Volatile Organic Compounds and Odor Emissions from Alkyd Resin Enamel-coated Particleboard

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To solve the odor emission problem of lacquer veneer particleboards, as well as to identify the key odor compounds and their sources, alkyd resin enamel-coated particleboards were studied *via* gas chromatography-mass spectroscopy-olfactometry. A single-factor experiment was established under various environmental conditions. The odor-causing compounds were identified, and the effects of environmental factors on the volatile organic compounds (VOCs) and odor emissions were investigated. The results showed that the main odor substances released from the alkyd resin enamel-coated particleboard were aromatic compounds, aldehydes, and alkanes, which are typically low in toxicity. The total volatile organic compounds concentration and total odor intensity increased as the temperature and relative humidity increased, but decreased as the air exchange rate to loading factor ratio increased. The temperature had a greater impact on the release of VOCs from the alkyd resin enamel-coated particleboard than either the relative humidity or air exchange rate to loading factor ratio. The odor substances concentration increased with an increase in the temperature, while the relative humidity and air exchange rate to loading factor ratio had little effect.

Keywords: Alkyd resin enamel-coated particleboard; Volatile organic compounds (VOCs); Odor characteristics; Environmental factors

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INTRODUCTION

In recent years, people have increasingly used wood materials in interior decorating because of its natural affinity and visual sense of harmony (Ada 2011). At the same time, indoor air pollution has become an invisible killer (Yang and Zhang 2014). Wood coatings are used to better protect and decorate woodworking (Kee 2001), and problems have arisen from the organic compounds that are released, which can be a threat to the health of people (Landry *et al*. 2010).

After changes in interior décor, many people perceive an obvious "decoration smell", which is composed of a variety of single compounds with each having a different odor. Research has shown that most of those odors are from an excess of formaldehyde or other substances containing volatile organic compounds (VOCs) (Tang 2008). However, many people are still aware of this odor, even when the formaldehyde and VOCs levels do not exceed current standards. Therefore, the authors decided to determine and understand the overall odor components and sources from an alkyd resin enamel-coated particleboard through the individual analysis of each compound. In this study, gas chromatography–mass spectroscopy–olfactometry (GC-MS-O) was used, which couples the excellent separation of GC with the abundant structural information of MS (Hsu and

Shi 2013) and the human sense of smell, which can exceed the ability of many chemical detectors (Xia and Song 2006). This method has been widely used to select and evaluate active odor substances from complex mixtures (Zhang *et al*. 2009).

Analysis with GC-MS-O is based on GC and was first proposed in 1964 (Fuller *et al*. 1964; Acree *et al*. 1976). This analysis is performed by smelling the outflow components from GC-MS directly. Acree *et al.* (1976) improved the original GC-MS-O technology by adding humid air and smelling the GC outflow after film chromatography processing. Acree *et al*. (1984) and Ullrich and Grosch (1987) used dilution-analysis methods to analyze the intensity of various odors at the same time, which made GC-MS-O technology widely applicable in many situations. Currently, there are four major GC-MS-O detection methods, which are dilution analysis, time-intensity analysis, detection frequency methods, and posterior intensity evaluation (Maarse and van der Heij 1994). With development of extraction and separation technology, GC-MS-O has been used in the study of fruits (Guillot *et al*. 2006), vegetables (Ruth *et al*. 2007), dairy products (Frank *et al*. 2004), and mephitis treatment (Bulliner *et al.* 2006; Chen *et al.* 2008).

EXPERIMENTAL

Materials

In this experiment, particleboard samples that were produced by Suofeiya (Guangzhou, China) were chosen as the experimental material. The particleboard density was 0.60 g/cm^3 and the moisture content of the particleboard was 8%, which was determined *via* a testo-616 moisture detector (Grows Instrument international trade Co., Ltd, Shanghai, China). After hot pressing and a veneer treatment (*Fraxinus mandshurica* with a 0.25-mm thickness; urea formaldehyde resin and white latex at a ratio of 6:4; a coating amount of 100 g/m^2; a hot-pressing time of 3 min with a temperature of 100 °C), the boards were coated using Beijing Red Lion alkyd varnish with a special diluent, brush construction methods, two coats of sealer, and two barrier coats with a coating quantity of 100 g/m^2. The samples were cut into round pieces (60-mm diameter) for the microchamber/thermal extractor apparatus, with an exposed area of 5.65×10^{-3} m^2. After the edges of the specimens were sealed with aluminum foil to prevent the release of compounds, the samples were stored in polytetrafluoroethylene bags and refrigerated until needed, four specimens were made for an identity condition, and the total number of specimens was 24. The experimental schemes are detailed in Table 1.

Table 1. Experimental Scheme

Experimental Scheme	Research Direction	Temperature (°C)	Relative Humidity (%)	Air Exchange Rate to Loading Factor Ratio (m^3/m^{2}h)
A	Standard environment	23	40	0.5
B	Influence of the temperature	23, 30, 40	40	0.5
C	Influence of the relative humidity	23	40, 60	0.5
D	Influence of the ratio of air exchange rate & loading factor	23	40	0.2, 0.5, 1.0

Temperature values are ± 1 °C; Relative humidity values are ± 5%; Air exchange rate to loading factor ratio values are ± 0.05 m^3/m^{2}h

Equipment

Sampling

A Micro-Chamber/Thermal Extractor μ-CTE 250 (Markes International Inc., Llantrisant, UK) was connected to a cylindrical volumetric flask to provide moisture and regulate the carrier gas humidity. This device had an adjustable temperature range from 0 °C to 250 °C. Tenax TA tubes (Markes International Inc., Llantrisant, UK), with stainless-steel bodies, contained 200 mg of 2,6-dibenzofurans porous polymer, which efficiently adsorb and desorb VOCs.

Detection and analysis

A unity thermal analysis desorption unit (Markes International Inc., Llantrisant, UK) used nitrogen as the carrier gas and had the following related parameters: a thermal desorption temperature of 280 °C, cold-trap adsorption temperature of -15 °C, thermal analysis time of 10 min, and injection time of 1 min. Using a DSQ II series GC-MS (Thermo Scientific, Dreieich, Germany), GC was performed with a DB-5 quartz capillary column (3000 mm × 0.26 mm × 0.25 μm).

Helium was used as the carrier gas with a constant velocity of 1.0 mL/min *via* a splitless injection. The chromatographic column was initially kept at 40 °C for 2 min, and then the temperature was increased to 50 °C (2 °C/min) and kept constant for 4 min. Finally, the temperature was increased to 250 °C (10 °C/min) and kept constant for 8 min. The injection port temperature was 250 °C.

The following GC-MS conditions for a Sniffer 9100 olfactory detector (Brechbühler, Schlieren, Switzerland) were used: an electron ionization source, ion energy of 70 eV, transmission line temperature of 270 °C, ion source temperature of 230 °C, and mass scan range of 50 amu to 650 amu. The odor intensity of the compound was determined directly and recorded. The effluent of the GC capillary was divided into two parts, where one part entered the mass spectrometer and the other part was used for sensory evaluation (1:1 ratio). The transmission line temperature was 150 °C, and nitrogen was used as the carrier gas *via* a purge valve. Humid air was added to prevent the dehydration of the nasal mucosa.

Methods

Sampling

The experiment used Tenax TA sampling tubes to adsorb 2 L of VOCs from alkyd resin enamel-coated boards under various experimental conditions. The cell volume was 1.35×10^{-4} m^3. Four samples were collected with a sampling cycle of 8 h. The amount of each sample was 2 L. After sampling, the Tenax TA sampling tubes were wrapped in Teflon plastic bags until needed.

Analytical method

The GC-MS and built-in software were used to analyze the VOCs. The MS detection peaks were compared with the spectral library of the National Institute of Standards and Technology (NIST, Gaithersburg, MD, USA), which matched samples with a degree of 800 or more. An internal-standard method was used in this experiment, with deuterium substituted for toluene at a concentration of 200 ng/μL, which added 2 μL to the total solution. The internal-standard quantitative-analysis method used the following equation,

$$M_i = A_i \times \left(\frac{M_s}{A_s} \right) \qquad (1)$$

where M_s is the mass of the internal standard added to the calibration standard (μg), A_i and A_s are the peak areas of the products tested and internal standard (mvAmin), respectively, and M_i is the mass of the sample to which the internal standard is added (μg).

A time-intensity method was chosen for analysis of the compounds. When the sample was injected and the chromatogram yielded peaks, the human sensory-evaluation assessors simultaneously perceived and described the column outflow from the odor port, and recorded the time, type, and intensity of the odor. After specific training, four assessors (between 20 years and 30 years old, with no history of smoking and no olfactory organ disease) formed an odor-analysis evaluation group, and the experimental environment was set according to the reference standard I. S. EN 13725 (2003). The room was well ventilated and there were no peculiar smells within the room. The temperature was kept at 23 ± 2 °C throughout the entire experiment. Activities, such as eating, which might influence indoor odors, were forbidden for 5 h before the experiment.

The experimental results were recorded when the same odor characteristics were described by at least two assessors at the same time. The intensity value was based on the average value from the different assessors. The grade division of the odor concentration of a substance was based mainly on the human sense of smell. The discrimination of the odor intensity was done according to the Japanese standard (Law No. 91 1971). The odor intensity was divided into the five grades of 1, 2, 3, 4, and 5, which represented barely perceptible, slightly perceptible, obviously perceptible, strong smell, and extremely strong smell, respectively.

The primary odor compounds were identified *via* MS, retention index, and odor characterization. The retention index value was calculated through the retention time of *n*-alkane under the same conditions (van Den Dool and Kratz 1963). The experiment was repeated four times under fixed conditions.

RESULTS AND DISCUSSION

Identification and Source Analysis of the Odor Components of Alkyd Resin Enamel-coated Particleboard

Figure 1 shows the odor-time intensity spectrum under scheme A (the standard environment). The odor of the alkyd resin enamel-coated particleboard appeared from 5 min to 35 min, was focused from 15 min to 35 min, and reached its maximum odor intensity value at 26.30 min.

The main components emitted from the alkyd resin enamel-coated particleboard are listed in Table 2. While there were more components in the alkane and aromatic hydrocarbon categories, there were still small levels of aldehydes, esters, and alcohols.

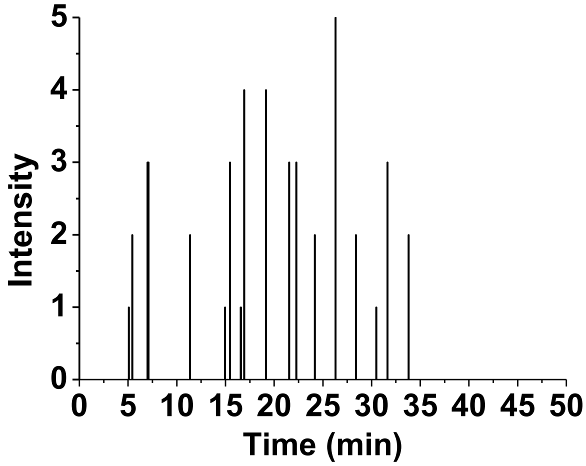


Fig. 1. Odor-time intensity spectrum of the alkyd resin enamel-coated particleboard

Table 2. Main Components Emitted from the Alkyd Resin Enamel-coated Particleboard

Component	VOCs of the Nitro-lacquer-coated Particleboard
Aromatics	Ethylbenzene; p-xylene; 1,3-dimethyl-benzene; trans-decahydro-naphthalene; trans-4a-methyl-decahydronaphthalene; 2-methyl-naphthalene
Alkanes	Pentane; 1,2-dichloro-propane; heptane; octane; 1,2,3-trichloro-propane; nonane; propyl-cyclohexane; 3,6-dimethyl-octane; 2-methyl-nonane; 3-methyl-nonane; 1-methyl-3-propyl-cyclohexane; decane; 2,6-dimethyl-nonane; 4-ethyl-heptane; 4-methyl-decane; 2-methyl-decane; 3-methyl-decane; undecane; 3-methyl-undecane; dodecane; 2,6-dimethyl-undecane
Aldehydes	Butanal; pentanal; hexanal; (z)-2-heptenal; (e)-2-decenal; 2-undecenal
Esters	Ethyl acetate
Alcohols	1,3-dichloro-2-propanol

Referring to the acute toxicity classification of compounds given by the World Health Organization (WHO/IPCS 1996) (Table 3), the authors were able to determine the odor compound characteristics and relevant parameters of the alkyd resin enamel-coated particleboard (Table 4).

Table 3. Acute Toxicity Classification of the Compounds by the World Health Organization

Toxicity Classification	Rats Orally LD_{50} (mg/kg)	Rat Inhalation and 1/3 to 2/3 Death in 4 h	Rabbit Transdermal LD_{50} (mg/kg)
Severe Toxicity	≤ 1	≤ 10	≤ 5
Highly Toxicity	1 to 50	10 to 100	5 to 43
Moderate Toxicity	51 to 500	101 to 1000	44 to 350
Low Toxicity	501 to 5000	1001 to 10000	351 to 2180
Slight Toxicity	5001 to 15000	10001 to 100000	2181 to 22590
Non-toxic	> 15000	> 100000	> 22600

Among the 35 species of VOCs, there were 18 species that were odorants. Aromatic hydrocarbons, aldehydes, and alkanes were the main sources of odors from the alkyd resin enamel-coated particleboard. Small amounts of odor substances were also found in the esters. The detailed odor characteristics and intensities are shown in Table 4. From the compounds emitted from the alkyd resin enamel-coated particleboard, ethylbenzene, p-xylene, and 2-decyl aldehyde were included in the list “VOCs from office furniture in greater than 10% of all products” from UL 2821 (2013). There was no direct correlation between the odor intensity and concentration of the different odorant compounds. However, the concentration can affect the odor intensity for certain types of compounds. Both p-xylene, with a with mass concentration of 1548.39 μg/m^3, and 1,3-dimethyl-benzene, with a mass concentration of 599.36 μg/m^3, presented the same odor intensity of 3.5.

Except for 1,2,3-trichloro-propane being moderately toxic and ethyl acetate being slightly toxic, the odor substances released from the alkyd varnish veneer particleboard had low toxicity levels. The odor substances released from the alkyd resin enamel-coated particleboard were primarily from the paint solvent. However, odor compounds also came from solubilization (surface active agents, dispersants, and plasticizers), pigments, spices, adhesive solvents, and particleboard emissions. The aldehydes were derived from the volatile by-products produced by the self-oxidation of unsaturated fatty acid methyl esters in the alkyd varnish. Related research has shown that self-oxidation by-products of hydroperoxide that only contain a single non-full double bond are mainly saturated aldehydes, ketones, and alcohols, and the compounds that contain two unconjugated unsaturated double bonds may produce a mixture of saturated and unsaturated aldehydes, ketones, and alcohols. Hexanal, heptenal, and octyl aldehyde had the highest contents (Hancock *et al*. 1989).

Table 4. Composition of Odorant Compounds of the Alkyd Resin Enamel-coated Particleboard

Compound Retention Time	RI	Mass Concentration (μg/m^3)	Toxicity Classification	Odor Character	Odor Intensity	Possible Source(s)
Aromatics						
Ethylbenzene	848	366.25	Low toxicity	Aromatic	1	Particleboard emission, paint solvent
p-xylene	857	1548.4	Low toxicity	Irritation, Aromatic, Sour	3.5	Paint solvent
1,3-dimethyl-benzene	879	599.36	Low toxicity	Aromatic and sweet	3.5	Particleboard emission, paint solvent, spice
trans-decahydro-naphthalene	1052	356.68	Low toxicity	Withered grass	2	Paint solvent
trans-4a-methyl-decahydronaphthalene	1110	326.88	Low toxicity	Grass	4.5	Paint solvent (reaction action)
2-methyl-Naphthalene	1336	240.85	Low toxicity	Almond	-	Solubilization (surfactants, dispersants)
Alkanes						
1,2-dichloro-propane	673	2165	Low toxicity	Sweet	3	Paint solvent

1,2,3-trichloro-propane	885	156.08	Moderate toxicity	Nausea and stench	4.5	Paint solvent
1-methyl-3-propyl-cyclohexane	985	157.47	Low toxicity	Cool	3.5	Paint solvent (reaction action)
Decane	1001	1824.3	Low toxicity	Lipid oxidation	3	Paint solvent
3-methyl-undecane	1173	151.9	Low toxicity	rancid flavor	2	-
Aldehydes						
Butanal	< 600	56.20	Low toxicity	Fresh	1	Volatile by-products, plasticizers, spice
Pentanal	668	514.96	Low toxicity	Irritation and scorched	3	Volatile by-products, accelerator, spice
Hexanal	778	3900.4	Low toxicity	Grass	2.5	Volatile by-products, plasticizers, the decomposition of the wood fibers
(z)-2-Heptenal	932	208.62	Low toxicity	Pine oil	4	Volatile by-products, spice
(e)-2-Decenal	1263	188.41	Low toxicity	Traditional Chinese medicine	1.5	Volatile by-products, spice
2-undecenal	1422	62.69	Low toxicity	Flower	2	Volatile by-products, spice
Esters	-	-	-	-	-	-
Ethyl acetate	< 600	831.4	Slight toxicity	Fresh and sweet	2	Paint solvent, Adhesive solvent

RI – retention index

Effect of Environmental Factors on the Emissions of VOCs

Figure 2a shows the changes in the total VOCs (TVOCs) concentration and total odor intensity under scheme B from day 1 to day 35. In the early stage, the TVOCs and total odor intensity reached their maximum values. However, they decreased over time until a stable phase was achieved (Liu *et al*. 2017).

When the temperature increased, the TVOCs concentration and total odor intensity also increased. As time proceeded, the distinction in the TVOCs concentration gradually diminished from the initial total odor intensity and the emissions gradually reached equilibrium. At 23, 30, and 40 °C, from day 1 to day 28, the average decline of the TVOCs was 95.30, 93.49, and 90.38%, respectively. When the temperature increased, the average decline of the TVOCs decreased. There were two possible reasons an increase in the temperature promoted the emission of TVOCs from the alkyd resin enamel-coated particleboard. An increase in the temperature could have promoted greater thermal motion in the TVOC molecules in the boards and surface paint, and this could have caused a rapid and massive emission of TVOCs if the material diffusion, desorption, evaporation, and chemical reaction rates increased at the same time (Li *et al*. 2013). Additionally, an increase in the temperature could have caused the mass transfer resistance to decrease, which benefitted the release of TVOCs from the material. This could have increased the mass transfer flux and release coefficient, which could have led to an increase in the TVOCs concentration (Li *et al*. 2008).

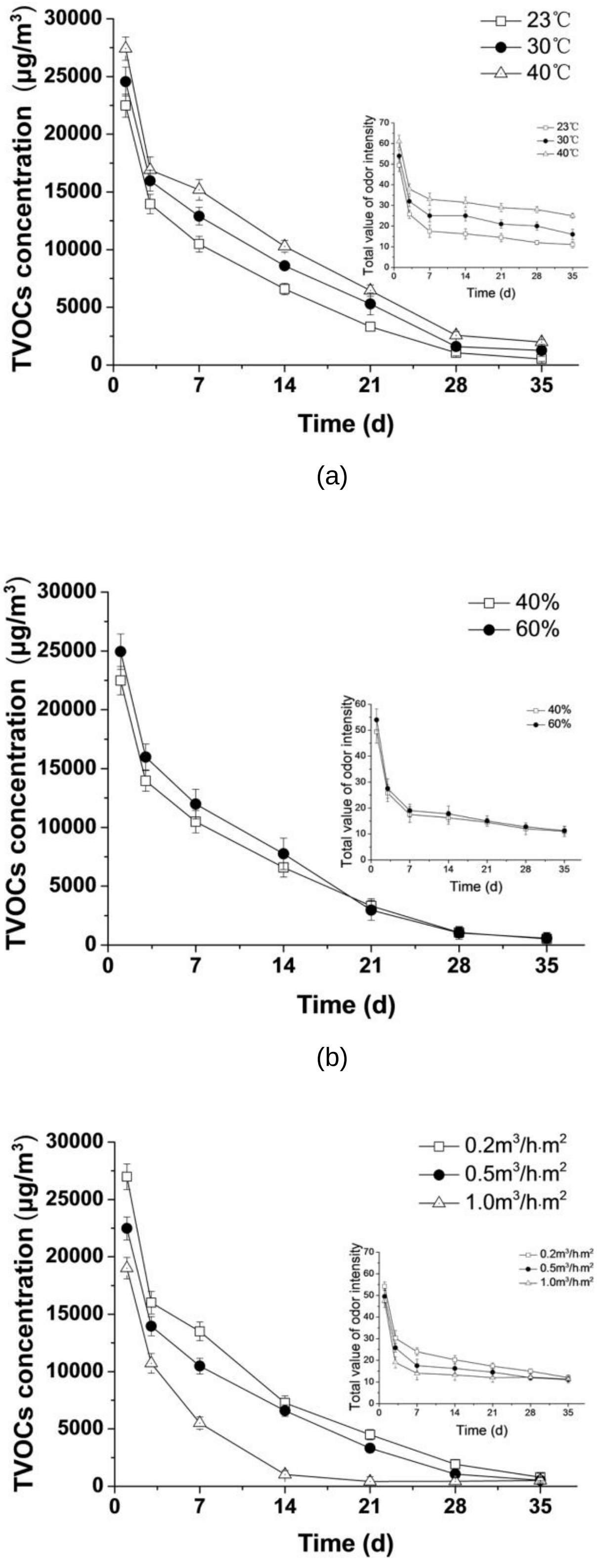
Figure 2b shows the influence of the relative humidity on the TVOCs concentration and total odor intensity under scheme C. On the 1st, 3rd, 7th, and 14th days, the concentrations at 60% relative humidity (2461.2 µg/m^3, 2031.3 µg/m^3, 1507.4 µgm^{-3}, and 1154.8 µg/m^3, respectively) were higher than those at 40% relative humidity, and the odor intensities were 4.5, 1.75, 1.5, and 1.5, respectively. After 21 d, the effect of the relative humidity on the release of TVOCs and total odor intensity of the alkyd resin enamel-coated particleboard gradually weakened. Previous experiments have shown that, in general, the TVOCs concentration and total odor intensity increase as the relative humidity increases, which may be explained by the change in the pore structure caused by the acceleration of hydrolysis, expansion of the pores in the drying layer, and hygroscopicity favoring the release of TVOCs (Shan *et al*. 2013; Zhu *et al*. 2013; Wang *et al*. 2017). Additionally, the moisture content of the oil coating increased with an increase in the relative humidity, which could have been caused by a decrease in the mass transfer resistance as the release coefficient increased, which led to an acceleration in the TVOCs emission (Li *et al*. 2008).

Figure 2c displays the relationship between the TVOCs concentration, total odor intensity, and air exchange rate to loading factor ratio under scheme D. In general, when the air exchange rate to loading factor ratio decreased, the TVOCs concentration increased and the time required for the TVOCs to reach equilibrium was shortened. The reason for this was that the increase in ventilation reduced the thickness of the material and air boundary layer, the mass transfer coefficient of the TVOCs in the air boundary layer increased, the mass transfer resistance decreased, and the release coefficient increased, which resulted in an acceleration of the TVOCs emission.

Effect of Environmental Factors on the Components of VOCs Emissions in the Equilibrium State

Figure 3 shows the TVOCs and odor components concentrations in the alkyd resin enamel-coated particleboards for different environmental factors in the equilibrium state. The TVOCs and odor components concentrations in the stable phase changed with changes in the single factors. An increase in the temperature and relative humidity or a decrease in the air exchange rate to loading factor ratio promoted the emission of most VOCs. The main odor substances released from the alkyd enamel-coated particleboard were aromatic compounds, aldehydes, and alkanes. The amounts of aromatic compounds and alkanes increased with an increasing temperature and relative humidity (Xue *et al*. 2016) and decreased as the air exchange rate to loading factor ratio increased. The emission of odorant aldehydes increased when the temperature increased, but it decreased when the relative humidity and air exchange rate to loading factor ratio increased.

At 23 °C, 30 °C, and 40 °C, the proportion of the odor substances concentration to TVOCs concentration was 51.7%, 53.7%, and 75.8%, respectively, while at 40% and 60% relative humidity, the proportion was 60.8% and 59.1. The proportion was 44.5%, 51.7%, and 50.6% when the air exchange rate to loading factor ratio was 0.2 m^3/m^{2}h, 0.5 m^3/m^{2}h, and 1.0 m^3/m^{2}h, respectively. The proportion of the odor substances concentration to TVOCs concentration increased with an increase in the temperature. The relative humidity and air exchange rate to loading factor ratio had little effect. The temperature had a greater impact on the release of VOCs from the alkyd resin enamel-coated particleboard than the relative humidity or air exchange rate to loading factor ratio.

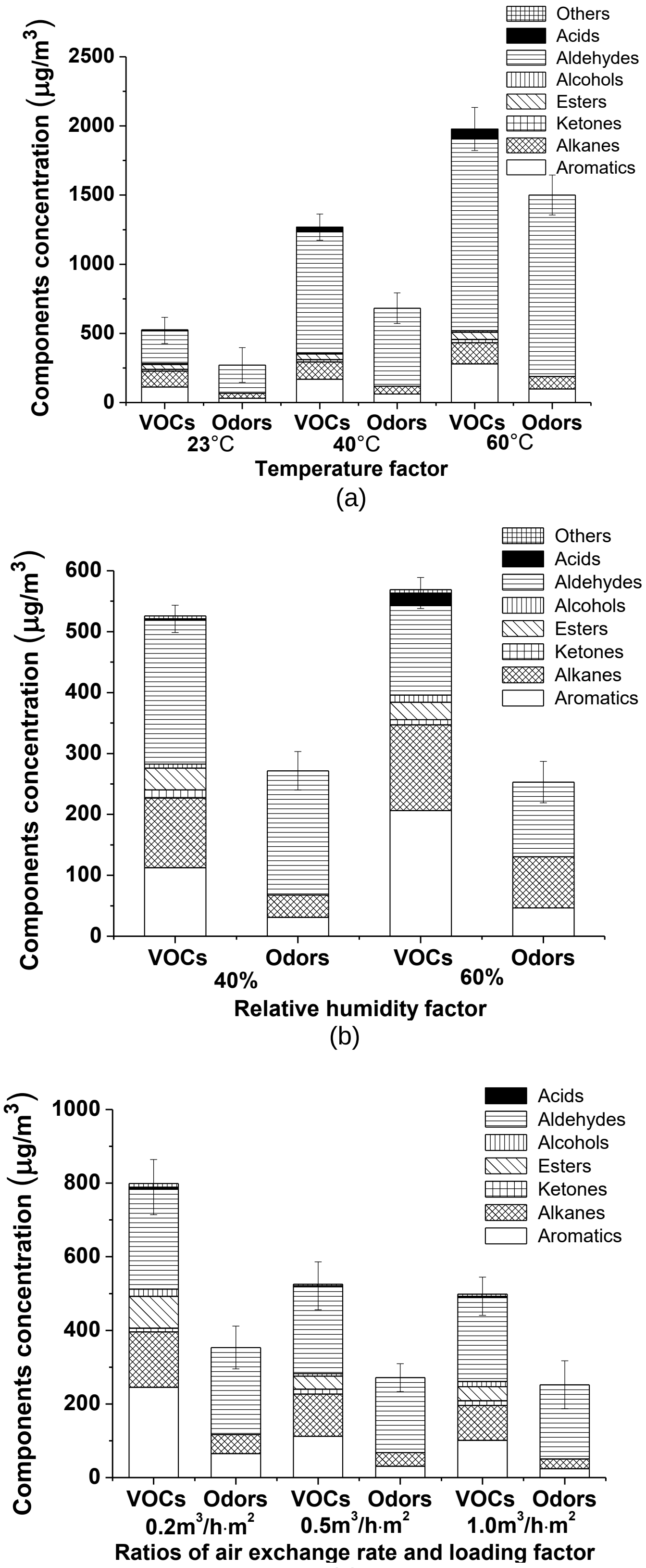


(a)

(b)

(c)

Fig. 2. TVOCs concentration and total odor intensity of the alkyd resin enamel-coated particleboards at (a) different temperatures, (b) different relative humidity values, and (c) different air exchange rate to loading factor ratios



(a)

(b)

(c)

Fig. 3. TVOCs and odor components concentrations from an alkyd resin enamel-coated particleboard under different conditions at an equilibrium state: (a) different temperatures; (b) different relative humidity values; and (c) different air exchange rate to loading factor ratios

CONCLUSIONS

1. The main odor substances released from the alkyd resin enamel-coated particleboard were aromatic compounds, aldehydes, and alkanes, which are primarily low in toxicity. At an equilibrium state, the amounts of aromatic compounds and alkanes increased with an increasing temperature and relative humidity and decreased as the air exchange rate to loading factor ratio increased. While the emission of odorant aldehydes increased when the temperature increased, it decreased when the relative humidity and air exchange rate to loading factor ratio increased.
2. The TVOCs concentration and total odor intensity increased as the temperature and relative humidity increased, but they decreased as the air exchange rate to loading factor ratio increased. The temperature had a greater impact on the VOCs released from the alkyd resin enamel-coated particleboard than the relative humidity or air exchange rate to loading factor ratio.
3. The percentage of the odor substances concentration increased with an increase in the temperature, while the relative humidity and air exchange rate to loading factor ratio had little effect.
4. There was no direct correlation between the odor intensity and odorant compounds concentration, but the odorant compounds concentration affected the odor intensity to a certain extent. The GC-MS-O analysis can be used as a method to detect the odor from wood-based panels and furniture materials.

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