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D6.5 – Emission properties of BRIMEE ECO-innovative panels

Organisation name of lead contractor for this deliverable: BAM

Due date of deliverable: M42

Dissemination Level		
PU	Public	**x**
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

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Glossary

AgBB	Ausschuss für die gesundheitliche Bewertung von Bauprodukten
CPR	Construction Products Regulation
DNPH	2,4-dinitrophenylhydrazine
GC-MS	Gas Chromatography with Mass Spectrometer
HPLC-DAD	High-Performance Liquid Chromatography with Diaode Array Detector
IAQ	Indoor Air Quality
LCI	Lowest Concentration of Interest
NCC	Nano-Cristalline Cellulose
SVOC	Semi-Volatile Organic Compound
TD	Thermal Desorption
THF	Tetrahydrofuran
VOC	Volatile Organic Compound

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1 Introduction

Deliverable 6.5 reports on the results of the tests on emissions of VOCs and SVOCs from BRIMEE NCC panel with the formulation agreed on the M36 project meeting. Since phenol emissions were determined in the previous panel version, the flame retardant – identified as its source – was changed. The overall aim of this deliverable is the success control of this measure.

In Chapter 2 the experimental programme is described, involving the description of the sample preparation, the test equipment, sampling and analysis of the air samples. Based on these results a health-related evaluation according to the German AgBB evaluation scheme was carried out. Those evaluations are currently required in only few European member states but will be mandatory in the future in the whole European Union in view to countering the requirements of the Construction Products Regulation (CPR) in terms of hygiene, health and the environment.

The results of the measurements and their discussion take place in Chapter 3 including an assessment of the material's impact on the indoor air quality.

2 Experimental programme

2.1 Sample preparation

The test samples were produced by SILCART and shipped to BAM shortly after production in an air-tight package as is required by prEN 16516. As agreed at the 36M GA meeting, a panel sizing 600 mm × 400 mm should be tested. Due to shortage of material, two broken pieces were sent (Figure 2.1).

Figure 2.1 – Test samples after arrival from SILCART

The test material had no constant dimensions. Therefore, mean areas and thicknesses were determined (Table 2.1)

The product loading in the test chamber is area specific (cf. section 2.2), and the area facing indoors is relevant. Because of the porosity of the material and the non-constant dimensions, it was decided to resign sealing back and edges, assuming that emissions migrate from inside the material to the surface.

Table 2.1 lists the resulting test parameters.

Table 2.1 – Test parameters

Sample	**Mean thickness [mm]**	**Mean surface area A_{mean} [m²]**	**Air flow rate $\dot{V}_{ch}$ [L/h]**	**Area spec. air flow rate q_{ch} [m³/(m²h)]**
1	6.5	0.17		
2	6.5	0.15		
total	6.5	0.32	162	0.5

The test samples were loaded into an emission test chamber with a volume of 250 L as described in section 2.2.

2.2 Test procedure

The test chamber was operated dynamically. This means that an air change rate n_{ch} is applied, defined as the ratio of air volume $\dot{V}_{ch}$ fed into the test chamber to the free volume V_{ch} of the test chamber. According to the test standard prEN 16516 [1], n_{ch} can be set to values between 0.25 and 2.0 h^{-1}. Representativeness with regards to the intended use of the test samples is assured by applying a product loading factor L between 0.5 and 2.0 m^2/m^3 for wall materials. L is defined as the ratio of the exposed area of the test specimen and the volume of the test facility. The ratio of n and L makes the area specific air flow rate q_{ch}. The relations between the variables are given in equation (1).

$$q_{ch} = \frac{n}{L} = \frac{\dot{V}_{ch} \times V_{ch}}{V_{ch} \times A} = \frac{\dot{V}}{A} \quad (1)$$

with $\dot{V}_{ch}$ as the flow rate of the supply air in the emission test chamber in L/h and V_{ch} as the volume of the test chamber in L.

The area specific emission rate $SER_{A,i}$ of the material i is calculated following equation (2).

$$SER_{A,i} = c_{ch,i} \times q \quad (2)$$

with $c_{ch,i}$ as the measured concentration in test chamber air of substance i in µg/m³.

The test was designed such that the measured chamber air concentration $c_{ch,i}$ is equal to the one that can be expected, if all walls of the European reference room are covered with NCC panel ($c_{ref,i}$). The reference room is sized 3 m × 4 m × 2.5 m (W × L × H) making a room volume of 30 m³. The rate n_{ref} of ventilation with fresh air is specified at 0.5 h^{-1}. By convention, this is considered to represent normal indoor air conditions and results in an area specific air flow rate q_{ref} of 0.5 $m^3/(m^2h)$ with a loading factor L_{ref} of 1.0 m^2/m^3.

Emission tests for the evaluation of construction products are normally lasting 28 days at a temperature of 23 C ± 1 K and a relative humidity of 50 % ± 5%. Then either steady-state emissions have been reached or the decay of emissions has at least slowed down significantly. If such a situation is achieved much earlier, the test can be stopped.

Figure 2.2 – Test chamber loaded with BRIMEE material

In Figure 2.2, the test chamber loaded with the BRIMEE material is depicted. It is produced from electro polished stainless steel with a volume of 250 L and equipped with an agitator to ensure homogenisation of test chamber air. The front door of the chamber provides ports for air sampling.

2.3 Sampling and analysis

Air sampling and analysis were carried out according to ISO 16000-3 [2] and ISO 16000-6 [3] at days 3, 7, and 14 after sample installation into the test chamber. As it will be explained below the test was stopped before the 28th sampling day because the abort criterion was fulfilled.

Cartridges filled with the adsorbent DNPH (2,4-Dinitrophenylhydrazine) were used for the determination of carbonyl compounds (aldehydes and ketones), particularly formaldehyde, and glass tubes filled with the adsorbent Tenax TA® for the determination of VOCs and SVOCs. The sampling volume on DNPH cartridges was 30 L. Afterwards they were eluted with a mixture of acetonitrile and water, and this solution was analysed using high pressure liquid chromatography equipped with a diode array detector (HPLC-DAD) on a ULTRASEP ES ALD column (2.0 mm × 125 mm, SEPSERV) with acetonitrile, water and tetrahydrofuran (THF) as mobile phase. For Tenax, duplicate sampling was carried out with volumes of 1 and 3 L at the 3rd and 2 and 5 L at the 7th and 14th day. Tenax sampling followed by thermal desorption (TD) of the tubes and analysis using gas chromatography on a slightly polar column (DB-5ms, 60 m × 0.25 mm × 0.25 µm, Agilent) and with a mass selective detector (GC-MS).

All identifiable VOCs that can be found on the list of Lowest Concentration of Interest (LCI) (cf. section 2.4) were quantified by using their individual response factor. Compounds which are not listed or show a mass spectrum that cannot specifically be assigned to a certain compound were quantified by use of the response factor for toluene (toluene equivalent). The blank value of the adsorbent tube was determined by analysis of the unloaded tube prior to sampling and subtracted. SVOCs were quantified by integrating the chromatogram

between the elution range of n-hexadecane and n-docosane with toluene equivalents giving the sum parameter ΣSVOC.

2.4 Evaluation of test results

Although prEN 16516 provides a harmonised procedure for the determination of materials emissions, there is still a lack of a harmonised regulation for the evaluation of test results. In Germany, the so-called AgBB scheme (**A**usschuss für die **g**esundheitliche **B**ewertung von **B**auprodukten – *Committee for health-related evaluation of building products*) is used for the health-related evaluation of single building materials and currently mandatory for the approval of flooring materials and decorative wall materials to be used in buildings with public access. The application of this evaluation scheme is under discussion as a model for a harmonised evaluation scheme in the EU [4, 5].

The evaluation is based on the analysis of test chamber air sampled at least after the 3rd and 28th day after loading [6]. When specific abort criteria are reached the measurement can be stopped after the 7th day. The following parameters are checked:

- TVOC (total VOC): Sum of the concentration of all individual substances with concentrations equal to or greater than 5 µg/m³ within the retention range C_6-C_{16}. It may not exceed 1000 µg/m³ at the 28th sampling day.
- ΣSVOC: Sum of the concentration of all individual substances with concentrations equal to or greater than 5 µg/m³ within the retention range > C_{16}-C_{22}. It may not exceed 100 µg/m³ at the 28th sampling day.
- Carcinogenic substances belonging to EU categories 1 and 2 or EU categories 1A and 1B.
- Assessable compounds: All VOCs with an LCI value; those compounds are listed in the appendix of the scheme; R ≤ 1.
- Non-assessable compounds: Sum of VOC not identifiable and with unknown LCI. It may not exceed 100 µg/m³ at the 28th sampling day.

The so-called R-value is based on the results of the assessable compounds at the 28th sampling day or earlier, when the test can be prematurely terminated. It is a sum parameter calculated according to Equation (3) and may not be greater than 1.

$$R_i = \sum_i \left(\frac{c_i}{LCI_i}\right) \quad (3)$$

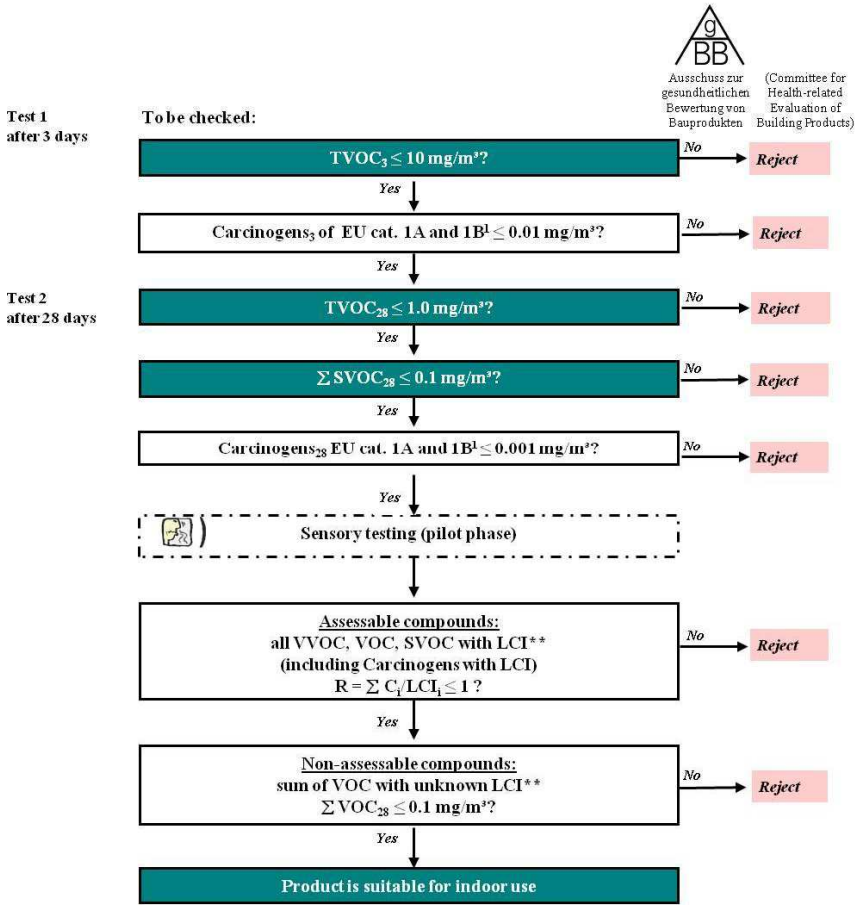

Figure 2.3 - Flow chart for the evaluation of VVOC, VOC and SVOC emissions from building products [6].

3 Results and discussion

The results of the emission test of the NCC panels are depicted in Figure 3.1 as mass concentrations. Due to the selected test parameters they correlate to concentrations that would be expected in the European reference room (cf. section 2.2) and need not to be additionally converted.

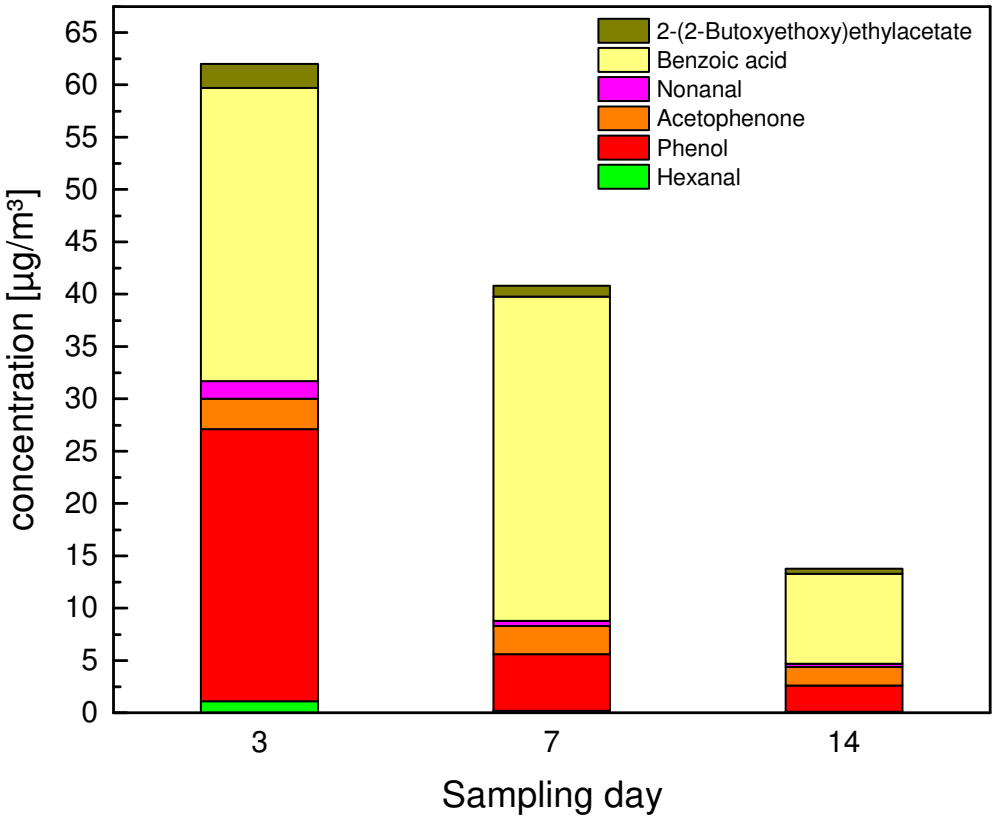

Figure 3.1 – Emission test results of NCC-foam sample

In general, the material is poor of emissions. Three VOCs (phenol [CAS 108-95-2], acetophenone [CAS 98-86-2] and benzoic acid [65-85-0]) were mainly emitted from the NCC material. The phenol emission is of particular interest, since in the previous material version it would have led to a rejection of the material against the AgBB evaluation. At that time, it was originated from the flame retardant, which was changed now. This time, the source was not identified but it can be said that the concentrations here are at a much lower level and decrease significantly during the 14 days of testing.

As it was described in section 2.4, the German AgBB scheme was used for a health-related evaluation of the materials emissions. Because the abort criteria were fulfilled at the 7th day after installation of the sample into the test chamber, the test was stopped after 14 days. It is obvious that all emissions further decreased from the 7th to the 14th sampling day. The

evaluation according to the rules as described in section 2.4 is as follows (Table 3.1).

Table 3.1 – Evaluation results and criteria according to the AgBB scheme. The measured concentrations are equal to the ones in the European reference room.

Parameter	**Day 3**					**Day 7**				
	measured conc.		✓	➔	✖	**measured conc.**		✓	➔	✖
	(µg/m³)	(mg/m³)	(mg/m³)	(mg/m³)	(mg/m³)	(µg/m³)	(mg/m³)	(mg/m³)	(mg/m³)	(mg/m³)
TVOC	26	0.0	0.3	10.0	>10.0	5	0.0	0.5	>0.5	-
ΣSVOC	28	0.03	0.03	>0.03	-	31	0.03	0.05	>0.05	-
R value	2.600	2.6	0.5	>0.5	-	0.540	0.5	0.5	>0.5	-
ΣVOC w/o LCI	0	0.00	0.05	>0.05	-	0	0.00	0.05	>0.05	-
Σ cancerogens	0	0.000	0.001	0.01	>0.01	0	0.000	0.001	>0.001	-
Evaluation				➔				✓		

✓ Abort criterion fulfilled

➔ Abort criterion not fulfilled, further testing required

✖ Material rejected

In this deliverable the raw insulation material was investigated. In reality, the surfaces that were exposed to the test chamber air will be coated or appropriately covered. So, new emittants from the coatings will occur. However, the material as it was tested here would be suitable for the indoor use from the IAQ point of view.

4 Conclusions

In this deliverable the emissions of the BRIMEE insulation material was tested for possible indoor emissions. Two broken pieces of NCC panel with an average surface area of 0.32 m² and larger than a standard panel (0.24 m²) were investigated in a 250 L emission test chamber, operated at test conditions defined in prEN 16516. The test was designed such that measured concentrations correlate to concentrations expected to be obtained in the European reference room assuming that all walls are covered. Hence, no further data treatment was necessary. The results were evaluated against the German AgBB scheme for the health-related evaluation of building products.

The AgBB scheme was chosen since it is a scheme currently applied for the evaluation of emissions from flooring materials and decorative wall coverings in Germany, and a scheme based on it is under discussion for the EU-wide harmonized health-related evaluation of building materials. However, it is not mandatory yet but it is recommended to be taken into consideration.

The test results attested the insulation material generally low emissions. Although the previous flame retardant was changed, phenol emissions were detected but on a much lower level than before (cf. D6.1 report). The overall VOC release was so low that the abort criteria as defined in the AgBB scheme were fulfilled at the 7th day. Regardless of the fact that in its future application the raw material will not be facing indoors, it can be summarized that the BRIMEE material at its current state is suitable for the indoor use from the IAQ point of view.

5 References

[1] prEN 16516, *Construction products - Assessment of release of dangerous substances - Determination of emissions into indoor air (prEN 16516:2015)*. 2015: Beuth, Berlin.

[2] ISO 16000-3, *Indoor Air - Part 3: Determination of formaldehyde and other carbonyl compounds - Active sampling method (ISO 16000-3:2011)*. 2011, International Organization for Standardization Beuth, Berlin.

[3] ISO 16000-6, *Indoor air - Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID (ISO 16000-6:2011)*. 2011, International Organization for Standardization: Beuth, Berlin.

[4] ECA report no. 27, *Harmonisation framework for indoor products labelling schemes in the EU*, in *European Collaborative Action*. 2012, Publications Office of the European Union: Luxembourg.

[5] ECA report no. 29, *Harmonisation framework for health based evaluation of indoor emissions from construction products in the European Union using the EU-LCI concept*. 2013, Publications Office of the European Union: Luxembourg.

[6] AgBB, *Health-related Evaluation of Emissions of Volatile Organic Compounds (VVOC, VOC and SVOC) from Building Materials* 2015.