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EFFECTS OF TEMPERATURE ON VOLATILE ORGANIC COMPOUNDS AND ODOR EMISSIONS OF POLYVINYL CHLORIDE LAMINATED MDF

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ABSTRACT

The objective of this study was to investigate odor active compounds of polyvinyl chloride (PVC) laminated medium density fiberboard (MDF) and explore the effect of temperature on total volatile organic compound (TVOC) and odor emissions. A micro thermal extractor was used based on the technology of gas chromatography–mass spectroscopy/olfactometry. The results showed that fruity, sweet, fragrant, and aromatic were the dominant odor impressions of PVC laminated MDF and were primarily concentrated in aromatics. Decoration treatment could effectively prevent the release of some odor compounds from MDF, yet these could add new odor substances. In the test period, the total odor intensity of MDF decreased more rapidly than that of PVC because of the characteristic of exposure. The TVOC from PVC increased when the temperature increased, and the effect was more significant early in the test period. Increasing temperature could accelerate the appearance of some odor active substances. The fastest release of odorant compounds occurred at 40°C. At higher temperature (60°C in this experiment), some substances could be enhanced, causing an increase in TVOC and odor. The temperature contrast between 40°C and 60°C contributed greatly to the release of alkanes, alcohols, esters, and ketones.

KEYWORDS: PVC laminated MDF, temperature, volatile organic compounds, odor active compounds, decoration, gas chromatography–mass spectrometry/olfactometry.

INTRODUCTION

The volatile organic compounds (VOC) from wood (Stachowiak-Wencek 2014) and wood based panels have been well investigated in recent years (Shen and Jiang 2018). However, limited information is available on the odor active substances contained in the wood based panels. The American Society of Heating, Refrigerating and Air-Conditioning Engineers (ANSI/ASHRAE 2003) defined qualified indoor air quality in standard 62-2001, which pointed out that a qualified environment should meet the conditions that the concentration of harmful substances should be below the limit and that the people in the surrounding environment must feel comfortable. According to the requirements of the standard, people should not be disturbed by odors indoors. The olfactory techniques, such as vision and olfaction need to be better employed (Sioma 2019).

In the field of wood, some research has been conducted on the odor characteristics of solid wood, such as American, French, Hungarian, and Russian oak (Díaz-Maroto et al. 2008); poplar (*Populus* L. spp.), pine (*Pinus* L. spp.), and basswood (*Tilia* L. spp.) (Wang et al. 2017a); incense cedar (*Calocedrus decurrens* (Torr.) Florin) (Schreiner et al. 2017); and Cathy poplar (*Populus cathayana* Rehd.) and rubberwood (*Hevea brasiliensis*) (Liu et al. 2018). Schreiner et al. (2018) investigated Scots pine (*Pinus sylvestris* L.) wood samples grown in Germany for their main odorant composition and found most identified odorants were fatty acid degradation products, plus some terpenoic substances and odorous substances resulting from the degradation of lignin. These studies mostly focus on the identification of odor substances and the exploration of the method. Few reports pay attention to the odor from decorated wood based panels and the influence of environmental factors.

Wang et al. (2018) studied VOC and odor emissions from alkyd resin enamel coated particleboards and found that temperature had an impact on the release of VOC and odor. Research about VOC release from three layer plywood (Wang et al. 2017b) also showed that temperature is the key factor for VOC emissions. Therefore, it is necessary to investigate the effects of temperature on VOC and odor emissions. As a commonly used wood based panel, polyvinyl chloride (PVC) laminated medium density fiberboard (MDF) was widely used in interior decoration and furniture. In this study, the characteristics of the odor compounds were identified, and the release trends were obtained. At the same time, the effect on VOC and odor emissions of PVC decoration and temperature was explored.

MATERIALS AND METHODS

Materials

The wood based composite panels used in this study were E1 grade MDF (tree species, *Eucalyptus robusta* Smith; hot pressing temperature, 220°C; pressing pressure, 3.2 MPa; moisture content, 6.25%; density, 0.00074 $kg \cdot m^{-3}$; adhesive, melamine modified urea-formaldehyde (UF) resin) from Suofeiya Home Collection Co. (Guangdong, China), with specifications (length by width by thickness) of 1200 × 1200 × 8 mm. The parameters of the overlay process were as follows: hot pressing temperature, 190°C; pressing pressure, 19.5 MPa; and adhesive, melamine modified UF resin. The experimental panels were as follows: MDF with no surface lamination and MDF overlaid with PVC. The samples were cut into round pieces (60 mm in diameter), with an exposed area of 5.65×10^{-3} m^2. The edges of the samples were wrapped with aluminum foil to prevent the release of compounds, and then they were stored in polytetrafluoroethylene bags and refrigerated until needed.

Sampling collection

For each sample, 2 L of VOC was collected from materials using a micro thermal extractor (Markes International, South Wales, UK). The VOC released from the specimen surfaces were absorbed by Tenax TA tubes (containing 200 mg of 2,6-dibenzofuran porous polymer) and were recorded at days 1, 3, 7, 14, 21, and 28. The main experimental conditions were set under temperatures of 23°C, 40°C, and 60°C with a constant relative humidity (40%) and an air exchange rate to loading factor ratio (0.5 $m^3 \cdot m^{-2} \cdot h^{-1}$) based on standard GB/T 18883-2002 (temperature ± 1°C, moisture content ± 5%, and air exchange rate to loading factor ratio ± 0.05 $m^3 \cdot m^{-2} \cdot h^{-1}$).

Sampling analysis

Gas chromatography–mass spectroscopy (GC-MS) was used to analyze the samples. This technology was applied using unity thermal analysis desorption of unit II (Markes International), DSQ_II series GC-MS (Thermo Fisher Scientific, Schwerte, Germany). The basic GC-MS parameters were as follows: For the thermal extractor, the carrier gas was nitrogen, the desorption temperature was 280°C, the cold trap adsorption temperature was –15°C, the thermal analysis time was 10 min, and the injection time was 1 min. For the GC-MS, chromatography was performed with a DB 5 quartz capillary column of 3000 mm (length) × 0.26 mm (inner diameter) × 0.25 µm (particle size). The carrier gas was helium of 99.996%, and the distribution flux rate was 1 ml/min. The chromatographic column was initially kept at 40°C for 2 min; then, the temperature was increased to 150°C (in 2°C min^{-1} increments) and was held at that temperature for 4 min. Finally, the temperature was increased to 250°C (in 10°C min^{-1} increments) and held at that temperature for 8 min. The injection port temperature was 250°C. The following GC-MS parameters were used: ionization mode, electron ionization; ion energy, 70 eV; transmission line temperature, 270°C; ion source temperature, 230°C; and mass scan range, 40 to 450 atomic mass units.

A Sniffer 9100 olfactory detector from Brechbühler AG (Echallens, Switzerland) was used in this experiment. Moist air was added to prevent dehydration of the nasal mucosa of the odor assessors. Direct intensity methods were chosen for analysis of the compounds. The experimental environment was set to reference standard EN 13725-2003 (NSAI 2003). The room was well ventilated, with no peculiar smells. During a GC run (described earlier), the human sensory evaluation assessors recorded the odorants by characteristic and intensity value, as well as retention time. A six-point scale ranging from 0 to 5 was used for the intensity judgment according to Japanese standards (Ministry of the Environment 1971): 0 = none, 1 = very weak, 2 = weak, 3 = moderate, 4 = strong, and 5 = very strong. Experimental results were recorded when the same odor characteristics were described by at least two assessors. The odorants were identified by comparing the MS spectra to the National Institute of Standards and Technology (NIST, Gaithersburg, MD) and Wiley (Hoboken, NJ) MS libraries (matching degrees up to 800 or above). The intensity value was based on the average value from the different assessors.

RESULTS AND DISCUSSION

Comparison of PVC laminated MDF with MDF

The odor active compounds from PVC laminated MDF and nonlaminated MDF were identified under the environmental conditions as follows: 23°C, 30% moisture content, and the 1.0 $m^3 \cdot h^{-1} \cdot m^{-2}$ ratio of air exchange rate to loading factor. Fig. 1 shows the comparison of odor active compounds in the early stage from PVC and MDF. In the early stage, in total, 10 kinds of common odor active compounds were identified, while 9 kinds of odor active compounds were found in MDF only and 8 kinds of odor active compounds were found in PVC only.

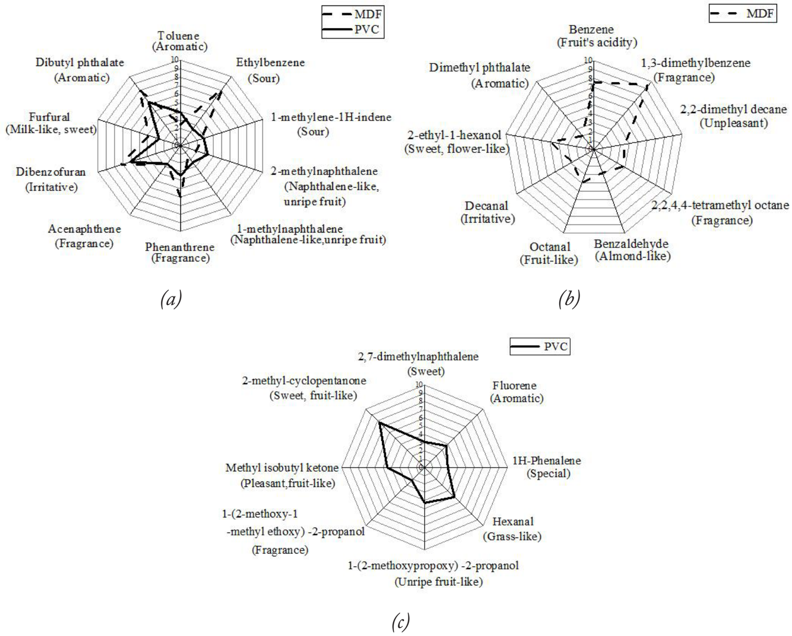


Fig. 1: Comparison of odor active compounds from PVC laminated MDF and MDF: (a) Common odor active compounds, (b) Only MDF contains, (c) Only PVC contains.

The results showed surface decoration could effectively prevent the release of odors from MDF. Some odor active compounds, such as benzene, 1,3-dimethylbenzene, 2,2-dimethyldecane, 2,2,4,4-tetramethyl octane, benzaldehyde, octanal, decanal, 2-ethyl-1-hexanol, and dimethyl phthalate (Fig. 1c), disappeared after decorating, which including aromatics, alkanes, aldehydes, and alcohols. The veneer could partially prevent the emission of ethylbenzene, phenanthrene, dibenzofuran, furfural, and dibutyl phthalate; their odor intensities decreased to varying degrees after facing. Incompleteness of prevention was caused by the void structure of MDF; as a result, some odorants found in the MDF were still detected in the PVC. However, the intensities of toluene, 1-methylene-1H-indene, 2-methylnaphthalene, and 1-methylnaphthalene increased with decorative lamination, which may be caused by the superposition of odor from MDF and PVC. The odor intensity of acenaphthene did not change. The common odor active compounds mainly concentrated in aromatics. Some new odor active compounds arose after facing because of the VOC emission of PVC and the adhesives used in the veneer process. Li et al. (2018) also found the melamine impregnated paper veneer can inhibit the emission of odor characteristic compounds of MDF, but because of veneer materials and adhesives, the mass concentration of aromatics increased. Tab. 1 shows that after decorating, the total odorant mass concentrations increased 31.98%, while the total volatile organic compounds (TVOC) decreased 27.86% in the equilibrium state. The odorant constituents of aromatics and alkanes were reduced, while the odorant concentrations of alkenes, aldehydes, alcohols, esters, and ketones rose.

The trend of VOC and odor emissions from PVC and MDF was explored. Fig. 2 shows that the TVOC of these two boards generally decreased over time until a stable phase was achieved.

Tab. 1: Component analysis of MDF and PVC laminated MDF in the equilibrium state.

Panel	Mass conc (μg·m^{-3})	Aromatics	Alkenes	Alkanes	Aldehydes	Alcohols	Esters	Ketones	TVOC
MDF	VOC	117.36	53.87	3.99	5.42	/	24.85	/	205.49
	Odor compounds	79.43	/	1.98	5.42	/	/	/	86.83
PVC	VOC	72.12	7.2	/	14.09	9.11	23.09	22.64	148.25
	Odor compounds	38.47	7.2	/	14.09	9.11	23.09	22.64	114.60

The TVOC mass concentrations sharply decreased from day 1 to day 3; after day 3, the decline rate slowed, and after day 14, it gradually reached equilibrium. That trend was caused by the larger concentration difference between the VOC and the external environment during the early release. According to the theory of mass transfer, the VOC inside the particleboard continued to release until the concentration difference disappeared (Liu et al. 2017). The composition of odorous substances is complex, and different odors can interact. Considering the complex interaction among various odorant compounds, the effect of fusion on the total odor intensity was chosen for this experiment. Over time, the total odor intensity of MDF decreased approximately linearly from day 1 to day 21, while the decline began to slow on day 28. However, the total odor intensity of PVC increased from day 1 to day 3, showing that although the TVOC concentration decreased, the odor active compounds increased. From day 1 to day 7, the total odor intensity of PVC was lower than that of MDF, but its value exceeded that of MDF after day 7. The veneer could prevent odor emissions in the early stage, but because of exposure, the odor of MDF decreased more rapidly than that of PVC. Odor release from PVC was irregular because of the sealing effect of the veneer.

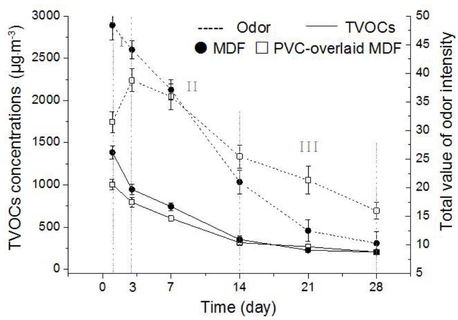


Fig. 2: The emission trend of TVOC and odor from MDF and PVC laminated MDF.

Characterization of odorous compounds from PVC laminated MDF

The key odor components of PVC laminated MDF were primarily from aromatics. Odorant components were also found in alkenes, aldehydes, alcohols, ketones, and esters. The change of odor intensity of PVC laminated MDF at different stages is shown in Fig. 3.

Dibenzofuran (No. 8), hexanal (No. 13), nonanal (No. 14), 2-methyl-cyclopentanone (No. 19), and dibutyl phthalate (No. 20) were the key odor compounds, and they had a greater impact on odor during the release process. As time went on, the total intensity of most odor active compounds (No. 1, 4–10, 12, 13, 15, and 17–20) decreased, and some odor characters disappeared in this process. Ethylbenzene (No. 2), nonanal (No. 14), and 2-ethyl-1-hexanol (No. 16) exhibited different properties. From the early stage to late stage, their total intensity first increased and then decreased. The intensity of 1-methylene-1H-indene (No. 3) fell and then rose. The intensity of 2-propyl cyclobutene (No. 11) gradually increased.

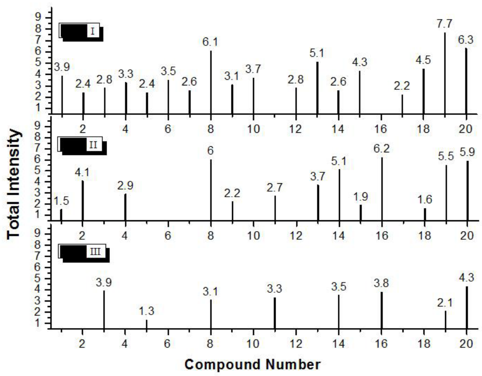


Fig. 3: Odor active compounds from PVC laminated MDF at different stages (I: earlier stage; II: middle stage; III: later stage).

Key odors were identified as potent smell contributors for the comparative analysis of mass spectral data, and identified odors were compared with relevant reference compounds (Xiao et al. 2016). The odor active compounds of PVC laminated MDF are listed in Tab. 2.

Tab. 2: Odor active component from PVC laminated MDF.

Constituents	No.	Compounds	Odor description	Earlier stage (I)		Middle stage (II)		Later stage (III)	
				Odor Intensity	Mass concentration (μg·m^{-3})	Odor Intensity	Mass concentration (μg·m^{-3})	Odor Intensity	Mass concentration (μg·m^{-3})
Aromatics	1	Toluene	Aromatic	3.9	261.48	1.5	17.01	–	–
	2	Ethylbenzene	Sour	2.4	20.58	4.1	26.88	–	–
	3	1–methylene–1H–indene	Sour	2.8	49.27	–	–	3.9	39.63
	4	2–methylnaphthalene	Mothball, unripe fruit	3.3	53.38	2.9	65.56	–	–
	5	1–methylnaphthalene	Mothball, unripe fruit	2.4	69.95	–	–	1.3	48.21
	6	Phenanthrene	Fragrant	3.5	64.75	–	–	–	–
	7	Acenaphthene	Aromatic	2.6	31.26	–	–	–	–
	8	Dibenzofuran	Irritative	6.1	40.15	6	121.38	3.1	43.63
	9	2,7–dimethyl naphthalene	Sweet	3.1	18.3	2.2	3.17	–	–
	10	Fluorene	Aromatic	3.7	98.85	–	–	–	–
Alkenes	11	2–propyl cyclobutene	Gunpowder	–	–	2.7	29.17	3.3	13.96
	12	1H–Phenalene	Special	2.8	84.78	–	–	–	–
Aldehydes	13	Hexanal	Grass	5.1	50.53	3.7	17.22	–	–
	14	Nonanal	Sweet	2.6	13.48	5.1	31.31	3.5	18.85
Alcohols	15	1–(2–methoxypropoxy) –2–propanol	Unripe fruit	4.3	34.43	1.9	10.59	–	–
	16	2–ethyl–1–hexanol	Sweet, flower	–	–	6.2	90.91	3.8	20.72
	17	1–(2–methoxy–1–methyl ethoxy) –2–propanol	Fragrant	2.2	7.61	–	–	–	–
Ketones	18	Methyl isobutyl ketone	Pleasant, fruit	4.5	30.36	1.6	9.54	–	–
	19	2–methyl–cyclopentanone	Sweet, fruit	7.7	404.48	5.5	86.83	2.1	22.64
Esters	20	Dibutyl phthalate	Aromatic	6.3	50.4	5.9	43.52	4.3	47.63

Odor compound characteristics were identified as follows: toluene was reported as aromatic, similar to the sweet, pungent, benzene-like odor reported by the U.S. National Institute for Occupational Safety and Health (NIOSH 2010). Ethylbenzene was found to have a sour character. Our testing found that phenanthrene has a fragrant odor, similar to the faint aromatic odor reported by the hazardous materials database of CAMEO Chemicals (2017). The hexanal in this experiment was reported to have a grasslike characteristic; Furia (1980) also found it had strong, green grass odor, whereas its odor was described as sharp and aldehyde by Lewis (2007). Burdock (2010) also found it presented a fruity odor on dilution. In this experiment, nonanal was reported to have a sweet character, similar to the orange and rose odor described by Lewis (2007). 2-Ethyl-1-hexanol was reported to have a sweet, flowerlike smell; other researchers found it presented a mild, oily, sweet, slightly rose odor (Burdock 1993). Dibutyl phthalate showed aromatic characteristics; it was the same as the slight, aromatic odor reported by the NIOSH (2010). The dibenzofuran detected in this experiment had an irritative characteristic.

Research showed that various factors could influence differences in odor characteristics. An odor's characteristic is related not only to its concentration but also to the medium, in which it exists, which indicates that in different concentrations and media, the same compound may show different odor characteristics. Some compounds' odors were initially detected in this research as follows: 1-methylene-1H-indene (sour), acenaphthene (aromatic), 1-methylnaphthalene (mothball-like, unripe fruit), 2-methylnaphthalene (mothball-like, unripe fruit), 2,7-dimethyl naphthalene (sweet), fluorene (aromatic), 2-propyl cyclobutene (gunpowder-like), 1-(2-methoxy-1-methyl ethoxy)-2-propanol (fragrant), 1-(2-methoxypropoxy)-2-propanol (unripe fruit), methyl isobutyl ketone (pleasant, fruitlike), and 2-methyl-cyclopentanone (sweet, fruitlike).

Effect of temperature factors on the odorous compounds from PVC laminated MDF

Fig. 4 shows the changes in TVOC concentration and total odor intensity under different temperatures. VOC and odor generally decreased with time until a stable phase was reached. The temperature had a significant effect on VOC release from PVC laminated MDF. The TVOC from PVC laminated MDF increased when the temperature increased. The effect was more significant in the active period and was reduced in the stable release period. Temperature influences VOC emissions through its effect on the diffusion coefficient. With increased temperature, the mass transfer resistance decreases and promotes the release of the VOC within the material; this may result in an increase in the mass transfer flux and the release coefficient, which could lead to an increase in the concentration of VOC (Wang et al. 2017b).

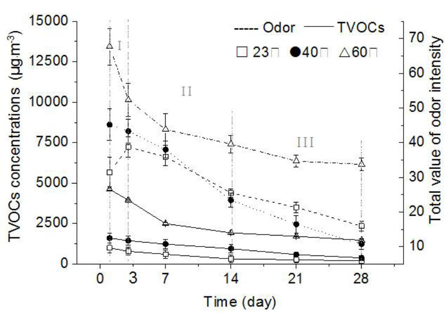


Fig. 4: TVOC concentration and total odor intensity of the PVC laminated MDF at different temperatures.

On the first day, the TVOC increased 3,008.75 μg·m^{-3} from 40°C to 60°C; however, the TVOC increased only 607.58 μg·m^{-3} when the temperature rose from 23°C to 40°C. On days 1, 3, 7, 14, 21, and 28, the TVOC increments from 40°C to 60°C were 4.95, 3.85, 1.99, 1.53, 3.53, and 6.10 times the TVOC increments from 23°C to 40°C, showing the temperature had greater influence on the VOC emissions under higher temperatures. Wolkoff (1998) came to a similar conclusion. He studied the release rule of VOC and found the total mass concentration and release rate of a PVC board under 60°C was far higher than occurred under a low temperature. Zhu and Geng (2005) also found temperature has a great effect at 60°C. In the current study, from day 1 to day 7, the total value of the odor intensity of PVC increased when the temperature rose. The total value of the odor showed the fastest rate of decline at 40°C. As a result, the total value of the odor intensity at 40°C was lower than that at 23°C from day 14 to day 28. The value of the intensity of PVC under 60°C was consistently higher than it was at 23°C and 40°C. At a high temperature, some substances that could not be detected under a low temperature from PVC may be enhanced, causing the odor to increase. Results showed the odor could be quickly emitted at a suitable temperature (40°C in this experiment). A higher temperature (60°C in this experiment) could promote the emission of TVOC and odor; however, the high temperature would slow the descent rate.

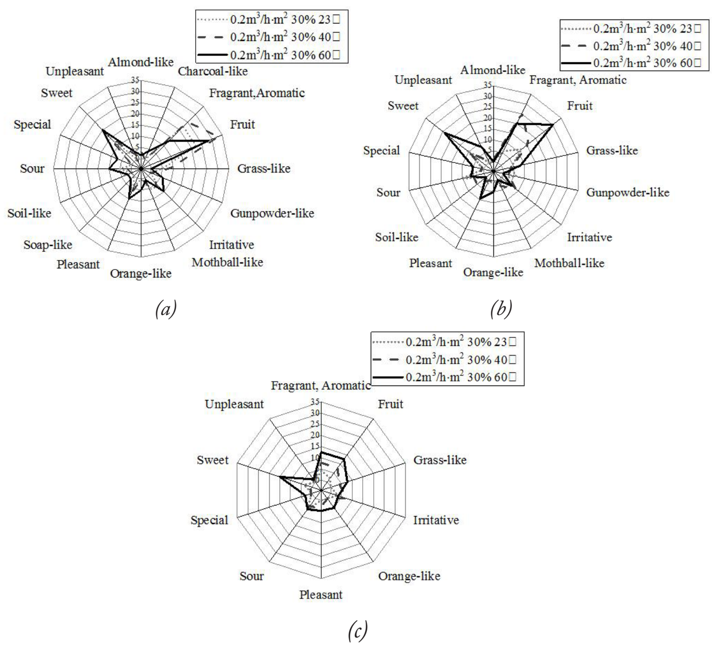
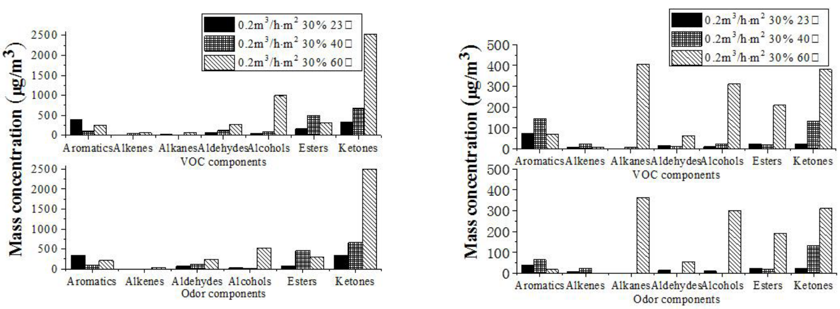


Fig. 5: Odor profile of PVC laminated MDF at different temperatures: (a) I earlier stage, (b) II middle stage, (c) III later stage.

Fig. 5 shows the odor profile of PVC laminated MDF at different temperatures during the release process. Fruity, sweet, fragrant, and aromatic were the dominant odor impressions during the entire release process. Considering subjectivity differences, the intensity of the dominant sweet character changed little from 23°C to 40°C. However, when the temperature rose to 60°C, its intensity rose rapidly in the middle and late stages. The fruit odor generally increased with the rise in temperature. Some substances were detected only in a certain period, when their release was accelerated by increasing temperature, such as the soil-like odor that appeared in the middle stage at 40°C and was detected in the early stage at 60°C. When the temperature increased to 60°C, orange-like (propyl 1-methylacetate), charcoal-like (E-2-octenal), and special (2-methyl-2-butyl acrylate and 2,2-dimethyldecane) odor characteristics appeared even though they were not detected at 23°C and 40°C, showing a high temperature could promote the release of some substances.

Fig. 6 shows the TVOC and odor component concentrations in the PVC under different temperatures in the initial and equilibrium states. In the initial state, the main odor components were found to be ketones, esters, and alcohols, and the mass concentration of odorant ketones and alcohols increased with the rise in temperature. The mass concentration of esters showed an upward trend when the temperature increased from 23°C to 40°C, but it declined when the temperature continued to rise to 60°C. In the equilibrium state, the mass concentration of odorant ketones always showed an increase from 23°C to 60°C. When the temperature increased from 40°C to 60°C, the mass concentrations of odorant alkanes, aldehydes, alcohols, and esters increased by 362.62, 53.78, 299.85, and 172.51 μg·m^{-3}, respectively. The concentrations of aromatics and alkanes were reduced by 46.54 and 21.85 μg·m^{-3}. The temperature contrast between 40°C and 60°C greatly contributed to the release of alkanes, alcohols, esters, and ketones.



(a) *(b)*

Fig. 6: VOC and odor components concentrations from PVC laminated MDF under different conditions: (a) Initial stage; (b) Equilibrium state.

CONCLUSIONS

The aim of this paper was to explore the effect of temperature on the VOC and odor emissions of PVC laminated MDF; at the same time, we sought to investigate the influence of decoration. For this purpose, the characteristics of the odor-active compounds were identified, and release trends were obtained.

Fruity, sweet, fragrant, and aromatic were the dominant odor impressions during the release process. Dibenzofuran, hexanal, nonanal, 2-methyl-cyclopentanone, and dibutyl phthalate were the key odor compounds. A veneer could prevent odor emissions in the early stage, but because of exposure, the odor of MDF decreased more rapidly than that of PVC. Some odor active compounds, including aromatics, alkanes, aldehydes, and alcohols, disappeared after decorating, while some new odor active compounds appeared and others were partially prevented from emitting from PVC. At a higher temperature, some substances could be enhanced, causing an increase in TVOC and odor. The TVOC from PVC increased when the temperature increased. The effect was more significant in the active period, but the effect was reduced in the stable release period. The total value of the odor showed the fastest rate of decline at 40°C.

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