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Application of Field and Laboratory Emission Cell (FLEC) to Determine Formaldehyde and VOCs Emissions from Wood-Based Composites*1

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ABSTRACT

The Korean Ministry of Environment started controlling indoor air quality (IAQ) in 2004 through the introduction of a law regulating the use of pollutant emitting building materials. The use of materials with formaldehyde emission levels above 1.25 mg/m^2 · h (JIS A 1901, small chamber method) has been prohibited. This level is equivalent to the E_2 grade (>5.0 mg/ℓ) of the desiccator method (JIS A 1460). However, the 20 ℓ small chamber method requires a 7-day test time to obtain the formaldehyde and volatile organic compound (VOC) emission results from solid building interior materials. As a approach to significantly reduce the test time, the field and laboratory emission cell (FLEC) has been proposed in Europe with a total test time less than one hour. This paper assesses the reproducibility of testing formaldehyde and TVOC emissions from wood-based composites such as medium density fiberboard (MDF), laminate flooring, and engineered flooring using three methods: desiccator, perforator and FLEC. According to the desiccator and perforator standards, the formaldehyde emission level of each flooring was ≤E_1 grade. The formaldehyde emission of MDF was 3.48 mg/ℓ by the desiccator method and 8.57 g/100 g by the perforator method. To determine the formaldehyde emission, the peak areas of each wood-based composite were calculated from aldehyde chromatograms obtained using the FLEC method. Formaldehyde, acetaldehyde, propionaldehyde, butyraldehyde and benzaldehyde were detected as aldehyde compounds. The experimental results indicated that MDF emitted chloroform, benzene, trichloroethylene, toluene, ethylbenzene, m,p-xylene, styrene, and o-xylene. MDF emitted significantly greater amounts of VOCs than the floorings did.

Keywords : field and laboratory emission cell (FLEC), formaldehyde, VOCs, wood-Based composites

1. INTRODUCTION

Since the 1980s, the formaldehyde emissions have been characterized (Hawthorne and Matthews, 1987). The environmental chamber technique for the determination of volatile organic

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compounds (VOCs) was introduced in the 1980s and has been standardized in Europe (European Collaborative Action, 1991; European Collaborative Action, 1993; ISO 16000-9, 2006a and ISO 16000-10, 2006b). A corresponding method exists in the US (American Society for Testing Materials, 1997). Due to their superb bonding properties and inexpensive cost, formaldehyde-based resins are used extensively as adhesives in the manufacture of a variety of household products. One prominent use of urea-formaldehyde (UF) resin is in the manufacture of particleboard (PB), plywood, and MDF. Several thin sheets of wood are glued together by the UF resin to produce plywood, whereas PB and MDF are manufactured by mixing wood chips and sawdust with the resin and then pressing the mixture into its final form at a high temperature (Kim and Kim, 2005a; Kim and Kim, 2005b).

The standard method for measuring emissions from wood-based panels is to use a test chamber. Three different chamber sizes of $\geq$ 12, 1 and 0.225 m^3 are proposed in the new European standard prEN 717-1 (prEN 717-1, 1997) for the determination of formaldehyde emission. In Sweden, emission testing is performed in a 1 m^3 chamber according to standard SS 27 02 36 (SS 1988). As such measurement of formaldehyde emission in a chamber takes time and requires specialized and expensive equipment, simpler laboratory methods which can be used for heterogenous products with good correlation to the chamber methods are needed. Of the several methods used for the determination of formaldehyde emission from PB, a good correlation has been found between the chamber, perforator method and flask method (Risholm-Sundman and Wallin, 1999). The Korean government started controlling indoor air quality (IAQ) in 2004. The law prepared by the Ministry of Environment regulates the use of building materials which emit pollutants. The use of materials with total VOC (TVOC) emission levels above 4.0 $mg/m^2.h$ (JIS A 1901, small chamber method) is prohibited. Most suppliers and consumer are concerned about how to reduce pollutants from building materials and how to control IAQ (Kim and Kim, 2005c; Kim *et al.*, 2006).

In renovated or completely new buildings, levels of indoor air pollutant emissions from construction and building materials, especially of VOCs, are often several orders of magnitude higher than the VOC levels in buildings under normal use (Brown, 1999; Rothweiler *et al.*, 1992; Tuomaninen *et al.*, 2001; Wolkoff *et al.*, 1991). Furthermore, the formaldehyde and VOCs emission test has been standardized and chemical analyses using the 20 ℓ small chamber method have been conducted by the Ministry of Environment. The 20 ℓ small chamber method was developed in Japan and its performance complies with ASTM (1996, 1997), ECA reports (1989, 1991, 1993, 1995), and ENV 13419-1 (1999).

The use of small-scale environmental chambers with volumes ranging from a few liters to a few cubic meters has been increasing (Wensing, 1999). The weakness of these traditional chamber techniques is that they cannot be used to investigate emissions from existing real building structures. The emission of VOCs from a material in a real building structure is affected not only by the material but also by the environmental conditions and other surrounding materials (Wolkoff, 1998; Wilke *et al.*, 2004; Wirtanen, 2005). Secondary emissions can develop under the influence of humidity, ozone, UV-light, etc. (Weschler and Schields, 1997; Wolkoff *et al.*, 2000). Hydrolysis reactions in the floor structure (PVC/adhesive/casein containing leveling agents) can produce 2-ethylhexanol, butanol, and ammonia (Karlsson *et al.*, 1989;

Gustafsson, 1990; Bornehag, 1991). Thus, the emission measured on site can differ considerably from the emission measured from a single material under laboratory conditions (Järnström *et al.*, In Press). The impact of the increased consciousness about indoor environment has created a demand for low-emitting (healthy) building materials, and hence also for standardized methods to characterize and quantify the VOC emissions from building materials and consumer products. Furthermore, methods for easy source identification of VOCs from potential emitting from building materials on site and for their quantification are required. Therefore, the field and laboratory emission cell (FLEC) has been proposed and has become a European standard for emission testing (prENV 13419-2, 1998). This is a kind of micro emission cells featuring a high sensitivity due to the large loading ratio (surface area/volume). Nowadays, a large percentage of emission tests for various materials are performed with FLEC (Wolkoff and Nielsen, 1996; Risholm-Sundman, 1999; Clenø *et al.*, 2001).

In this study, we apply a field and laboratory emission cell (FLEC) to the measurement of formaldehyde and VOCs emission levels from wood-based composites and compare the results with those obtained with two other typical methods using desiccator and perforator.

2. EXPERIMENTAL

2.1. Material

Among the various 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 the core material, with overlay paper, deco paper and valance paper. Each paper is impregnated with melamine-papers pressed at about 200°C. Finally, the edges of the product are machined to produce a tongue and groove profile. In the case of engineered flooring, 0.5 mm-thick fancy veneer of a wood such as birch, oak, beach, cherry, or maple is glued to a 7.2 mm-thick plywood sheet and pressed at about 160°C. A ultra-violet (UV) curable coating is coated on this fancy veneer. For comparison with flooring, we used non-veneered 18 mm-thick medium density fiberboard (MDF) as furniture material and other wood-based composites. The moisture contents of these materials are shown in Table 1.

Table 1. Moisture contents of wood-based composits

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

2.2. Typical Formaldehyde Emission Tests by Desiccator and Perforator Methods

The Japanese standard desiccator method (JIS A 1460) was used to determine the formaldehyde emissions from the laminate flooring, engineered flooring, and non-veneered MDF. The formaldehyde emission test by a desiccator method for wood-based composites utilizes 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 water or deionized water. The principle for determining the formaldehyde concentration absorbed in the distilled or deionized water is

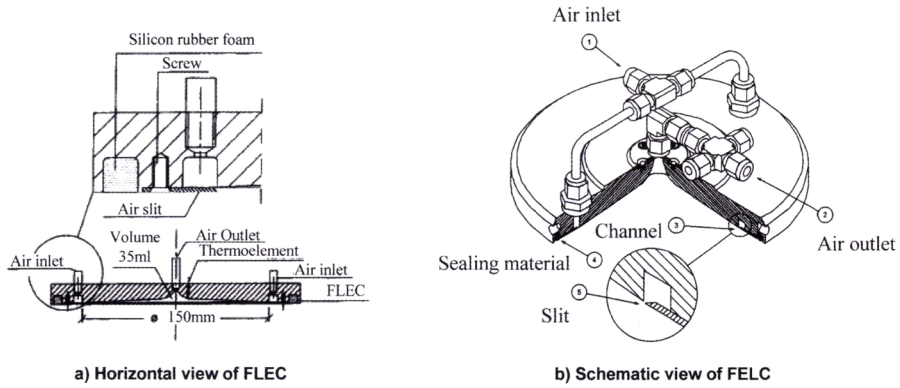


Fig. 1. Schematic view of the field and laboratory emission cell.

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-hour desiccator method uses a common glass desiccator with a volume of 10 ± 1 liters. Eight test specimens with dimensions of 5 × 15 cm were positioned in the desiccator. The emission test lasted 24 hours 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 (Kim and Kim, 2005c).

The perforator value of formaldehyde emission was determined using the DIN EN 120 (European Committee for Standardization, 1991) method which primarily used in Europe. A specific perforator apparatus is required for this method. A sample (110 g) and 600 ㎖ of toluene were placed in a flask, and the perforator was filled with 1,000 ㎖ of distilled water. The boiled toluene was passed through the distilled water for two hours. In this process, the distilled water absorbed the formaldehyde and other VOCs 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.

2.3. Field and Laboratory Emission Cell (FLEC)

2.3.1. Outline of FLEC

The FLEC design is shown in Fig. 1. The inner surface of the stainless steel FLEC was formed with a lathe and was hand polished. The cell was circular with a diameter of 150 mm, providing a maximum test material surface area of 177 cm^2 and a volume of 35 ㎖. By placing the FLEC on top of the material specimen, the surface becomes the bottom part of the cell. The loading factor (test material area to emission cell volume) was a maximum of 506 m^2/m^3. For emission-free condition, silicon rubber foam was used to seal the interface between the FLEC and the test material surface. All tubes and couplings were made of high quality stainless steel. The air (or nitrogen) was introduced through two diagonally positioned inlets into a circular shaped channel (depth 7 × 7 mm) at the perimeter of the cell, from where the air was distributed over the test material surface through the circular air slit (1 mm). The air exited the cell at the top of its center. Such an arrangement provided a constant and efficient air velocity over the entire surface, apart from a smaller part near the center, because the

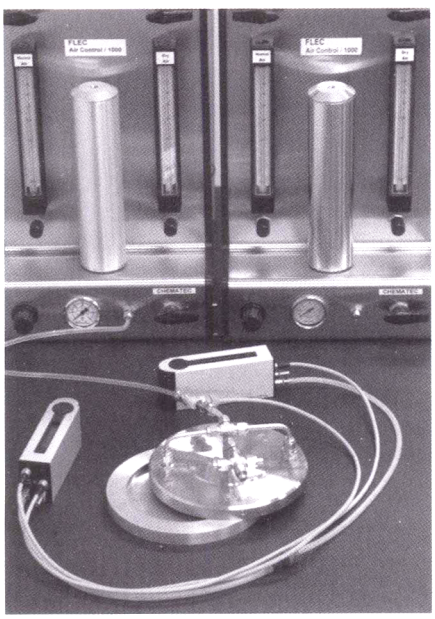


Fig. 2. Air supply and sampling pump of FLEC.

cylindrical cross section area remains constant from the perimeter. The FLEC air supply unit and sampling pump are shown in Fig. 2. The FLEC was supplied with clean and humidified air (or nitrogen) from an air supply control unit. The unit was coupled to the FLEC with teflon tubing. The outlet of the FLEC was connected to a 90° union coupled to a union cross with a 90 mm outlet tube protruding to avoid false air intake during testing and sampling. The two sample outlets were closed with metal rods during conditioning of the test material. The standard cleaning procedure for the FLEC was heating to 75~100°C for one hour in a vacuum oven at about 50 mBar. The detailed protocol for the cleaning and sampling procedures can be found in two reports by Wolkoff *et al.* (1991; 1995).

2.3.2. Collection of Formaldehyde and VOCs by FLEC

The advantage of FLEC is a shorter test time than the 20 ℓ small chamber method. A comparison between the FLEC and 20 ℓ small chamber test methods is listed in Table 2. A diagram of the apparatus used for emission testing is shown in Fig. 3. The FLEC was used as a micro emission cell in this experiment. When the circular stainless steel cell is put onto the surface of the planar test material, the material surface becomes an integral part of the cell itself. The material surface area exposed to airflow inside the FLEC is 177 cm^2 and the internal volume of cell is 35 mℓ. Fig. 4 shows the FLEC test of laminate flooring.

The FLEC was supplied with purified and humidified air at a given ventilation rate. The temperature and relative humidity inside the chamber were kept constant. The emission sample was collected after 5 min of equilibration time and 5 min of cleaning time under the FLEC lid at an airflow of 250 mℓ/min. For formaldehyde, 4.5 ℓ of gas was collected in a 2,4-DNPH cartridge for 30 min under a gas flow rate of 150 mℓ/min while 1.5 ℓ of gas was collected in a Tenax-TA tube for 30 min under a gas flow rate of 50 mℓ/min. The condition of the correction gas is listed in Table 3.

2.3.3. Emission Factor

Formaldehyde and VOCs were analyzed by HPLC and TDS/GC-MS, respectively, as listed in Tables 4 and 5. In this paper, TVOC was defined as the conversion of all peak areas 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 with Tenax-TA and 2,4-DNPH cartridges 7 days after the sample specimens were installed into the 20 ℓ small chamber, according to the regulation of the Ministry of Environment, Korea.

The calculation of the emission factor (EF) is explained in ASTM D5116. Two technical terms

Table 2. Comparison of test condition between FLEC and 20 ℓ small chamber

Test condition	FLEC	20 ℓ small chamber
Sample area	0.0177 m^2	0.0392 m^2
Volume	0.035 ℓ	20 ℓ
Loading factor (Area of sample/volume, m^2/m^3)	504.64 m^2/m^3	1.96 m^2/m^3
Air change rate (h-1)	428.57/h	0.5 ± 0.05/h
Air Supply (ℓ/min)	250 mℓ/min	167 mℓ/min
Equilibration time	Sampling after 15~30 minutes	Sampling after 7 days
Temperature / humidity	23 ± 2.0°C / 50 ± 5%	25 ± 1.0°C / 50 ± 5%
Compounds, sampling flow and total sampling	VOC : 50 mℓ/min, 1.5 ℓ Formaldehyde : 150 mℓ/min, 4.5 ℓ	VOC : 167 mℓ/min, 3.2 ℓ Formaldehyde : 167 mℓ/min, 10 ℓ
Inlet air	High purity air	Room air
Background concentration	VOC : 2 μg/m^3 TVOC : 20 μg/m^3	VOC : 2 μg/m^3 TVOC : 10 μg/m^3
Cleaning process	Vacuum oven or cleaning by methylene then high purity air for 1 day	Cleaning by pure water then oven for above 15 minutes in 260°C
Analysis method	VOC : GC/MS Formaldehyde : HPLC	VOC : GC/MS Formaldehyde : HPLC

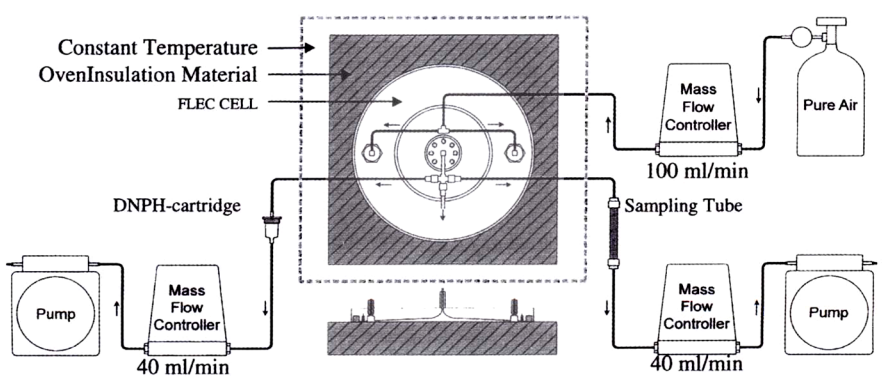


Fig. 3. Schematic drawing of the apparatus for identification and measurement of chemical compounds emitted from the surface of polymeric foam insulation materials (Uchiyama *et al.*, 2001).

of EF and ER are commonly used to describe the rate of emissions from indoor materials, and are related as follows:

$$ER = A(EF) \quad \cdots\cdots \text{eq. 1}$$

Where:

ER = emission rate (mg/h)
A = source area (m^2)
EF = emission factor (mg/m^2h)

Thus, ER can be applied to both area and non-area sources, whereas EFs are reported as mass/mass/time, or in the case of caulk beads,

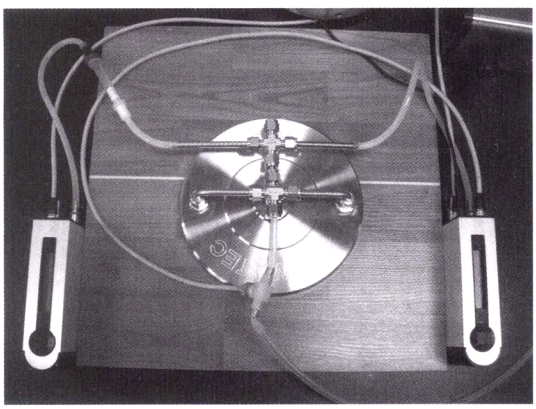


Fig. 4. Test of laminate flooring by FLEC.

Table 3. Condition for FLEC

Variables	FLEC condition
Chamber volume	0.035 ℓ
Sample size	0.0177 m^2
Air flow rate	50 mℓ/min (VOCs), 150 mℓ/min (Formaldehyde)
Air collects	1.5 ℓ (VOCs), 4.5 ℓ (Formaldehyde)
Ventilation rate	428.57/h
Sample loading factor	506 m^2/m^3
Temperature	25 ± 1°C
Humidity	50 ± 5°C

Table 4. Analysis conditions for formáldehyde

Variables	Formaldehyde analysis condition
HPLC	Agilent HP1100
Detector	UV/Vis 365 (Bw.30), ref. 590 (Bw.10)
Column	Supelco C18. 4.6 × 250 mm
Mobile phases	Acetonitrile : Water = 45 : 55
Analysis time	25 min
Injection volume	20 μℓ
Column temperature	40°C
Mobile phase flow rate	1.0 mℓ/min

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 the non-veneered MDF are shown in Fig. 5. Each material was tested three times.

Table 5. Analysis conditions for VOCs

Variables		VOCs analysis 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

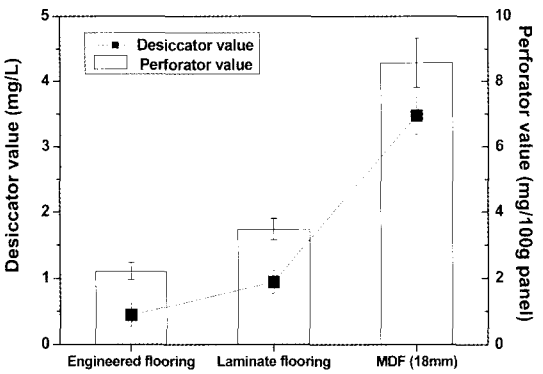


Fig. 5. Comparison of the desiccator and perforator values of the formaldehyde emission from wood-based composites.

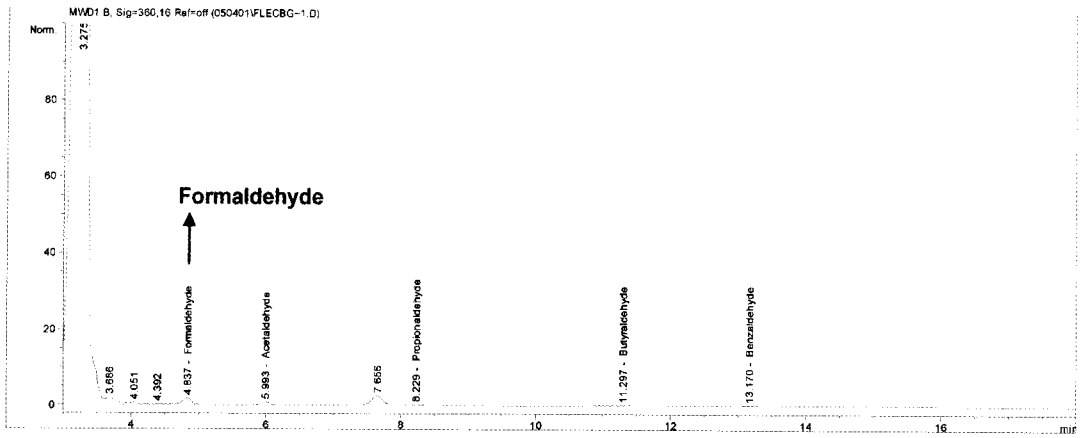
The desiccator and perforator values were 0.94 mg/ℓ and 3.47 mg/100 g panel for the laminate flooring, and 0.44 mg/ℓ and 2.21 g/100 g panel for the engineered flooring, respectively. According to both standards, the formaldehyde emission level of each flooring was $\leq E_1$ (below 1.5 mg/ℓ) grade. Generally laminate flooring is manufactured to satisfy the E_1 grade in Europe. The greatest influence 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 is used as the core in plywood flooring is glued with phenol-formaldehyde (PF) resin, its formaldehyde emission is lower than that of laminate flooring. E_1 grade of wooden flooring materials has been circulated in Korean flooring market.

On the other hand, the MDF furniture materials in the experiment were veneered with decorative paper foil, with a formaldehyde emission of E_2 grade. The MDF emission was 3.48 mg/ℓ and 8.57 g/100 g panel by desiccator and perforator methods, respectively. These results exceeded the E_2 (1.5~5.0 mg/ℓ) grade. The sample used for this study emitted a lot 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 the wooden board is used in the perforator method, the dimensions of the wooden board are taken into consideration in the desiccator method. Although the formaldehyde emission values from the same boards were slightly different because of the difference in measuring methods, these two methods produced proportionally equivalent results.

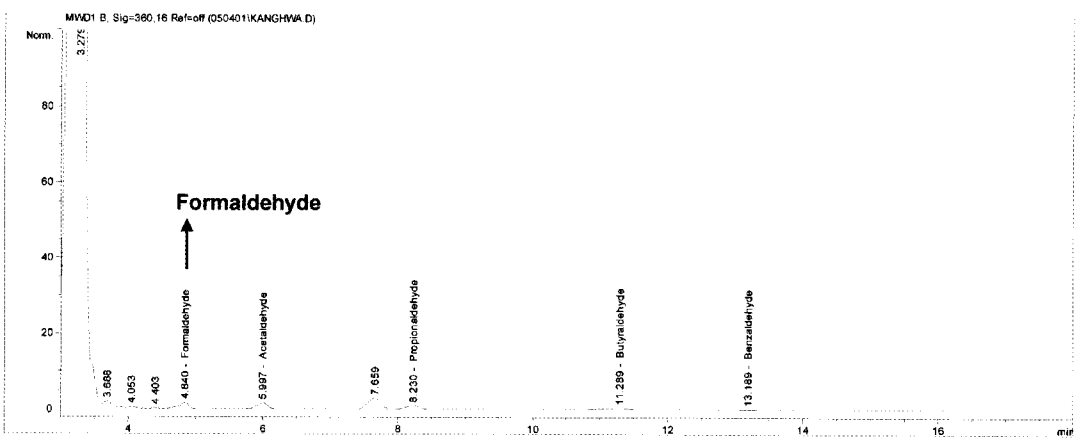
3.2. Field and Laboratory Emission Cell (FLEC)

The HPLC analysis results of aldehyde emissions from the wood-based composites are shown in Fig. 6 in the form of aldehyde chromatograms. From the aldehyde chromatograms, formaldehyde was the firstly detected aldehyde at the early retention time of 4.8 min because of its simple molecular structure of HCOH. Differences of each peak height and area for the wood-based composites were measured. Table 6 presents the peak areas from all wood-based composites calculated from these chromatograms. As the wood-based composites were made with formaldehyde-based resins such as UF and PF which are commonly used in industry, formaldehyde registered the highest emission among the various aldehydes. The other detected aldehyde compounds were acetaldehyde, acrolein/acetone, propionaldehyde, methacrolein, 2-butanone/butyraldehyde, benzaldehyde and isovaleraldehyde. The order of aldehyde emissions from the MDF and engineered flooring was formaldehyde > acetaldehyde > butyraldehyde > propionaldehyde > benzaldehyde, whereas propionaldehyde was detected at higher levels than butyraldehyde from the laminate flooring.

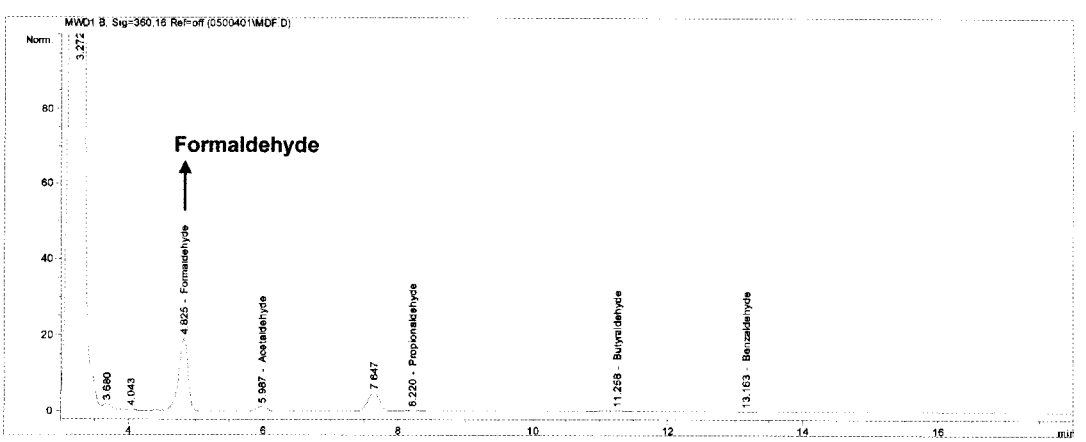
Formaldehyde EFs were calculated from these



1) Engineered flooring



2) Laminate flooring



3) MDF

Fig. 6. Aldehyde chromatograms of wood-based composites by FLEC.

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

Aldehyde	Retention time (min)	Peak area		
		Engineered flooring	Laminate flooring	MDF
Formaldehyde	4.84	18.94	18.20	185.91
Acetaldehyde	5.99	9.17	17.92	13.10
Propionaldehyde	8.23	3.90	14.97	6.36
Crotonaldehyde	9.85	ND*	ND	ND
Butyraldehyde	11.30	7.09	11.48	8.24
Benzaldehyde	13.17	1.63	1.82	1.69

ND*: non-detected

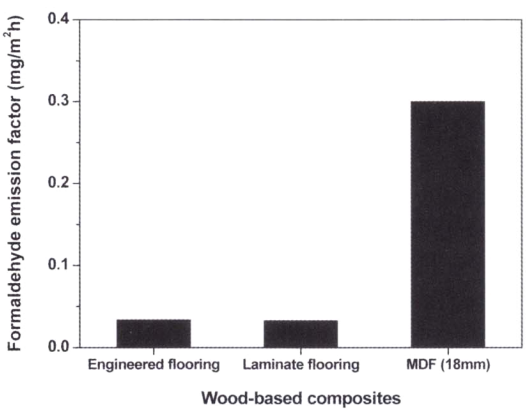
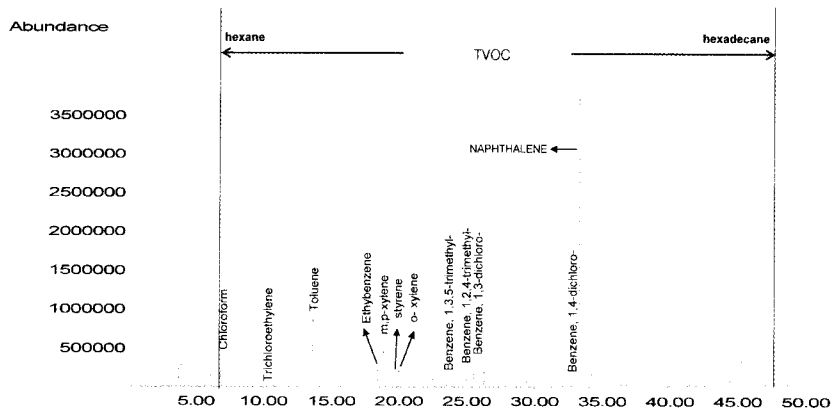


Fig. 7. Formaldehyde emission factor of wood-based composites as determined by FLEC.

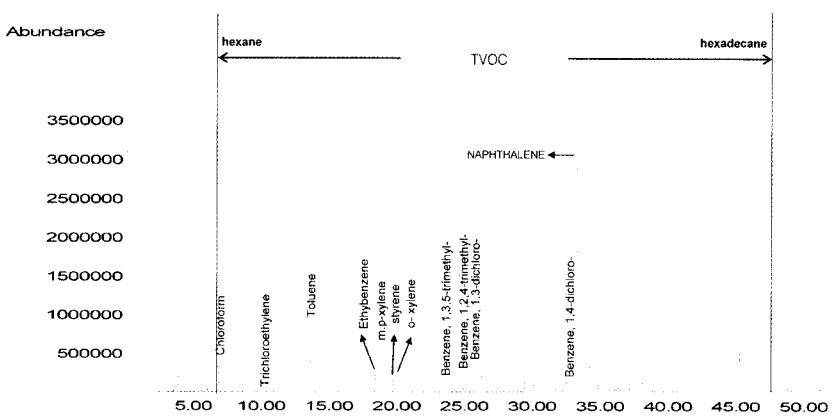
peak areas and are shown in Fig. 7. As expected given the composition of the typical, wood-based composite building materials, the formaldehyde EF of MDF was higher than that of flooring materials made to satisfy the E_1 grade, although that of engineered flooring was a little higher than that of laminate, in contrast to the results from the typical desiccator and perforator methods. This small difference in the formaldehyde emission data can be explained by the structure of the wood-based composites. A correction is performed to account for the formaldehyde emitted from the entire faces of the sample for the desiccator test, but only from the upper surface for the FLEC method, according to similar results from other studies with the 20 ℓ small chamber (Kim *et al.*, 2006). Non-veneered MDF is not coated by materials such as low-pressure melamine laminates, decorative film and UV curable varnish, which greatly increases the formaldehyde emission level. Furthermore, no consideration is made for emissions from the sample edges. On the other hand, flooring materials are composed with a UV-cured coating for engineered flooring and MF resin-impregnated paper for laminate flooring. Despite these differences of test principle between the two typical methods (desiccator and perforator) and FLEC, the overall formaldehyde emission results showed a similar trend.

TVOC chromatograms of the wood-based composites are shown in Fig. 8. Koontz and Hoag [26] reported that unfinished PB and MDF from North America emitted many different VOCs in addition to formaldehyde, and often at greater VOCs concentrations than formaldehyde. The major VOCs reported were (in approximate order of emissions): acetone, hexanal, pentanal, benzaldehyde, pentanol, heptanal, pinenes, nonanal and octanol. In this experiment, the MDF specimens emitted chloroform, benzene, trichloroethylene, toluene, ethylbenzene, m,p-xylene, styrene, and o-xylene. TVOC EFs between C_6 and C_{16} for each wood-based composite are shown in Fig. 9.

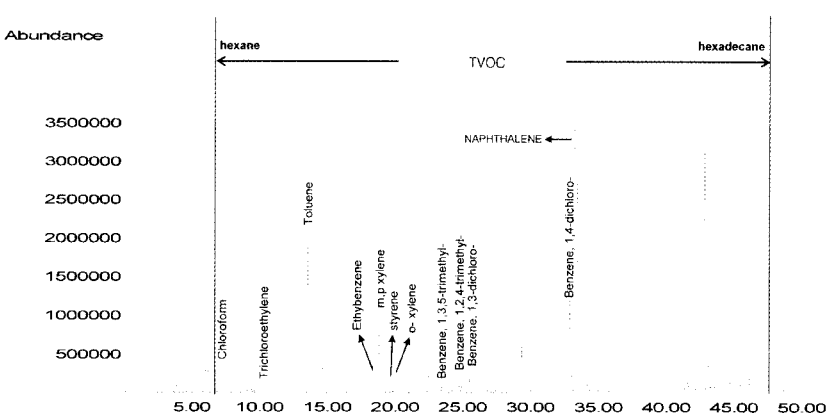
In a trend similar to that observed with formaldehyde EF, the MDF specimens emitted significantly more VOCs, almost two times of the engineered flooring and laminate flooring speci-



1) Engineered flooring



2) Laminate flooring



3) MDF

Fig. 8. TVOC chromatograms of wood-based composites by FLEC.

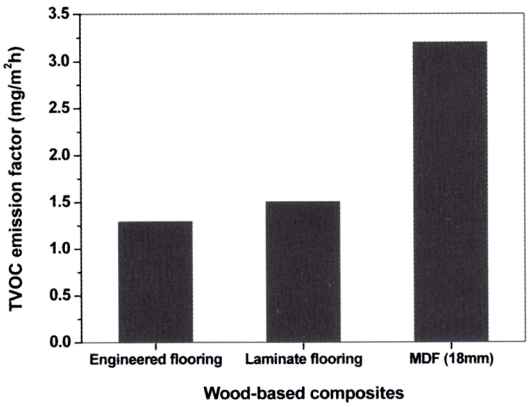


Fig. 9. TVOC emission factor of wood-based composites as determined by FLEC.

mens. However, many natural VOCs such as α- and β-pinene are emitted from MDF (Kim *et al.*, 2006). As TVOC calculated between C_6 and C_{16} includes these harmless, natural VOCs, the TVOC EF from PB was higher than that from engineered flooring. In Korea, the Ministry of Environment provides guidelines for VOC emissions from building materials in terms of TVOC; i.e., even natural VOCs from wood are considered 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.

4. CONCLUSION

Formaldehyde and VOC emissions from wood-based composites can adversely affect indoor air quality. In Korea, standard test methods have been developed to determine formaldehyde and VOC emission levels from building products. The Ministry of Environment has regulated the use of pollutants emitted from building materials following the Korean government's decision to control IAQ in 2004. The 20 ℓ small chamber test was developed in Japan and its performance has been ratified to comply with ASTM, ECA reports, and ENV 13419-1. However, the 20 ℓ small chamber method requires a long testing time of 7 days. For international compliance using this method, the formaldehyde and VOCs must be gathered into a DNPH-cartridge and Tenax-TA tube at 7 days after sample installation in the 20 ℓ small chamber. In comparison, FLEC was successfully applied as a test procedure for formaldehyde and VOC emission with the advantage of much simpler equipment and procedure, as well as, a significantly shorter total test time less than one hour.

To determine the formaldehyde emission levels, the peak areas of each wood-based composite were calculated from the aldehyde chromatograms obtained by FLEC. The order of the peak areas of aldehydes from the wood-based compounds was formaldehyde > acetaldehyde > butyraldehyde > propionaldehyde > benzaldehyde. As expected from the results of the two typical methods, desiccator and perforator, the formaldehyde emission factor of MDF was higher than that of flooring materials satisfying the E_1 grade, however, that of engineered flooring was slightly higher than that of laminate flooring, in contrast to the results obtained from two typical methods. Although the formaldehyde emission results from the FLEC method showed the similar trends as those from the typical methods, many factors considered including the sample size, collection method for the emitted formaldehyde and analysis procedure should be. For exact quantification of the correlation between the two typical methods and the FLEC procedure, these factors must be considered in conjunction with the performance results of many tests.

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