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Hydroxide ion concentration at an interface between concrete and a self-levelling flooring compound

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ABSTRACT

This article presents results from measurements of hydroxide ion transport between a concrete and a floor screed of a lower alkalinity than the concrete. As many floor coverings and floor adhesives are sensitive to high alkaline conditions it is important to know how hydroxide ions are transported to be able to evaluate the long-term function of floor constructions. It was found that only minor transport of hydroxide ions occurs in the hygroscopic moisture range. One conclusion is therefore that a floor screed of lower alkalinity may protect sensitive floorings from the high alkalinity present in concrete.

Key words: alkalinity, concrete, SLC, flooring compound, moisture.

1. INTRODUCTION

During the last decades there has been an increased attention to indoor air quality. Poor indoor environments and “damp” buildings are suspected to contribute to adverse health effects, such as headaches, tiredness, mucous membrane symptoms, as well as asthma and allergy [1-3]. Indoor pollution has also been correlated with lowered productivity in office environments [4].

Organic compounds in indoor air are suspected to contribute to poor indoor air quality [5, 6]. Studies have shown that secondary emissions (emissions produced by reactions in materials) are important for the indoor air quality [7] and secondary emissions from floor constructions with bonded plastic coverings on concrete are suspected to cause health problems [8, 9]. Such emissions occur when floor adhesives and floor coverings are degraded forming volatile products that are emitted to the indoor air. The degradation occurs under the influence of high pH and moisture (alkaline hydrolysis). Common floor constructions subjected to degradation are PVC and linoleum floorings bonded on concrete substrates. Typical degradation products

are 2-ethyl-1-hexanol and butanol [10-12], which both have an unpleasant odour. Correlation between increased levels of 2-ethyl-1-hexanol and “sick building syndrome” (SBS) symptoms have been reported by for example [13-15].

As many of these floor coverings are almost impermeable to moisture, it is important that the concrete substrate is sufficiently dry prior to applying the floor covering. Generally the moisture state of a substrate, measured as the relative humidity (RH), the moisture content or the moisture flow from the surface, is used as criterion for when floor coverings may be applied. The prediction of the long-term state of a construction from such measurements is very uncertain. Besides, it is not the moisture as such, but the combination of moisture and the alkaline nature of inorganic binders that causes problems.

The surface often has lower pH due to that it has been exposed to atmospheric carbon dioxide resulting in carbonation. This is a process where CO_2 from air is dissolved in the pore solution and then mainly reacts with dissolved $Ca(OH)_2$ in the pore solution forming solid $CaCO_3$ and water thereby decreasing the amount of hydroxide ions in the pore solution. The rate of penetration of carbonation varies with e.g. the concrete quality and the moisture state, but is generally said to be proportional to the square root of time at constant moisture conditions [16] and the front has a non sharp profile [17]. Carbonation lowers the pH of a Portland cement based concrete from around 13 to less than 9 [18]. Carbonation is therefore a way to decrease emissions from concrete floors. However, when a floor covering is applied, the moisture and hydroxide ions may be redistributed in the slab and the pH level of the top part of the slab may therefore increase again due to convection of ions with the moisture flow and ion diffusion in the pore water. The long-term effect of carbonation of the surface is therefore uncertain.

Generally, hydrolysis is any process where a chemical compound reacts with water. In the present context, hydrolysis is the reaction between an ester group and a hydroxide molecule, leading to the formation of an alcohol and an acid [11]. The rate of this process is dependent on the pH level: the more alkaline the solution the higher the rate of hydrolysis. The rate of ester hydrolysis in solution is proportional to the hydroxide ion concentration [19]. For, e.g., adhesives on alkaline substrates the trend is probably the same, even if the situation is much more complex.

Self-levelling flooring compounds (SLCs) are generally laid in thin layers, normally 1-50 mm, to give horizontal smooth surfaces. The cement based SLCs described in this article normally contain a mixture of calcium aluminate cement, calcium sulphate and Portland cement as binders. Quartz sand and finely ground limestone are normal filler materials. To improve flexural strength, tensile strength, and abrasion resistance a redispersible polymer powder is added. The polymer particles coalesce into films or membranes that bind the cement hydrates together in a network [20]. Usually a polymer primer is applied on the substrate before applying the SLC. The primer gives a better and more uniform bonding between substrate and SLC, better flow, prevents trapped air in the concrete surface to give rise to bubbles on the SLC surface, and also prevents excessive water to be absorbed in the concrete.

Several investigations have shown lower rates of emissions from constructions where floor coverings were bonded on SLCs than when the floor coverings were bonded directly on concrete [10, 12, 21]. The reason for this is believed to be the lower alkalinity (pH) of SLCs.

The pH of a non-carbonated SLC is about 11 [22] so its concentration of hydroxide ions is about two orders of magnitude lower that in a non-carbonated concrete.

Hydroxide ion transport requires a transport medium, moisture. It is therefore of interest to investigate what levels of moisture that are necessary to obtain a transport of hydroxide ions from concrete to SLC as well as within an SLC. At RH levels above 90%, emissions seems to increase with time from this type of floor constructions. One proposed reason for this is that hydroxide ions from the concrete are transported through the SLC, thereby increasing the pH in the contact zone between the floor adhesive and the floor covering [10].

Björk and Eriksson [23] investigated pH in specimens where flooring compounds were cast on top of concrete. They did not see any transport of hydroxide ions, measured as pH, from concrete to SLC during 12 months of time on specimens with moisture levels estimated to 85-95% RH. The concrete was exposed to atmospheric environment while drying and was therefore carbonated to some extent in the surface. An increase in pH in the top layer of the concrete was however noted, which indicates that transport from inside the concrete towards the carbonated surface occurred.

The pore solution of concrete mainly consists of potassium, sodium and hydroxide ions, but also to some extent calcium and sulfate ions [18]. Transport of a mixture of ions in the pore solution in concrete is influenced by five main mechanisms: diffusion, convection, binding, and electro-migration due to external electrical fields or induced internal electric fields [24]. Diffusion occurs when a concentration gradient of the individual ion is present. As the concrete has a higher concentration of hydroxide ions than SLC, hydroxide ion transport should go from concrete to SLC. However, the concrete surface has normally been subjected to carbonation prior to applying the SLC why hydroxide ions initially may be transported from SLC to concrete. After a while the higher concentrations of hydroxide ions from the non-carbonated part of the concrete will reach the top of the concrete and the redistribution continues into the SLC.

Convection is mass flow of ions due to the motion of the pore solution. Convection occurs directly after casting, but ceases when a vapour tight floor covering is applied and the moisture has been redistributed. Convection has been shown to be important when describing transport of chloride ions in concrete [25] and Wengholt Johnsson [26] studied concentrations of potassium ions in concrete specimens that were drying by surface evaporation and concluded that transport of these ions occur towards the surface while drying. Binding (precipitation or dissolution) of ions is also an important process in this context. For example will calcium hydroxide dissolve if hydroxide ions are removed from the pore solution in Portland cement based concrete [27]. External electrical field are generally not present, but induced internal electrical fields may be present at small scales of length and time [24].

SLCs contain a rather large amount of polymers that form polymer films throughout the material. Polymer-modified mortars have in investigations shown to have lower transport of chloride ions [28-30]. Chemical interactions between concrete and polymer and the fact that polymer films can seal larger pores are two proposed explanations for the reduced transport.

This article presents results from measurements of hydroxide ion levels in specimens that consisted of concrete substrates and SLCs at different moisture levels, both with and without a surface treatment (primer) on the concrete. Attempts are also made to extrapolate the experimental results in time to study the possible long-term effect of the ion transport. The

concrete was initially dried to two different moisture levels to study how initial moisture levels influence the transport of hydroxide ions by giving rise to different convective moisture flows.

2. Materials

One Portland cement based concrete, one SLC and one primer were chosen for the experiments. The concrete had 410 kg/m^3 CEM I 52.5 R cement, 1520 kg/m^3 aggregate size 0-8 mm and W/C 0.65. No additives were used. The SLC was cement based and normal drying, see [31] for a more detailed description of the product and [32] for a more comprehensive description of this kind of products. The primer was a dispersion of styrene acrylate in water. In these tests the primer contained about 12% styrene acrylate and 0.4 kg of this solution was applied per m^2 of substrate as recommended by the manufacturer. Deionised water was used throughout this study.

3. Method

3.1 Casting and conditioning

The concrete was cast in 40 mm thick layers in 40x100 mm^2 moulds and cured at 20 ºC. The specimens were stored one week at 100% RH followed by three months at either 95% RH or 75% RH in climate boxes. After this SLC was cast on the specimens in 20 mm layers. Five days later, the specimens were de-moulded and stored in four different climates, 85, 91, 95 and 98% RH at 20 ºC, see Table 1. The RH in the climate boxes was generated by saturated solutions of KCl, $BaCl_2$, KNO_3 and K_2SO_4. To avoid three-dimensional moisture flow, the test specimens were treated on the sides (not top and bottom) with a mixture of beeswax and paraffin before placing them in the climate boxes. One week prior to casting the SLC, some of the specimens were surface treated with the primer. Although stored in climate boxes, some carbonation occurred in the specimens as no special care was taken to prevent this. The carbonation depth was measured on separate specimens before casting the SLC.

Table 1. Description of the test specimens and RH in climate boxes before and after casting SLC on the concrete.

Sample nr	RH first three months	Primer	RH after casting SLC
1	75%	yes	85%
2	75%	yes	91%
3	75%	no	91%
4	95%	yes	91%
5	95%	yes	95%
6	95%	no	95%
7	95%	yes	98%

3.2 pH indicator

A pH-indicator, phenolphthalein in ethanol solution that changes colour in the pH-range of 8,2 to 10 [33], was sprayed on specimens to visually check the depth of carbonation on the

concrete before applying the SLC. Phenolphthalein was also sprayed on each specimen at the measurements at 1, 6 and 12 months (se below). The sprayed specimens were photographed with a high-resolution camera for visual evaluation of the “pH-front”. Four measurements were performed on specimens stored at 75 and 95% RH in connection to casting the SLC and two measurements were performed on each specimen at 1, 6 and 12 months.

3.3 pH measurements

After 1, 6, 12 and 15 months the pH profile in the specimens was measured. Measurements after 1 and 6 months were mainly made in order to study the influence on ion transport resulting from the convective flow that is present after casting. Layers of 1-2 mm (4 to 12 grams) were milled off from top and downwards. Thinner 1 mm layers were used close to the boarder between concrete and SLC. The milling was made with a flat tool, 32 mm diameter over approximately 60 mm. The resulting particles were immediately collected and stored in sealed ampoules until the pH-measurements were made. The pH of the particles was measured after stirring the particles for 10 min with a magnetic stirrer in twice their mass of deionised water. A glass-body pH-electrode (Orion 9172 BNWP) connected to a pH-meter (Orion 720A Benchtop pH/ISE) was used. Calibration was made in three buffer solutions, pH 7, 10 and 12.5 each day when measurements were performed. A thorough investigation and comparison of measurements performed with pH-electrode in solution and by pore extraction has previously been performed by [22]. It was found that the two methods correlate well for the SLC and the CEM I based concrete used. Slightly higher water to powder ratio than recommended by Räsänen and Penttala, 2 instead of 0.7-1.5, was used in this study to get enough solution for the pH-sensor. The measurements of pH in this study are meant to indicate whether a transport of hydroxide ions occur and should not be seen as true values of the pH in the materials.

3.4 RH measurements

RH was measured on pieces cut out from the material using Vaisala HMP44 humidity probes after 1 and 12 months. The humidity probes were calibrated in a two-pressure humidity generator (Series 2500, Thunder Scientific Corporation, USA) at four humidity levels, 75%, 85%, 90% and 95% RH.

4. RESULTS

4.1 pH indicator

The pH front as indicated by the phenolphthalein was between 1.2 and 0.4 mm from the concrete surface, with a tendency of moving closer to the interface with time. The depth of carbonation on the concrete specimens prior to casting the SLC was thus about 1 mm.

4.2 pH measurement

Results of measurements of pH on particle solutions from specimens after 6 and 15 months are presented in Figure 1. The pH shows rather steep jumps from concrete to SLC and a small

gradient in the SLC close to the interface to the concrete. The results show that only minor transport of hydroxide ions has occurred in the specimens between 6 and 15 months, irrespective of the humidity level.

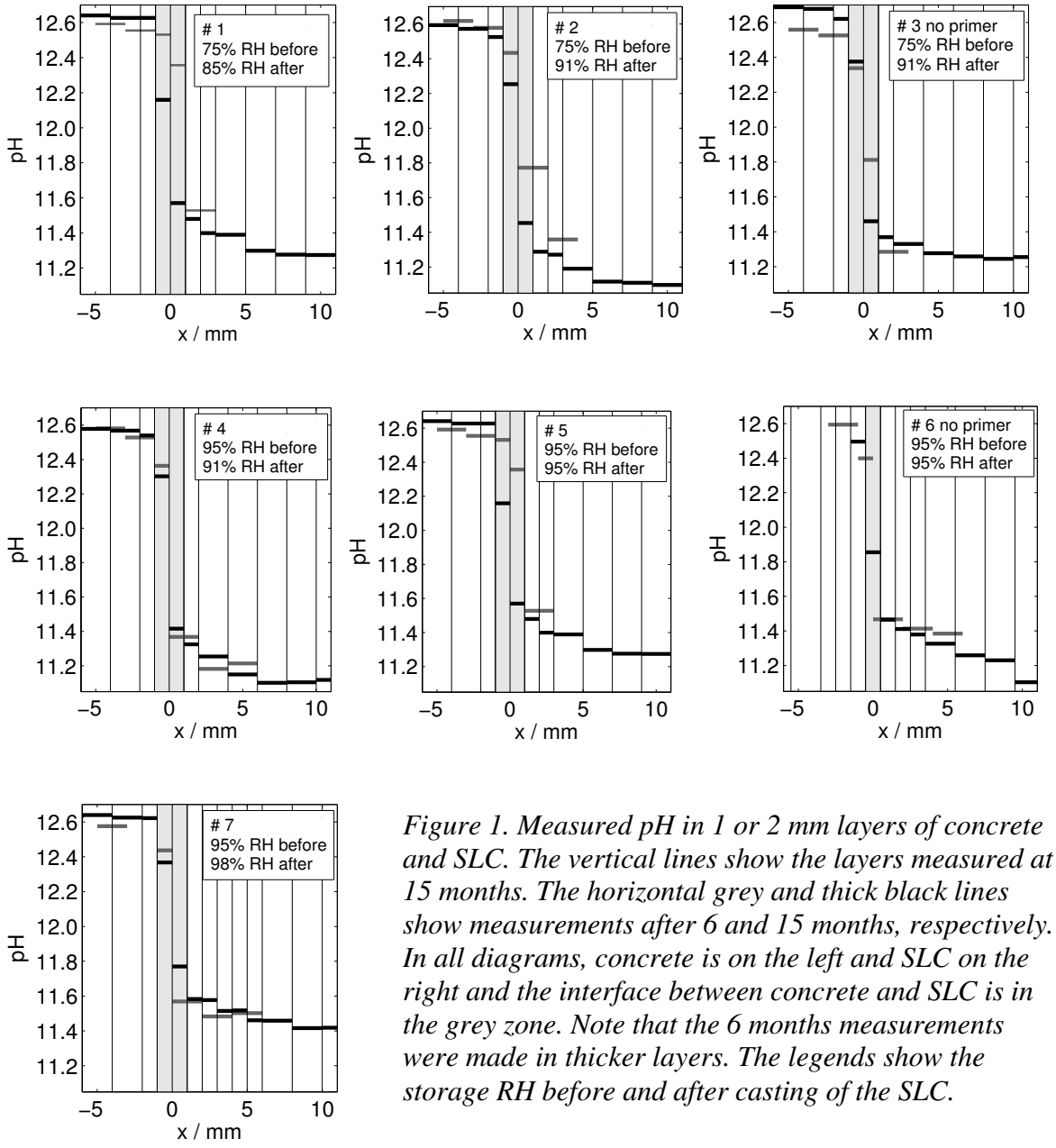

Figure 1. Measured pH in 1 or 2 mm layers of concrete and SLC. The vertical lines show the layers measured at 15 months. The horizontal grey and thick black lines show measurements after 6 and 15 months, respectively. In all diagrams, concrete is on the left and SLC on the right and the interface between concrete and SLC is in the grey zone. Note that the 6 months measurements were made in thicker layers. The legends show the storage RH before and after casting of the SLC.

4.3 RH measurements

The results from the RH measurements performed on pieces cut out from specimens are presented in Table 2. The results of the measurements performed 1 month after casting the SLC agreed well with the values from the salt solutions except for samples 1 and 4 that deviate 4 %-units. Measurements performed after 12 months generally show about 2 %-units lower than would have been expected from the salt solutions, except for samples 5 and 6 that both show 6 %-units lower values. One reason for the lower RH than expected by 12 months may be that the climate boxes were not vapour tight.

Table 2. Measured RH-values in specimens 1 and 12 months after casting the SLC compared with the RH of the saturated salt soutions in the climate boxes.

Sample	RH 1 month	RH 12 months	RH salt
1	89	83	85.1
2	92	88	91
3	92	89	91
4	95	89	91
5	95	89	94.6
6	95	89	94.6
7	95	96	97.6

5. DISCUSSION

A qualitative description of the development of the moisture and pH state in a combination of concrete and SLC is given in Fig. 2. The newly cast concrete has pH 13 and a high moisture state (A), but will immediately start to dry and carbonate in the surface (B). After some time, SLC with pH 11 will be cast on top of the concrete (C). Moisture from the SLC will both evaporate and be absorbed by the concrete, often resulting in rather complicated moisture profiles (D). The SLC will carbonate in the surface before the vapour tight floor covering is applied. The moisture will now redistribute from the more humid parts in the bottom of the construction towards the drier parts in the top, possibly bringing ions in convective flow (E). When the moisture is redistributed, diffusion will continue to transport hydroxide ions from the concrete to and through the SLC (F).

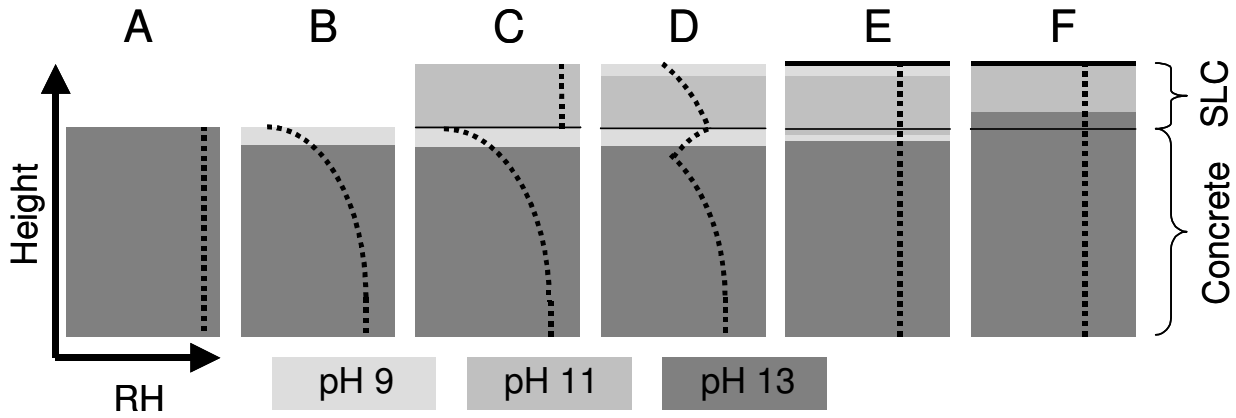

Figure 2. Qualitative illustration of moisture and pH development in a material combination of concrete and SLC in time. Dotted line represents the moisture profile, the thick black horizontal line the floor covering and the different grey shades the different pH-levels.

The present measurements show that only a very limited transport of hydroxide ions occurred during the first 15 months and it is probable that the pH profile seen is a result of the early convective water movements after the SLC had been cast. There is no discernible difference between the results after 6 and 15 months so the diffusion of hydroxide ions is low. As there is no reason to believe that the rate of diffusion will accelerate it is reasonable to believe that a

layer of SLC may act to protect the floor covering against the highly alkaline concrete. No conclusion regarding the influence of the primer can be drawn as the transport was low irrespective of using a primer or not.

Even if the present measurements show very low hydroxide ion transport by diffusion, this transport should not be ignored and may be an explanation of why emissions sometimes increase in time. This may, for example, be the case where carbonation has given a thin layer of lower alkalinity in the surface that may be protective for a while, but given time, perhaps several years, the transport of hydroxide ions may re-alkalise the carbonated layer and degrade the flooring. Another case is when the humidity of a floor construction increases e.g. due to construction changes. One should also be aware of that for example PVC-flooring plasticisers might migrate down into the alkaline substrate.

A simple attempt to fit the obtained hydroxide ion concentration data and extrapolate the results forward in time is here made. We have used the error function solution to Fick's law of diffusion [34] for the semi-infinite case with constant diffusivity and no convective flow. For the present case it can be used for simple modelling of ion transport assuming that the pH-level in the non-carbonated concrete is constant and that the boundary, the top of the SLC, does not influence the profile significantly. A rough estimate of the diffusivity of 10^{-14} m^2/s was obtained by fitting measured profiles, assuming that the hydroxide ion profiles measured after 15 months were the result of only diffusion. This value is used in Figure 3 to predict how the pH-profile may develop in time. This is a worst-case scenario as the profile from which the diffusivity was calculated was not a result of only diffusion, but also of initial convection as discussed above.

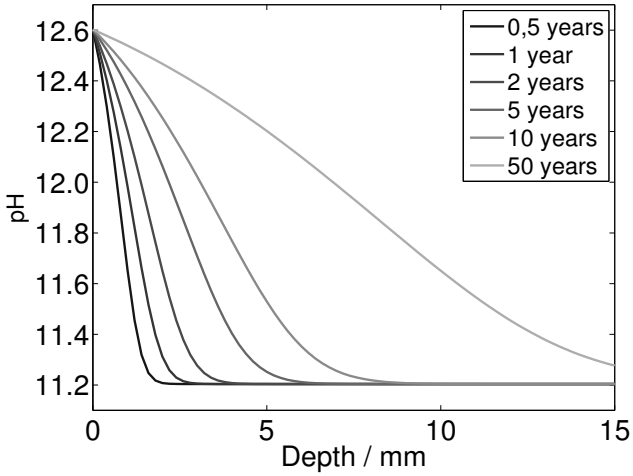

Figure 3. Results of simulations of hydroxide ion diffusion using a diffusivity of 10^{-14} m^2/s. Note that the hydroxide ion concentration has been recalculated into pH on the y-axis.

Concrete constructions often need long drying times to prevent for example degradation of floor coverings. It may be possible to speed up the building process by shortening drying times if SLCs are used as these are less aggressive than concrete and therefore can allow higher moisture levels. However, to use SLCs in this way further studies of ions transport and degradation processes are necessary.

6. CONCLUSIONS

The measurements show only low transport of hydroxide ions in the specimens (diffusion coefficients lower than 10^{-14} m^2/s), even after 15 months of storage at a moisture state above 95% RH. This indicates that SLC can be used as alkaline barriers between concrete and flooring materials also on a long-term basis. We found the present method suitable for these kind of measurements, but much longer exposure times are needed to quantify transport coefficients.

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