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Research Article

GC-FID and Olfactometry-Assisted Assessment of Odors from Polymeric Foams under Normal and Repeated-Use Conditions

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Polymeric foams are the primary components of upholstered furniture, and their emissions play a decisive role in the acceptability of the final furniture product. This study is focused on passive emissions and odors from commercial foams under normal and repeated-use conditions. Six different types of foams, viz., highly elastic foam K5040, standard PU foam N5063, bonded polyurethane foam R100, viscoelastic foam V5020, self-extinguishing foam KF5560, and foam rubber, were used. The samples were collected at the intervals of 72 hours and 672 hours (28 days) to identify the odors due to chemical reactions in the material or slowly released due to its porous structure. Additionally, repeated-use studies were done to understand the effect of prolonged usage/natural ageing on emitted odors from the foams. The samples were tested as per ČSN EN 13 725 (2005) and ISO 16000-6 (2011) criteria using GC-FID (gas chromatography-flame ionization detector) and olfactometry. The most unpleasant substance was found to be nonanal, with an average score of -4 (unpleasant). A total of 23 compounds were identified (5 unidentified) using olfactometry; however, only 11 of them were confirmed by GC-FID-based testing. Any new compound or increase in odor intensity was not observed in long-term measurements and simulated repeated-use conditions.

1. Introduction

Odors are gaseous compounds of organic or inorganic origin [1]. The odor is an organoleptic (sensory) property that is perceived by the olfactory organ after inhalation of a specific volume of a substance [2]. Many compounds are recognized by a trigeminal nerve stimulus that causes nose, eye, and throat irritation [3]. In indoor air, many sources can be responsible for different odors (pleasant or unpleasant) like wooden furniture [4–6], fabrics, composites [7], and finishing materials [8]. A wide range of technologies have also been developed to address this issue [9]. The odors and scents can influence our mood and performance, promote creativity, or

improve the quality of sleep [10]. The hedonic phenomenon, which describes the perception of the odor by the individual, also depends on his experiences, memories, attitude to the problem, and mental state (fatigue, irritation, hunger, and nervousness). It is usually described using a scale, wherein the +5 and -5 ratings represent an extremely pleasant and extremely unpleasant feeling, respectively. The fragrances of strawberries and apples are classified as about +3, and the odor of urine, fertilizer, and dead animals are described with a value between -3 and -4 [11]. The effects of odorous substances do not directly threaten the health of human beings, but they negatively affect the psychological and physical well-being of human beings. Odors are closely related to

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VOCs (volatile organic compounds), which affect the health of an individual [12]. Brain reaction studies, EEG (electroencephalogram), and ERP (event-related potentials) suggest that VOCs and VVOCs (very volatile organic compounds) present in the ambient air at subliminal concentrations may affect the nervous system without our knowledge [13]. Odor sensitivity was also suggested to be associated with migraines, poor sleep, and intolerance of certain foods (dairy products) [14]. In a previous study using the GC-MS-O method on four viscoelastic foams (PU, polyurethane), assessing which type of odor (fruity, burnt, ammonia, and others) is due to which chemical compound was tried, and no long-term and repeated-use scenarios were tested [15, 16].

Upholstered furniture is typical of modern houses and constitutes an essential part of the indoor environment. Therefore, odors from upholstered furniture due to primary and secondary emissions have been studied widely [15]. The issue of odors can also be addressed at the manufacturing stage, using novel blowing agents and additives [17–21] or using the masking agent [12, 22]. Additionally, at later stages, air purification and other technologies can be utilized.

Furthermore, in the context of secondary emissions (due to porous nature and chemical reactions), long-term testing and repeated use are crucial. In this study, six commonly used foams in upholstered furniture (commercially available) were characterized for their characteristic odors and repeated-use-driven mechanical stress to understand their impact on odor quality. Measurements were done at the intervals of 72 and 672 hours (28 days). This study adds the important data (otherwise missing) on passive emission caused by long-term use-induced mechanical stress. Long-term measurements (28 days) were done to differentiate the primary emissions (physically released compounds present in new products) and secondary emissions (compounds produced by a chemical reaction in a product and are released gradually due to the mechanical stress).

2. Materials

The parameters of samples tested for assessment and identification of odor content can be found in Table 1. The size of the measured sample was $0.65 \text{ m} \times 0.65 \text{ m} \times 0.05 \text{ m}$, $S = 0.98 \text{ m}^3$.

Calibration was performed by injecting standard solutions and an internal standard solution of known concentration in a sorption tube (spike) and desorption onto a gas chromatograph. The analysis was performed under the same conditions for all the samples. The calibration line is the line between the ratio of the areas of the analyte peaks and the peak areas of the internal standard with respect to the ratio of the amount of analyte and the amount of internal standard in ng per tube. Standard mixtures of certified calibration solutions were Indoor Air Standard 50 Components, $1000 \, \mu \text{g/ml}$ (TraceCert, Sigma Aldrich); BTEX, 2000/g/ml or $1000 \, \mu \text{g/ml}$ (Sigma Aldrich); an independent standard for measuring control charts; and cyclic hydrocarbons, $2000 \, \mu \text{g/ml}$ (TraceCert, Sigma Aldrich).

The individual pure chemicals or certified solutions (supplied by Sigma unless specified otherwise) used were

pentanal, hexanal, 1-methoxy-2-propanol, butoxyethanol, myrcene, α -phellandrene, 3- δ -carene, bornyl acetate, n-hexane, n-hexadecane, and D10-o-xylene (internal standard for GC) as represented in Table 2. The gases used were helium (purity 5.5), nitrogen (purity 5.0), and compressed air (technical).

3. Methodology

3.1. Sampling of Released Emissions. Prior to the sampling of odors, each foam sample $(0.65 \text{ m} \times 0.65 \text{ m} \times 0.05 \text{ m})$ was airconditioned for 72 hours (23°C, RH 50%, and an air velocity of 0.1 to 0.3 ms⁻¹) by placing it in a small-space emission chamber VOC TEST 1000 with a volume of 1 m³. Followed by conditioning, samples were subjected to wait in the sampling chamber for different periods (72 h and 28 days), with no waiting period for repeated-use samples. The sampling was carried out via a splitter by pumping air through two pumps with an airflow of 12l·h⁻¹ through a sorption tube with Tenax TA (Scientific Instruments Inc., USA, supplied by Labicom SRO, Czech Republic) sorbent for 180 min, where the organic components were adsorbed on the sorbent. The specifications of Tenax TA tubes (as provided by suppliers) were size, 6×70 (outer dimeter \times length in mm); number of sections, 2; glass open ends; and with foam and glass wool separators. The Tenax tube is recommended by ISO standard to capture SVOC, VOC, and VVOC compounds as defined by the World Health Organization (WHO) [23, 24]. All the sampling parameters were followed as per ISO 16000-6:2011 [25]. A flow diagram of the complete process is represented in Figure 1.

3.2. GC-FID System and Assessment of Odors. The contents of the sampling tube were passed through a thermal desorption tube and gas chromatograph (GC). The sample, after passing through the chromatographic column, was split (1:1) into two parts. One part was analyzed for qualitative analysis (flame ionization detector (FID), Table 3), and the other part leads the air sample to the sniffer (electronic nose) heated through the humidifier. Sniffer provides a sample to the panelists for the assessment of hedonism and intensity. The assessment of hedonism was +/-, while 0-5 is the scale of the intensity. These two factors represent a scale with a range of -5 (extremely unpleasant) to +5 (very pleasant). The odor was evaluated by four panelists (two men and two women). The panel met the requirements of ČSN EN 13 725 (2005) and ISO 16000-6 (2011) [25]. The sample was administered to the panelists via the electronic nose after separation on the chromatographic column. All emission estimates to determine the hedonic effect were performed in parallel to the analysis of VOCs using an FID detector. The recorded odor was defined by the retention time, the hedonic tone, and the odor intensity according to the scale (Figure 2).

3.3. Repeated Use (Mechanical Stress) and Release of VOCs from Samples. Simulation of repeated use (mechanical stress) was performed using a device for testing the functional characteristics of the upholstery (Zdeněk Životský, Brno, Czech Republic) based on EN 1957 (2012). It was used to observe

Table 1: Parameters of various samples used in the study (also published in part 1 of the study [15].

Material	Density (kg·m ⁻³)	Cell diameter	Manufacturer and supplier
Highly elastic foam K5040	46.5-51.5	3 mm	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic
Standard PU foam N5063	46.5-51.5	0.8-1.5 mm	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic
Bonded polyurethane foam R 100	90-120	_	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic
Viscoelastic foam V5020	45-55	_	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic
Self-extinguishing foam KF5560	51.5-59.5	_	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic
Foam rubber	_	_	Eurofoam GmbH (Austria) and BPP spol. s.r.o., Czech Republic

Table 2: List of different VOCs with their characteristic features.

S. no	Compound	RT	MW	Tg	Q1	Q2	Q3	
0	D10-o-Xylene (I.S.)	10.186	116	98	116	114		
1	Ethyl acetate	2.109	88	43	70	88		
2	Benzene	3.588	78	78	77	79		
3	1-Methoxy-2-propanol	3.891	90	45	47	75		
4	Pentanal	4.558	86	44	58	29		
5	Trichloroethylene	4.725	130	130	132	95	95 97	
6	Toluene	6.821	92	91	92			
7	Hexanal	7.664	100	56	44	72	82	
8	Tetrachlorethylene	8.096	164	164	166	129	131	
9	n-Butyl acetate	8.093	116	43	56	73		
10	Ethylbenzene	9.514	106	91	106	105		
11	m,p-Xylene	9.724	106	91	106	105	77	
12	Styrene	10.282	104	104	103	77		
13	o-Xylene	10.359	106	91	106	105		
14	Butoxyethanol	10.521	118	57	87	100	45	
15	α -Pinene	11.396	136	93	121	136		
16	Camphene	11.796	136	93	121	136		
17	3-Ethyltoluene	12.032	120	105	120	91		
18	4-Ethyltoluene	12.077	120	105	120	91		
19	1,3,5-Trimethyl-benzene	12.195	120	105	120	91		
20	eta-Pinene	12.476	136	93	121	136		
21	2-Ethyltoluene	12.482	120	105	120	91		
22	Myrcene	12.592	136	93	121	136		
23	1,2,4-Trimethyl-benzene	12.812	120	105	120	91		
24	lpha-Phellandrene	13.048	136	93	121	136		
25	3-δ-Carene	13.199	136	93	121	136		
26	1,2,3-Trimethyl-benzene	13.492	120	105	120	91		
27	Limonene	13.600	136	68	93	136		
28	γ-Terpinene	14.224	136	93	121	136		
29	Bornyl acetate	18.805 196 95			136	196		

the gradual changes in the properties of the tested foams with repeated use under the conditions of minimum natural aging of the material. This device consists of a cylinder and a mechanism that horizontally pulls the roller on the surface in a to and fro motion (Figure 3). The cylinder on the sample was subjected to a force of 1.400 (\pm 7) N (measured in a static state) for 200 cycles. The material sample was reintroduced into the small-space sampling chamber immediately after

being mechanical stress air-conditioned for 24 hours, and then the emission samples were collected.

4. Results and Discussions

The representative chromatogram including internal standard peak and other VOCs can be found in Figure 4. The olfactometric assessment of the samples collected from

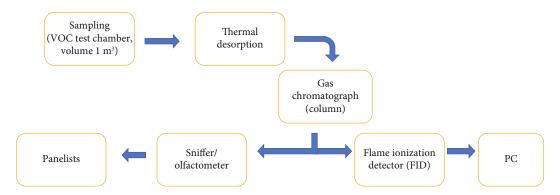


Figure 1: Flow diagram of different steps used in study.

Table 3: Thermal desorption and GC-FID parameters used in the study (same GC used in part 1 of the study [15]).

Thermal desorption: short path thermal	desorption controller model TD-4, serial J210							
Temperature and time conditions of th	*							
Operation	Time (s)	Temper	ature (°C)					
Gas purge time	300	——————————————————————————————————————						
Injection time	30	_						
Desorption time	180	200 (initial)-250 (final)						
GC start delay	30 200 (IIIIIai) -230 (IIIIai)							
Gas chromatograph: Agilent Technologi	es with FID HPST 4890 GC system							
Thermostat								
Initial temp (°C)		40						
Initial time (min)		2						
	Temperature rise rate (°C⋅min ⁻¹)	Final temp (°C)	Final time (min)					
Ramp	8	240	2					
Run time (min)	29	29	29					
Front inlet								
Mode		Split						
Temp (°C)		250						
Pressure (kPa)	62.9							
Split ratio	40:1							
Split flow (mL·min ⁻¹)		47.9						
Total flow (mL·min ⁻¹)	52.6							
Gas type	Nitrogen							
Column		8						
Capillary column type:	Agilent HP-5MS	(5% phenyl methyl siloxane	·)					
Max. temperature (°C)	325							
Nominal length (m)	30							
Nominal diameter (μ m)	320							
Nominal film thickness (µm)	0.5							
Initial flow (mL·min ⁻¹)	1.2							
Average velocity (cm·sec ⁻¹)	40							
Nominal initial pressure (kPa)	63							
Outlet	MSD							
Outlet pressure		Vacuum						
Detectors								
Detector A		FID						
Detector B	Olfacto	metry; panel assisted						

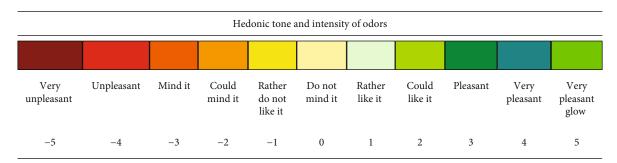


FIGURE 2: Degrees of evaluation of odors.



Figure 3: Images of the equipment used to simulate mechanical stress caused by repeated use.

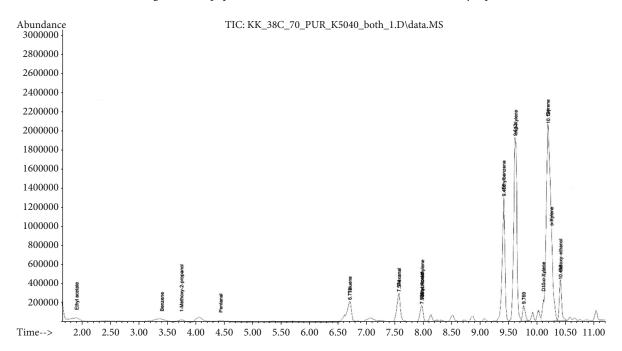


FIGURE 4: Representative chromatogram with internal standard and VOC peaks.

individual foams can be found in Table 4. It follows the scale mentioned in Figure 2; all of these ratings of individual substances were in negative values, and therefore, these compounds had an unpleasant hedonic tone. The intensity of individual substances ranged from -1 to -5 units of the classi-

fication scale. Substances that were not detected by the panelist (no sense of smell) were too low in intensity to score or odorless. The average values of individual ratings are listed (Table 4), which was recorded by at least three panelists in 72 hours, 672 hours, and simulated repeated-use samples. These

	K5040 N5063			KF5560 V5020							R100			Foam rubber					
Substance	R.T.	72 h	672 h	M.s.	72 h	672 h	M.s.	72 h	672 h		72 h nsity	672 h	M.s.	72 h	672 h	M.s.	72 h	672 h	M.s.
ETA	0.8	-1	_	_	_	_	_	_	_	_	_	_	_	-1	_	_	_	_	_
Benzene	2.0	_	_	_	_	-1	-2	_	_	_	-1	_	-1	_	_	_	-1	_	_
MP	2.8	_	-1	_	-1	_	_	_	_	_	_	_	_	_	-1	_	_	-1	-1
NNDA	4.5	-2	_	_	_	_	_	-1	_	_	-2	_	_	-2	_	-1	-2	_	_
UI	5.0	-1	_	_	_	_	_	_	-1	_	_	_	_	-1	-1.5	_	_	_	_
Toluene	5.7	-2	-3.5	-2.5	_	-3	-3	_	-3	-3	-2	-2	-3	-2	-3	-1.5	-2.5	-1	-2.5
Hexanal	6.5	_	-1	_	-1	-0.5	-1	1	_	-1	_	_	_	_	_	_	_	_	-1
BTA	7.4	_	-1	_	_	-2	-1	_	-1	_	_	_	_	_	_	_	_	_	_
UI	8.1	_	_	_	_	-1	-2	_	_	_	-1	_	_	_	_	_	_	-2	_
MPX	8.9	-3	-2	-2.5	-1.5	-3.5	-3	_	_	_	-1	_	-2	-3	_	-1	-2	-2	_
Styrene	9.5	-3	_	_	-3.5	-3.5	-3	-2	_	_	-2	_	_	-3	_	_	-3.5	-3	-4
BX	10.2	_	-3	_	_	-4	-3.5	-2	-3	-3.5	-3	-4	_	_	_	-3	-4	-4	_
α-Pinene	10.6	_	_	_	-2	_	-3	_	_	_	_	_	_	_	_	_	_	_	-1
BD	11.7	-1.5	-4	-4	-2.5	-3.5	-3.5	-2	-1.5	-2	-3	-3	-3	-1.5	-3	_	-2.5	-4	-2.5
Limonene	13.4	_	-2	_	-2.5	_	-2	-1	_	_	-3	-2	-3	_	_	-1	-4	_	-3
NNDBA	13.6	_	_	_	_	_		-3	-4	-2	_	_	_	_	_	_	_	-2	_
UI	14.2	_	-3.5	-2	-2	-3	-3	_	-2	-1	-2	_	_	_	_	_	_	_	_
EA	14.6	-3	-3	_	-3	-4	-3.5	_	-4	-2	-2.5	-1.5	-4	-3	_	_	-4		-4.5
Nonanal	15.2	-4	-4	-4	-4.5	-3.5	-3	-3.5	-3.5	-4.5	-3.5	-5	-4.5	-4	-4.5	-4.5	-3.5	-4	-4
UI	15.8	_	-2	_	_	_	-1	_	-2	_	_	_	_	_	_	_	_	_	-1.5
UI	16.0	_	_	-2	-2.5	-2	-3.5	_	-2	-1	_	-2	-3	_	-2	_	-2	-2	-1
Decanal	17.4	-1	-3.5	-3	-3	-3.5	-2.5	-2	-3	-2.5	_	-2	-3	-1	-2	-1	-3	-3.5	-1
RΔ	19.0	-3			-2				_				_	-3					-3

Table 4: Results of the olfactory assessment of polymeric foam materials.

VOCs were identified based on the retention times of the FID detector chromatogram recording and the evaluation of the odor ratings by the panelist. The least unpleasant compound was identified only by a noninferior classification with -4 (unpleasant). Class rated most compounds -3 and -2 ratings. Rankings "-3" (bad) were styrene, butoxyethanol and 2ethylhexanoic acids, benzaldehyde, N,N-dimethylbenzylmethaneamine, and boron acetate. The classification "-2" were toluene, m,p-xylene, α -pinene, limonene, and decanal. Rating 1 ("I do not like") was ethyl acetate, benzene, 1-methoxy-2-propanol, N,N-dimethyl-2-aminoethanol, hexanal, and butyl acetate. The negative results (unpleasant) assessed by panelists could not be directly compared with a similar study by Hillier et al. (2009), in which they used the terminologies like amine, acrid, fruity, fishy, burnt, and caramel with no inclusion of intensity criteria [16].

Representative substances for individual polymeric foam materials are those recorded by the panelist in all samples of the material, i.e., after 72 h, 672 h, and simulated repetitive usage; samples are presented in Table 5. These were predominantly alcohols or aldehydes (polar substances with higher electronegativity). Not all substances identified by the panelist could be identified and are therefore identified as unidentified substances. These are substances that are not characterized by any peak in the chromatogram and thus have such small concentrations that they are not registered by the chromatograph detector (FID) but still have an odor

detectable by the panelist. This was due to the much higher ability of the human nose to detect the odors as compared to the chromatograph detector [26].

In Tables 4 and 5, substances were observed at 72 hours, 672 hours, and simulated repeated use of materials using their time-based and characteristic compound-based classifications, respectively. Nonanal was recorded by the panelists for all materials. Benzaldehyde was recorded for all the materials in all three scenarios (except R100 under mechanical stress). Also, decanal was recorded for all materials except the V5020 for 72 hr measurements. Toluene was recorded in all scenarios for K5040, foam rubber, R100, and V5020. It was also recorded for the KF5560 and N5063 except for the post 72-hour measurements. It indicates its presence with time and natural aging. Hexanal was recorded in all conditions only for N5063. It was not detected for R100 and V5020. In the rest of the samples, no consistent trend was observed. The m,p-xylene was recorded for K5040 and N5063 in all three scenarios; however, it too did not show any consistent behavior in other samples. Butoxyethanol was recorded in KF5560 (increase in hedonic tone with time and aging) with an inconsistent trend in other samples and scenarios.

MP: 1-methoxy-2-propanol; NNDA: N,N-dimethyl-2-aminoethanol; NNDBA: N,N-dimethylbenzylmethaneamine: EA: 2-ethylhexanoic acid; UI: unidentified; BD: benzaldehyde; BX: butoxyethanol; BA: bornyl acetate; BTA: butyl

TABLE 5: Characteristic substances recorded by olfactometry for specific materials.

Substance	Measured polymeric foam materials						
Toluene	K5040, V5020, R100, rubber foam						
Hexanal	N5063						
m,p-Xylene	K5040, N5063						
Styrene	N5063, rubber foam						
Butoxyethanol	KF5560						
Benzaldehyde	K5040, N5063, KF5560, V5020, rubber foam						
Limonene	V5020						
N,N-Dimethylbenzylmethaneamine	KF5560						
2-Ethylhexanoic acid	N5063, V5020						
Nonanal	K5040, N5063, KF5560, V5020, R100, rubber foam						
Decanal	K5040, N5063, KF5560, R100, rubber foam						

acetate; RT: retention time; ETA: ethyl acetate; MPX: m,p-xylene.

Limonene was recorded for V5020 in all scenarios, and it was not recorded for N5063 and foam rubber after 672 h. For NNDA, at least one sample showed positive results in at least one scenario except N5063. The EA was recorded for N5063 and V5020 in all three scenarios, and the rest of the samples and scenarios were inconsistent.

The odorous compounds are either a component of foam formulation or formed as a byproduct during the polymerization reaction. The presence of toluene can be attributed to diethyltoluenediamine and dimethylthiotoluenediamine, which are commonly used as chain extenders and crosslinkers. Additionally, toluene diisocyanate is also commonly used as raw materials in foam manufacturing [27]. The hexanal, nonanal, decanal, and other aldehydes originating from the original natural oil-based polyol production process are among the common emissions from PU foams. The odor effect of these and other odor materials is lessened or eliminated during the stage at which the natural oil-based polyol is transformed into the isocyanate-modified polyol in a pretreatment step [28]. The xylene derivative styrene (monomer) is a common component of highly elastic PU foams [29]. Styrene is also a crucial component of rubber foams [30]. N,N-Dimethylbenzylmethaneamine is used as a catalyst and butoxyethanol (fire retardant by increasing the flashpoint) for self-extinguishing foam synthesis [31, 32]. Limonene is a tackifier resin and antibubbling component of viscoelastic foams [33].

The concentration or peak area did not affect the hedonistic evaluation of individual materials. Even if the minimum concentration or peak area was found, the assessment did not vary depending on the factor. Assessment by the panelist has a similar rating scale as when the substance had a high concentration or a large peak area. It should be noted that the most prominent peak in the FID is not necessarily the most critical odorant. It can be expected that compounds with a high hedonic effect exhibit a low FID response. Five compounds were unidentified in the olfactometric assessment. It may be due to too low concentrations or a small peak area in the sample that could not be detected by FID. How-

ever, they are substances with a strong hedonic effect and odor intensity. This observation is similar to Hillier et al. (2009); they studied the odors released from PU foams made with viscoelastic foams and the character of the odor was judged by three panelists. Several odors were reported in the olfactory assessment, but it was not possible to analyze with the GC-FID; these were N, N-dimethylethanolamine, 4-ethyl morpholine, m-xylene, p-xylene, benzaldehyde, limonene, undecane, and propylene [16]. The issue of odorous compounds should be addressed at the formulation stage of polymeric foams (using functional additives). Moreover, new approaches like fabrics that are active and modified to counter odors and additional deodorizing solutions can be used in the built environment. Additionally, sustainedrelease odor masking agents can also be added in the formulation to mask unpleasant odors that are emitted from foams or absorbed from the surroundings.

5. Conclusion

The olfactory assessment found that most of the released compounds had a negative hedonistic tone with an odor intensity of -1 to -5. Out of the compounds with negative hedonistic tone, most of the substances were rated by panelists as classification -3 (bad) and classification -2 ("I could mind"). The most unpleasant substance was found to be nonanal, with an average score of -4 (unpleasant). From an olfactory evaluation, the specific substances were identified as toluene, m,p-xylene, styrene, butoxyethanol, limonene, nonanal, and decanal, which were also recorded due to significant concentrations or peak area in the VOC analysis. The N,Ndimethylethanolamine, 4-ethyl morpholine, benzaldehyde, undecane, and propylene were only identified by olfactometry due to their characteristic odor and higher sensitivity of the human nose. Based on the amount of individual VOCs detected in the olfactory assessment for long term studies (28 days), it could not be concluded if their concentrations in emissions will increase or decrease over time. Additionally, the quantity of identified substances was not found to increase or decrease under simulated repeated-use conditions.

Data Availability

The data used to support the findings of this study are included in the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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