Blistering of epoxy industrial floor on concrete substrate: phenomena and case study

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Abstract:

Blistering of polymer coating and flooring systems on concrete slab substrates are a well known phenomenon. However, the reasons for and the origin of the blisters are much less understood. The paper gives an overview of available experience and theoretical knowledge on the blistering phenomenon: osmotic effects, moisture and damp problems in the substrate or in the environment, adhesion imperfections, polymer mixing problems.
The available knowledge will be checked on a real damage case, studied by the authors, where intense blistering occurred notwithstanding all necessary precautions seemed to have been taken. Because a complete record of the successive construction phases was available, it was possible to reconstruct the time history of the floor construction, and to compare experimental findings on the presence of the blisters and on the chemical analysis of the moisture in the blisters with specific actions in the construction process of the concrete floor and the epoxy industrial floor on top of it. In this case, two specific polymers were found in the blisters: pine-oil and phenol resin. The pine-oil was predominantly present in blisters that occurred at the transition zone between epoxy-mortar used as joint and hole filler and the epoxy-floor, whereas the phenol-resin only occurred in blisters between the concrete and the epoxy-floor. The paper evaluates and discusses the findings on the site and in the laboratory, and tries to determine the causes of the blistering, and the ways to avoid it.

Keywords: blistering, osmotic effects, polymer flooring, alkali-aggregate reaction

Introduction [1]

Polymer overlays on concrete have been used for more than 35 years. The problem of blistering underneath or within the polymer layers is as old as the application of these materials for coatings and flooring systems. Concrete is a porous material through which water or other fluid materials can easily be transported, together with the water-soluble materials of the concrete. By that, the pore water consists of inorganic compounds and has a rather high pH.

Three different types of pressure can be distinguished when looking at the concrete-flooring system. The first one is the hydrostatic pressure. This is the case when there is water pressure underneath the concrete floor and air pressure at the coating site. Normal bond strength of the polymer flooring can easily be higher than 1,5 MPa. To cause blistering, the hydrostatic pressure must be at least this value. In practice, this will almost never be the case, since a 10 m water column only represents a pressure of 0,1 MPa.

The second type can be the pressure due to capillarity. Theoretically, this pressure rises, when pore size becomes smaller. Since the pore sizes and forms in the concrete are very various, it is very difficult to calculate the pore pressure due to capillarity underneath the flooring. Experimental research on cement mortars [2] resulted in pressures due to capillarity of 0,2 MPa, which also is too small to cause blistering.

Considering the fact that blistering also occurs on dry concrete or on concrete without an hydrostatic pressure, it must be clear that the above mentioned phenomena could not be the reason for blister formation.

Research revealed that only osmotic processes could cause sufficiently high pressures for blistering to occur. Osmotic pressures can be higher than 2,5 MPa [3].

Osmosis

Osmosis is the phenomenon by which solutions try to reach equilibrium across a semi-permeable membrane. Water will flow from a more dilute solution to a more concentrated one when separated by a semi-permeable membrane. Such membranes allow for the passage of water but not of a dissolved substance.

Osmotic blistering can occur in new construction because the bond interface of the newly coated concrete slab acts as a semi-permeable membrane. The osmosis process continues until either equilibrium is met or until the hydrostatic pressure generated by the increase in volume of the more concentrated solution equals that of the osmotic pressure. In the case of a coated concrete floor, the pressure builds up at the interface between the coating and the concrete and causes blistering and progressive debonding.

Four elements are needed for osmotic blistering:

- A semi-permeable membrane, which in most construction cases is the bond interface or the extreme upper layer of the concrete.

- A concentration of water-soluble material, which can be anything from the resin ingredients in the epoxy coating to the material that can form at the surface of concrete when it is acid etched. Adding to the problem is the fact that Portland cement also has a naturally occurring soluble salt content that can also act as a catalyst for osmotic blistering.

- Water. Even when the concrete looks dry, it still contains up to 5 percent of free water by mass. This is enough moisture to start the osmotic blistering process. Adding to the problem are other outside moisture sources like groundwater, drains, water left over from cleaning processes or condensation.

- A deformable, impervious coating

The osmotic process is schematically presented in figure 1.

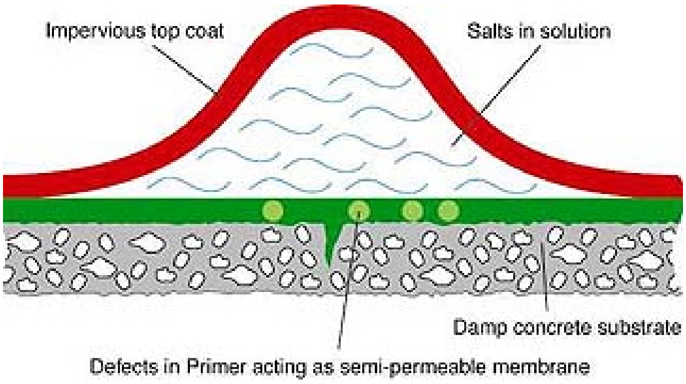

Fig. 1. The osmotic blistering process [4]

Semi-permeable membrane

The semi-permeable membrane can be the upper layer of the concrete itself, even when the pore size is too large. The penetration of (components of) the used primer can reduce the pore section in such a way that a semi-permeable membrane is formed.

Experimental research [5] indicated that this penetration did not occur for concrete with almost no pores (capillarity), for instance concrete C50/60 or concrete where the pores are completely filled with water (wet concrete). Therefore no catalyst for future osmotic blistering is present and osmotic blistering is not likely to occur.

Also the primer can act as a semi-permeable membrane and even the coating it self can act as a semi-permeable membrane. For instance in a water canal where water from the canal can penetrate through the semi-permeable coating and can cause blistering.

Blister fluid

The fluid in the blisters consists out of water with soluble compounds. These compounds are mainly inorganic coming from the concrete itself, but also organic compounds can be found in most cases. These organic compounds are base materials for the primer or the flooring itself. They can penetrate in the pore system of the upper layer of the concrete. Due to different pore sizes and also to the different molecule sizes of the compounds, a kind of separation on micro scale occurs. This is called the chromatographic effect. Also other organic compounds can be found in the blister fluid, compounds which are not chemically bonded, like solvents or softeners. In most cases these compounds are highly hygroscopic and so they attract water in addition to the osmotic effect.

In some cases, no fluid is found inside the blister [6]. When the concrete surface underneath the blister is closely examined an amorphous deposit can be observed. Chemical analysis reveals the presence of water glass. The incidence of blisters caused by an alkali-silica reaction in aggregates of a cement-bound base is extremely rare. The blistering is due to reactions directly on the surface of the substrate caused by a volume-enlarging transformation of alkali-hydroxide containing additives like amorphous opal sandstone and flint into water glass, which is highly hygroscopic. The reaction is represented by the following scheme:

$SiO_{2\ amorph}$ + 2 NaOH (KOH) => Na_2SiO_3 (K_2 SiO_3) + H_2O

This reaction can only occur if silicic acids are present in the additives, soluble alkali-hydroxide can escape from the concrete pores and sufficient humidity is present. Even than it is very difficult to distinguish between these blisters and the typical elevations caused by osmosis, because sometimes a white deposit can be observed underneath the blister, which is caused by a deposit of an oversaturated calcium-hydroxide solution in the blister [7].

Case study: conductive epoxy floor layer at ACE Electronics N.V., Diest [8]

The conductive floor at the production hall of ACE Electronics consist of the following parts:

- concrete base (fibre reinforced concrete and a PE-foil underneath)
- levelling layer of epoxy-mortar, near the expansion joints (Episol EMT)
- epoxy primer (Episol primer)
- electro-statically conductive layer (Episol Leitlack)
- epoxy flooring with added carbon (Episol SL EL)
- poly-urethane top coating

The concrete had a quality of C20/25. The cement used is a Portland cement CEM I 42,5R. No additives where used. The concrete was poured during the winter of 2002. At the end of April 2002, the epoxy flooring was applied. The surface humidity was still 3,7 %, but due to planning reasons it was suggested to put in place the epoxy flooring. Also, the temperature during placement of the epoxy system was rather low.
Due to a PE-foil underneath the concrete floor, no water transport from the soil must be considered.

Within a month after the placement of the epoxy floor, blistering was observed. First, the blisters could only be observed near the expansion joints which were filled with epoxy-mortar. At a later stage, blisters could also be observed at other places.

Figure 2 shows the marked blisters near an expansion joint.

Fig. 2. Blistering near expansion joint

It is remarkable that the blistering does not occur directly above the expansion joints, which would be the most obvious, but in a range of 5 to 10 centimetres next to this joint. This must be related to the fact that when filling the joints with epoxy-mortar a kind of smearing of the mortar occurred around the joint. So the blistering occurred in the transition zone between the smeared epoxy-mortar and the epoxy-floor.

To determine the cause of blistering three core drillings were executed and some blister fluid was sampled. During the sampling of the fluids, two differences could be observed and smelled. The fluid in the blisters in the zone of the expansion joints had a typical pine-oil smell. The fluid in the other blisters had a phenol resin smell.

The fluid samples where tested at the chemical laboratory of K.U. Leuven. A GC/MS TurboMass analysis was performed, after which the data was analysed with a NIST and Wiley Library search. The results of these tests proved the presence of the above mentioned organic substances (together with other organic substances).
The manufacturer of the epoxy flooring system confirmed the presence of pine-oil in the used epoxy-mortar and phenol in the used epoxy-primer.

The results of the core drilling revealed that the debonding always occurred in the contact surface between the concrete and the epoxy floor. In none of the cores a debonding within the floor system could be observed. A typical view of a concrete core with blisters in the epoxy flooring is shown in figure 3. A microscopic view of a spliced core is shown in figure 4. In this figure the debonding can be observed.

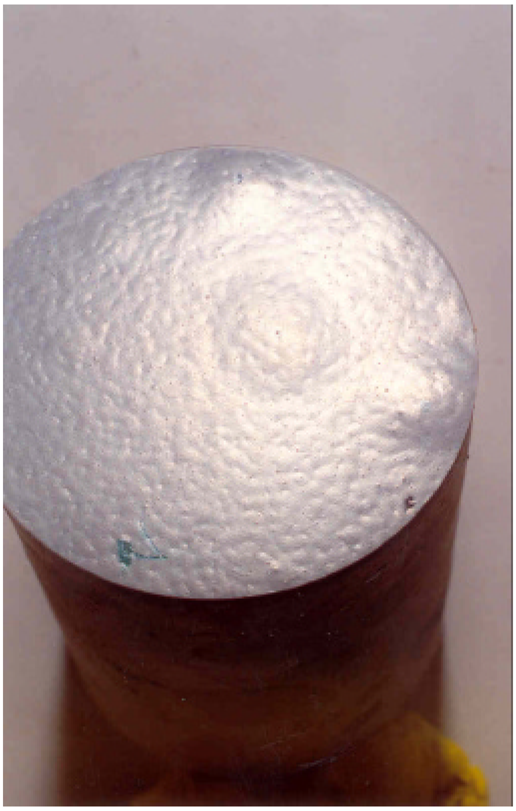

Fig. 3. Core with blisters

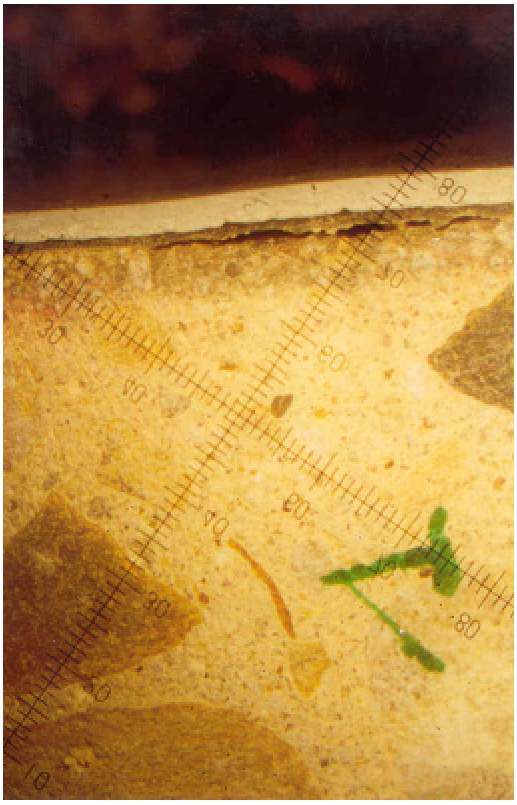

Fig. 4. Debonding

A core, drilled by the contractor revealed a white deposit underneath the debonded surface of the epoxy flooring. The blister fluid was no longer present.

The core is shown in figure 5.

Fig. 5. Core with white deposit

An XRD analysis of the deposit revealed that it was amorphous. A further EDX-analysis revealed the presence of potassium and silicon in the amorphous deposit. Only a small amount of calcium could be observed in the deposit. The presence of both potassium and silicon in an amorphous deposit could indicate the presence of reactive aggregates in the concrete resulting in the formation of highly hygroscopic water glass (alkali-aggregate reaction), which could also cause blistering, as already mentioned.

The main reason for blistering occurrence at this site was osmosis. The penetration of primer in the porous concrete created a semi-permeable membrane. Also the smearing of the epoxy mortar near the expansion joints caused the formation of a semi-permeable membrane. Due to the difference in concentration of soluble material in the concrete and in the epoxy-floor layers, a diffusion of water occurred, which resulted in the formation of blisters. Due to the low temperature during the placement of the epoxy system, the epoxy system could not react fast enough to ensure a proper binding. By that, the concentration difference was present for a longer period, thus the formation of osmotic blisters was more likely to occur.

Conclusions

Blistering is mainly caused by osmotic effects. Alkali-aggregate reaction can also sometimes cause blistering.

The worst part about osmotic blistering is that there really is no simple litmus like test to determine whether or not it will occur. It has been reported to occur after six months but also after only six days.
To prevent blistering to occur or at least reduce the chance of occurrence, the following actions can be taken [1], [4], [5]:

- If possible, minimize the amount of soluble salts in the concrete mix design;
- Take all necessary precautions to prevent alkali-aggregate reactions;
- Let the concrete dry for at least two months and/or ensure a fully functional water vapor barrier is underneath the slab when placed;
- Do not wash the concrete with detergent before applying the coating;
- Ensure that the epoxy coating resin constituents are accurately proportioned and mixed;
- If possible, apply the epoxy-system at high temperatures (20 °C to 30 °C), so a fast and proper curing of the floor can occur (this may effect the workability);
- Avoid using primers with non-chemically bonded compound, such as solvents, softeners, etc …;
- If possible, apply a dense layer, for instance an epoxy-mortar, on top of the primer on the whole surface to make the primer layer impermeable;
- If compatible with the primer system, a hydrophobic treatment can be considered to prevent moisture transport through the pores;

Obviously, some of those options are not viable when it comes to timely construction. Some options are also very costly. Worse yet, most of the extreme cases of osmotic blistering may occur even if these suggestions are followed.

A lot of research has already been performed to describe the phenomenon of osmotic blistering, still more research is needed to acquire a good understanding of the causes of blistering in floorings and of the prevention of it.

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