/h$ after 3 days and dropped to 160 $\mu g/m^2/h$ after 28 days.

As a summary, coating materials and sheep wool were shown to have the lowest emissions rates of formaldehyde and TVOCs. TVOC emissions of wood fibre material would be of slight concern as this has the potential to exceed the current IAQ guidelines. In a European Reference Room (CEN/TS 16516)[37] of 30 m^3 with all four walls covered entirely with wood fibre material, airborne concentrations of up to around 600 $\mu g/m^3$ of TVOC, due to emissions from the material, could be found. Besides wood fibre, all materials are well below the recommended guidelines.

3.2 Adsorption and re-emission behaviour

Figure 6 presents the curves of toluene, limonene, dodecane and formaldehyde concentrations with time in the reference chamber (empty chamber). VOCs and formaldehyde reached their maximum concentrations after 24 h of doping time at which point they remained stable until the adsorption phase finished after 144 h. Once the re-emission phase started, all concentrations dropped to 0 $\mu g/m^3$ after 1 h, except for dodecane, which may be due to its higher molecular weight compared to the other chemical compounds. The maximum concentration of each VOC and formaldehyde in the reference chamber was limited by the emission rate from the respective source.

3.2.1 Materials discussion

The comparison of the behaviour of four selected building materials to adsorb and re-emit each individual VOC and formaldehyde is presented in Figure 7. The materials studied in this experiment were uncoated MDF, coated MDF, Lime A and sheep wool.

Uncoated MDF showed similar concentrations to the reference chamber during the experiment for the three VOCs. This means that this wood panel has low capacity to adsorb gases. On the other hand, coated MDF adsorbed limonene, dodecane and formaldehyde until 48 h of the experiment had elapsed, and then showed the similar value of concentration as the reference chamber, meaning that the material reached saturation.

The Lime A sample showed a high adsorption rate and a low desorption rate – *i.e.* a good sink effect, which means that it is a good material to adsorb VOCs quickly and store them, thereby

improving the IAQ in a real case. The highest sink effect of Lime A was observed for dodecane and formaldehyde, followed by limonene and toluene. This sink effect can be correlated to the large surface area of the porous matrix of the lime mortar and to the affinity between VOCs and SiO_2 (silica from the sand aggregate).[38,39]

Sheep wool showed a high capacity to adsorb formaldehyde, confirming the chemisorption processes between its proteins and formaldehyde as observed by Curling *et al.*[29] In the desorption phase, the effect of the toluene, limonene and dodecane molecular mass can be observed in all materials. VOCs with small molecular mass such as toluene (92.14 g/mol, Table 2) were completely desorbed from the materials' surface after 24 h. On the other hand, dodecane (170.34 g/mol) was not totally desorbed from Lime A and uncoated MDF even after 48 h of the desorption phase.

4. Conclusions

Volatile organic compounds and formaldehyde emissions of 16 different building materials (insulation, coatings and wood panels) were successfully analysed after 3 and 28 days of testing as required by the BS EN ISO 16000-9:2006. The results, for the first time, definitively show that the majority of the materials can be classified as low emitting in terms of formaldehyde and TVOCs and emissions are related with chemical composition.

Wood panels showed higher formaldehyde emissions due to the urea-formaldehyde resin used to bind the wood fibres. The degradation of the hemicellulose led to the emission of acetic acid and furfural. This demonstrated the secondary emission process. Inorganic materials such as lime mortars and clays showed negligible emissions of formaldehyde and VOCs. Coated wood panels

showed lower formaldehyde emissions than the uncoated ones because the paper coating acted as a barrier to formaldehyde diffusion. Furthermore, chipboard panels showed higher formaldehyde emissions compared to MDF which was attributed to its larger microstructural pores.

Adsorption/desorption behaviour of two wood panels, one lime mortar and sheep wool was successfully analysed and compared. The adsorption mechanism of VOCs and formaldehyde is mainly physisorption however, sheep wool proved to be a good sink for formaldehyde, confirming the chemisorption processes between its proteins and formaldehyde. The high sink effect of Lime A can be due to two factors: good porous microstructure leading to a high surface area and interactions between SiO_2 and VOCs or formaldehyde. Sorption mechanism depends on a material's properties, mainly chemical composition and porosity/microstructure and the VOC/VVOC's physicochemical properties.

The 15 building materials showed to be of low-impact in an indoor environment due to their low emissions of formaldehyde and volatile organic compounds. As shown in this paper, materials such as lime mortar and sheep wool show potential to improve indoor air quality due to their ability to adsorb formaldehyde and volatile organic compounds, also called sink effect. The passive improvement of the Indoor Air Quality is a very important feature for the future of buildings design and construction. Unlike control of VOC's using photocatalytic coatings, passive improvement does not require a source of light for the adsorption of VOCs and formaldehyde therefore broadening the range of applications where it may be effective.

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Table 1. Impact of TVOC concentration on human health.[9]

TVOC concentrations (μg/m^3)	Health impact
< 200	No irritation or discomfort expected
200 to 3,000	Irritation and discomfort may be possible
3,000 to 25,000	Discomfort expected and headache possible
> 25,000	Toxic range where other neurotoxic effects may occur

Table 2. Physicochemical properties of formaldehyde, toluene, limonene and dodecane.

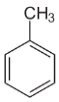
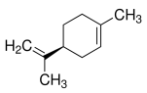
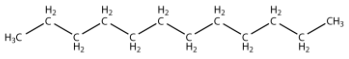
	Formaldehyde	Toluene	Limonene	Dodecane
Molecular structure				
Chemical conformation	Simple	Aromatic	Cyclic	Straight chain
Polarity	Polar	Non-polar	Non-polar	Non-polar
Molar mass (g/mol)	30.03	92.14	136.24	170.34
Boiling point (°C)	-19	111	176	216

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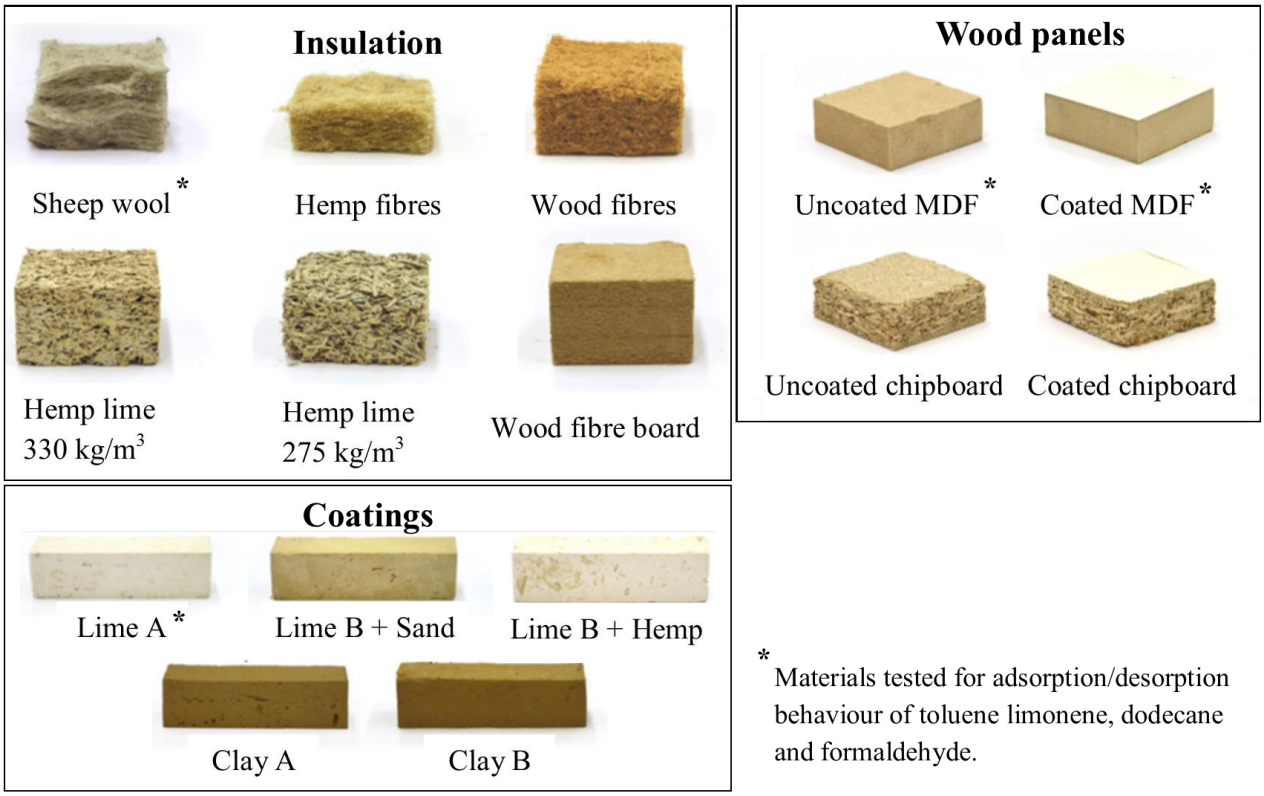
Figure 1. Materials studied for emissions of VOCs and formaldehyde and adsorption/desorption behaviour (indicated with *).



* Materials tested for adsorption/desorption behaviour of toluene limonene, dodecane and formaldehyde.

Figure 2. a) Schematic of the rig for emissions testing and adsorption/re-emission experiment. b) Samples placed in aluminium boats for testing. (i) uncoated MDF, (ii) coated MDF, (iii) lime A, (iv) sheep wool.

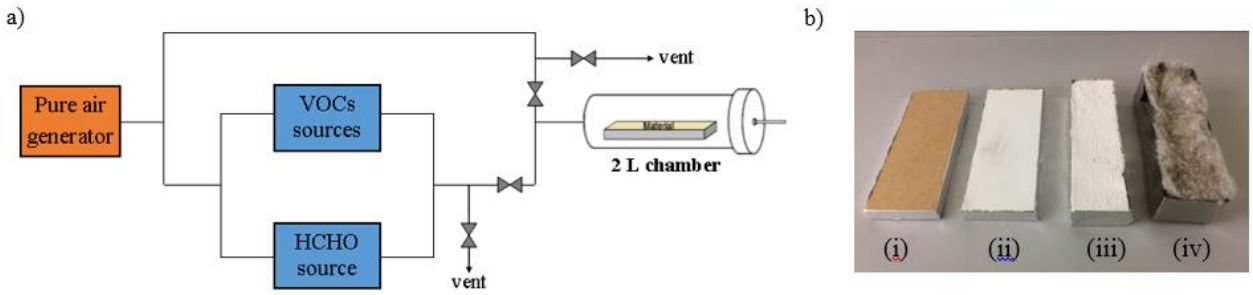


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Figure 3. Schematic of the 2 L chambers experiment.

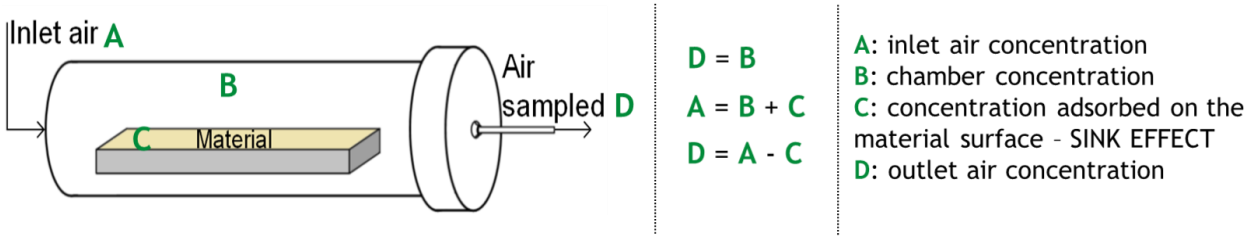

Figure 4. Area specific emissions rates of formaldehyde by the insulation, coating and wood based panels materials.

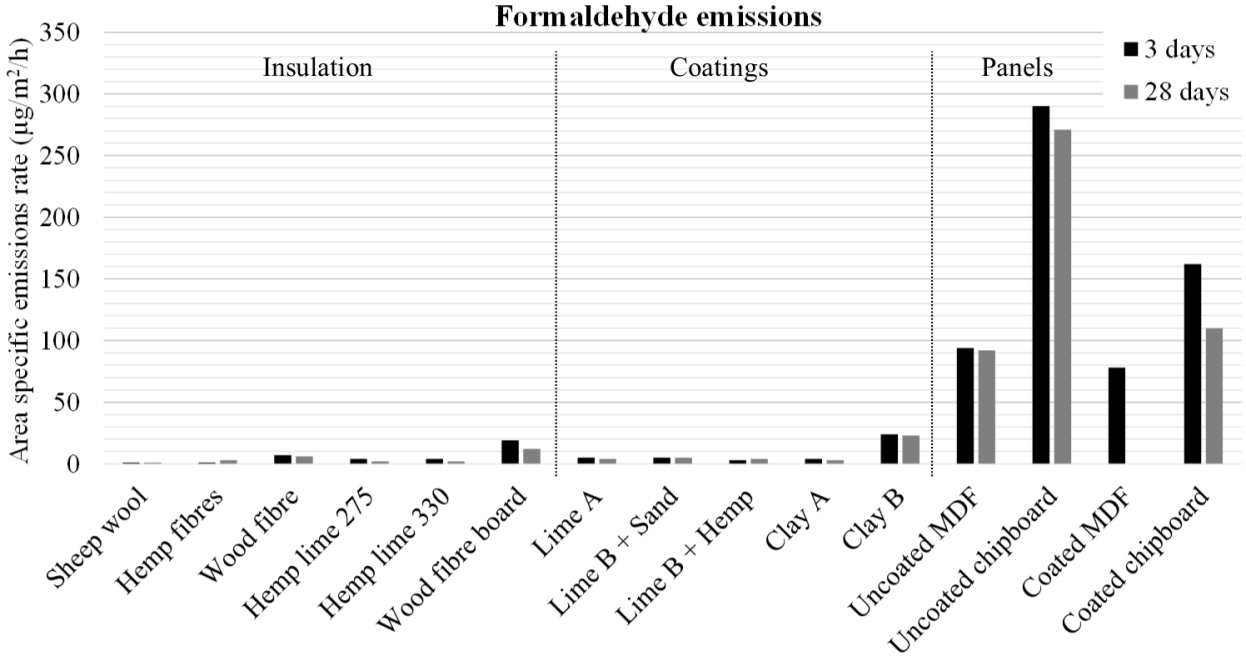

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Figure 5. Area specific emissions rates of VOCs by the insulation, coating and wood based panel materials.

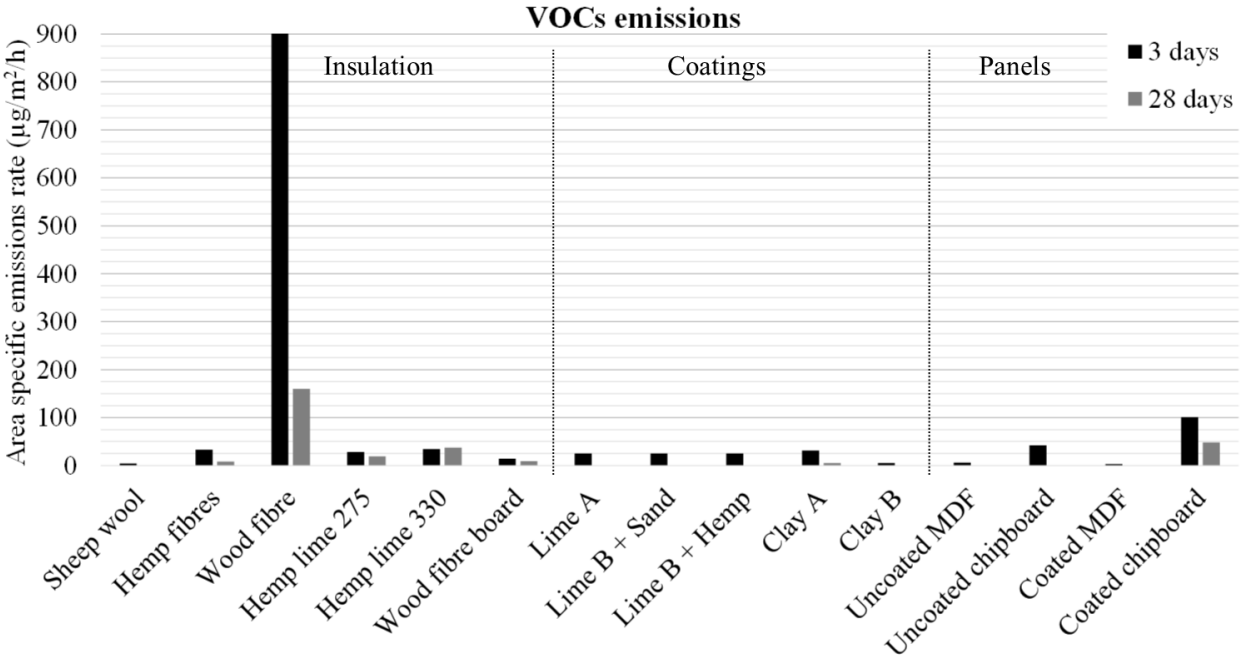

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Figure 6. Reference chamber adsorption and re-emission curves.

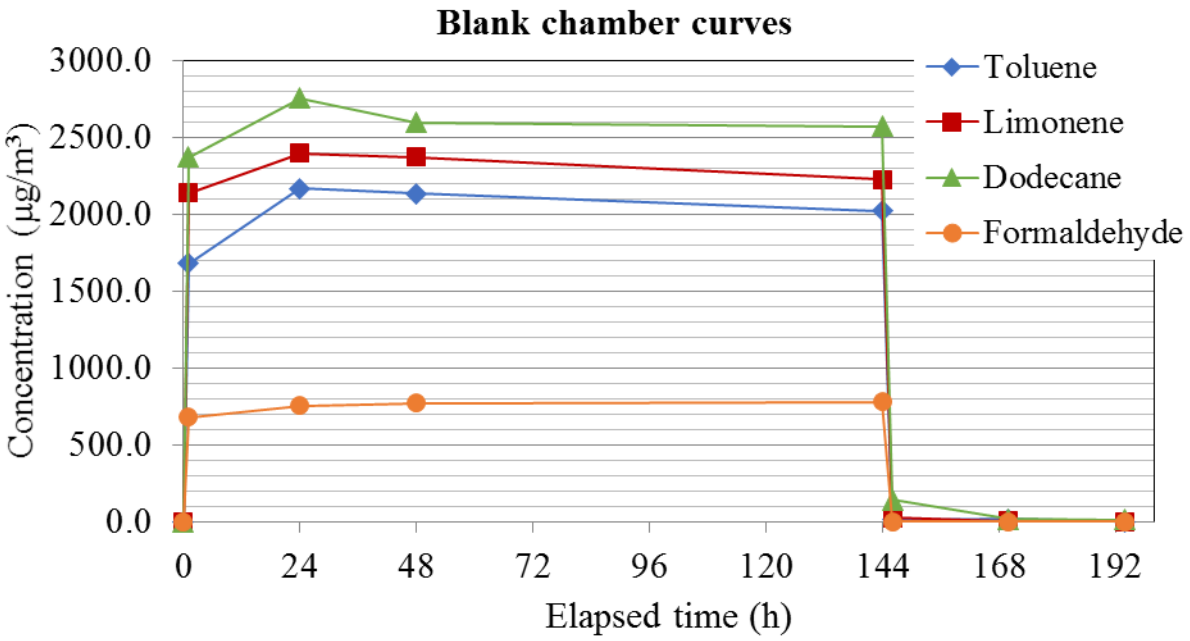

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Figure 7. Adsorption/desorption curves of toluene, limonene, dodecane and formaldehyde from uncoated MDF (U MDF), coated MDF (C MDF), Lime A and sheep wool.