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Test Method

Determination of formaldehyde and TVOC emission factor from wood-based composites by small chamber method

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Abstract

This paper assesses the reproducibility of testing formaldehyde and TVOC emissions from wood-based composites such as medium density fiberboard (MDF), particleboard (PB), laminate flooring, and engineered flooring using desiccator, perforator and 20 L small chamber methods. According to desiccator and perforator standards, the formaldehyde emission level of each flooring was $\leqslant E_1$ grade. The formaldehyde emission of MDF and PB was 3.48 mg/L PB and 5.38 mg/L by the desiccator method, and 8.57/100 g and 10.21/100 g PB by the perforator method, respectively.

A 20 L small chamber was developed in Japan with performance in compliance with ASTM, ECA reports, and ENV 13419-1. To determine formaldehyde emission, the peak areas of each wood-based composite were calculated from aldehyde chromatograms obtained using the 20 L small chamber method. Formaldehyde, acetaldehyde, acrolein/acetone, propionaldehyde, methacrolein, 2-butanone/butyraldehyde, benzaldehyde and isovaleraldehyde were detected as aldehyde compounds. In this experiment, it was found that MDF and PB emitted hexanal, pinenes, pentanal, nonanal, heptanal, octanol, etc. MDF and PB emitted significantly greater amounts of volatile organic compounds (VOCs) than the flooring materials did.

Keywords: Formaldehyde; TVOC; Small chamber; Wood-based composite

1. Introduction

Many building materials emit volatile organic compounds (VOCs) which have the potential to affect health and comfort. Formaldehyde is a suspected human carcinogen that is known to be released from pressed-wood products used in home construction, including products made with urea-formaldehyde (UF) resins (e.g., particleboard (PB), hardwood plywood, medium density fiberboard (MDF), and paneling) and those made with phenol-formaldehyde (PF) resin (e.g., softwood plywood, oriented strand board) [1–5]. The toxicity of wood-based panels bonded with UF resin, due to the emission of formaldehyde and the associated possible health hazard, could act as an obstacle to their acceptance by the public, given the prevailing climate of environmental awareness and concern.

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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 [6].

To prevent "sick house syndrome", suitable ventilation rates and reduction of emission rates (ERs) from building products are required. Over the past decade, researchers have developed various techniques for measuring emissions of VOCs from building materials. Several small-scale chambers for measuring aldehyde and VOC ERs have been proposed [7–10] and have often been used to determine chemical ERs from building materials [11]. 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. The emission testing techniques for building materials are important for manufacturers, indoor air quality investigators and researchers. Such validation is important for the quantification of the impact of construction products on indoor air quality [12].

The standard method for measuring emission from wood-based panels is to use a test chamber. Three different sizes, $\geqslant 12\,m^3$, $1\,m^3$ and $0.225\,m^3$, are proposed in the new European standard prEN 717-1 (prEN 717-1, 1997) for formaldehyde emission determination. In Sweden the emission test is performed in a $1\,m^3$ chamber according to standard SS 27 02 36 (SS 1988). As measuring the formaldehyde emission in a chamber takes time and requires specialized and expensive equipment, simpler laboratory methods which can be used for inhomogenous products with good correlation to the chamber methods are needed. Several methods have been mainly used for the determination of formaldehyde emission from PB and a good correlation has been found between the chamber, perforator and flask methods [13].

The Korean government started controlling indoor air quality in 2004. The law from the Ministry of Environment regulates the use of pollutant emission building materials. The use of materials with formaldehyde emission level above $1.25\,mg/m^2\,h$ (JIS A 1901, Small chamber method) is prohibited. This is E_2 grade ($>5.0\,mg/L$) when converted to the desiccator method (JIS A 1460).

In renovated or completely new buildings, levels of indoor air pollutants, especially of VOCs, from emissions from construction and building materials are often several orders of magnitude higher than the VOC levels in buildings with normal use [14–17].

In this study, a 20 L small chamber was employed to measured formaldehyde and VOC emissions from wood-based composites as building materials. ERs of formaldehyde and VOCs from wood-based composites were measured quantitatively and compared to typical methods, such as desiccator and perforator methods.

2. Experimental

2.1. Materials

For wood-based composites, we chose laminate flooring and engineered flooring. Currently, these are extensively used in new apartment interiors and in the remodeling market in Korea. Laminate flooring is composed of waterproof, high-density fiberboard (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 Fig. 1. Finally, the edges of the product are machined to produce tongue and groove profile. In the case of engineered flooring, a fancy, 0.5 mm-thick veneer of a wood

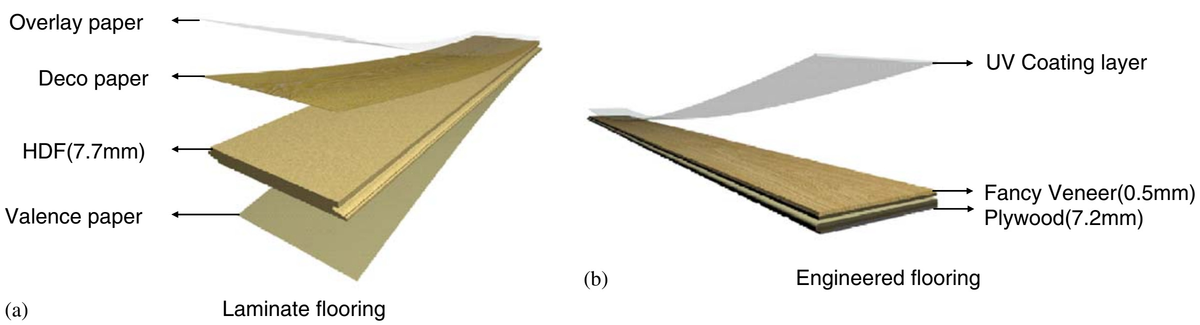

Fig. 1. Structures of laminate and engineered floorings.

such as birch, oak, beach, cherry, or maple is glued to a 7.2 mm-thick plywood sheet and pressed at about 160 °C. Ultra-violet (UV) curable coating is applied to this fancy veneer. The structures of both flooring products are shown in Fig. 1. 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 contents of these materials are shown in Table 1.

2.2. Typical formaldehyde emission tests: desiccator and perforator methods

The Japanese standard method with a desiccator (JIS A 1460) was used to determine the formaldehyde emissions from the laminate flooring, engineered flooring, non-veneered MDF and PB. The formaldehyde emission test for wood-based composites by the desiccator method is carried out using a glass desiccator. The emitted quantity of formaldehyde is obtained from the concentration of formaldehyde absorbed over a 24 h period in distilled or deionized water when the test pieces of a specified surface area are placed in the desiccator with the specified amount of distilled or deionized water. The principle for determining the formaldehyde concentration absorbed in the distilled or deionized water is based on the Hantzsch reaction in which the formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL) (Japanese Industrial Standard, 2001). The 24-h desiccator method uses a common glass desiccator with a volume of 10 ± 1 l. Eight test specimens, with dimensions of 5×15 cm, were positioned in the desiccator. The emission test lasted 24 h in the covered desiccator at a temperature of 20 °C. The emitted formaldehyde was absorbed in a water-filled petri dish and was analyzed by the chromotropic acid method [18].

The perforator value of formaldehyde emission was determined using the DIN EN 120 (European Committee For Standardization, 1991) method, primarily used in Europe. A specific perforator apparatus is required for this method. A sample (110 g) and 600 ml of toluene were placed in a flask, and the perforator was filled with 1000 ml of distilled water. The boiled toluene was passed through the distilled water for 2 h. In this process, the distilled water absorbed the formaldehyde and other volatile organic compounds stripped by the boiling toluene. The formaldehyde trapped by the water was then quantitatively determined using an UV spectrophotometer after treatment with acetyl acetone and acetyl acid ammonium.

Table 1
Moisture contents of wood-based composits

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

2.3. Twenty liter small chamber method

A 20 L small chamber was developed in Japan with its performance in compliance with ASTM [19,20], ECA reports [21–24], and ENV 13419-1 [25]. Fig. 2 shows the main chamber which is made

Fig. 2. The 20 L small chamber.

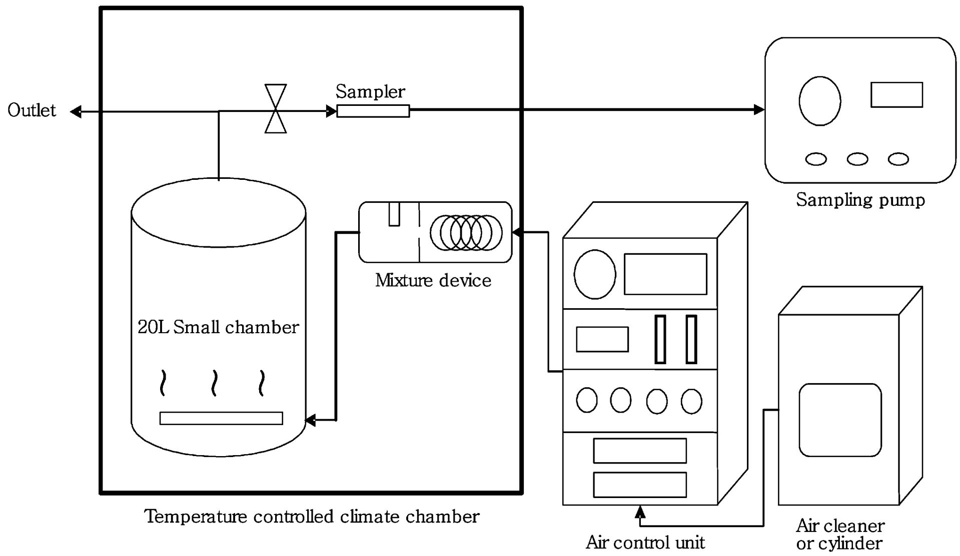

Fig. 3. Schematic diagram of the 20 L small chamber

of stainless steel (SUS304) and the air control unit. Although there are larger chambers, the 20 L chamber was used in this study because it has been standardized in Korea. The air control system consisted of an air supply unit, a humidifier and pumps. The 20 L chamber was set up in a temperature-controlled climate chamber. Purified air was used for ventilation. Fig. 3 shows a schematic diagram of the 20 L small chamber system. The stainless steel seal box was used to prevent the cut edge effect, which allowed chemical emission only from one side surface of the test piece. When two seal boxes were used, the total surface area was 0.044 m^2 and the loading was 2.2 m^2/m^3 [11].

Before setting up the chamber and seal 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 given ventilation rate. The temperature and relative humidity inside the chamber were kept constant. The test conditions are shown in Table 2. Test pieces, laminate flooring, engineered flooring and non-veneered MDF and PB, all sealed with seal boxes, were set in the chamber, and the air inside the chamber was sampled after 12 h. Sampling conditions are shown in Table 3. Throughout the measurements, the air temperature and relative humidity inside the test chamber were kept constant at 25 ± 1 °C and 50 ± 5%, respectively, and ventilated at 0.5 h^{-1}. Aldehydes were analyzed by HPLC, and TDS/GC-MS was used for VOCs, as shown in Tables 4 and 5. In this paper, TVOC was defined with the conversion of all areas of the peaks between C_6 and C_{16} to concentrations using the toluene response factor. A peak area under 10 was defined as the limit of detection. The sample gas was taken by Tenax-TA and 2,4-DNPH cartridge 7 days

Table 2
Test conditions in the 20 L small chamber method

Variables	Condition
Chamber volume	20 L
Sample size	0.0432 m^2 (0.147 m × 0.147 m × 2)
Air flow rate	0.01 m^3/h
Ventilation rate	0.5/h
Sample loading factor	2.16 m^2/m^3
Temperature	25 ± 1 °C
Humidity	50 ± 5%

Table 3
Sampling condition in the 20 L small chamber method

	Formaldehyde	VOCs
Sampler	2,4-DNPH Cartridge (Supelco, USA)	Tenax-TA (Supelco, USA)
Air flow rate	167 mL/min	167 mL/min
Total volume	10 L	3.2 L

Table 4
Analysis conditions for formaldehyde

Variables	Condition
HPLC	Acme HPLC
Detector	UV/Vis 360 nm
Column	Nova-Pak C_{18} (3.9 m × 150 mm), Waters
Mobile phases	Acetonitrile : Water = 60: 40
Analysis time	10 min
Injection volumn	20 μL
Column temperature	25 °C
Mobile phase flow rate	1.0 mL/min
Purge gas and flow rate	He (99.99%), 100 mL/min

Table 5
Analysis conditions for VOCs

Variables	Condition
TDS	Perkin Elmer ATD400
GC/MS	HP6890/Agilent5973
Column	RTX-1 (105 m × 0.32 mm × 3 μm)
Carrier gas and flow	He (99.99%)
Temperature program	40 °C (5 min) → 70 °C (5 min) → 150 °C (5 min) → 200 °C (5 min) → 220 °C (5 min) → 240 °C (5 min)
MS condition	
Mode	EI (Electron ion)
Electron energy	70 eV
Detection mode	TIC (scan), *m/z*: 35/350

after the sample specimens were installed into the 20 L small chamber, according to the regulation of the Ministry of Environment, Korea.

2.4. Emission factor

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

$$ER = A\,(EF), \qquad (1)$$

where ER is the emission rate (mg/h), A the source area (m^2) and EF the emission factor ($mg/m^2\,h$).

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.

3. Results and discussion

3.1. Desiccator and perforator methods

The formaldehyde emission results obtained by desiccator and perforator methods for each flooring, and from non-veneered MDF and PB, are shown in Fig. 4. Each material was tested three times. The desiccator and perforator values were 0.94 mg/l and 3.47 mg/100 g panel for the laminate flooring, and 0.44 mg/l and 2.21 g/100 g panel for the engineered flooring, respectively. According to both standards, the formaldehyde emission level of each flooring was $\leqslant E_1$ (below 1.5 mg/L) grade. Generally, laminate flooring is manufactured as E_1 grade in Europe. The greatest effect on formaldehyde emission in laminate flooring is exerted by HDF, which is the core of laminate flooring. This grade of laminate flooring can be used for residences. Because the plywood that was used as the core in plywood flooring was glued with PF resin, its formaldehyde emission was lower than that of laminate flooring. E_1 grade of wooden flooring materials has been circulated in Korea.

On the other hand, the MDF and PB furniture materials in the experiment were veneered with decorative paper foil, with a formaldehyde emission of E_2 grade. The emission for MDF and PB was 3.48 and 5.38 mg/l by desiccator method, and 8.57/100 g and 10.21/100 g panel by perforator method, respectively. These results were over E_2 (1.5–5.0 mg/L) grade. The sample used for this study emitted a lot

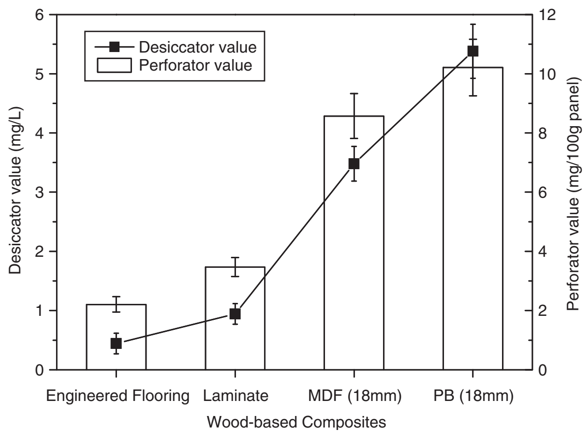

Fig. 4. Comparison of desiccator and perforator values of formaldehyde emissions from wood-based composites.

of free formaldehyde. Although the perforator value was directly proportional to the desiccator value in the case of the E_1 grade level, it increased at a lower rate than the desiccator value did. Whereas the weight (100 g) of wooden board is used in the perforator method, the dimensions of the wooden board are taken into consideration in the desiccator method. In spite of the formaldehyde emission values from the same boards being slightly different because of the difference in measuring methods, these two methods produced proportionally equivalent results.

3.2. Twenty liter small chamber method: formaldehyde and TVOC

The results, in the form of aldehyde chromatograms, of aldehyde emission from wood-based composites by HPLC analysis are shown in Fig. 5. These results were recorded 7 days after sample installation as this is the period mandated by the Ministry of Environment in Korea. Formaldehyde, detected at a retention time of 5.2 min, was the first detected compound from the aldehyde chromatograms. Table 6 presents the peak areas from all wood-based composites calculated from these chromatograms.

Formaldehyde was the aldehyde with the highest emission from the wood-based composites, because these wood-based composites were made with formaldehyde-based resin such as UF resin and PF resin, as is common in industry. In addition, formaldehyde was detected at the early retention time of 5.2 min because it is an aldehyde with a simple molecular structure: HCOH. Not only formaldehyde, but also acetaldehyde, acrolein/acetone, propionaldehyde, methacrolein, 2-butanone/butyraldehyde, benzaldehyde and isovaleraldehyde were detected as aldehyde compounds. The order of peak area of aldehydes from the wood-based compounds was formaldehyde > acrolein/acetone > acetaldehyde > propionaldehyde. However, from the flooring materials, 2-butanone/butyraldehyde and crotonaldehyde were detected at much higher levels than acrolein/acetone and acetaldehyde.

From these peak areas, formaldehyde EFs were calculated and are shown in Fig. 6. As expected from the typical method, the formaldehyde EFs of MDF and PB were higher than those of flooring

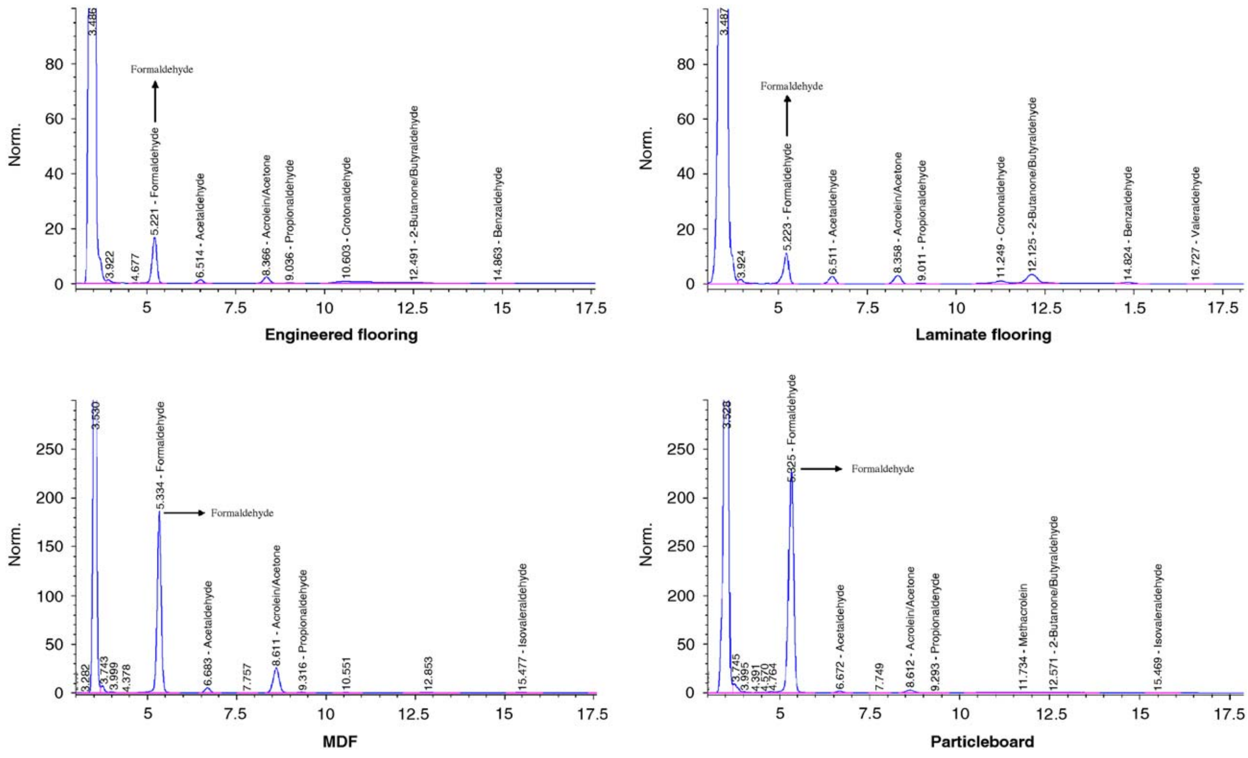

Fig. 5. Aldehyde chromatograms of wood-based composites.

Table 6
Aldehydes detected by HPLC analysis of wood-based composites

Aldehyde	Retention time (min)	Peak area			
		Engineered flooring	Laminate flooring	MDF	PB
Formaldehyde	5.221	152.19	118.41	1567.11	2146.77
Acetaldehyde	6.514	14.15	34.49	56.37	24.20
Acrolein/Acetone	8.366	29.58	44.84	319.84	48.68
Propionaldehyde	9.036	3.57	5.43	12.35	4.04
Crotonaldehyde	10.603	48.10	27.00	ND	ND
Methacrolein	11.928	ND	ND	ND	35.37
2-Butanone/butyraldehyde	12.491	30.66	76.98	ND	17.47
Benzaldehyde	14.863	2.26	8.05	ND	ND
Isovaleraldehyde	15.962	ND	ND	11.93	3.55
Valeraldehyde	17.474	ND	1.85	17.06	ND
Hexaldehyde	25.322	ND	ND	3.84	ND

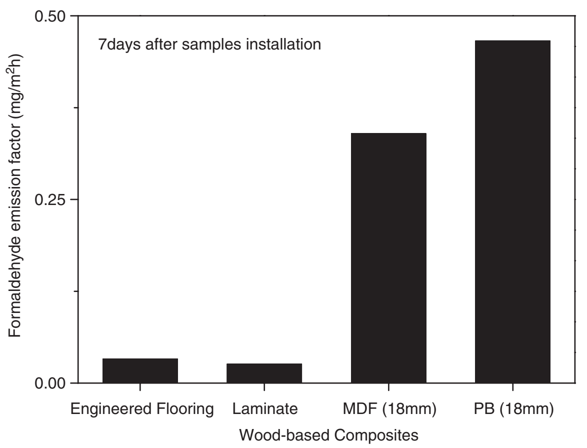

Fig. 6. Formaldehyde emission factor of wood-based composites as determined by the 20 L small Chamber.

materials made as E_1 grade, although engineered flooring was a little higher than laminate in contrast to that from the typical desiccator and perforator methods. This small difference on formaldehyde emission data can be explained by the structure of wood-based composites. A correction is performed for the formaldehyde emitted from the entire faces of the sample for the desiccator test, but only from the upper surface for the 20 L small chamber method. Non-veneered MDF and PB were not coated by materials such as low-pressure melamine, decorative film and UV curable vanish, so the formaldehyde emissions can be much higher. Furthermore, there is no consideration of emissions from sample edges. On the other hand, flooring materials are coated with a UV-cured coating for engineered flooring and MF resin impregnation paper for laminate flooring. Despite these differences of test principle between the typical method and 20 L small chamber method, the results for formaldehyde emission showed a similar trend.

TVOC chromatograms of wood-based composites, 7 days after sample installation, are shown in Fig. 7. Koontz and Hoag [26] 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 surface of engineered flooring was coated with UV-cured material while the unfinished surface of PB was uncoated, unusual (from 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 Fig. 8. The PB specimens emitted significantly greater amounts of VOCs than the engineered flooring specimens did. This result was similar to the tendency with formaldehyde EF. However, there are many natural VOCs emitted from PB such as α-pinene and β-pinene. When TVOC is calculated between C_6 and C_{16}, these harmless, natural VOCs are included, which explains why TVOC EF from PB was higher than that from engineered flooring. In Korea, the Ministry of Environment provides guidelines for

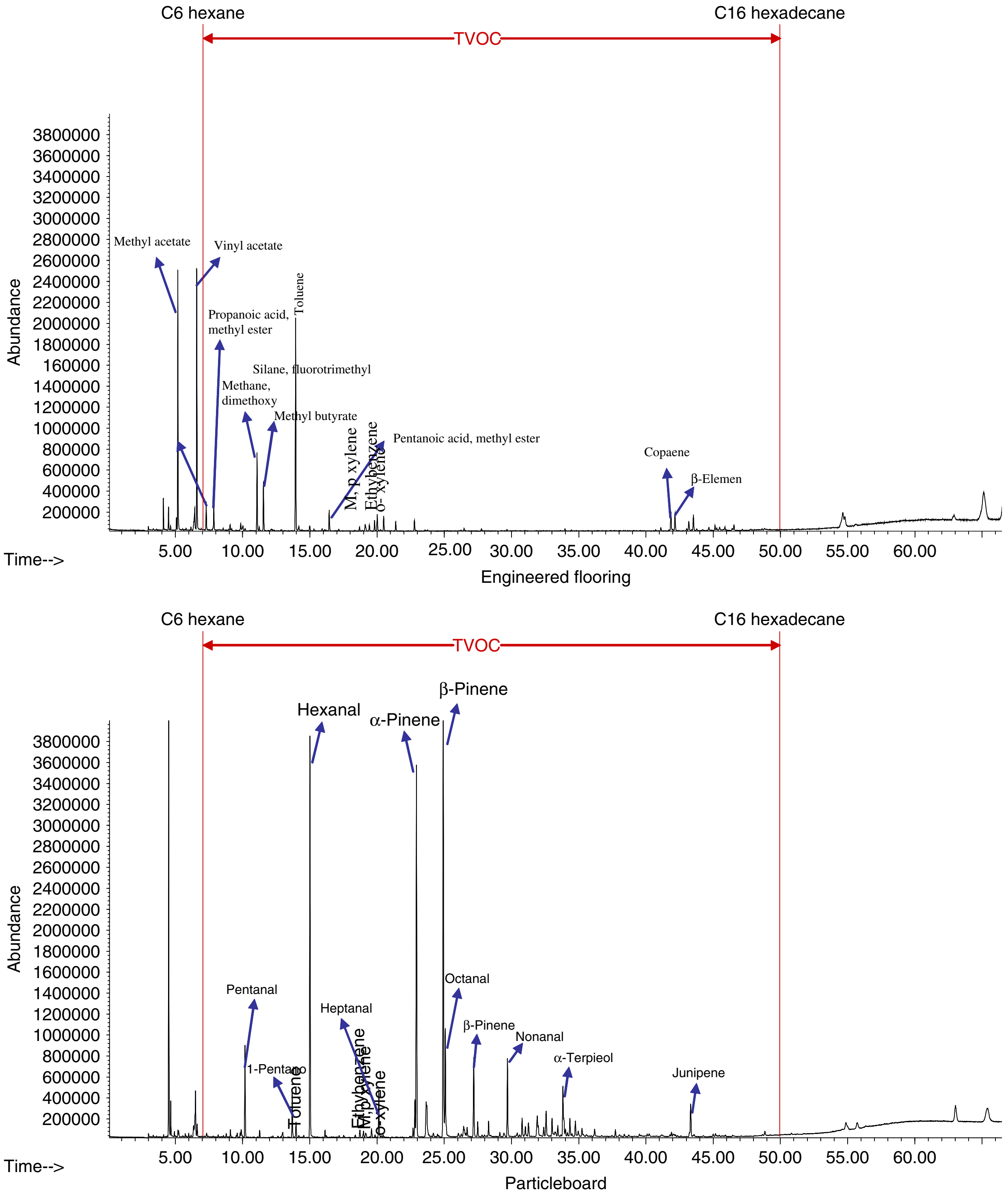

Fig. 7. TVOC chromatograms of engineered flooring and particleboard.

VOC emissions from building materials 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.

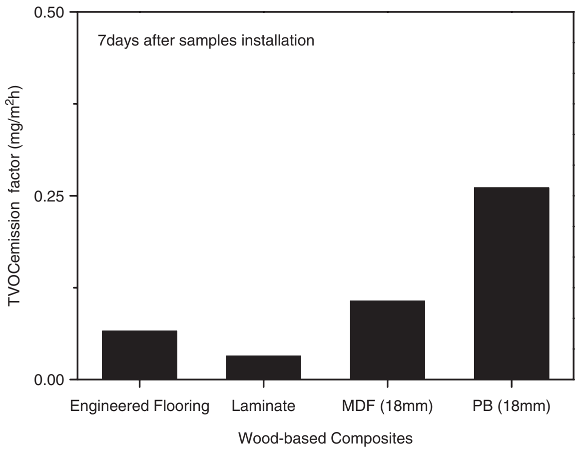

Fig. 8. TVOC emission factor of wood-based composites as determined by the 20 L small chamber; 7 days after samples installation in the chamber.

4. Conclusions

Emissions of formaldehyde and VOCs from wood-based composites can adversely affect indoor air quality. In Korea, standard test methods have been developed to determine formaldehyde and VOC emissions from building products and the Ministry of Environment regulates the use of pollutant emission from building materials.

The 20 L small chamber test was developed in Japan with its performance in compliance with ASTM, ECA reports, and ENV 13419-1. To determine the formaldehyde emission levels, the peak areas of each wood-based composite were calculated from aldehyde chromatograms attained with the 20 L small chamber. The order of peak areas of aldehydes from wood-based compounds was formaldehyde > acrolein/acetone > acetaldehyde > propionaldehyde. As expected, the formaldehyde EFs of MDF and PB were higher from flooring materials made as E_1 grade, although engineered flooring was slightly higher than laminate, in contrast to the results obtained from typical methods with desiccator and perforator. Although the trend of formaldehyde emission results from desiccator and perforator methods were seen in the small chamber method, there were many factors that had to be considered; for example, the size of sample, method for collection formaldehyde emitted and analysis. To make correlation between desiccator and perforator methods and the small chamber method, these factors must be considered and investigated with many repeat tests.

The small differences in formaldehyde emission data can be explained by the different structures of wood-based composites. 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-pinene and β-pinene. These harmless, natural VOCs should be considered in the TVOC calculation between C_6 and C_{16} in the Korean government regulations.

Acknowledgements

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