

# VOC emissions of PVC flooring - long-term analysis

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## SUMMARY

In this study, different screed, adhesive and PVC floor covering combinations together with different floor covering conditions were used in a long-time laboratory test series. VOC emissions were then measured from material samples (Bulk-VOC) and from surface emission samples (FLEC-VOC). The test specimens have been measured for 3.5 to four years after installing the floor covering. The test results show changes of primary and secondary VOC emissions in both deteriorated and flawless flooring samples. In some test specimens where the flooring was installed directly above moist concrete the deterioration was noticed quite soon after installing the flooring. The emissions of C9- to C10-alcohols started increasing later than other common emissions (1-Butanol, 2-ethyl-1-hexanol). According to the VOC analysis some peak values of the primary emissions of 2-EH and 1-Butanol were noticed at about one year after installation the floor covering, C9-C10-alcohol emissions even until two years.

## KEYWORDS

VOC emissions, PVC flooring, IAQ

## 1 INTRODUCTION

Concrete as alkaline (pH ~12.5) and moist material can cause deterioration of PVC floor coverings and adhesives through alkaline hydrolysis, causing harmful volatile organic compound (VOC) emissions into indoor air. The efficiency of the reaction, the hydrolysis of esters (Uhde and Salthammar, 2007), depends on the moisture content or relative humidity (RH), temperature and pH. Both PVC flooring and some commonly used flooring adhesives, such as acrylate-based copolymer adhesives, consist of esters and can therefore deteriorate causing VOC emissions. The VOCs in indoor air have hazardous effects on human health and well-being. The most typical symptoms are odour nuisance and eye and upper airway irritation (Wolkoff et al, 2006). Attempts to prevent this phenomenon have been made by installing floor coverings on top of 'dry-enough' concrete or using a low-alkali layer (screed) between the concrete and the floor covering. The pH of low-alkali screed is typically less than 11.5. Many new materials can produce primary emissions, meaning the physical release of compounds of a new product. Deterioration of materials cause secondary emissions, which are defined as 'compounds produced by chemical reactions in the product or in the indoor environment'.

The screed, adhesive and PVC floor covering combinations have been varied together with different floor covering conditions (RH of the concrete) in this study. The measured long-time primary and secondary VOC emissions in deteriorated as well as flawless flooring samples have been presented in this paper.

## 2 MATERIALS/METHODS

The screed, adhesive and PVC floor covering combinations have been varied using different floor covering conditions (RH of the concrete) in a full-scale thickness laboratory test series. The structure of the laboratory test samples corresponds with a typical slab-on-ground structure, with thermal insulation (EPS) beneath a 80 mm concrete slab, capacity of drying only upwards, and a glued PVC covering on top. Moisture level of concrete at the installing of floor covering ( $RH_{crit}$  in 32 mm depth from the top) has been varied: RH80%, RH85% and RH93%. The RH85% equals the current guideline's flooring criterion. Some samples were prepared with a 5 mm self-levelling screed layer. The pH of the screed material or the pH of the flooring underlay have been varied: a gypsum-based screed (pH less than 10), a low-alkali screed (pH about 11.5) and a cement-based screed or no screed (pH ~12,5). Two different types of adhesives were used: a common acryl copolymer dispersion or an alkali resistant adhesive. Three floor coverings have also been used: two homogenous floor coverings, intended for public spaces, one containing DINCH (diisonyl cyclohexane-1.2-dicarboxylate) plasticizer and the other DINP (diisononyl phthalate) plasticizer, and one heterogenous floor covering, for housing rooms, containing DINCH plasticizer. The heterogenous floor covering typically consists foamy bottom and thin uniform PVC cover. The wear resistance and water vapor resistance is lower than homogenous floor coverings. We have totally 23 different test specimens.

The RH of the concrete slab at the equivalent depth (equals the maximum RH in the area close to the surface between the flooring and the interface of concrete or screed (Hedenblad, 1997)) was measured using computer-controlled Vaisala HMP110 temperature and RH sensors and handheld Vaisala HMP40 T and RH sensors, accuracy of the sensors being  $\pm 2$  RH%.

Test samples were cast into  $530 \times 325 \times 10$  mm<sup>3</sup> steel boxes. At the bottom of the box, a 20 mm layer of EPS (expanded polystyrene) thermal insulation layer was installed. An 80 mm layer of concrete, with a water/cement ratio of 0.5, was cast on top. The test samples were stored under constant conditions,  $T = +21$  °C and  $RH = 50\%$ , until the RH of the concrete at the equivalent depth reached the agreed floor covering criterion. The self-levelling screed layer was applied to some samples and dried under constant conditions before the floor covering was glued.

VOC emissions were measured from material samples and surface emission samples. Material samples or Bulk-VOC samples were collected from a sample of about  $40 \times 40$  mm<sup>2</sup>, including floor covering and some adhesive and screed, into a Tenax TA tube using a Micro-Chamber/Thermal Extractor (Micro-Chamber,  $\mu$ CTE) and then the emissions were analysed using the TD-GC-MSD instrument (European Committee for Standardization, 2004). The method has been presented by Hughes et al. (2006). In Finland, the Bulk-VOC analysis using  $\mu$ -CTE analysis is the most common method in practical cases where possible deterioration of PVC flooring structures has been investigated, because it is quicker and cheaper than FLEC sampling. The surface emission samples were collected using the Field and Laboratory Emission Cell (FLEC) method (Nordtest, 1996) and analysed using the TD-GC-MS. The main VOC analysing method in this study was to analyse emissions from material samples (Bulk-VOC using  $\mu$ -CTE analysis) because the possible deterioration could be first noticed in materials and their interfaces and only later in surface emissions.

The pH of the concrete and the screed was defined from material samples using a method introduced by Björk and Eriksson (2002). A small sample was drilled from the concrete, or the screed, and the  $0.5 \pm 0.01$  g drill dust was mixed with  $5 \pm 0.2$  g ion purified water and let

to blend for 20 minutes, mixed together in every 5 minutes. After the solid matter settled in a laboratory tube, the pH of the liquid was measured using a pH electrode.

### **3 RESULTS**

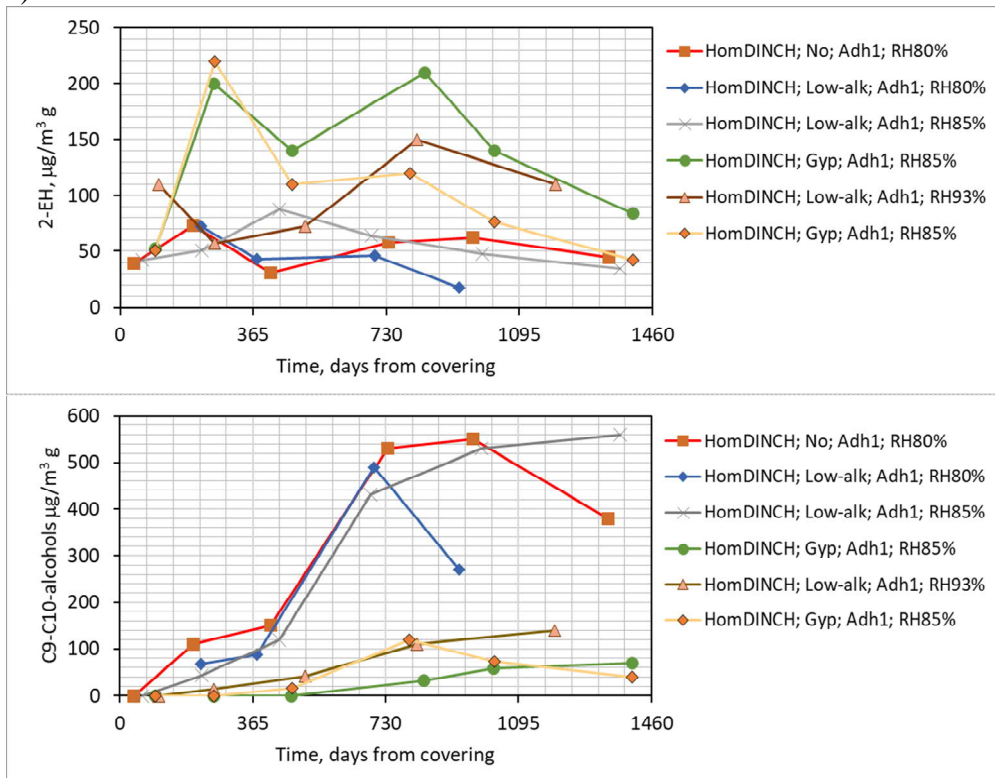
Altogether 117 Bulk-VOC analysis from the flooring material samples have been made, three to four samples from each test specimen. The first samples have been collected about three months after installing the floor covering and the latest ones 3.5 or four years after.

In 58% of the taken samples the floor covering was homogenous with DINCH plasticizer, in 32% heterogenous with DINCH and in 10% the floor covering was homogenous with DINP plasticizer. The most common VOC emission compounds in all test samples were 2-ethyl-1-hexanol (2-EH), long-chained alcohol isomers (C9-C10) and 1-Butanol, as expected. The dominant compound in the samples where the floor covering was homogenous with DINCH plasticizer and the adhesive being an acryl copolymer dispersion was 2-EH (43%), in the samples of heterogenous DINCH 1-Butanol (95%) and in the samples of homogenous DINP both 1-Butanol and 2-EH were equally common (38%). In the samples where the floor covering was homogenous with DINCH plasticizer and the adhesive was alkali resistant, the dominant compounds were C9-C10-alcohols (71%).

#### **Homogenous floor covering with DINCH plasticizer and acryl copolymer dispersion adhesive**

Among the 10 test samples, clear deterioration was noticed in four (Figure 1, b). The VOC emission levels of material samples (Bulk) were clearly high and so were the surface emissions (FLEC-VOC) the maximum TVOC being  $330 \mu\text{g}/\text{m}^2 \text{ h}$  and the maximum 2-EH being  $220 \mu\text{g}/\text{m}^2 \text{ h}$ . The high 2-EH emissions of the material samples (Bulk) were already measured a few months after installing floor covering. In all four samples the floor covering was installed directly above concrete (no screed) or the used screed was cement based and the measured pH of the flooring underlay was above 12. The RH of concrete during the floor covering was RH85% or RH83%. Figure 1 shows the temporal changes of 2-EH and C9-C10-alcohol concentrations. Typically, the highest 2-EH emissions were measured about one year after the floor covering. There were also some 2-EH emissions in the “flawless” flooring (Figure 1, a), partly due primary emissions. The C9-C10 alcohol emissions reached the “peak” values about two years after installing the floor coverings.

a)



b)

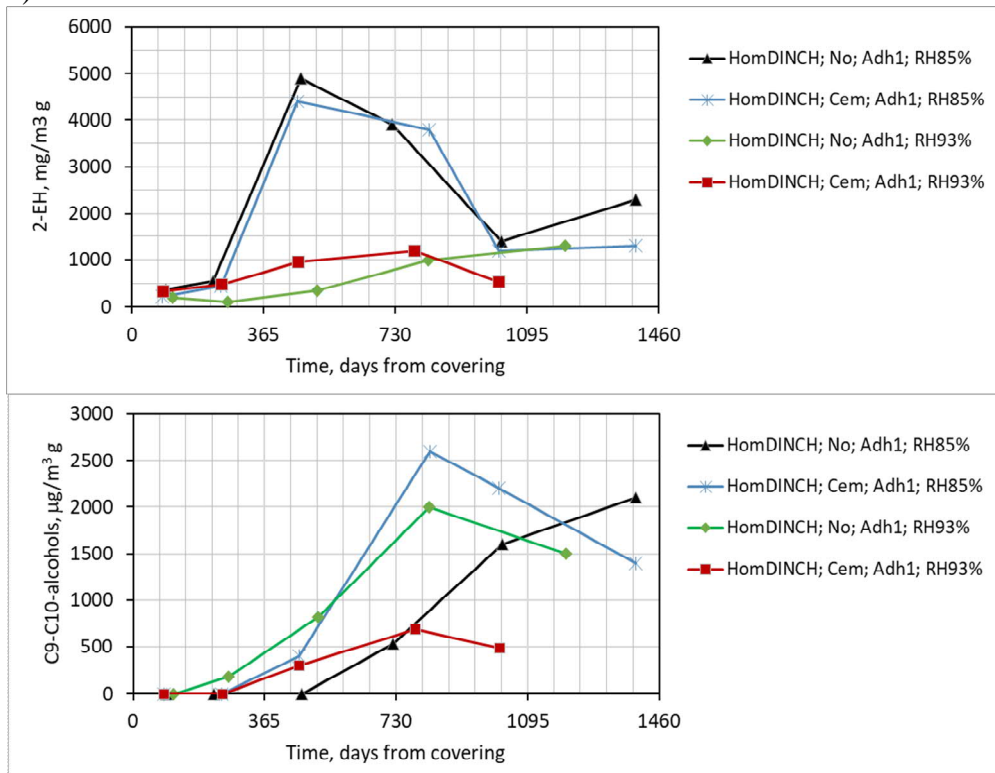


Figure 1. Temporal changes of 2-EH and C9-C10-alcohols, Bulk-VOC. a) deteriorated flooring, b) flawless flooring. Homogenous floor covering with DINCH plasticizer (HomDINCH) and acryl copolymer dispersion adhesive (Adh1) and a gypsum-based screed (Gyp), a low-alkali screed (Low-alk), a cement-based (Cem) or no screed (No). Note: different scaling in each diagram!

## Heterogenous floor covering with DINCH plasticizer and acryl copolymer dispersion adhesive

The VOC emission levels of material samples (Bulk) from the 8 test specimens were usually lower than the samples from specimens with a homogenous floor covering. The water-vapor resistance of heterogenous flooring, used in housing is relatively low and therefore the emissions penetrate the floor covering quite easily compared to specimens with a homogenous covering. There was no clear difference in the VOC emissions of material samples between deteriorated and flawless specimen because the emissions penetrate through the permeable floor covering. Though, the measured FLEC-emissions were quite high in some samples (maximum TVOC  $240 \mu\text{g}/\text{m}^2 \text{h}$  and maximum C9-C10-alcohols  $200 \mu\text{g}/\text{m}^2 \text{h}$ ), which indicate deterioration. Those samples were installed directly above concrete (no screed), or the used screed was cement based, with a high pH. Figure 2 shows the temporal changes of the concentrations of 2-EH, C9-C10-alcohols and 1-Butanol.

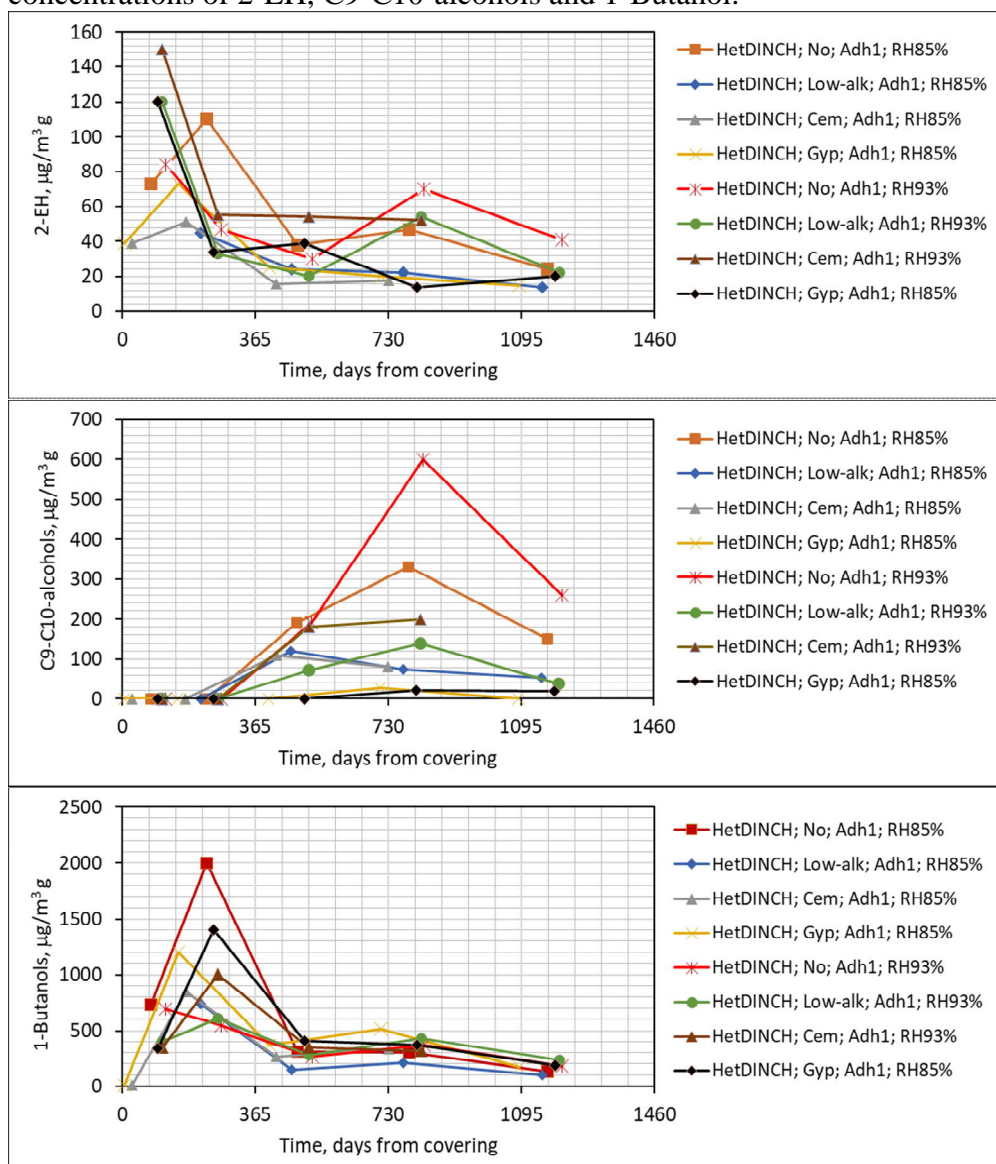


Figure 2. Temporal changes of 2-EH, C9-C10-alcohols and 1-Butanol, Bulk-VOC. Heterogenous floor covering with DINCH plasticizer (HetDINCH) and acryl copolymer dispersion adhesive (Adh1) and a gypsum-based screed (Gyp), a low-alkali screed (Low-alk), a cement-based (Cem) or no screed (No). Note: different scaling in each diagram!

### Homogenous floor covering with DINCH plasticizer and alkali resistant adhesive

Figure 3 shows the temporal changes of the concentrations of 2-EH, C9-C10-alcohols and 1-Butanol of three test samples of material samples (Bulk). The moisture of concrete was RH93% (exceeding the guideline's floor covering criterion) when the floor coverings were installed in all three samples. The 2-EH emissions were very low, since there is no material which has 2-EH as a reaction product. The smallest C9-C10-alcohol and 1-Butanol emissions are from the specimen with the floor covering on a gypsum-based screed layer (pH lower than 9). The C9-C10-alcohol emissions of one sample, where floor covering was installed directly above concrete (no screed) are still growing. The FLEC-emissions are still quite low (maximum TVOC less than 20  $\mu\text{g}/\text{m}^2 \text{ h}$  and maximum 1-Butanol 16  $\mu\text{g}/\text{m}^2 \text{ h}$ ).

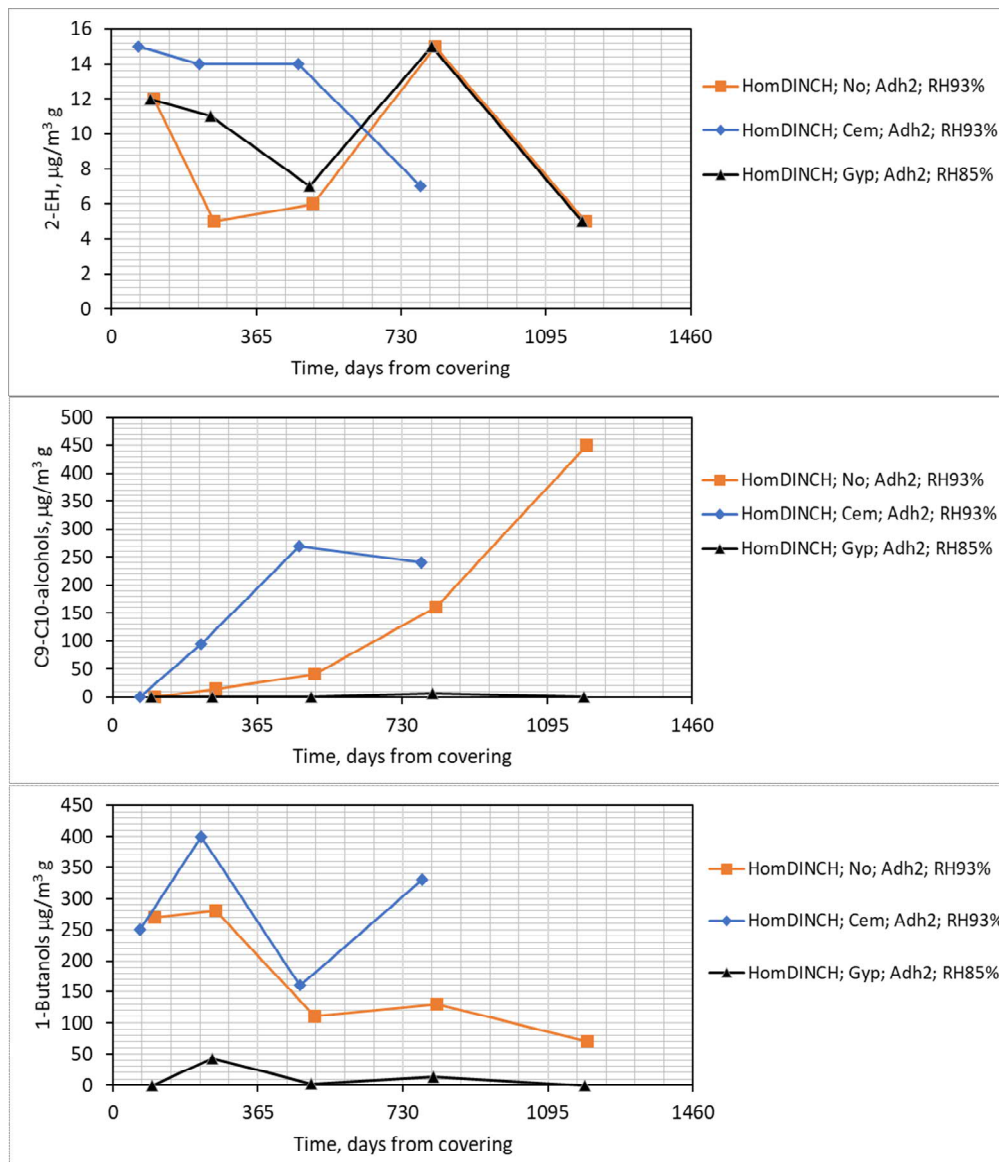


Figure 3. Temporal changes of 2-EH, C9-C10-alcohols and 1-Butanol, Bulk-VOC. Homogenous floor covering with DINCH plasticizer (HomDINCH) and alkali resistance adhesive (Adh2) and a gypsum-based screed (Gyp), a cement-based (Cem) or no screed (No). Note: different scaling in each diagram!

#### **4 DISCUSSION**

One clear finding from the research, based on both FLEC- and Bulk-VOC analysis, is that a 5 mm layer of low-alkali screed (pH less than 11,5) will effectively protect PVC flooring against alkaline hydrolysis. The previous studies considering low-alkali layer, mainly performed in Sweden in 1990's and 2000's (Björk et al, 2003) had the same conclusion. This previously discovered finding is also valid for new PVC flooring materials with new type of plasticizers.

According to the VOC analysis from material samples (Bulk) up to 3.5 or four years after the floor covering the highest emission peak of 2-EH and 1-Butanol was noticed about one year after installing the floor covering, for C9-C10-alcohol emissions about two years after. After that the emissions levels started decreasing. These emissions are partly/mainly primary emissions. There is a risk of misleading interpretation of floor covering deterioration if material samples for Bulk-VOC analysis are taken from recently installed floor coverings. We suggest that another round of sampling and analysis should be performed two or even three years after installing the floor covering when investigating an actual case with suspicions of floor problems. Furthermore, a more reliable assessment of possible VOC emissions into indoor air can then be obtained from a FLEC-VOC sampling.

Castagnoli (2019) studied the surface (FLEC) and bulk emissions of two DEHP-free PVC floorings over three years. The floorings were glued on the screed layer above concrete slab which RH was 75%, 85%, or 95%. The highest emissions of 2-EH from Bulk-VOC analysis were measured in first months in samples with 75% RH and about 8 months after covering in samples with 95% RH. The highest emissions of C9-alcohol from Bulk-VOC analysis were measured about 8 months after covering in samples with 75% RH and in samples with 85% or 95% RH the C9-alcohol emission levels were more or less increasing in the last measurement 36 months after covering. These results are quite congruent with our results.

The research will continue until at least five years has passed from installing the floor coverings of all the samples.

#### **5 CONCLUSIONS**

VOC emissions of totally 23 different test specimens where the moisture level (RH80%, RH85% and RH93%) of concrete, flooring underlay material (gypsum-based, low-alkali or cement-based screed or no screed), adhesive (acryl copolymer dispersion and alkali resistant adhesive) and floor covering (homogenous floor covering, containing DINCH or DINP plasticizer and heterogenous floor covering containing DINCH plasticizer) have been studied 3.5 to four years after installing the floor coverings. The highest VOC emissions and deterioration were measured in samples where the floor coverings were installed directly above concrete (no screed) or where the used screed was cement based with the measured pH of flooring underlay above 12 and the moisture level of concrete was RH85 or RH93%.

According to the VOC analysis from material samples the highest emission peak of 2-EH and 1-Butanol, mainly due primary emissions, was noticed about one year after installing the floor covering, for C9-C10-alcohol emissions even two years after.

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