

# DEVELOPING A RAPID TEST METHOD FOR MEASURING VOLATILE ORGANIC COMPOUNDS FROM THREE-LAYER PARQUET WITH MICROCHAMBER

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**Abstract.** The volatile organic compounds (VOCs) released from wooden floors can cause poor indoor air quality; therefore, the 1-m<sup>3</sup> chamber method is usually used with wooden floors to test for those VOC emissions. However, that method is costly, time consuming, and has limited application in various test environments. In this study, a rapid test method was used to detect VOC emissions from three-layer parquet under various test conditions and to compare the results with those from the traditional 1-m<sup>3</sup> chamber method. The VOC constituents and mass were confirmed by gas chromatography-mass spectrometry. Results showed that the VOCs emitted from three-layer parquet comprised aromatic hydrocarbons and esters. The optimum conditions when sampling VOCs emitted from three-layer parquet using the rapid test method were 80°C, 60% RH, and 0.2 m<sup>3</sup>·m<sup>-2</sup>·h<sup>-1</sup> area-specific airflow rate. The time required for the total VOC emissions to reach a state of equilibrium was 10 da for the rapid test method, which was 18 da shorter than the time required by the 1 m<sup>3</sup> chamber method. Both methods detected the same VOCs and the R<sup>2</sup> of two methods was 0.9598, which, being close to one, showed good correlation. Therefore, the rapid test method may be helpful in improving efficiency and reducing the costs associated with detection of VOCs released from wood flooring.

**Keywords:** Chamber method, rapid test method, microchamber, three-layer parquet, VOCs.

## INTRODUCTION

Wood-based panels, such as plywood, medium density fiberboard, and particleboard, are ordinarily used in manufacturing furniture, flooring, and other wood products (Kim et al 2010; Sun and Shen 2010; Liu et al 2015). However,

wood products bonded with urea formaldehyde have become a major source of indoor volatile organic compounds (VOCs) and formaldehyde emissions (Salthammer et al 2010; Yrieix et al 2010; Wang et al 2014). The VOC emissions from wood products affect indoor air quality and can cause a variety of symptoms, such as dry coughs, tiredness, headaches, dizziness, and difficulty concentrating (WHO, 1989; US EPA,

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1990; Kim et al 2001; Xu and Zhang 2003; Kim et al 2006). Therefore, the study of VOC emission characteristics released from wood products is important.

Recently, various methods for detecting VOCs from wood-based panels have been introduced, such as full-size environmental chambers, field and laboratory emission cells (FLECs), and the desiccator methods. The desiccator method was used to collect VOCs in Germany in the 1990s (Kim and Kim 2005). The FLEC method, with microemission cells that feature high sensitivity, has become the European standard for emission testing (Kim 2010). At present, small-scale environmental chambers are widely used to detect VOCs emitted from wood-based panels (Brown 1999; Funaki and Tanabe 2002; Kim et al 2007). ASTM D 5116 (2010) stipulates that a small-scale environmental chamber should be used to determine organic emissions from indoor materials and products. ASTM D 6330 (2014) provides guidelines for determining VOC (excluding formaldehyde) emissions from wood-based panels using a small environmental chamber under defined test conditions.

Environmental chambers are widely used in China to study VOCs (Shen et al 2009, 2012). The Chinese standard HJ 571 (2010) stipulates that 1-m<sup>3</sup> environmental chamber should be used for the detection of VOC emissions. This method accurately simulates the release into the environment from wood-based panels. However, the chambers are expensive (millions of Chinese Yuan) and time required for testing with a 1-m<sup>3</sup> chamber is about 28 da, which is inefficient for both researchers and enterprises. Therefore, a speedy, highly accurate and low-cost detection method is needed urgently.

Scholars have confirmed that various external factors, such as RH, temperature, and the rate of air exchange rate can affect the release of VOCs (Sollinger et al 1993; Van der Wal et al 1997; Wolkoff 1998; Wiglusz et al 2002; Liu et al 2010; Zhang et al 2010). Researchers frequently use thermal extraction methods to obtain the emission characteristics of VOCs from the

plastic products, but the method still needs to be verified for use with wood-based panels (Kieliba et al 2007). The aim of this work was to develop a rapid, low-cost test method for detecting VOCs released from wood-based panels. Furthermore, the reliability of the rapid test method was analyzed by studying the correlation between the results from the rapid test method and those when using a 1-m<sup>3</sup> chamber.

## MATERIALS AND METHODS

### Materials

Because three-layer parquet is widely used by Chinese families, a three-layer parquet product produced in Zhejiang, China, was chosen as the experimental material and was obtained directly from the manufacturing line. Table 1 lists the detailed parameters of the three-layer parquet used in this study. After being cut into cylinders with diameters of 60 mm, the specimens were covered with aluminum foil to prevent the release of VOCs. The specimens were subsequently stored in Telfon bags and placed in a refrigerator until needed.

### Methods

***The rapid test method for volatile organic compound collection.*** The sampling device for the rapid test method was composed of a Micro-Chamber/Thermal Extractor M-CTE250 (Markes International, Llantrisant, UK) connected to a cylindrical water tank to regulate the humidity of the carrier gas (Fig 1). The increased control functions allowed the temperature to be adjusted between 0°C and 250°C and the RH to be adjusted between 30% and 80%. Otherwise, the unit maintained a constant airflow with low background concentrations.

The inner surface of the microchamber was cleaned with acetone; then, clean nitrogen, which was used as the carrier gas, was supplied for approximately 1 da. The specimens were put into the micropools, and the humidified carrier gas entered the microchamber, with its humidity controlled by a Digital Thermo-Hygrometer TY-9700

Table 1. Detail parameters of the three-layer parquet.

Balance veneer	Middle layer	Hardwood surface	MC	Length × width × thickness (mm × mm × mm)	Adhesive	Resin content (g/m <sup>2</sup> )	Primer	Finishes
1.5 mm poplar	7 mm pine	3.5 mm Oak	8%	910.0 × 132.0 × 12.1	Melamine-modified UF resin	180-220	Polypropylene glycol (PPG)	Treffert paint

(Tian Yue Environmental Protection Technology, Beijing, China). The cycle time was 8 h·da<sup>-1</sup>, and VOCs emitted from the specimen surfaces were collected in Tenax-TA tubes (200mg, 60-80 mesh; SKC, Shanghai, China). Airflow was sampled 19, 47, and 94 mL·min<sup>-1</sup>, with 2 L of air collected in total each day. Specimens were maintained continuously under constant conditions within the microchamber until the VOC emissions reached a state of equilibrium. Steady-state equilibrium was determined when the relative deviation in VOC concentration between 1 da and the next was less than 5%. Table 2 presents the operating parameters maintained during the experiment.

**The 1-m<sup>3</sup> chamber method for volatile organic compound collection.** For comparison, a 1-m<sup>3</sup> environmental chamber V-1000 (Simplewell Scientific Co., Dongguan, China) was used to detect VOC emissions, which both the device and the sampling method maintained in accordance with ISO 16000-9(2006) and ISO 16000-6(2011). The three-layer parquet was spliced to ensure the exposed specimen area was 1 m<sup>2</sup>, and the edges were covered with aluminum foil. Before the

start of the experiment, the inner chamber was washed with distilled water. During the experiment, purified, humidified air was supplied at an airflow rate of 16.7 L·min<sup>-1</sup>, and the air temperature and RH inside the chamber were kept at a constant at 23 ± 0.5°C and 50 ± 3%, respectively. The VOCs emitted from the surface of the specimens were captured in Tenax-TA tubes and sampled on day 1, 3, 7, 14, 21, and 28. The sample airflow was 150 mL·min<sup>-1</sup> and 3 L of gas was collected each time.

VOCs absorbed by Tenax-TA tubes were thermally desorbed with a thermal desorption sample injector TP-5000 (Beifen Instrument Technical, Beijing, China) at 280°C for 5 min and were subsequently characterized and quantified with a DSQ II gas chromatography-mass spectrometer (GC-MS; Thermo Fisher Scientific, Waltham, MA). Table 3 presents the basic parameters for the GC-MS. The GC temperature program was 40°C for 2 min, raised to 50°C in 2°C·min<sup>-1</sup> increments and maintained for 5 min, followed by 150°C raised in 5°C·min<sup>-1</sup> steps and maintained for 4 min, and finally, 250°C raised at 10°C·min<sup>-1</sup> increments, maintained for 8 min.

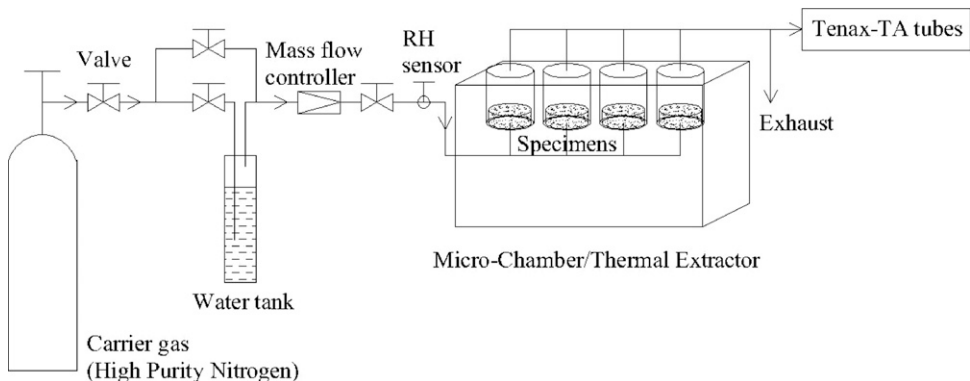


Figure 1. Schematic diagram of the experimental system.

Table 2. Microchamber/thermal extractor operating parameters of the rapid test method.

Variables	Conditions
Specimen area	$5.65 \times 10^{-3} \text{ m}^2$
Cell volume	$1.16 \times 10^{-6} \text{ m}^3$
Loading factor	$4.87 \times 10^3 \text{ m}^2/\text{m}^3$
Area specific airflow rate	0.2/0.5/1.0 $\text{m}^3 \cdot \text{m}^{-2} \cdot \text{h}^{-1}$
Temperature	60°C/80°C
RH	40%/60%

According to GB/T 18883-2002 Interior Air Quality Specification standards, the VOCs were determined as all areas of the peaks at the range of hexane ( $\text{C}_6$ ) and hexadecane ( $\text{C}_{16}$ ). The internal standard method of quantitative analysis was used to determine the mass of the VOC constituents, and toluene- $\text{D}_8$  was treated as the internal standard. The individual VOC constituents were identified by retention time with a standard mass-spectra library and a match quality of not less than 90%, and they were quantified based on response factors derived from the standard curves.

## RESULTS AND DISCUSSION

### The Rapid Test Method for Detection of Volatile Organic Compounds

The main components of VOCs emitted from the three-layer parquets were confirmed as aromatic hydrocarbons and esters; in addition to alkanes, alkenes, aldehydes, and ketones, there were also some small amounts of ethers, alcohols, and acids (Table 4).

The components extracted from the wood were complex and were mainly composed of terpenes, flavonoids, fatty acids, and lignophenol. The alkenes released from the three-layer parquet were primarily extracted from the wood, whereas the alkanes were generated by chemical reactions

among the wood extracts. However, the aromatic hydrocarbons were not derived from the wood extracts or from other wood components, so their source was inferred to be the adhesives used in the manufacturing process as well as the organic solvents used in finishing the surface of three-layer parquet.

Total VOC emissions from the specimens gradually decreased with time and reached an equilibrium after 10 da. Higher temperature and humidity had a significant effect on VOC emissions (Li et al 2007; Zhu et al 2013). Increased temperature and RH, within a certain range, increased initial VOC emissions; however, high airflow rates decreased total VOC emissions (Fig 2). Compared with an increase in RH, an increase in temperature increased VOC emissions more significantly. As shown in Fig 2, the optimum conditions for the rapid test method when sampling of VOCs emitted from three-layer parquet were 80°C, 60% RH, and  $0.2 \text{ m}^3 \cdot \text{m}^{-2} \cdot \text{h}^{-1}$  area-specific airflow rate.

The downward trends in the VOC concentrations over time showed that the internal VOC concentration of the three-layer parquet formed a concentration gradient with the external environment at the initial stage. According to the mass-transfer principle, internal VOCs from the parquet are released rapidly into the external environment until the point at which the VOC concentration within the parquet reaches an equilibrium with that of the external environment. The emission curves of the three-layer parquets were not smooth, which might be the result of systematic or random error during the process of detection and acquisition of the VOCs.

High temperatures increased total VOC emissions from the three-layer parquet (Fang et al 1999; Lin et al 2009). Because the compound

Table 3. The basic parameters of gas chromatography-mass spectrometer.

Variables	Condition
Column	DB-5 (3 m × 0.26 mm × 0.25 μm)
Carrier gas	He (99.99%)
Temperature program	40°C (2 min)→50°C (4 min)→150°C (4 min)→250°C (8 min)
Ion source	Electron ion
Ion source temperature	230°C
Scan mode	full scan (40-450 amu)

Table 4. The main constituents of volatile organic compounds emitted from the three-layer parquet by the rapid test method and 1-m<sup>3</sup> chamber method.

Category	Main components
Aromatic hydrocarbons	Toluene, Ethylbenzene, o-Xylene, p-Xylene, acetophenone, 1,3-dimethyl benzene, 1-methylene 1H-Indene, butylated hydroxytoluene
Esters	Acetic acid butyl ester, 3-methylheptyl acetate, 2-propenoic acid 2-ethylhexyl ester
Alkenes	2-propenylidene cyclobutene, 1,3,5-cycloheptatriene, 3,7-dimethyl 1-octene, tetradecene, copaene, cedrene
Aldehydes and ketones	Hexanal, benzaldehyde, nonanal, 2-methyl cyclopentanone, benzophenone
Alkanes	Octane, decane, undecane, dodecane, tridecane, tetradecane, pentadecane, hexadecane
Others	n-butyl ether, 2-ethyl 1-hexanol, $\alpha$ -hydroxy- $\alpha$ -methyl benzeneacetic acid

vapor pressure increased with increasing temperature, the air pressure difference between the internal and external air also increased. However, high temperatures also increased the diffusion coefficient of the VOCs in the three-layer parquet, which could increase the release of VOCs, according to the mass-transfer principle. Increases in RH also accelerated the release of

VOCs because it promoted adhesive hydrolysis and produced moisture expansion within the pore structure of the wood.

Under optimum conditions, the main VOC constituents detected by the rapid test method were esters and aromatic hydrocarbons (Table 5). The initial and equilibrium concentrations of the

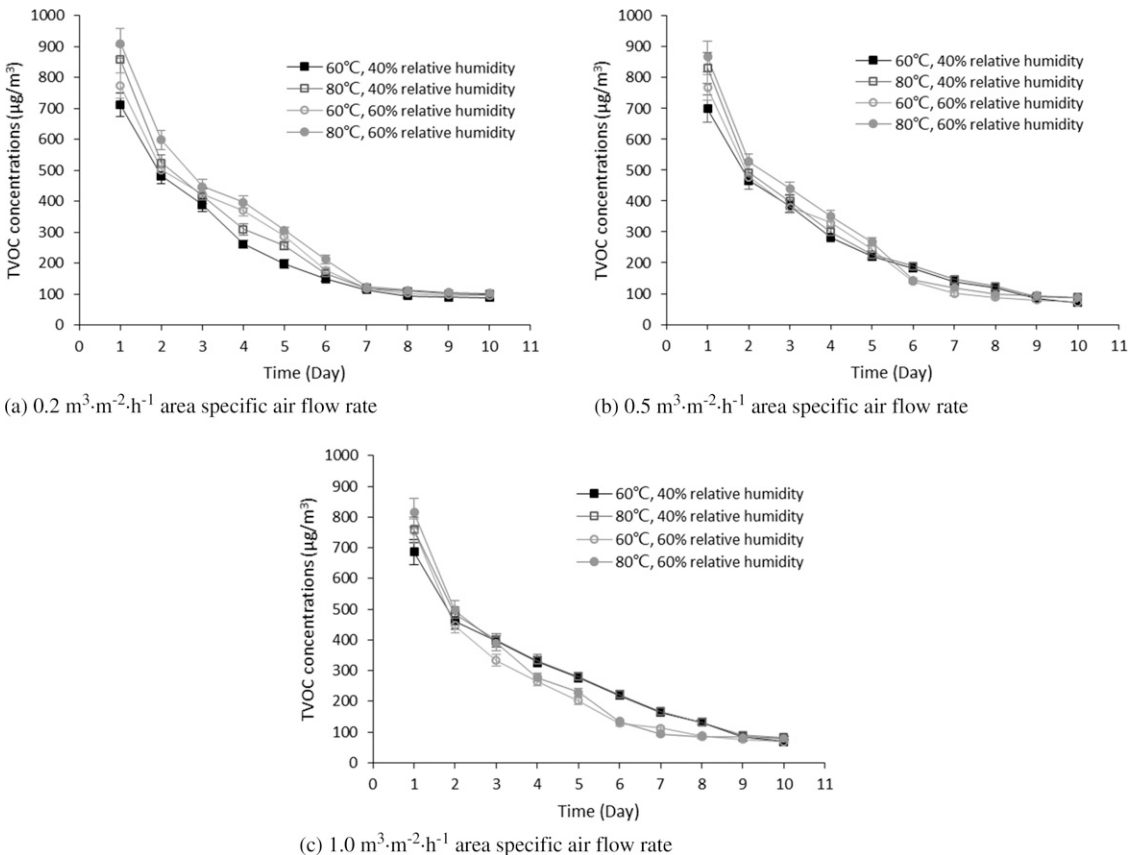


Figure 2. TVOC concentrations of the three-layer parquet by the rapid test method.

Table 5. The concentrations of the main constituents of volatile organic compounds (VOCs) released from three-layer parquet by rapid test method.

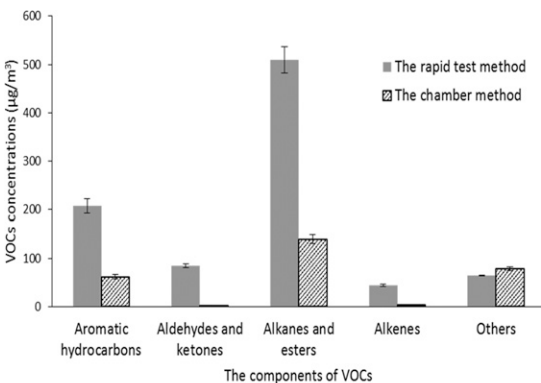
Time (da)	VOC concentrations ( $\mu\text{g}/\text{m}^3$ )					
	Category					
	Aromatic hydrocarbons	Aldehydes and ketones	Alkanes	Esters	Alkenes	Others
1	207.8	84.6	37.6	472.1	43.6	64.7
2	160.1	73.6	30.6	269.8	32.4	32.4
3	132.1	50.7	18.5	203.0	20.5	22.9
4	117.1	48.6	15.5	156.1	15.6	45.1
5	103.1	43.7	10.6	104.6	10.3	35.0
6	84.3	30.5	7.5	70.7	7.3	12.7
7	51.4	23.7	4.9	30.2	6.0	7.7
8	48.9	22.8	4.3	27.4	5.2	4.6
9	45.9	22.6	3.1	23.5	4.3	6.8
10	44.8	21.2	2.5	21.1	4.1	9.7

total VOC emissions from the three-layer parquet were  $910.5 \mu\text{g}\cdot\text{m}^{-3}$  and  $103.3 \mu\text{g}\cdot\text{m}^{-3}$ , respectively. Meanwhile, the initial concentrations of the esters and aromatic hydrocarbons were  $472.1 \mu\text{g}\cdot\text{m}^{-3}$  and  $207.8 \mu\text{g}\cdot\text{m}^{-3}$ , and their total was  $679.9 \mu\text{g}\cdot\text{m}^{-3}$ , which accounted for 74.7% of the total VOC concentration. When the VOCs were at equilibrium, the primary component was confirmed to be aromatic hydrocarbons at a concentration of  $44.8 \mu\text{g}\cdot\text{m}^{-3}$ , accounting for 43.4% of the total VOC concentration.

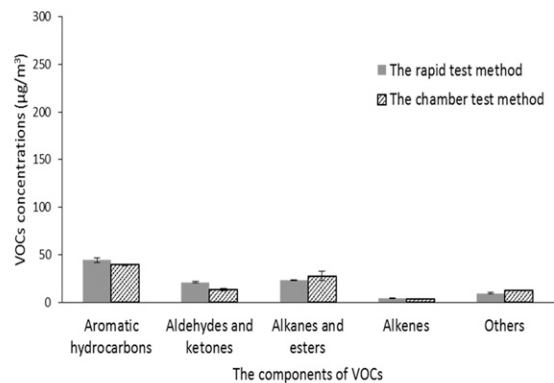
### Correlation between the Rapid Test and 1-m<sup>3</sup> Chamber Methods

The main constituents of the VOCs released from the three-layer parquets as determined by the 1-m<sup>3</sup> chamber method were alkanes and esters, which had

a total initial emission concentration of  $139.6 \mu\text{g}\cdot\text{m}^{-3}$  (Fig 3). The aromatic hydrocarbons, which had an initial emission concentration of  $61.5 \mu\text{g}\cdot\text{m}^{-3}$ , were less than one half of the total amount of the alkanes and esters. The main components, as well as their proportions within the total VOC, were largely consistent with the data from the rapid test method. When a state of equilibrium was reached, the primary final residue from the VOCs was confirmed to be aromatic hydrocarbons, the same as found with the rapid test method, and the emission concentration was  $39.6 \mu\text{g}\cdot\text{m}^{-3}$ , accounting for 40.8% of the total VOC concentration. At a state of equilibrium state, there was no obvious variance in the release amounts from each constituent between the two methods. In addition, the same types of VOCs were detected by both methods.



(a) The initial concentrations of VOCs



(b) The equilibrium concentrations of VOCs

Figure 3. The initial and equilibrium emission concentrations of volatile organic compounds (VOCs) by two methods.

According to the emission trends of total volatile organic compounds (TVOC) released from the three-layer parquet, the result can be divided the curves into four stages (stages I, II, III, and IV) (Fig 4). Stages I-III were the rapid emission period, and stage IV was the stable emission period. In stage I, the rapid test method required 1 da for stage completion, whereas the 1-m<sup>3</sup> chamber method needed 3 da. The rate at which the TVOC decreased with the rapid test method was 12.98  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$ , much faster than that of the 1-m<sup>3</sup> chamber method (1.58  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$ ). To complete stage II, the rapid test method took 2 da, whereas the 1-m<sup>3</sup> chamber method required 4 da, with the TVOC decrease rate of 4.19  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$  and 0.39  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$ , respectively. For stage III, the rapid test method required 5 da for completion, and the 1-m<sup>3</sup> chamber method took 14 da at decrease rates of 2.43  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$  and 0.09  $\mu\text{g}\cdot\text{m}^{-3}\cdot\text{h}^{-1}$ , respectively. Stage III took the most time for completion by either method. During the stable emission period (stage IV), the total emission values of the VOCs had no significant decreases and tended to be stable.

Under optimum conditions, we analyzed the correlation between the TVOC release values from the three-layer parquet as measured by the rapid test method and the 1-m<sup>3</sup> chamber method (Fig 5). The rapid test method was used to detect TVOC emission concentrations from the 1 da to

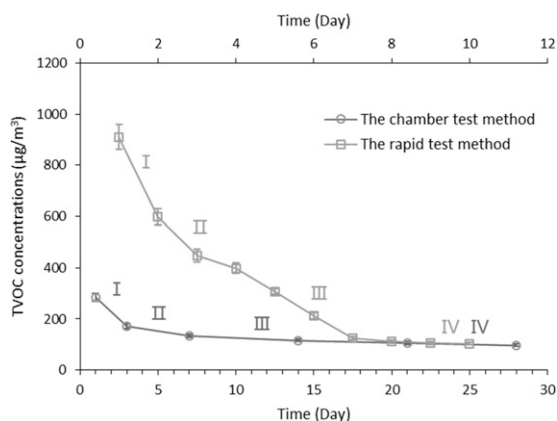


Figure 4. Comparison of two methods of TVOC emission concentrations from three-layer parquet.

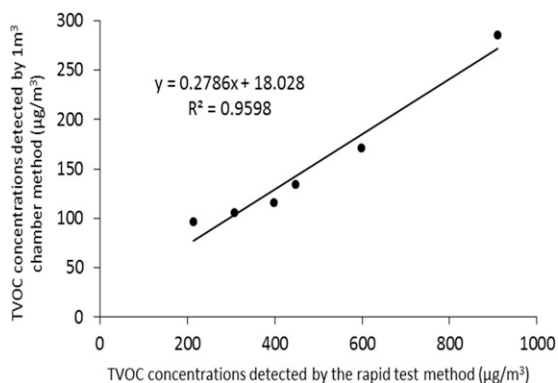


Figure 5. Correlation of volatile organic compounds (VOCs) released from the three-layer parquet detected by the rapid test method and 1-m<sup>3</sup> chamber method.

6 da and the 1-m<sup>3</sup> chamber method was used to detect TVOC emission concentrations on 1, 3, 7, 14, 21, and 28 da, respectively. The fitting equation was  $y = 0.2786x + 18.028$  for the six groups of data, and the degree of fit was 0.9598. Therefore, results from the rapid test method correlated well with those from the 1-m<sup>3</sup> chamber method. At the same time, the TVOC release values of the three-layer parquet by the 1-m<sup>3</sup> chamber method could be obtained with the fitting equation, and the test time was shortened 18 da.

The release trends for the TVOC for the three-layer parquets using either the rapid test method or the 1-m<sup>3</sup> chamber method were basically the same; moreover, the same species of VOCs were detected. The initial emission concentrations for the TVOC detected by the rapid test method were greater than those of the 1-m<sup>3</sup> chamber method, but the equilibrium emission concentrations were lower. TVOC emissions reached a state of equilibrium state in 10 da with the rapid detection method, a significantly shorter period than required for the 1-m<sup>3</sup> chamber method. The rapid detecting device provided reliable performance, easy operation, and low cost.

## CONCLUSIONS

The VOC emissions from three-layer parquet were detected within a microchamber under various test conditions. The types of VOC released

from the wood panels, as determined by the two test methods, were basically the same, primarily aromatic hydrocarbons and alkenes. The initial release rate of TVOC was higher with the rapid test method than it was with the 1-m<sup>3</sup> chamber method, and the results of both methods correlated well. The time required by the rapid test method was shorter than that required by the 1-m<sup>3</sup> chamber method. Screening the VOC emissions from three-layer parquet in shorter times can improve the quality of the products.

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