# Physico-mechanical Properties and Formaldehyde/TVOC Emission of Particleboards with Volcanic Pozzolan[*1]

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## ABSTRACT

The purpose of this study was to investigate the physico-mechanical properties and characteristics on reduction of formaldehyde and total volatile organic compound (TVOC) emission from particleboard (PB) with added volcanic pozzolan. Pozzolan was added as a scavenger at the level of 1, 3, 5, and 10 wt.% of urea formaldehyde (UF) resin for PB manufacture. The moisture content, density, thickness swelling, water absorption and physical properties of PBs were examined. Three-point bending strength and internal bond strength were determined using a universal testing machine. Formaldehyde and TVOC were determined by desiccator and 20L small chamber methods. With increasing pozzolan content the physical and mechanical properties of the PBs were not significantly changed, but formaldehyde and TVOC emissions were decreased. Because pozzolan has a rough and irregular surface with porous form, it can be used as a scavenger for PBs at a content up to 10 wt.% without any detrimental effect on the physical and mechanical properties.

*Keywords* : pozzolan, formaldehyde emission, TVOC, particleboard, scavenger

## 1. INTRODUCTION

With the increasing standard of living, the more prosperous consumers, who are health conscious, are increasingly turning to natural, wood-based construction materials for interior decoration and flooring. As the consumption of wood-based raw materials increases, the need for wood substitutes is also growing [1].

In the past, urea formaldehyde (UF) and phenol-formaldehyde resin binders have contributed greatly to the progress made by the wood industry. These adhesives are widely used as a major component in the production of building and furniture materials, such as medium density fiberboard (MDF), particleboard (PB), and plywood. However, decreasing the emission levels of formaldehyde fumes from PBs manufactured using UF resins has now become one of the major concerns of the timber and wood adhe-

sives industry, particularly in the case of adhesively bonded, wood products. Recently, attention has turned to other volatile organic compounds that may be emitted from wood products. These VOCs include chemicals naturally present in the wood, as well as those added during processing. In new energy-efficient buildings, air exchange rates are low, permitting concentrations of VOCs to accumulate to detectable and possibly harmful levels. The adverse health effects associated with these increased VOC concentrations include eye and respiratory irritation, irritability, inability to concentrate and sleepiness. Moreover, health and the environment constitute two key concerns of the 21$^{st}$ century [2,3,4].

Many building materials emit VOCs which have the potential to affect health and comfort. The formaldehyde emission from wood-basedmater- ials is usually determined in a reaction chamber at a predefined temperature, humidity and ventilation rate. The concentration of formaldehyde in the air within the chamber is measured until a constant concentration is reached. This is a time consuming method and it also requires special equipment [5,6].

The Korean government has started controlling indoor air quality since 2004. The law from the Ministry of Environment regulates the use of pollutant emitted building materials and prohibits the use of materials that emit formaldehyde more than 1.25 mg/ m$^2$ · h (JIS A 1901, small chamber method). This is equivalent to E$_2$ grade ( >5.0 mg/L) when changed to the desiccator method (JIS A 1460). Most suppliers and people are concerned about how to reduce pollutants from building materials and how to control indoor air quality [7].

Post-treatment methods to decrease and minimize formaldehyde release are based on compounds like ammonia, ammonium salts, or urea. Another effective way to reduce formaldehyde

release is the addition of formaldehyde-binding substances ("scavengers") to the resin or to the wood particles [8,9]. A conventional formaldehyde scavenger added to UF resins is urea, which is often used in combination with ammonium chloride (about 20:1). Urea also acts as a buffer to control the pH and improvethe stability of UF resins. Ammonium chloride acts as an acid catalyst of the curing reaction and as a scavenger. The urea/ammonium chloride system apparently has no effect on the resin curing and bonding properties of the PB product if it is added up to 10% of the UF resin dry weight. Other approved formaldehyde scavengers are organic amines. Especially in Sweden, dispersion systems based on formaldehyde-binding paraffins were developed [10]. These wax systems are often added to the wood particles before drying. The amounts necessary to obtain perforator values below 10 mg depend on plant conditions and on the perforator value of the untreated board. The scavenger does not negatively influence the board strength and pressing time.

Natural pozzolans are defined as either raw or calcined natural materials that have pozzolanic properties (e.g., volcanic ash or pumicite, opaline chert and shales, tuffs, and some diatomaceous earths). Historically, they are among the oldest materials used in combination with lime for construction purposes. For example, a volcanic eruption around 1500 BC on Santorin Island, Greece, left a large deposit of natural pozzolans there. In modern construction technology, natural pozzolans are still used in various applications [11]. Pozzolanic materials can be natural in origin or artificial and are available widely. They have been used throughout the world to make good quality concrete in recent years. Although they have been used successfully in many countries, research attention continues to focus on finding new and improv-

Table 1. Chemical composition of pozzolan by weight percent [14]

| Element | $SiO_2$ | $Al_2O_3$ | $TiO_2$ | $Fe_2O_3$ | MgO | CaO | $Na_2O$ | $K_2O$ | MnO | $P_2O_5$ | LOI[a] |
|---------|---------|-----------|---------|-----------|-----|-----|---------|--------|-----|----------|--------|
| Pozzolan | 59.5 | 17.7 | 0.9 | 7.6 | 1.3 | 0.2 | 0.08 | 4.7 | 0.1 | 0.08 | 5.0 |

[a] Loss on ignition

ed ways to form high strength concrete with new pozzolanaddi- tives. Due to the strength it imparts to concrete and its durability, the silica fume by-product from industrial waste materials is a well-known and very efficient pozzolan [12].

We used volcanic pozzolan to reduce VOCs emitted from PB. Volcanic pozzolans are poorly crystallized materials rich in $SiO_2$ and $Al_2O_3$, with a porous form and a high content of ab- sorbed water [13]. Therefore, the porous form of volcanic pozzolan is expected to absorb gen- erated odor and VOCs of PB.

The purpose of this study on wood-based UF PBs was to investigate the effect of adding vol- canic pozzolan as a scavenger on the phys- ico-mechanical properties and on the character- istics on reduction of formaldehyde and TVOC emission.

# 2. EXPERIMENTAL

## 2.1. Materials

The wood particles used for manufacturing PB were donated by the Donghwa Enterprise Co., Ltd. in South Korea, and consisted of re- cycled chips used for the core, which had 3% moisture content. UF resin was used to produce the PB with a molar ratio of 1.25 (F/U). This resin was supplied by Tae Yang Chemical Co. Ltd. Before PB manufacture, 3 parts (to resin) of 25% ammonium chloride as a hardener and 13 parts of 44% wax solution for water-proof- ing were added. Pozzolan was added as a scav- enger at the level of 1, 3, 5 and 10 wt.% of UF resin.
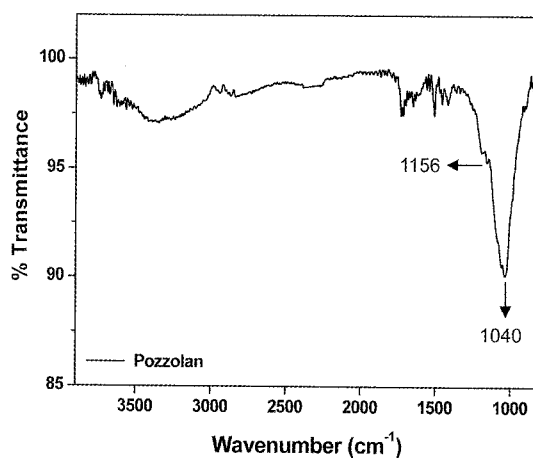


Fig. 1. FTIR spectra of volcanic pozzolan [14].

Volcanic pozzolan was obtained from Auto- Win Co. The chemical composition of pozzolan, as determined using an X-ray fluorescence spec- trometer (Shimadzu XRF-1700, Japan), is shown in Table 1 which presents the high concen- tration of $SiO_2$ and $Al_2O_3$ elements in pozzolan [14]. The particle size of pozzolan is over 70 μm. Fourier transform infrared (FTIR) spectra of pozzolan were obtained using a Thermo Nicolet Nexus 870 FTIR spectrophotometer and are shown in Fig. 1. The absorption bands in the region $3600 \sim 3100$ cm$^{-1}$ were assigned to hydroxyl (OH) groups bonding to silicon of pozzolan [13]. The absorption band at 1040 cm$^{-1}$ was attributed to the Si-O(Al)-O vibrations of pozzolan. The shoulder absorption band at the 1156 cm$^{-1}$ region was due to amorphous silica [15]. These results were confirmed by the functional groups of pozzolan. Scanning elec- tron microscopy (SEM) was used to measure the porous surfaces of the pozzolan using a SIRIOM SEM instrument (FEI Co., USA). The

acceleration potential used during this investigation was 20 kV. The total scanning time during elemental map generation was 20 min. Prior to the measurement, the specimens were coated with gold (purity, 99.99%) to eliminate electron charging.

## 2.2. Manufacturing Particleboards

The PBs were fabricated using UF resin with pozzolan added to obtain a specific gravity of 0.8 and dimensions of 270 mm × 270 mm × 8 mm (length × width × thickness). The wood particles were placed in a rotary drum mixer and rotated as they were sprayed with the UF resin used as the composite binder. The quantity of adhesive was calculated to be 14 wt.% of the raw material based on the oven-dried weight. The mixture of particles and adhesives was cold pressed at 2 kg/cm$^2$ for 2 minutes, to ensure the stability of the mat and to obtain the proper density gradient of the composites prior to hot pressing.

The mixture was then hot pressed to form composite boards at a peak pressure of 30 kg/cm$^2$ and temperatures of 160°C. The press time was 5 minutes, with the pressure being released in two steps of 1 minute each. The fabricated PBs were pre-conditioned at 25°C and 65% RH for two weeks before testing.

## 2.3. Physical and Mechanical Test

Moisture content, density, thickness swelling and water absorption were examined using the ASTM D 1037-99 method. Density was controlled by quality control testing, wherein each value represents the average of five samples. Three-point bending strength and internal bond strength were determined using a Universal Testing Machine (Zwick Co.) using the ASTM D 1037~99 method. Each value represents the

average of five samples. The results were compared with the ANSI A208.1-1999 standard.

## 2.4. Desiccator Method for Formaldehyde Emission Test

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 compositesby 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 samplesof a specified surface area are placed in the desiccator filled with the specified amount of distilled water or deionized water. The 24-h desiccator method uses a common glass desiccator with a volume of 10 ± 1 liters. Eight test specimens, with dimensions of 5 cm × 15 cm, were positioned in the desiccator. The emission test lasted 24 hour in the covered desiccator at a temperature of 20°C. The emitted formaldehyde was absorbed in a water-filled petri dish and was analyzed with the chromotropic acid method. 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 (Japanese Industrial Standard, 2001).

## 2.5. 20L Small Chamber Test and TVOC Emission Factor

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 20L small cham-

Table 2. Test condition of 20L small chamber method

| Variables | Condition |
|---|---|
| Chamber volume | 20L |
| 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°C |

Table 3. Sampling condition of 20L 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 |

ber 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 listed in Table 2. The test samples, 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 hours. Sampling conditions are listed 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%, relatively, and the chamber was ventilated at 0.5/h. Aldehydes were analyzed by HPLC, and TDS/GC-MS was used for VOCs, as listed in Table 4 and Table 5. In this paper, TVOC was defined by converting all areas of the peaks between $C_6$ and $C_{16}$ to concentrations using the tolueneresponse factor. A peak area under 10 was defined as the limit of detection.

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 |

The sample gas was taken by Tenax-TA and 2,4-DNPH car- tridge 7 days after the sample specimens were installed into the 20L small chamber, according to the regulation prescribed by the Korean Ministry of Environment.

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

Thus, ERcan 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 case examples considered here, only EF is used [16].

Table 5. Analysis conditions for VOCs

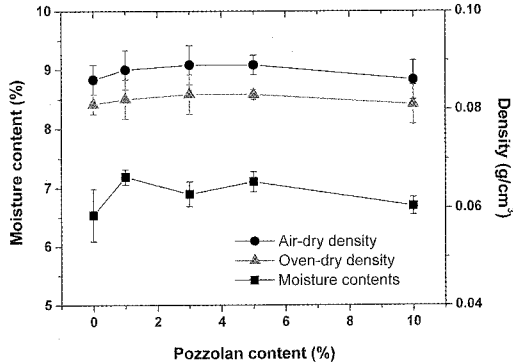| Variables | | Condition |
|---|---|---|
| TDS | | Perkin Elmer ATD400 |
| GC/MS | | HP6890/Agilent5973 |
| Column | | RTX-1 (105 m × 0.32 mm × 3 μm) |
| Carrier gas and flow | | He (99.99%) |
| Temperature program | | 40°C (5 min) → 70°C (5 min) →150°C (5 min) → 200°C (5 min) → 220°C (5 min) → 240°C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



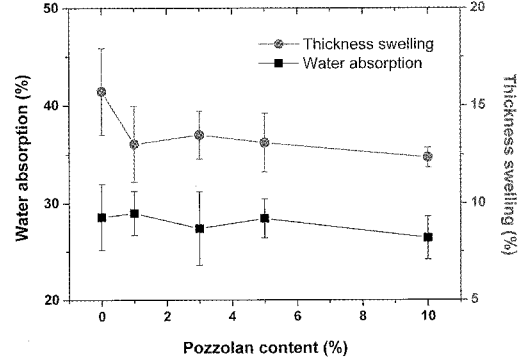Fig. 2. Moisture content and density of particleboard at different pozzolan contents.



Fig. 3. Water absorption and thickness swelling of particleboardat different pozzolan contents.

# 3. RESULTS and DISCUSSION

## 3.1. Physical and Mechanical Properties

No significant difference was found in the moisture content and density of the PBs made with pozzolan at the investigated concentrations. The moisture content of the different pozzolan was in the range of 6.5~7.2 wt.%, as shown in Figure 2. Because a low pozzolan content of 1 ~10% wt.% of UF resin was added in order to avoid any detrimental effects on the normal properties of UF resin, the moisture content and density were not effected. The physical proper-

ties (thickness swelling and water absorption) of the investigated PBs are shown in Fig. 3. The thickness swelling of PB were decreased with increasing pozzolan content. The thickness swelling was decreased by 6% with 1% added pozzolan. However, water absorption was not significant affected by pozzolan content. On the whole, the PB physical properties were not markedly changed from those of control PB (0% of pozzolan) with pozzolan content ranging from 1~10 wt.%.

The same tendency was evident in the mechanical roperties. Fig. 4 shows the bending and internal bonding strength of PBs with different pozzolan content. The bending and internal
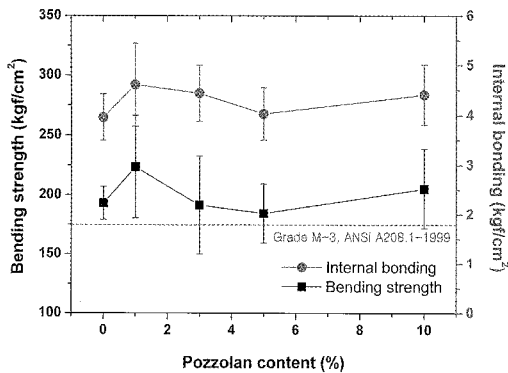
Fig. 4. Internal bonding and bending strength of particleboard at different pozzolan contents.

bonding strength of the PBs were not significantly changed by changing pozzolan content. However, the strength of the PBs was slightly increased at 1% pozzolan content. Normally, the addition of inorganic materials such as silica and zeolite degrades the physical and mechanical properties of composites because of interfacial failure between hydrophilic and hydrophobic materials. Although functional and environmentally friendly properties were increased, basic properties such as physical and mechanical properties must not be decreased. The low pozzolan content of only 10%.wt of the UF resin restricted any decreasing of the physical and mechanical properties. Based on ANSI [17], the bending strengths of all the adhesive systems for all press times wereover Grade M-3.

## 3.2. Formaldehyde and TVOC Emission

Formaldehyde is a well known emission from wood-based products such as PB, MDF and plywood bonded with formaldehyde-based resin. Especially, UF resin has the highest formaldehyde emission rate because of incompletely cured, UF resin bonded, wood-based products. To reduce formaldehyde emission, pozzolan was added to UF resin at various contents in
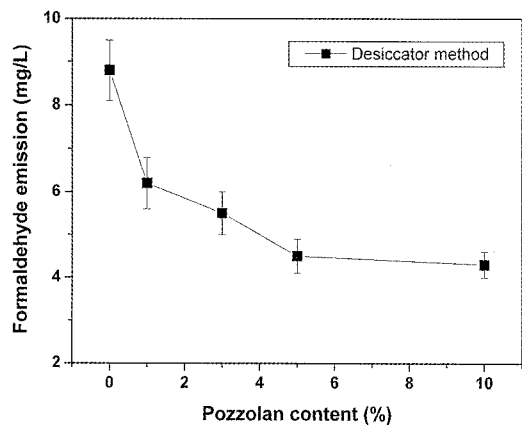


Fig. 5. Formaldehyde emission from particleboard at different pozzolan contents by desiccator method.

this study.

Fig. 5 shows the reduction of formaldehyde emission with increasing pozzolan addition. The formaldehyde emission of PBs, determined by desiccator method, was decreased with increasing pozzolan content. However, all PBs still satisfied the $E_2$ grade ($1.5 \sim 5.0$ mg/L). Pozzolan has been reported to have a rough and irregular surface with porous form [14]. This porous pozzolan structure was considered to act by capturing and absorbing formaldehyde, thereby reducing the formaldehyde emission of the PBs during the manufacturing process. These results were confirmed by the reduced formaldehyde emissions of the PBs with increasing pozzolan content.

The most basic testing method used to test formaldehyde emission in the furniture and flooring industries is the desiccator method, as defined in KS F 3104 and KS F 3200, which are similar to JIS A 1460 (Building Boards Determination of Formaldehyde Emission-Desiccator Method) [18]. This method is inexpensive and simple to carry out. Considering the small-scale nature of the building, flooring and furniture-related industry in Korea, several practical testing methods need to be developed
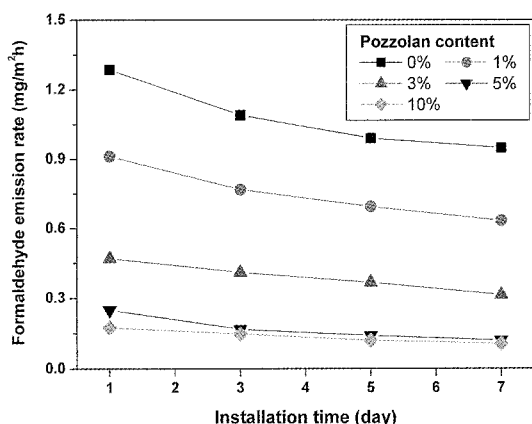
Fig. 6. Formaldehyde emission from particleboard at different pozzolan contents by the 20L chamber method as detected by GC/MS analysis recorded 7 days after sample installation.
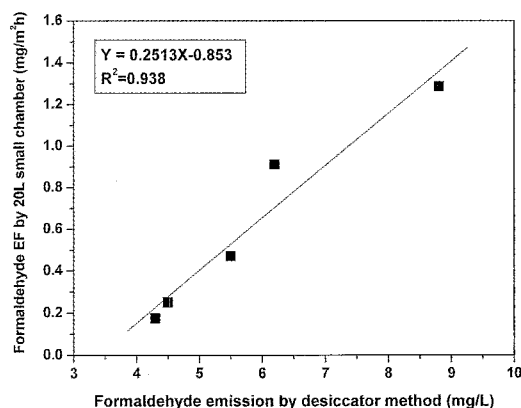


Fig. 8. Correlation between desiccator and 20L small chamber methods for the determination of formaldehyde emission from particleboard at different pozzolan contents.
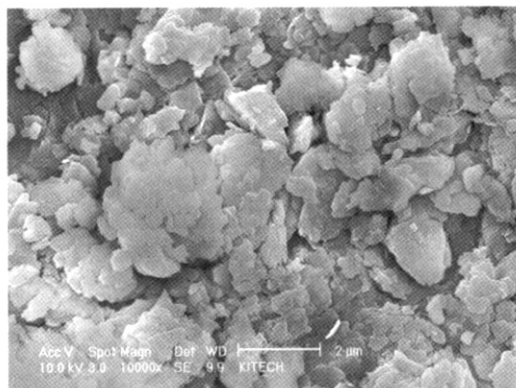


Fig. 7. SEM micrographs of the pozzolan: 10,000×.

and the correlations among them established. The Korean Ministry of the Environment started controlling IAQ in 2004 by regulating the use of pollutant emitting building materials. 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 E$_2$ grade (>5.0 mg/L) when converted to the desiccator method [19].

The formaldehyde EF results are shown in Fig. 6. The samples were tested for 7 days, as mandated by the Korean Ministry of Environ-

ment, in the 20L small chamber and the formaldehyde emissions in the 2,4-DNPH cartridge were sampled after 1, 3, 5, and 7 days. The results showed a similar tendency with those from the desiccator method in that pozzolan addition reduced the formaldehyde emissions. With increasing period after sample installation, formaldehyde EFs were decreased. At 7 days after sample installation, the formaldehyde emission was 0.949 mg/m$^2$ · h for UF resin, but was reduced to under 0.12 mg/m$^2$ · h with 5% pozzolan content. SEM micrographs of pozzolan are presented in Fig. 7. The SEM micrograph of pozzolanshows a rough and irregular surface with porous form. This porous pozzolan structure was considered to act by capturing and absorbing toluene, thereby reducing the formaldehyde emission during the PB manufacturing process.

The formaldehyde emission results of the desiccatorand 20L chamber methods showed good correlation, as shown in Fig. 8. From the report by Marutzky [20], wood-based panels show a correlation between the emission values, as determined in large-chamber tests, which are used for the fundamental classification proce-

Table 6. VOCs from particleboard at different pozzolan contentstested with the 20L chamber method, as detected by GC/MS analysis

| Resin | Pozzolan 0 % (mg/m$^2 \cdot$ h) | | | |
|---|---|---|---|---|
| Sample installation day | 1 | 3 | 5 | 7 |
| Chloroform | 0 | 0 | 0 | 0 |
| 1,2-Dichloroethane | 0 | 0 | 0 | 0 |
| Benzene | 0 | 0 | 0 | 0.004 |
| 1,2-Dichloropropane | 0 | 0 | 0 | 0 |
| Trichloroethylene | 0 | 0 | 0 | 0 |
| cis-1,3-Dichloropropene | 0 | 0 | 0 | 0 |
| trans-1,3-Dichloropropene | 0 | 0 | 0 | 0 |
| 1,1,2-Trichloroethane | 0 | 0 | 0 | 0 |
| Toluene | 0.126 | 0.065 | 0.071 | 0.300 |
| 1,2-Dibromomethane | 0 | 0 | 0 | 0 |
| Tetrachloroethylene | 0 | 0 | 0 | 0 |
| Chlorobenzene | 0 | 0 | 0 | 0 |
| Ethylbenzene | 0.045 | 0.019 | 0.029 | 0.036 |
| Styrene | 0.023 | 0.006 | 0.012 | 0.016 |
| 1,1,2,2-Tetrachloroethane | 0 | 0 | 0 | 0 |
| o-Xylene | 0.052 | 0.019 | 0.028 | 0.026 |
| m,p-Xylene | 0.140 | 0.054 | 0.080 | 0.073 |
| Benzyl chloride | 0 | 0 | 0 | 0 |
| 1,3,5-Trimethylbenzene | 0.010 | 0.004 | 0.008 | 0.005 |
| m-Dichlorobenzene | 0 | 0 | 0 | 0 |
| o-Dichlorobenzene | 0 | 0 | 0 | 0 |
| p-Dichlorobenzene | 0 | 0 | 0 | 0 |
| 1,2,4-Trichlorobenzene | 0 | 0 | 0 | 0 |
| Hexachlorobutadiene | 0 | 0 | 0 | 0 |
| Known Total (%) | 0.395(9.3) | 0.168(4.1) | 0.228(7.6) | 0.460(17.6) |
| Unknown Total (%) | 3.874(90.7) | 3.954(95.9) | 2.777(92.4) | 2.155(82.4) |
| Total Concentration (%) | 4.269(100) | 4.122(100) | 3.006(100) | 2.615(100) |

dures and for basic research on wood products, and their actual formaldehyde content sufficiently strong to allow the use of the formaldehyde content value as a basis for their classification. Based on the correlation between the large-chamber and perforator values, the perforator method was the second method to gain acceptance for the determination of the emission class of PBs in Germany. The desiccator methods have thus become the standard test method for determining formaldehyde emission. There-

fore, the chamber method which produced results well correlated with the standard methods can be successfully applied to the pre-test measurement of formaldehyde emission [19].

Representative TVOC chromatograms at 7 days after sample installation of PBs bonded with UF resin are listed in Table 6. Koontz and Hoag [21] reported that unfinished PB and MDF from North America emitted many VOCs in addition to formaldehyde, and often at greater concentrations than formaldehyde. Major
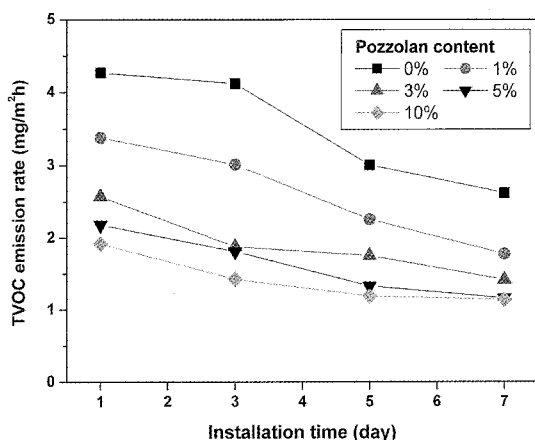
Fig. 9. TVOC emission of particleboard at different pozzolan contents from the 20L chamber method as detected by GC/MS analysis recorded 7 days after sample installation.

VOCs reported were (in approximate order of emissions): acetone, hexanal, pentanal, benzaldehyde, pentanol, heptanal, pinenes, nonanal and octanol. In our experiment, the PB specimens emitted hexanal, pinenes, pentanal, nonanal, heptanal, and octanol. However, many natural VOCs such as $\alpha$-pinene and $\beta$-pinene are also emitted from wood-based panels. The Korean Ministry of Environment provides guidelines for VOC emissions from building materials in terms of 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. Because more than 90% of the emissions are unknown VOCs, high emission VOCs need to be regulated and included in the TVOC emission calculations. Furthermore, non-harmful VOCs such as natural VOCs from wood, $\alpha$-pinene and $\beta$-pinene, are included as harmful VOCs when TVOCs are calculated. TVOC was calculated between $C_6$ and $C_{16}$, as shown in Fig. 9. The final data at 7 days were similar with the

formaldehyde data. TVOC emission from PBs was decreased with increasing pozzolan content. Especially, the samples with 5 and 10% of pozzolan gained a citation level in the Korean standard for environmentally friendly building material of one clover mark. Pozzolan addition to UF resin decreased the TVOC emission, but the effect was limited because of the low level of pozzolan addition.

# 4. CONCLUSION

Pozzolan was added to UF resin to reduce formaldehyde emission from PB. Because pozzolan has a rough and irregular surface with porous form, its addition to UF resin increased the formaldehyde absorption and reduced TVOC emission. Howev- er, with increasing pozzolan content, the internal bonding and bending-strengths, and general physical and mechanical properties of the PBs were not significantly changed because the maximum pozzolan content was low at 10%. According to the Japanese and Korean (JIS and KS) standards, although the achieved emission levels of the PB product could not be reduced to within the $E_1$ grade, the results presented here have clearly supported the effectiveness of pozzolan addition in PB for reducing the formaldehyde and VOC emissions of PB without any degradation in physical and mechanical properties.

# ACKNOWLEDGMENTS

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