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Evaluation of VOC Emissions from Building Finishing Materials Using a Small Chamber and VOC Analyser

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VOC analyser · VOCs · 20 L small chamber · building finishing materials

Abstract
A test chamber method was developed to provide a small and simple emission testing facility capable of testing construction products over a range of climatic parameters, such as temperature, ventilation rate and air velocity, that could be varied independently around typical indoor values. Volatile organic compounds (VOCs), specifically the aromatic hydrocarbons fraction, were measured using a "VOC analyser". This portable measuring equipment is a simple gas-chromatograph which is designed to measure certain aromatic compounds in indoor air. Compounds separated through the column are detected by a semiconductor gas sensor and the concentration of the target compounds automatically calculated and shown. Correlation of the results from this analyser with those from GC/MS was greater than 99%. We have examined the compounds emitted from various building materials, such as medium density fibreboard, particleboard (PB) and wood-based flooring (laminate flooring and engineered flooring), using test methods with a desiccator and 20 L small chamber for analysis of VOCs and formaldehyde emission levels. Over 2 and 7 days, we tested the change of VOC emissions with time and compared the results for each material.

Introduction

Wood-based panels bonded with urea formaldehyde resin emit formaldehyde which is toxic and has an associated possible health hazard. This could act as an obstacle to their acceptance by the public, given the prevailing climate of environmental awareness and concern. As a result, the European and Northern American governments have already, or are about to, impose regulations limiting the emission of formaldehyde from building materials and from the materials used for the manufacture of furniture and fittings [1]. Many consumer products containing formaldehyde-based resins release formaldehyde vapour, leading to consumer dissatisfaction and health-related complaints. In fact, those consumer products, specifically construction materials, are the major source of formaldehyde in the indoor environment [2]. Formaldehyde emissions have resulted in various symptoms, the most common of which is irritation of the eyes and the upper respiratory tract. High levels of formaldehyde have been found to produce nasal carcinomas in mice and rats after exposure to 14.1 $mg{\cdot}L^{-1}$

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and 5.6 mg·L^{-1} of formaldehyde, respectively, over a long period of time. These findings have led to an intensified interest in formaldehyde in the indoor environment.

Over the past decade, researchers have developed various techniques for measuring emissions of volatile organic compounds (VOCs) from building materials. An ASTM standard guide, a guideline from the Commission of the European Communities (ASTM, 1992; CEC, 1992) and a European preliminary standard ENV 13419, part 13 (CEN, 1998) have been published for such tests (see [3] for details). The techniques for testing emissions from building materials are important for manufacturers, indoor air quality investigators and researchers. Tests are necessary to quantify the impact of construction products on indoor air quality.

The standard method for measuring emission from wood based panels is to use a test chamber. Three different sizes, ≥12 m^3, 1 m^3 and 0.225 m^3, are proposed in the new European standard prEN 717-1 (prEN 717-1, 1997) for formaldehyde emission determination (see [4] for details). In Sweden the emission test is performed in a 1 m^3 chamber according to their standard SS 27 02 36 [5].

As measuring the formaldehyde emission in a chamber takes time and requires specialised and expensive equipment, simpler laboratory methods which can be used for in homogeneous products to give results with good correlation to the chamber methods are needed. Several methods have been used mainly for the determination of formaldehyde emission from particleboard (PB) and a good correlation has been found between the chamber, perforator and flask methods [4]. There is still a need for smaller, more flexible methods that correlate well with results from emission tests in chambers. Other methods like the perforator (EN 120), flask (EN 717-3) and gas analysis (EN 717-2) have been used, but they are hard to correlate to the chamber method as the tests are done using an extraction technique or are made at high humidity and temperature [6].

The Korean government started controlling indoor air quality in 2004. The law from the Ministry of Environment regulates the use of building materials that emit pollutants. The use of materials with a formaldehyde emission level above 1.25 mg·m^{-2}·h (JIS A 1901, small chamber method) is prohibited. This is the E_2 grade (>5.0 mg·L^{-1}) when the desiccator method is used (JIS A 1460).

The concern of most suppliers and people today is how to reduce pollutants from building materials and how to control indoor air quality [7]. In renovated or completely new buildings, levels of indoor air pollutants, especially VOCs, resulting from emission from construction and building materials are often several orders of magnitude higher than the VOC levels in buildings that have aged in normal use for a time after new works [8–11]. Furthermore, action has been taken on standardisation of emission testing and chemical analysis, conducted through the Ministry of the Environment, where the 20 L small chamber method has been applied. A 20 L small chamber was developed in Japan with its performance in compliance with ASTM (1996, 1997) [12,13], ECA reports (1989, 1991, 1993, 1995) [14–17], and ENV 13419-1 (1999) [18]. Although various chamber sizes have been used, the 20 L chamber was used in this paper because it will soon be the standard in Korea. Only the desiccator method (JIS A 1460) has been standardised so far. In this study a 20 L small chamber was used to measure VOC emissions from wood-based composites used as building materials. Furthermore, this article describes a method to determine formaldehyde emission with the VOC analyser and how this correlates to the desiccator method for different building products.

Experimental

The air control system for the test consisted of an air supply unit, a humidifier and pumps. A 20 L small chamber was set up in a temperature-controlled climate chamber. Purified air was used for ventilation. Figure 1 shows a schematic diagram of the 20 L small chamber system. The stainless steel sealed box was used to prevent the cut edge effect and so allow chemical emission only from the surface of one side of the test piece. When two sealed boxes were used, the total surface area was 0.044 m^2 and the loading was 2.2 m^2·m^{-3} [19].

The VOC analyser (ABILIT Corporation, Osaka, Japan) is a portable item of equipment designed to measure the four main aromatic hydrocarbon gases in petrol (BTEX): benzene, toluene, ethylbenzene and xylene. With the VOC analyser, a high resolution semiconductor gas sensor eliminates the need for a carrier gas which is required for conventional gas chromatographs. In addition, since the semiconductor gas sensor is supersensitive to gaseous components, it is not necessary to use a conventional gas concentrator or other complicated apparatus. A special column is used to separate the four aromatic hydrocarbon compounds. The overall principle of the system is similar to that of a gas chromatograph/mass spectrometer (GC/MS). Compared with other measurement methods, use of this analyser is useful for

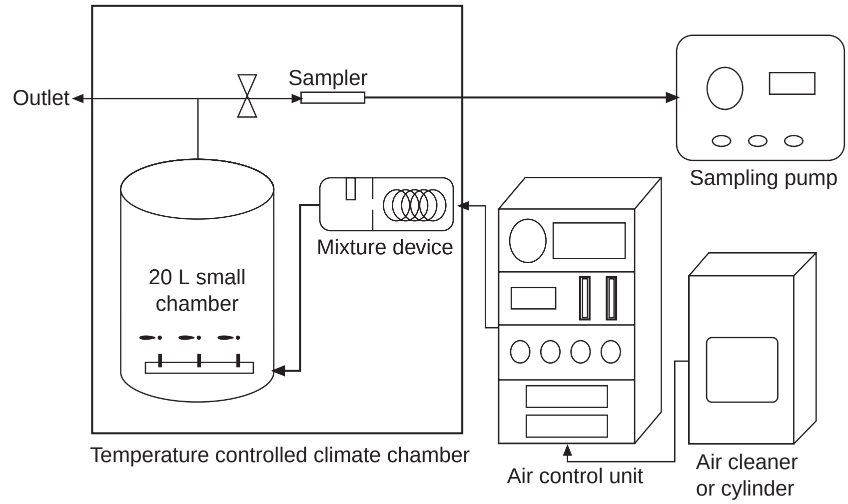


Fig. 1. Schematic diagram of a 20L small chamber.

measuring VOCs in new buildings because it is easy to get results directly in the field and do repeat tests. As an extension of its use it can be used to monitor the harmful emission of gaseous products during the manufacture of adhesives, paint, furniture and building materials.

The benefits of using the instrument include:

- Display of gas concentrations in standard units of ppm and mg per 10 ml
- Precise measurement accuracy with the simplified gas chromatograph method
- No carrier gas required
- Not affected by ambient temperature and humidity
- Short standby time of 30 min or less
- Short measuring time of 8 min.

Materials

For wood-based composites, we chose laminate flooring and engineered flooring. Currently, these are extensively used in new apartment interiors and in the remodelling market in Korea. Laminate flooring is composed of waterproof, high-density fibreboard (HDF) as core material, overlay paper, deco paper and valance paper. Each paper is impregnated with melamine-papers pressed at about 200 °C in the order shown in Figure 2. Finally, the side and edge of the product is processed by the tenoner. In the case of engineered flooring, a fancy, 0.5 mm veneer of a wood such as birch, oak, beach, cherry or maple is glued on a 7.2 mm plywood sheet and pressed at about 160 °C. An ultra-violet (UV) curable coating is applied to this fancy veneer. The structures of both flooring products are shown in Figure 2. For comparison with flooring, we used non-veneered MDF and PB as furniture material and other wood-based composites. Both were 18 mm thick. The moisture content of these materials is shown in Table 1.

Table 1 Moisture content of materials

Materials	Moisture content (%)
Laminate flooring	7.2 ± 0.7
Engineered flooring	6.5 ± 0.5
MDF	7.6 ± 0.8
PB	7.9 ± 0.9

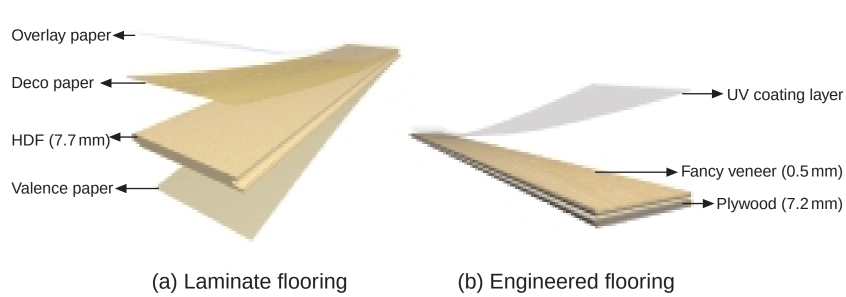


Fig. 2. Structure of laminate flooring and engineered flooring.

The Desiccator Method

The Japanese standard method with a desiccator (JIS A 1460) was used to determine the formaldehyde emissions from the laminate flooring, plywood flooring and MDF. This test to determine the quantity of formaldehyde emitted from building boards is carried out using a glass desiccator. The quantity of formaldehyde emitted is obtained from the concentration of formaldehyde absorbed in distilled water or deionised water when the test pieces with specified surface area placed in the desiccator which is filled with the specified amount of distilled water or deionised water and left for 24 h. Analysis to determine the concentration of formaldehyde dissolved in the distilled or deionised water is based on the Hantzsch reaction in which the formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine(DDL) [20].

The 24 h desiccator method uses a common glass desiccator with a volume of 10 L. Ten test specimens, with dimensions of 5 × 15 cm, were positioned in the desiccator. The edges of samples were sealed with polyethylene wax which melted at 60 °C. The test lasted for 24 h at a temperature of 20 °C. The formaldehyde emitted was absorbed in a water-filled Petri dish and then analysed by the chromotropic acid method. We set the temperature of the dry oven to 20 °C, 37 °C and 50 °C. First, the materials: laminate flooring, plywood flooring and MDF, were conditioned in the dry oven at 37 °C for 1 day and 7 days and then at 50 °C for a further day to mimic the condition found with the Korean under-floor heating system known as ONDOL. Second, we measured formaldehyde emission from these materials by JIS A 1460.

The 20 L Small Chamber Method for Collection of Gas Samples

Before setting up the chamber and sealed boxes, they were washed with water and baked out in an oven at 260 °C to eliminate any pollutants from the chamber itself. The 20 L small chamber was supplied with purified and humidified air at a ventilation rate of 0.5 ACH through an air-flow of $0.01\,m^3{\cdot}h^{-1}$. The temperature of 25 ± 1 °C and 50 ± 5% relative humidity (RH) inside the chamber were kept constant. The test was conducted using two samples of size 0.147 × 0.147 m (total $0.0432\,m^2$) at a sample loading factor of $2.16\,m^2{\cdot}m^{-3}$. The air inside the chamber was sampled after 12 h.

The VOCs were analysed using an Automatic Thermal Desorption/MS (TDS/GC-MS). The instruments used were the TDS (Perkin Elmer ATD400) and GC/MS (HP6890/Agilent 5973). Chromatography was through a RTX-1 capillary column (105 m by 0.32 mm by 3 μm) using helium (99.99%) as carrier gas, temperature programmed at 40 °C (5 min) then stepped up to 70 °C (5 min), then 150 °C (5 min), then 200 °C (5 min), 220 °C (5 min) and finally 240 °C (5 min).

Mass spectrometric analysis was in the electron ion (EI) mode at 70 eV with detection by total ion current (TIC scan) for m/z 35–350.

In this paper, TVOC was defined by conversion of all areas of the peaks between C_6 and C_{16} to concentrations using the toluene response factor. Any peak area under ten was defined as the limit of detection. The gas was sampled by absorption on Tenax-TA, 7 days after the sample specimens were installed into the 20 L small chamber, according to the regulation from the Ministry of the Environment, Korea.

Emission Factor

The calculation of the emission factor (EF) is explained in ASTM D5116 [12]. Two technical terms are commonly used to describe the rate of emission from indoor materials, EF and ER, which are related as follows:

$$ER = A(EF) \quad (1)$$

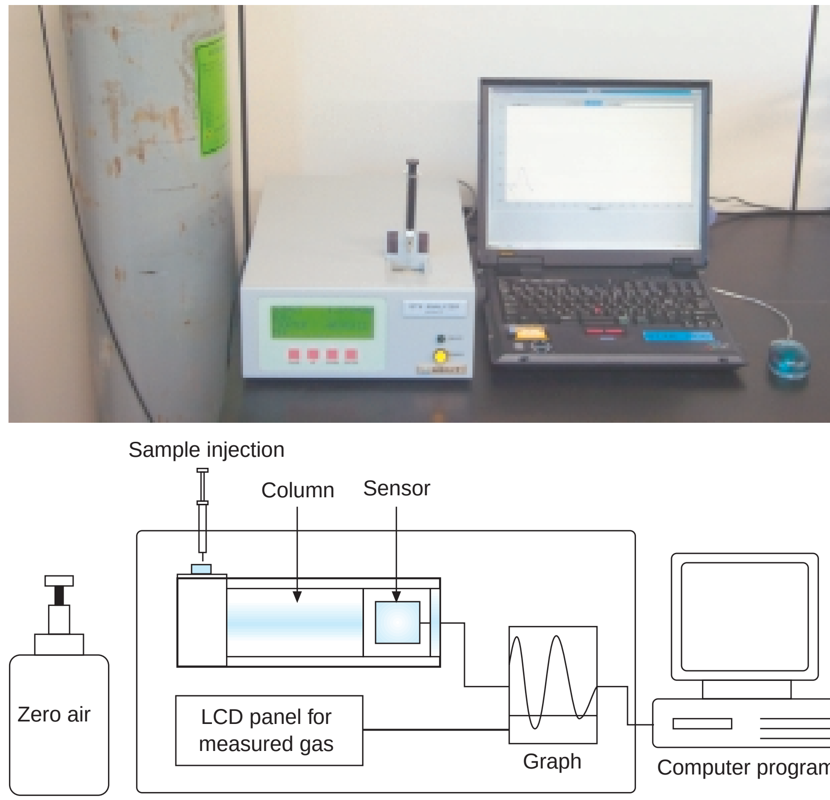
Where:
ER is the emission rate ($mg{\cdot}h^{-1}$)
A is the source area (m^2)
EF is the emission factor ($mg{\cdot}m^{-2}h$)

Thus, ER can be applied to both area sources and non-area sources, whereas EFs are reported as mass/mass/time, or in the case of caulk beads, mass/length/time, when a standard bead diameter is used. In the remainder of the cases, only EF is used in the examples.

VOC Analysis

The gases containing VOC emissions for the VOC analyser were collected from both the 20 L chamber and desiccator, that were used to measure formaldehyde in the laminate flooring, plywood flooring and MDF tests. In a variation of JIS A 1460, we placed a water-filled Petri dish in the desiccator used to collect the air sample for the VOC analyser (this removes formaldehyde). The VOC analyser and its structure are shown in Figure 3. There are three steps in the measuring procedures. First, the plastic syringe that comes with the product is inserted into the 20 L chamber or desiccator. A hole for the

Fig. 3. VOC analyser and a schematic diagram.



syringe is made on the top of the desiccator. Then, the plunger is slowly pulled, pushed in again, and pulled out for the second time before removing the syringe from the chamber and desiccator. If the top of the syringe is wet, it is wiped dry with a tissue. The dedicated needle is attached and 0.5 ml of the sampled gas is ejected by pushing the plunger. The remaining gas is then injected into the inlet on the main unit of the VOC analyser, after which the measurement starts automatically.

VOC Analysis by GC/MS for Correlation of the Methods

To get correlation data for the GC/MS technique and the other VOC analysis, the system was set up as shown in Figure 4. First, we collected gas containing standards diluted with air to give a concentration of 0.005, 0.1 and 1.0 ppm using a 5 L Teflon bag. The gases were then sampled through both a Tenax-TA tube and into the syringe used for analysis. The Tenax-TA tube was analysed using GC/MS while the air sample in the syringe was analysed through the VOC analyser.

Chemical compound measurements from analysis of the emissions were made according to a validated and accredited method for the thermal desorption of Tenax-TA tubes. Active sampling of VOCs collected in the 5 L teflon bag was performed by using a pump to draw chamber air through a Tenax-TA adsorbent tube at a rate of 1 L over 10 min. The Tenax-TA (100 mg) was held as the adsorbent in a steel tube designed to fit the Perkin Elmer ATD 400 equipment used for thermal desorption.

Results

The initial formaldehyde emission results obtained by the desiccator method for each flooring material and from MDF and PB, are shown in Figure 5. Each material was measured three times. The emission from the laminate flooring gave an air concentration of 0.92 ppm and from the engineered flooring 0.66 ppm. The formaldehyde emission of both these floor materials was under the standard for E1. Generally, laminate flooring is manufactured as E1 grade in Europe. The greatest effect on formaldehyde emission in laminate flooring is exerted by the high density fibreboard (HDF) core of the laminate. This is made using urea-formaldehyde resin or melamine-urea-formaldehyde (MUF) condensed resin. This grade of laminate flooring can be used for

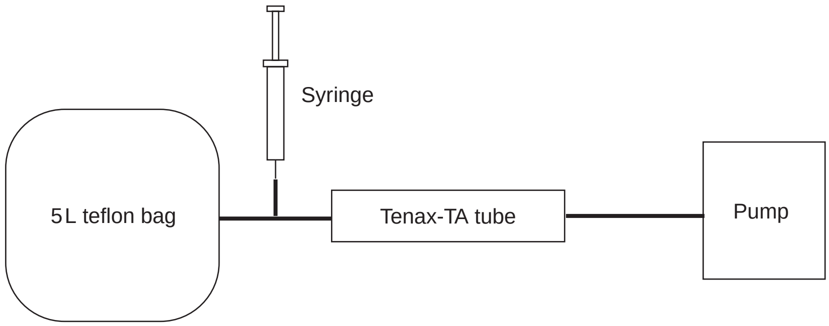


Fig. 4. Collection of gases for comparison between GC/MS and VOC analysis.

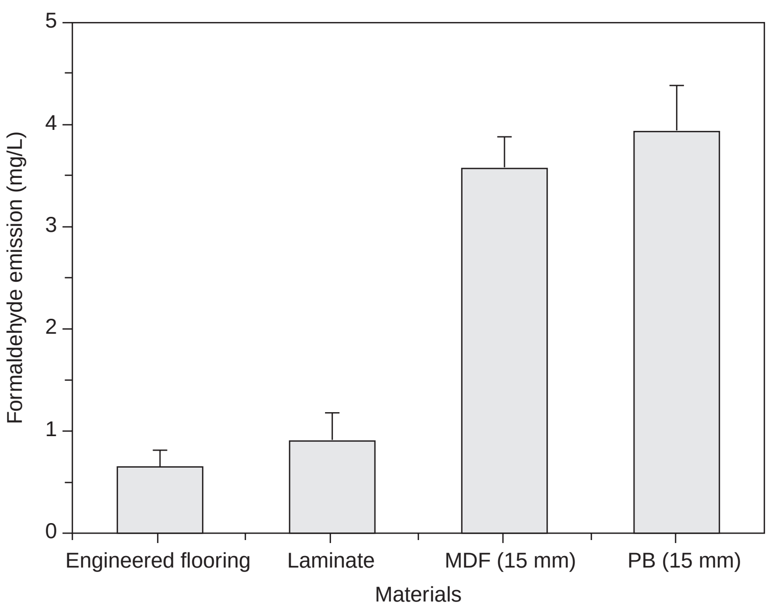


Fig. 5. Formaldehyde emission from each building and furniture material by the desiccator method [21].

residences. Because the plywood that was used as the core in engineered flooring was glued with phenol-formaldehyde resin, its formaldehyde emission was lower than that of laminate flooring. The E1 grade of wooden flooring materials has been in common circulation in Korea. To maintain low formaldehyde levels to achieve E1 grade, the manufacturers use MUF condensed resin for HDF and phenol-formaldehyde resin for plywood as adhesives. On the other hand, the MDF and PB used in the experiment, as manufactured had a formaldehyde emission that made it E2 grade. Emission was 3.59 ppm for MDF and 3.95 ppm for PB. In Korea, MDF and PB of an emission grade more than E2 are sold and used for furniture manufacture.

TVOC chromatograms of wood-based composites, 7 days after sample installation, are shown in Figure 6. Koontz and Hoag [22] reported that unfinished PB and MDF from North America emitted many VOCs in addition to formaldehyde, and often at greater concentrations than formaldehyde. Major VOCs reported were (in approximate order of amounts emitted): acetone, hexanal, pentanal, benzaldehyde, pentanol, heptanal, pinenes, nonanal and octanol. In this experiment, it was found that the PB specimens emitted hexanal, pinenes, pentanal, nonanal, heptanal, octanol and so on. This was comparable to engineered flooring. Because the UV-cured coated surface of the engineered flooring was bare like the unfinished surface of the PB, unusual (for wood-based materials) VOCs were emitted such as methyl acetate, vinyl acetate, toluene, methyl butyrate, pentanoic acid, methyl ester and copaene. TVOC EFs of each wood-based composite, between C_6 and C_{16}, are shown in Figure 7. The PB specimens emitted much greater amounts of VOCs than the engineered flooring specimens. This result was similar to the finding with formaldehyde EF. However, there are many natural VOCs emitted from PB such as α- and β-pinene. When TVOC is calculated between C_6 and C_{16}, these natural

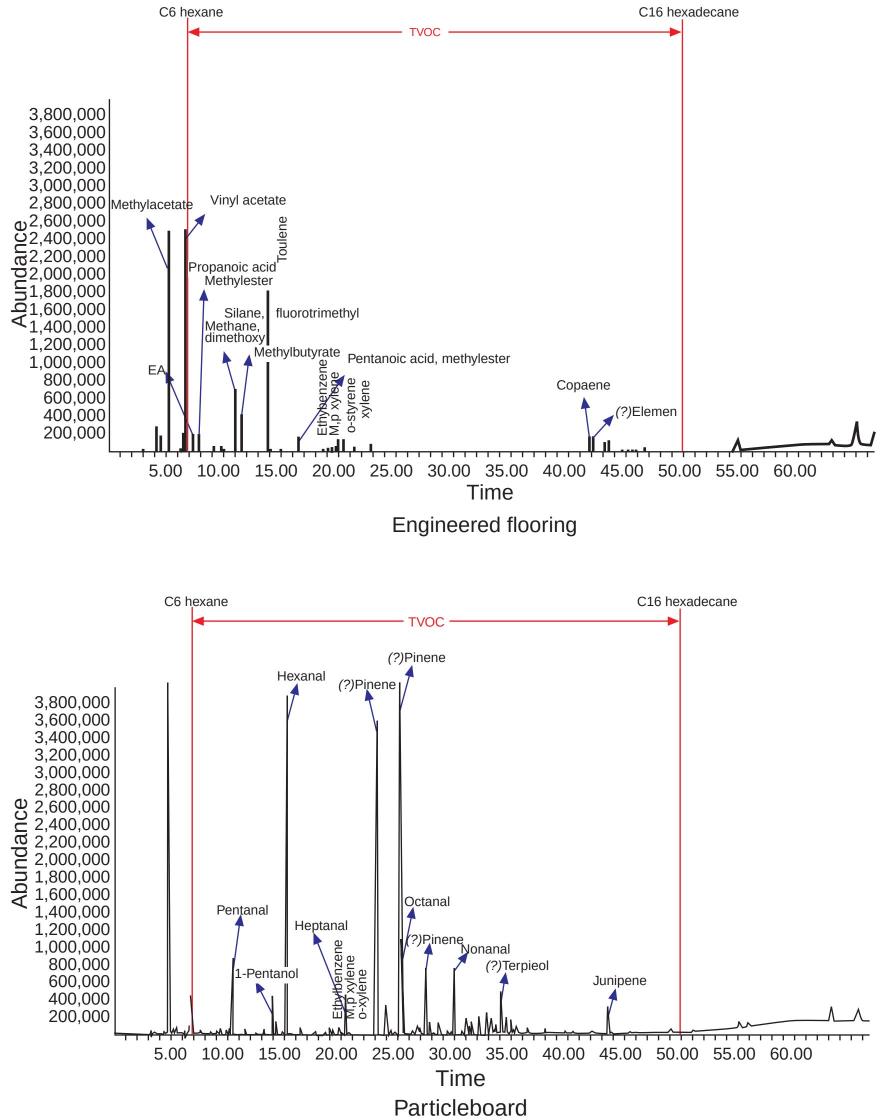


Fig. 6. TVOC chromatograms of engineered flooring and particleboard [21].

VOCs are included, which explains why TVOC EF from PB was higher than that from engineered flooring. In Korea, the Ministry of the Environment provides guidelines for VOC emissions from building materials presented as TVOC. Even natural VOCs from wood are considered to be harmful and are included in the TVOC calculation. Consequently, it is necessary to consider natural VOCs when reassessing the regulations governing VOC emissions from building materials.

To apply the results of the VOC analyser to this experiment, correlation between the results of the GC/MS and VOC analyser was important. We found that there was good correlation between the results from the GC/MS and the measurements made by the VOC analyser, as shown in Figure 8. BTEX measured by GC/MS were directly proportional to the results from the VOC analyser analysis of these compounds as indicated by the numerical formula $Y = 1.20X - 0.37$, where Y is the result from the GC/MS and X is the result from the VOC analyser.

To determine VOC emissions using the VOC analyser, we collected emitted compounds in the desiccator that had been set-up with MDF, laminate flooring and engineered flooring. The difference from the standard desiccator method for formaldehyde emission was that there was no 300 ml water dish to prevent the gases from dissolving in the water. Figure 9 shows results of 2-day VOC emissions from 15 mm MDF. For the first 5 h, the emission rates were high. The gases emitted from a board of dimension 1800 mm^2 filled the 10 L desiccator in 5 h and then slightly increased. The increase finally stopped after 1 day, after which the volume was maintained. Among the VOCs measurable by this analyser, the highest emission was from xylene. About 3500 μg/m³ of xylene was emitted from non-treatment MDF compared to under 1000 μg/m³ for the three other VOCs: toluene, ethyl benzene and styrene. Comparison of formaldehyde emission from each material at 20 °C, 37 °C and 50 °C by the desiccator method was previously reported by the authors with the finding that formaldehyde emission decreased with time and after high temperature baked-out [7,24,25]. This tendency also appeared in the present experiment with the VOC analyser for determination of VOC emissions.

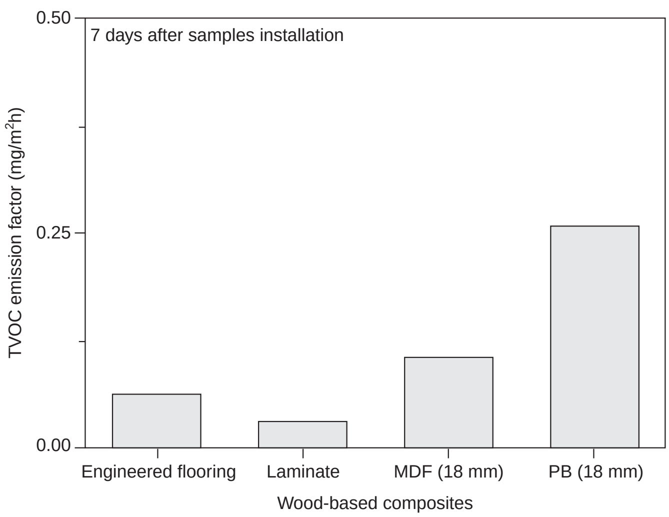


Fig. 7. TVOC emission factor of wood-based composites by a 20 L small chamber; 7 days after samples installation in the small chamber.

The results of laminate flooring and engineered flooring are shown in Figures 10 and 11. For the E2 grade in formaldehyde emission, the VOC emissions were considerably lower than for MDF. Especially in the case of flooring materials at 37 °C and 50 °C, very low VOC emissions were observed. Although related to the E1 grade of the laminate flooring and engineered flooring, this result is noteworthy in that the VOC emissions from wood-based materials were related to formaldehyde emission.

Generally, laminate flooring is manufactured as E1 grade in Europe. As noted, the greatest effect on formaldehyde emission from laminate flooring is exerted by the HDF, which is the core of laminate flooring. This residential grade of laminate flooring is made by urea-formaldehyde resin or urea-melamine-formaldehyde condensed resin. Because the plywood that was used as the core in plywood flooring was glued with phenol-formaldehyde resin, its formaldehyde emission was lower than that of laminate flooring. The E1 grade of wooden flooring materials is now in use in Korea. To maintain a level under E1 grade, the manufacturers use MUF condensed resin for HDF and phenol-formaldehyde resin for plywood as adhesives. For these results, the VOC analyser can be used with the standard desiccator method for VOC emission while formaldehyde emission is being measured.

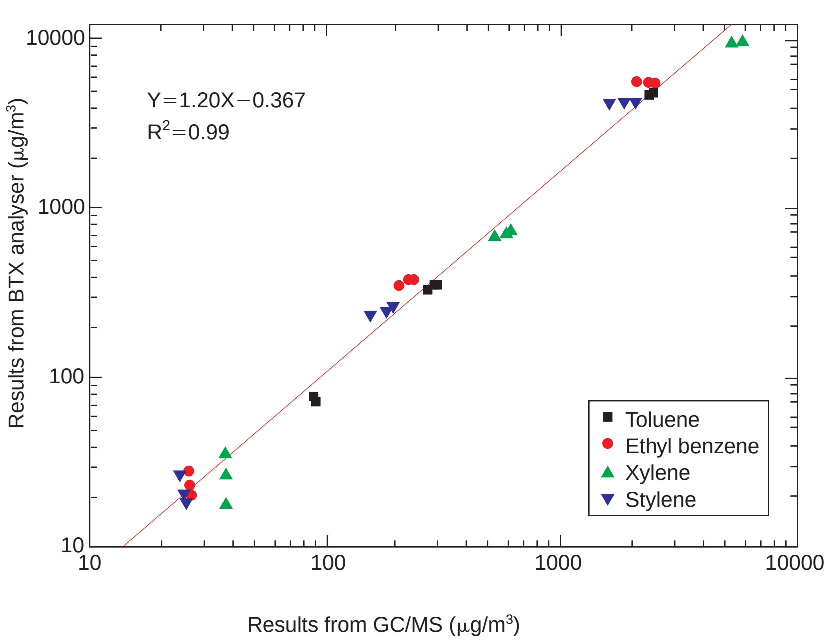


Fig. 8. Correlation between VOC emissions by GC/MS and the VOC analyzer [23].

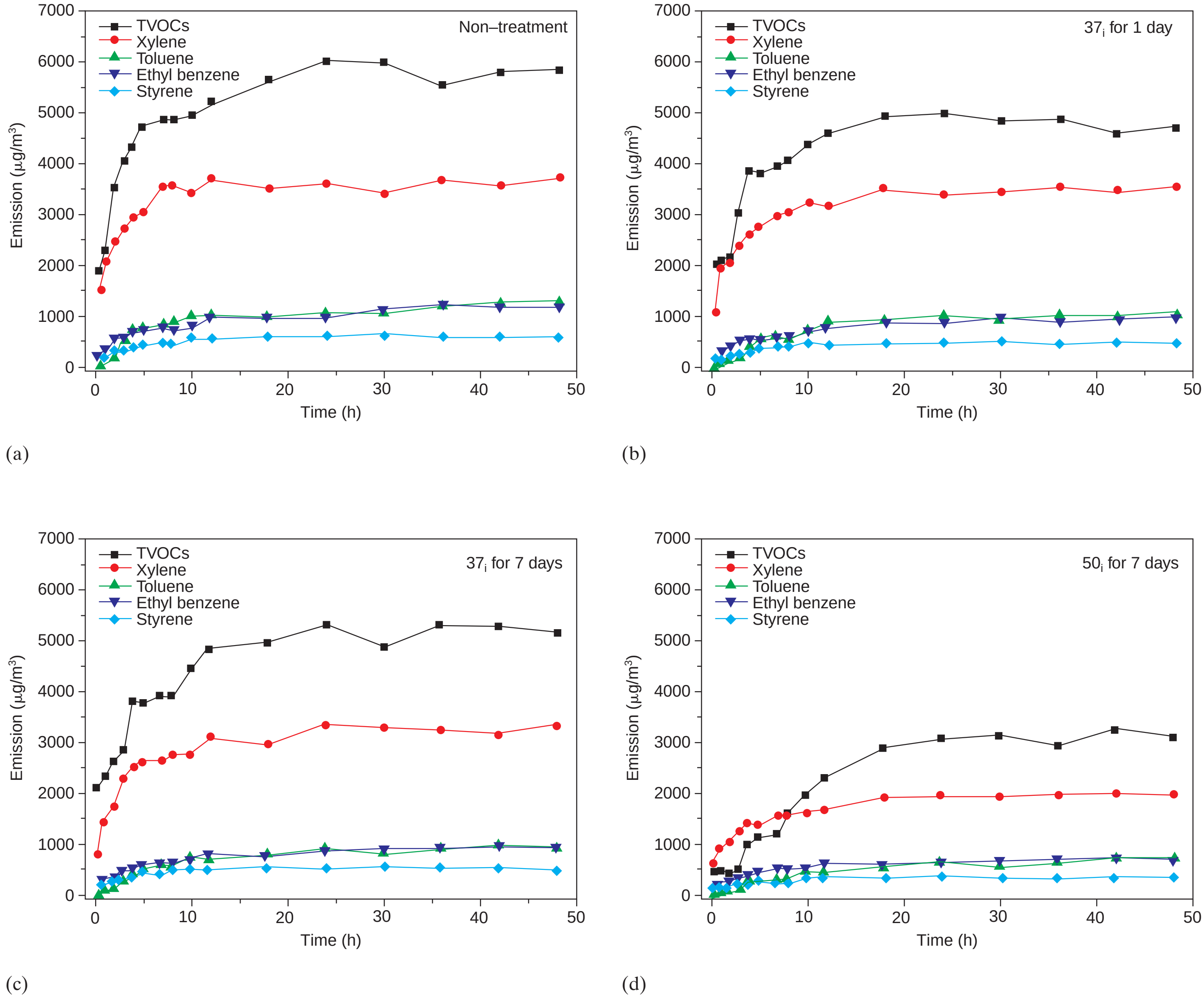


Fig. 9. VOC emissions results from MDF in the desiccator as time and sample treatment.

The VOC emissions results of gases collected from the 20 L small chamber for measuring formaldehyde and VOC emissions are shown in Figure 12 which compares the VOC emissions for the following materials: 15 mm PB, MDF and laminate flooring. Likewise, from the desiccator results which showed that xylene emission was higher than other compounds. This demonstrates that the concentration of each compound in the air is initially very high, but decays rapidly when the wood-based panel and laminate flooring are directly exposed to the chamber air. However, the concentration of each compound in the chamber air for PB was initially the same as that for MDF and they also decayed quickly. However, with time, the concentration of each compound in the chamber air, when the laminate flooring was being tested, decreased below that for PB or MDF, indicating that E1 grade laminate flooring has a lower emission rate than E2 grade wood-based panels.

After reaching their maximum value within 5–10 h, the concentrations decreased systematically with further exposure time. The fractions of VOC amounts lost after 7 days of exposure were relatively similar to the maximum values attained after 5–10 h. After 7-day exposure, the VOC concentrations were reduced by about 99% relative

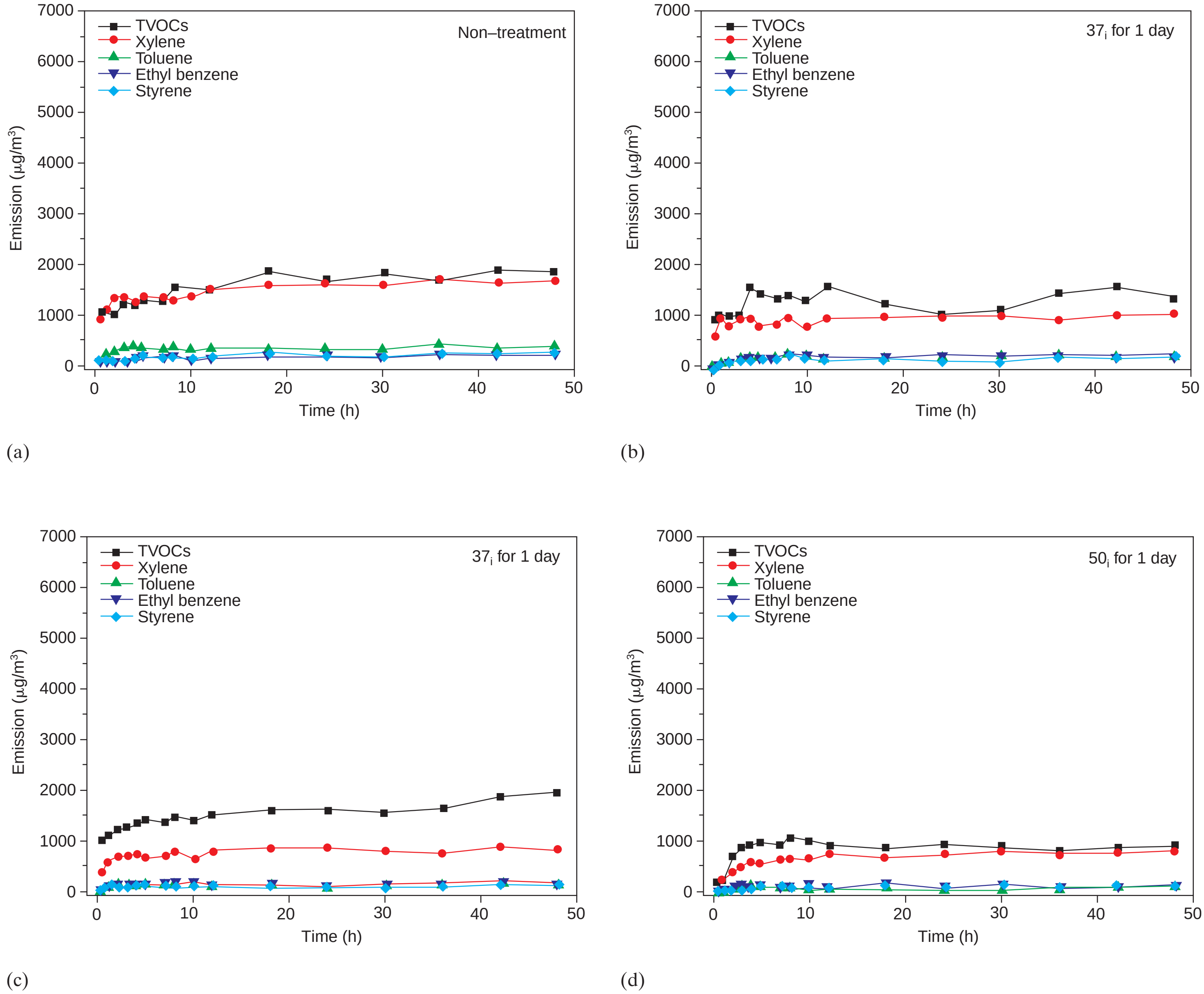


Fig. 10. VOC emissions results from laminate flooring in the desiccator as time and sample treatment.

to the peak concentrations. Over a fixed time of exposure, the concentration of each VOC in the atmosphere varied markedly with the substrate.

Discussion

Emissions of VOCs from wood-based composites can adversely affect indoor air quality. In Korea, standard test methods have been developed to determine VOC emissions from building products and the Ministry of Environment regulates the use of pollutant emission building materials because the Korean government has been controlling indoor air quality since 2004. The 20 L small chamber method was developed in Japan and has a performance in compliance with ASTM, ECA reports and ENV 13419-1. TVOC analysis found that MDF and PB emitted hexanal, pinenes, pentanal, nonanal, heptanal, octanol and so on. There are also many natural VOCs from MDF and PB such as α- and β-pinene. These natural VOCs should be considered for inclusion in the calculation of TVOCs between C_6 and C_{16} in the Korean government regulations.

The VOC analysis of the VOC emissions from various building materials, such as PB, MDF, laminate flooring and engineered flooring, are best done with a desiccator

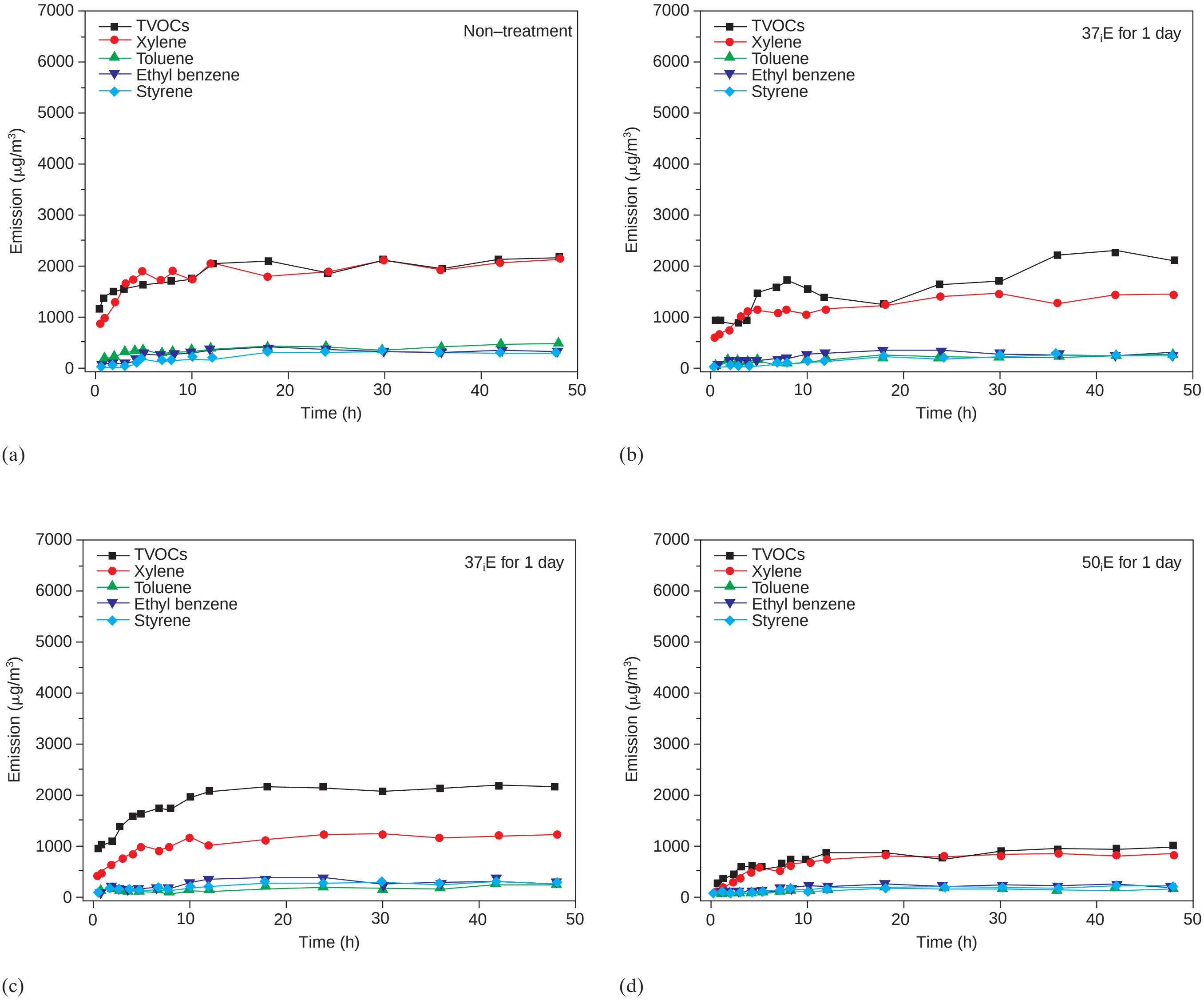


Fig. 11. VOC emissions results from engineered flooring in the desiccator as time and sample treatment.

and a 20 L small chamber. The correlation between the chamber and the VOC analysis and GC/MS method is good. For both the VOC analysis and GC/MS, conditions corresponding well with the desiccator and chamber method can be chosen. The VOC analysis has the advantage of being a proposed standard method for VOC emissions from building materials which is less expensive and simpler than GC/MS. The benefits of using the VOC analyser have been summarised above. This method of VOC analysis is therefore a good alternative to the traditional chamber method for determining VOC emission levels from building products.

Acknowledgements

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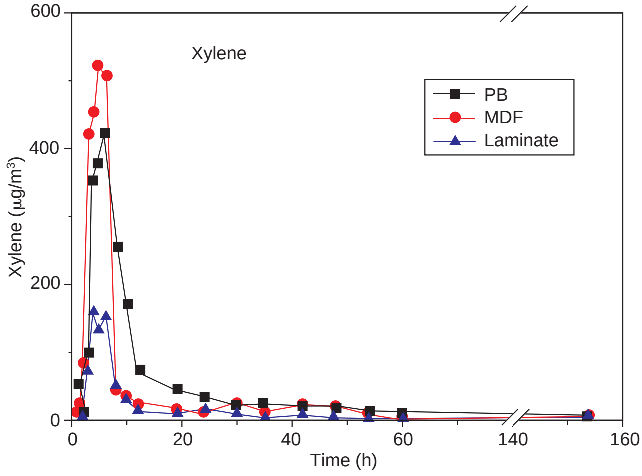

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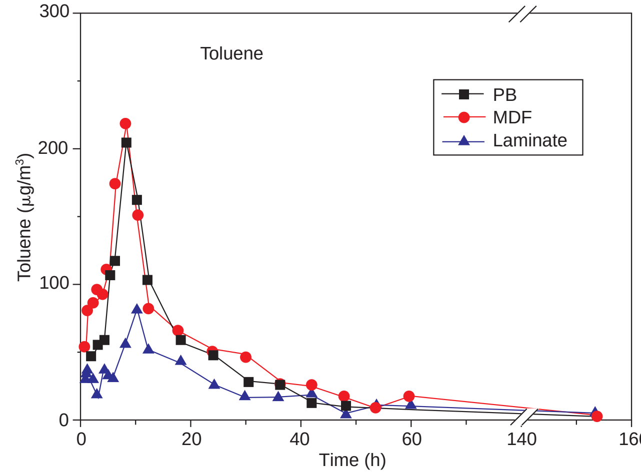

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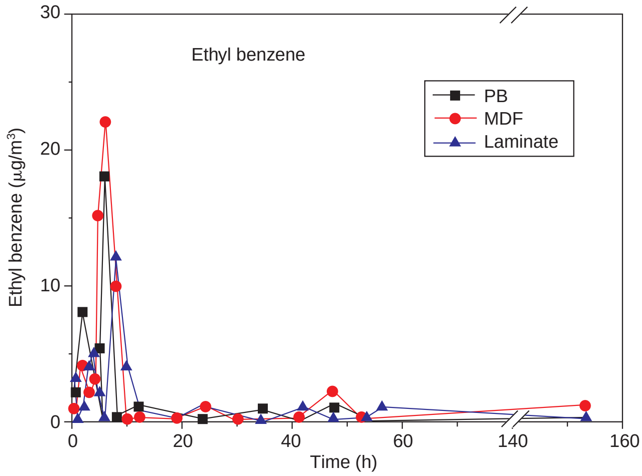

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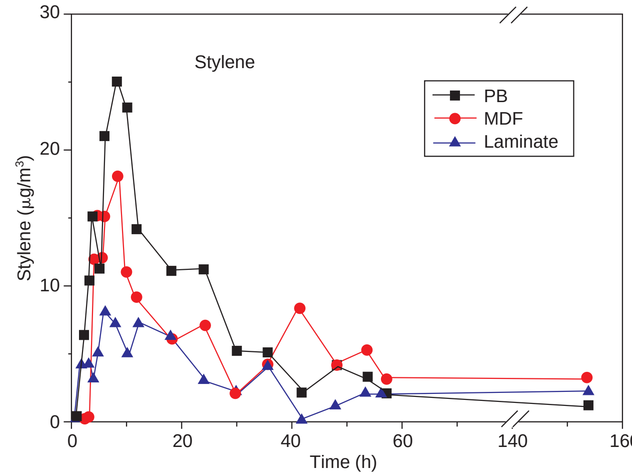

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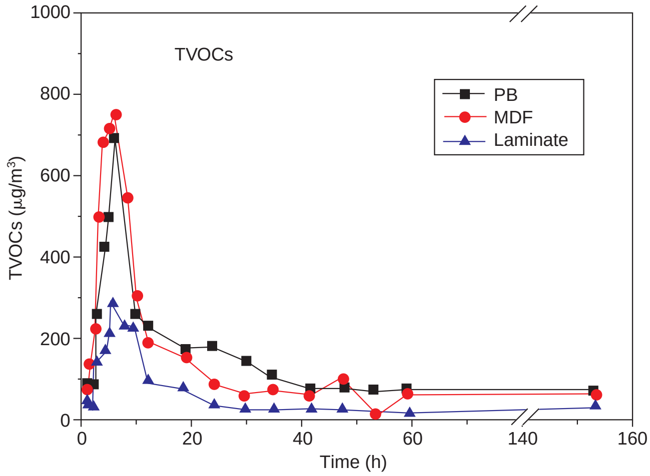

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Fig. 12. VOC emission results from PB, MDF and laminate flooring in the 20 L chamber as time and sample treatment.

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