THALE SOFIE WESTER PLESSER

Moisture in Concrete - Alkaline sensitive surface treatments

Literature study
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SINTEF Building and Infrastructure

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Moisture in Concrete - Alkaline sensitive surface treatments

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alkali sensitive, alkali resistance, VOC, test methods

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**Introduction**

Building materials deteriorate over time. Deterioration is inevitable, but is accelerated under some conditions and can be retarded if care and knowledge about the behavior of materials are put into the process of developing and using materials in building structures. Important environmental conditions that add to the stress that is put on a material include temperature, moisture, aggressive substances, ultraviolet radiation and microbes.

The ageing processes of the materials used in a building are not only of importance to those who are concerned with service-life and maintenance intervals, but may also be of immediate relevance to the health and feeling of comfort that its inhabitants experiences. Particularly moisture contributes to health related problems, and several studies have shown that dampness in buildings is a risk factor for health effects such as cough, wheeze and asthma (Bornehag et al. 2004, Bornehag et al. 2005). Some people experience inexplicable feelings of tiredness or headaches in some buildings (Sjögren 2001). These buildings are said to be sick. The reasons for specific health problems as well as more general feelings of discomfort are unknown, but probably related to the quality of the indoor air.

The quality of indoor air is highly dependent on the type and amount of emissions of volatile organic and inorganic compounds from building materials. As the number of materials used in buildings increase and the composition of products become ever more complex concerns for health effects and environmental effects have increased. Legislative measures are being introduced to lower emissions and standards for measuring and evaluating building materials have been developed. Emissions from building materials are commonly divided in two groups: primary and secondary emissions. The primary emissions are due to solvents and other compounds that are part of the production process. These will usually evaporate over the course of days, weeks or a few months. Primary emissions can be controlled by the manufacturer and are amendable to correction at the manufacturing stage unless they reach acceptable levels within a specified time after installation. The secondary emissions are caused by reaction between the finished product and its immediate environment. In moist environments a number of important building material types may degrade and cause emission of harmful or unpleasant volatiles. Secondary emissions are harder to predict as predictions require knowledge about the behavior of a material in sometimes unpredictable and less than ideal environments.

Concrete is a highly alkaline building material. The alkalinity, in combination with moisture, will sometimes cause degradation followed by secondary emissions from materials in contact with the concrete. This report investigates:

- deterioration of materials in contact with concrete and the emission products
- methods for protecting alkali-sensitive materials from alkaline moisture
- test methods for evaluating a material's resistance against alkaline moisture

Oslo, 2007

Thale Sofie Wester Plesser
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Moisture in concrete

The sources of moisture in concrete are construction errors, accidents, moisture remaining after pouring the concrete or moisture being introduced as a part of some normal step in the building process (e.g. the use of a water based surface treatment). Concrete contains calcium hydroxide and alkali oxides and as a result the pore water can reach a pH of up to 13.5. Carbonation of concrete leads to a lowering of the pH to a level between 8 and 9. The extent of the damage to an alkali sensitive material depends on the material composition, the pH in the water, contact time and temperature as well as the microstructure and the relative humidity in the concrete.

Critical relative humidity

Concrete is a porous material with a continuous system of pores and capillaries (Geving and Thue 2002). Porosity, i.e. the ratio between pore volume and total volume, and the sizes of the pores depend on the water to cement ratio (w/c). Both porosity as well as the pore sizes increases with increasing water to cement ratio.

The water in concrete is either chemically or physically bonded. Chemically bonded water is sufficiently inert that it will not easily be released and does not contribute to the humidity levels. Physically bonded water normally is of one of two different types:

- Adsorbed on the outer surface or pore surface of the material
- Condensed in the material capillaries.

If the material is in equilibrium with the surrounding air at a given temperature and air humidity all pores up to a certain size will be filled with water. At low relative humidity water is condensed only in the submicro pores. At higher relative humidity larger pores are filled as well. Dissolved salts in the pore water causes water to condense at a lower relative humidity.

Since water condenses in small concrete pores at low relative humidity and only condenses in larger pores at higher relative humidity, it follows that concretes dominated by small pores or with small pores at the concrete surface can cause problems at lower relative humidity than concretes with larger pores. The critical relative humidity is the level at which a material in contact with concrete will sustain damages due to alkaline moisture. The critical relative humidity is usually thought to be 85-95 % RH, although materials are believed to sustain damages at lower relative humidities if the concrete is dominated by small pores (Geving and Thue 2002). In response to the realization that concrete floors with smaller pore size (lower w/c ratio) create a more aggressive environment, the Swedish Adhesive and Sealants Association has recommended that alkali resistant adhesives are used when bonding floor coverings to concretes with low w/c ratios (w/c = 0.38-0.50), whereas normal adhesives may be used at concretes with higher w/c-ratios (Swedish Adhesive and Sealants Association 2006).

Alkaline hydrolysis of building materials

The reaction between a material and the hydroxide ion in an aqueous solution is called alkaline or basic hydrolysis. Alkaline hydrolysis is responsible for the degradation of materials due to contact with moisture in concrete.

Adhesives containing ester-based linkages are particularly sensitive to hydrolysis, while urethane-, amide- and urea-linkages also show some hydrolytic instability (Harper and Petrie, 2003, p. 269). Ester linkages can be present in some polyurethanes as well as some epoxies. Alkaline hydrolysis of an ester-linkage leads to the formation of an alcohol and the salt of a carboxylic acid (Roberts 1981, p. 821), see figure 1. This process is called saponification. Acrylate-, ethylene vinyl acetate
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(EVA)- or polyvinyl acetate (PVAc)-based adhesives all containing ester linkages that are susceptible to alkaline hydrolysis as shown in figure 1. However, only the hydrolysis of acrylates will lead to the formation of volatile alcohols, typically 2-ethyl hexanol or 1-butanol. In the case of EVA and PVAc a non-volatile alcohol and the salt of an acetic acid will be formed. In this case there may still be loss of adhesion (Blom et al. 1996).

Alkyd paints are also known to react with alkaline moisture, e.g. in contact with concrete (Weldon 2001). This process is called saponification and involves decomposition of the ester-linkage to form alcohol and the salt of a carboxylic acid. The reaction is essentially the same as when adhesives containing an ester-linkage come in contact with moist concrete. The alcohol is non-volatile, but the paint will debond and loose its protective properties.

Paints based on acrylates and styrene-acrylate copolymers generally have good alkali resistance, but should not be used on a new concrete. It is recommended that the concrete is allowed to cure for at least one month prior to applications, otherwise the coating may fail to perform as intended (Stadelmann 2002).

In the Nordic countries much attention has been given to flooring systems (adhesive and covering) in contact with moist concrete, see Table 1. Adhesives for floor are usually used on large areas, and the damages due to alkaline hydrolysis can be extensive. These studies have concentrated on flooring products commonly used on concrete substrates in Norway and Sweden. Typical reaction products are 2-ethyl hexanol, 1-butanol, heptanol, C9- and C10-alcohols. Persson (2003) also found 2-(2-butoxyethoxy)ethanol and 2-phenoxyethanol. 2-(2-Butoxyethoxy)ethanol and 2-phenoxyethanol are most likely not degradation products but rather components in the adhesive that are released when the adhesive decomposes.

Volatile degradation products are not only emitted to the air. A substantial amount of volatile degradation products is also absorbed into the pore system of the concrete and can be released long after the damaged adhesive has been removed (Sjöberg 2001b).

Björk (2003) and Sjöberg (2001a) found that a number of the materials used in a flooring system were stable towards alkaline hydrolysis at pH 11, but not at pH 13. One would therefore expect that materials in contact with old concrete that has a lower surface pH due to carbonization will not degrade because of alkaline hydrolysis.

Outside of the Nordic countries the effect of moist concrete on the degradation of building materials and the possible effect on indoor air quality has received little attention in the scientific literature.

![Figure 1. Alkaline hydrolysis of an ester linkage in an acrylic polymer leading to the formation of an alcohol. Typically the alcohol will be 2-ethyl hexanol or 1-butanol.](image-url)
Table 1. Components that are part of flooring systems. Resistance to alkali moisture.

<table>
<thead>
<tr>
<th>Product</th>
<th>Reaction with alkaline moisture</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Components in flooring adhesives:</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acrylate based flooring adhesives</td>
<td>Hydrolysis of the acrylate polymer in the adhesive leads to formation of alcohols and loss of adhesion. Volatile alcohols can have a detrimental effect on the indoor climate.</td>
<td>Blom et al. 1996, Alexanderson 2004</td>
</tr>
<tr>
<td>Acrylate polymer dispersion - component in flooring adhesives</td>
<td>Hydrolysis of the ester group in some acrylate polymers can lead to formation of C₈ hydrocarbons. The amount of hydrocarbon increases with pH. Some acrylates are stable even at pH 13.</td>
<td>Björk et al. 2003</td>
</tr>
<tr>
<td>Acrylate copolymer and acrylate-acetate copolymer based flooring adhesives</td>
<td>2-Ethyl hexanol and 1-butanol is formed when the adhesive comes in contact with synthetic concrete pore solution of pH 13. At pH 11 the adhesives are stable. The stability of different adhesives shows large variations. The flooring materials were not affected.</td>
<td>Sjöberg 2001a</td>
</tr>
<tr>
<td>Acrylate-based adhesive</td>
<td>2-Ethyl hexanol, 2-(2-butoxyethoxy)ethanol and phenoxyethanol.</td>
<td>Persson 2003</td>
</tr>
<tr>
<td><strong>Components in PVC flooring:</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PVC floor covering</td>
<td>Hydrolysis of the plasticizer (normally phthalates) leads to the formation of alcohols such as 2-ethyl hexanol, nonanol isomers and decanol isomers.</td>
<td>Blom et al. 1996, Alexanderson 2004, Persson 2006</td>
</tr>
<tr>
<td>DOP - dioctyl phthalate (plasticizer in PVC flooring)</td>
<td>Hydrolysis of the ester group leads to the formation of 2-ethyl hexanol. Some 2-ethyl hexanol can be observed at pH 11 and 70 % RH.</td>
<td>Björk et al. 2003</td>
</tr>
<tr>
<td>Jayflex DINA - diiso nonyl adiapate (plasticizer in PVC flooring)</td>
<td>Hydrolysis of the ester group leads to the formation of branched C₉ alcohols. Some alcohols can be observed at pH 11 and 70 % RH.</td>
<td>Björk et al. 2003</td>
</tr>
<tr>
<td>Jayflex DINP - diiso nonyl phthalate (plasticizer in PVC flooring)</td>
<td>Hydrolysis of the ester group leads to the formation of branched C₉ alcohols. Some alcohols can be observed at pH 11 and 70 % RH.</td>
<td>Björk et al. 2003</td>
</tr>
<tr>
<td>Jayflex 77 - diiso heptyl phthalate</td>
<td>Hydrolysis of the ester group leads to the formation of heptanol. Heptanol is observed at pH 11 and 70 % RH.</td>
<td>Björk et al. 2003</td>
</tr>
<tr>
<td>DIDP - diisodecyl phthalate (plasticizer in PVC flooring)</td>
<td>Hydrolysis of the ester group leads to the formation of C₁₀ alcohols. Some alcohols are observed at pH 11 and 70 % RH.</td>
<td>Björk et al. 2003</td>
</tr>
</tbody>
</table>
**Adhesives as a source of moisture**

Water based dispersions (latexes) based on acrylate, vinyl acetate or ethylene vinyl acetate copolymers typically contain 25-30 % water by weight, meaning that 55-60 g of water will be added to the substrate as part of the adhesive per square meter, see table 2. Water from the adhesive that cannot evaporate to the air will be absorbed into the concrete and cause the relative humidity of the concrete to increase. Experiments performed by Sjöberg (2001a) show that the relative humidity increases sharply at the concrete surface shortly after bonding the flooring material. The relative humidity in the top 3-5 mm of the concrete in figure 3 rose well into the range that is considered to be critical for alkaline hydrolysis (85-95 % RH). In an unlucky case the relative humidity under a tight flooring material that prevents evaporation (e.g. PVC) may rise above the critical relative humidity for alkaline hydrolysis of a particular adhesive or floor covering.

There are two principally different methods for bonding: wet- and dry-bonding. In the first case the adhesive is applied to the substrate and the flooring is applied directly onto the wet adhesive. Dry-bonding means that the adhesive is applied to the substrate and allowed to dry until it feels dry when touched lightly. The latter method allows some moisture to evaporate before the flooring is put in place. Dry-bonding is used for non-absorbing substrates with a tight covering. Examinations performed by SINTEF Building and Infrastructure (Blom 2007) of damaged floors indicate that erroneous use of the wet-bonding technique on concrete with decreased ability to absorb the water from the adhesive is suspected to cause alkaline hydrolysis of the adhesive and subsequent debonding of the floor covering.

Table 2. Adhesives that are used in Norway and Sweden for gluing PVC, linoleum and other floor coverings. The data is according to the specifications of the manufacturers.

<table>
<thead>
<tr>
<th>Name</th>
<th>Binder¹⁾</th>
<th>Dry solids content [%]</th>
<th>Density [kg/m³]</th>
<th>Surface covered [m²/l]</th>
<th>Moisture from the adhesive²⁾ [g/m²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cascoproff Extra 3444</td>
<td>Acrylate/EVA copolymer latex</td>
<td>72</td>
<td>1260</td>
<td>4-6</td>
<td>59</td>
</tr>
<tr>
<td>Cascoproff 3448</td>
<td>Acrylate copolymer latex</td>
<td>70</td>
<td>1300</td>
<td>3-6</td>
<td>65</td>
</tr>
<tr>
<td>Cascolin 3449</td>
<td>Acrylate/EVA copolymer latex</td>
<td>74</td>
<td>1400</td>
<td>3-5</td>
<td>73</td>
</tr>
<tr>
<td>MultiTac MT Golv och vägglim</td>
<td>Latex</td>
<td>71</td>
<td>1270</td>
<td>3-6</td>
<td>61</td>
</tr>
<tr>
<td>Attack golv och vägglim</td>
<td>Latex</td>
<td>71</td>
<td>1270</td>
<td>4-6</td>
<td>61</td>
</tr>
<tr>
<td>Linotack linoleumlim</td>
<td>Vinyl acetate copolymer</td>
<td>75</td>
<td>1380</td>
<td>3-6</td>
<td>58</td>
</tr>
</tbody>
</table>

¹⁾ EVA - Ethylene vinyl acetate polymers.
²⁾ Calculated from the maximum consumption.

**Emissions and indoor air quality**

Some individuals suffer non-specific complaints such as headache, nausea, cough or skin problems after exposure to certain indoor environments. This is known as "Sick-building syndrome". It is difficult to link the complaints to specific causes, but both primary and secondary emissions are suspected to contribute.
In a study of indoor air quality and asthma symptoms in daytime personnel at hospitals, 2-ethyl hexanol has been linked to asthma symptoms in adults (Norbäck et al. 2000). 2-Ethyl hexanol was only observed in the buildings with signs of dampness in the floor construction. The floors had PVC floor coverings and it was assumed that the 2-ethyl hexanol originated from alkaline degradation of the flooring materials. The concentration of 2-ethyl hexanol was found to be on the average 12 μg/m$^3$ (min-max concentrations: 2-32 μg/m$^3$).

A Japanese study of a particularly sensitive individual linked respiratory symptoms with 2-ethyl hexanol in the work environment (Kondo et al. 2007). The concentration of 2-ethyl hexanol was found to be 13-44 μg/m$^3$.

2-(2-Butoxyethoxy)ethanol is commonly used in detergents and paints. The acute toxicity of 2-(2-butoxyethoxy)ethanol due to a single instance of inhalation is low but the liquid substance is classified as an eye irritant, and may cause respiratory irritation at high vapour concentrations (European Union Risk Assessment Report 2000).
Protecting alkali sensitive materials against alkaline moisture

Damage due to alkaline moisture requires the presence of free water containing hydroxide (OH\(^-\)). When the conditions are sufficiently dry and/or the sufficiently free of alkalis no damages due to alkali moisture will occur. The methods of protection against alkaline moisture can be divided in two groups: products that prevent moisture from reaching the alkali sensitive material and products that prevent alkalis from reaching the sensitive material. Moisture barriers reduce the water transport to the surface and alkali barriers reduce the alkali transport through some other mechanism than by reducing the moisture transport. In practice there is an overlap between the groups and it is not always clear or known whether a product works as water barrier, alkali barrier or maybe both. Most likely most commercial barrier products currently available are primarily water barriers that prevent alkalis from being transported by hindering the transport of liquid water containing dissolved salts, rather than membranes or barriers that stop the alkalis through absorption, chemical reaction or other direct mechanism irrespective of water transport effects.

In the Nordic countries low-alkali self-leveling floor screeds and epoxies are commonly used as barriers against alkalis or moisture. Many floor screeds are known to effectively stop alkaline degradation of flooring adhesives due to the lower alkalinity of the screed (Alexanderson 2004). Floor screeds that have met the criteria for P-marking of floor screeds offered by SP Technical Research Institute of Sweden can be used as barriers.

A study on the effectiveness of five different barrier types in protecting a flooring adhesive against alkaline moisture revealed that epoxies are particularly effective. The study compared the following barrier types (Sjöberg 2001b):

1. Water barrier based on cement.
2. Thermoplastic polyethylene and acrylate latex.
3. Water glass.
4. Silone water repellent.
5. Two-component epoxies.

The barriers were applied on one concrete with w/c ratio = 0.39 and one with w/c ratio = 0.66. The alkaline degradation was monitored by measuring the emission of 1-butanol and 2-ethyl hexanol from the flooring surface of concrete specimens with adhesive and flooring material applied on one face. The amount of 1-butanol absorbed into the concrete was also measured. The study revealed that epoxies were by far the most effective irrespective of concrete type. Thermoplastic latex, waterglass and the silane also had some ability to reduce the alkaline degradation of the adhesive compared to the unprotected adhesive, while degradation increased in the presence of the cement-based water barrier, see figure 2.

Sjögren also investigated the ability of different barrier types to seal the concrete such that 1-butanol and 2-ethyl hexanol absorbed into the concrete could not escape. This is a relevant characteristic when decomposition products have been absorbed into the floor after water damage to the floor has occurred, as it can prove difficult to remove the decomposition products from the concrete. Alternatively to removing the decomposition products, a seal can be applied that locks the compounds into the concrete (emission barrier). Once more the epoxies proved particularly effective, see figure 3.
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Figure 2. The effectiveness of barriers toward preventing alkaline degradation. In the specimen without barrier the absorption/emission is set to 100%. The data for the specimens with barrier is relative to the specimen without barrier. The butanol absorption was measured at 5 mm depth in the specimens. The top graph shows data for concrete with w/c ratio = 0.66, while the bottom graph shows data for concrete with w/c ratio 0.39. The graphs are based on maximum emission/absorption data taken from Sjögren (2001b).

Figure 3. Vapor permeability of 1-butanol and 2-ethyl hexanol through concrete with a barrier placed on the top face of the concrete specimen. 1-Butanol and 2-ethyl hexanol were transported through the concrete substrate from the bottom of the test specimen. The specimen without barrier the absorption/emission is set to 100%. The data for the specimens with barrier is given relative to the specimen without barrier. a) W/c ratio = 0.66. b) W/c ratio = 0.39. The graphs are based on data taken from Sjögren (2001b).
Testing alkali resistance

Materials that are supposed to be in direct contact with concrete should be tested with regard to alkali resistance. The test procedure can be divided into three steps:

1. Choosing a test method that sufficiently simulates real conditions of use.
2. Evaluating the effect of the test on the test material.

In this section choosing a test method and evaluating the effect of the test on the test material will be discussed. Estimating life time will not be a topic in this report, however it should be noted that estimating life times is a difficult task and that the results from accelerated test are either not useful for this purpose or easily subject to incorrect interpretation.

Test methods - an overview

A number of methods designed for testing chemical resistance to any chemical can be used for testing alkali resistance as well, see table 3. Most test methods are of the same general format: the test material is contacted with a test solution at a given temperature for a specific duration of time. The temperature may be increased above the normal temperature of use for the material, thus obtaining accelerated testing. It is often necessary to test at extreme temperatures as the deterioration processes would otherwise be too slow for practical purposes. Most well formulated products are stable for a long period of time, sometimes many years, in their intended environments at room temperature.

The test solution is usually a dilute solution of sodium hydroxide (NaOH) in water. To simulate the concrete pore solution the pH should be adjusted to lie in the range between 13 and 14. Sometimes a synthetic pore water solution is used, see table 4. This simplification of the chemical environment that a material will experience in real use can lead to erroneous results. Experiments with polyethene show that deterioration of the polyethene in contact with fresh concrete was much faster than deterioration in contact with a synthetic pore solution, see table 5 (Jakubowicz and Klaesson 1997). A reason for this may be that in concrete the ions in the pore solution are in equilibrium with the cement phase so that when ions in the solution are depleted they are replaced by dissolution of the appropriate compounds from the cement phase. The experiments also showed that the age of the concrete used in the experiments were of importance. In contact with old concrete (age unspecified) the degradation process is slowed down. The results with old concrete may be due to carbonation of the concrete resulting in lowering of the surface pH.
Table 3. Methods for testing chemical resistance and alkali resistance.

<table>
<thead>
<tr>
<th>Method</th>
<th>Title</th>
<th>Short description</th>
</tr>
</thead>
<tbody>
<tr>
<td>EN ISO 2812-1</td>
<td>Paints and varnishes - Determination of resistance to liquids - Part 1: General methods</td>
<td>The method is intended for testing of paints and varnishes. The product is put onto a substrate and is allowed to dry. The test piece is immersed in the test liquid at room temperature. The immersion time is not specified. The paint is evaluated visually.</td>
</tr>
<tr>
<td>EN 13529</td>
<td>Products and systems for the protection and repair of concrete structures - Test methods - Resistance to severe chemical attack.</td>
<td>The product is spread over a concrete substrate. The test is performed at room temperature for up to 90 days. The surface treatment is evaluated visually with regard to degree of cracking, blistering, flaking, changes in color and gloss, adhesion and hardness.</td>
</tr>
<tr>
<td>NT Poly 161</td>
<td>Plastics - accelerated ageing in alkaline environment</td>
<td>The method is intended for testing of polyethylene film in contact with moist concrete. The film is placed between two concrete prisms. The lower prism is placed in water. The test piece is kept at 90 ºC for 24 weeks. The film is evaluated visually, by tensile strength testing or thermal analysis.</td>
</tr>
<tr>
<td>Golvbranschen - Industry protocol</td>
<td>Measuring the emission characteristics of composite floor structures</td>
<td>The method is intended to test the ability of floor screed to prevent decomposition of the adhesive due to alkaline moisture. A floor segment is built using moist concrete, floor screed, acrylate based adhesive and floor covering. After 6 months emissions of 2-ethyl hexanol and 1 butanol are measured.</td>
</tr>
<tr>
<td>Golvbranschen</td>
<td>GBR:metod 2005-1. Tätskikt av plastmattor. Golv/vägg. Alkalibeständighet.</td>
<td>The method is intended for testing of plastic films. The test material is kept in a 0.1 M NaOH-solution at 80 ºC for 28 days. Tensile test are performed on the test material at the end of the test period.</td>
</tr>
<tr>
<td>EN 13578</td>
<td>Products and systems for the protection and repair of concrete structure. Test method. Compatibility on wet concrete.</td>
<td>A water saturated concrete slab is treated with a coating on one face. The uncoated face is exposed to water. The treatment is evaluated visually (peeling, blistering and discoloration) and by use of a pull-off test. The results are compared to the performance of a coating on a dry concrete substrate. The test is performed over a period of 56 days at the minimum permitted application temperature.</td>
</tr>
</tbody>
</table>

Table 4. Examples of synthetic pore solutions.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>pH (theoretical)</td>
<td>13.8</td>
<td>13.05</td>
</tr>
<tr>
<td>NaOH (M)</td>
<td>0.1</td>
<td>0.028</td>
</tr>
<tr>
<td>KOH (M)</td>
<td>0.5</td>
<td>0.083</td>
</tr>
<tr>
<td>CaSO4 (M)</td>
<td>0.02</td>
<td>4 x 10^-6</td>
</tr>
<tr>
<td>Ca(OH)2 (M)</td>
<td>0.0135</td>
<td>-</td>
</tr>
</tbody>
</table>
Table 5. Time to 50 % retention of elongation at break. Measurements on polyethylene films aged in different environments (concrete and simulated concrete) at 90 ºC. The tensile strength was measured according to SS 24 10 20. Data from Jakubowicz and Klaesson (1997).

<table>
<thead>
<tr>
<th>Test environment</th>
<th>Time to 50 % retention of elongation, t_{50} [days]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh, moist concrete, C25</td>
<td>18</td>
</tr>
<tr>
<td>Deionised water</td>
<td>21</td>
</tr>
<tr>
<td>Saturated CaO-solution</td>
<td>28</td>
</tr>
<tr>
<td>5 weight % NaOH-solution</td>
<td>32</td>
</tr>
<tr>
<td>Synthetic cement pore solution(^{1)})</td>
<td>41</td>
</tr>
<tr>
<td>0.5 weight % FeSO(_4) solution</td>
<td>&gt; 100</td>
</tr>
</tbody>
</table>

\(^{1)}\) The composition is given in table 4.

NT Poly 161 (Nordtest method), see table 3, was developed specifically to test polymers in a moist concrete environment. The test is performed at 90 ºC for 24 weeks which is supposed to be the equivalent of 50 years at 22 ºC. This type of extrapolation, based on the Arrhenius equation, from behavior at an elevated temperature (accelerated environment) to ambient temperatures, is very uncertain. The extrapolation supposes that the deterioration mechanism is the same over the entire temperature interval and that the activation energy is not a function of temperature. Comparison of results from accelerated tests with behavior in real environments has shown that materials often are predicted to fail more quickly than they do in a natural environment (Croll and Hinderliter, 2007).

The Swedish organization GBR specifies a method for testing the ability of floor screeds to prevent alkaline decomposition of flooring materials used on a moist concrete substrate. The decomposition reaction is monitored by measuring the emissions from the test specimen. A variation of this method has been used in several studies to evaluate the performance of flooring materials in contact with moist concrete (Sjögren 2001, Persson 2003 and Alexanderson 2004). The decomposition process is monitored through the emissions of volatile decomposition products.

EN 13578, see table 3, specifies the testing of coating products on wet concrete. The coat is applied to a wet concrete substrate, and then kept in contact with moisture for the duration of the test period. As many coating types will perform well under wet conditions if they are applied to a dry substrate that subsequently comes in contact with moisture, while other coatings are unaffected by the moisture level in the substrate at the time of application (Liu and Vipulanandan 2005, Vipulandan and Liu 2005), it is important to differentiate between performance when the concrete is wet at the time of application and performance when the concrete is dry at the time of application. EN 13578 may be a useful method for testing coatings that are to be applied to old concrete structures that have become saturated with water or green structures, but other methods should be used to evaluate the performance on dry concrete.

Products that undergo simultaneous mechanical and chemical stress will often be more severely affected than the sum of the individual factor. This must be taken into consideration when choosing a test method. None of the methods in table 3 are suitable for this type of scenario.
Evaluating the alkali resistance

Choice of method for evaluating the alkali resistance of a material after exposure to an alkaline environment must to some degree be material dependent. Knowledge about the deterioration mechanism is essential to the choice. Visual inspection, if necessary aided by a microscope or magnifying glass, will often give an indication of how the material is doing. The inspection may reveal cracks, color and gloss changes or blistering. If the test material is attached to a substrate (e.g. paints, adhesives or membranes) adhesion tests may be used as an evaluation method. When the test material is a film, such as plastic foil, tensile strength measurements are normally performed. Decomposition can also be successfully monitored using chemical analysis methods such as infrared spectroscopy and thermogravimetric analysis.

Care must be taken in choosing the evaluation method because different testing methods, when used on the same material, can give different results. An example to this point taken from Jakubowicz (2004), shows that the effect of ageing on the material properties of a low density polyethylene film is observed at an earlier point when the elongation at break is measured than when the carbonyl index is measured by means of infrared spectroscopy. The acceptance and failure criteria depend on correctly determining the point of failure to perform as necessary in a given application or environment, and on relating this point to the measurement value using a particular test method. Although the criteria are of crucial importance to the successful evaluation of a material they are rarely established as part of the test method, but are generally left to the judgment of the manufacturer or others.

As mentioned earlier, the Swedes have developed a method for monitoring decomposition of flooring materials in alkaline environments by measuring emissions, see table 3. The GBR method allows the decomposition to be monitored non-destructively, but much care must be put into the experimental setup if one wants to obtain good results that can be interpreted correctly. When this method is being employed it is important to bear in mind that the amount of emissions from the surface depends on a number of factors that may contribute to make the amount of decomposition products to appear to be smaller or larger irrespective of the total amount of decomposition products:

- The concrete may absorb a substantial amount of the decomposition products, see figure 2. Different concrete qualities may absorb volatiles more or less readily.
- Decomposition products with low or no volatility will go undetected.
- A tight floor covering may prevent the volatiles from being released or at least reduce the emissions from the surface substantially. Floorings with different permeability towards a compound may produce different emission levels although the amount of a compound that is available for emission is the same.

When the GBR method is being employed to compare the decomposition patterns of different flooring systems it is essential to monitor both emissions from the surface and absorption of decomposition products into the concrete and perform material balance calculations, otherwise interpretation of the results become very difficult. The study performed by Persson (2003 and 2006) demonstrates the importance of measuring the amount of volatiles absorbed into the concrete. In the study it was found that the surface emissions of organic volatiles from floor samples based on a concrete substrate with low w/c ratio and low relative humidity were higher than from floor samples based on concrete substrates with high w/c and high relative humidity, see figure 4. This result is seemed somewhat surprising and two explanations were given:

1. In a low w/c concrete the moisture condenses at a lower relative humidity than in a high w/c concrete because the pores are smaller, i.e. al low w/c concrete provides a more aggressive environment to materials that are alkali sensitive because liquid water is available at lower relative humidity.
2. In a high w/c concrete more volatiles are absorbed into the concrete pores giving an appearance of a lower decomposition rate although the rate may in reality have been higher, due to a higher moisture level.

Additionally, it is difficult to quantify the emission products. The reproducibility of the GBR test is low, a fact that is pointed out in the foreword to the procedure description (Golvbranschen 2004), and random very high or very low measurement results are common.

![Graph](image-url)

Figure 4. Emissions of total volatile organic compounds (TVOC), 2-ethyl hexanol (2-EtHx) and 2-(2-butoxyethoxy) ethanol (2-(2-BuEt)Et) from the surface of floor specimens. No or very little butanol was detected. Relative humidity is measured at 30 mm below the surface. The concrete was poured as cylinders with dimensions height x diameter = 250 mm x 250 mm. After one month the top surface was covered with a PVC flooring using a non-alkali resistant acrylate-based adhesive. Similar results were obtained with other flooring types. The graph is based on data from Persson (2003).
Conclusion

Adhesives, floor coverings and paints in contact with moist concrete may undergo alkaline hydrolysis. Some decomposition products, such as 2-ethyl hexanol and butanol are volatile and are emitted to the air. The occurrence of 2-ethyl hexanol in indoor air is linked to respiratory problems. Substances that are part of an adhesive formulation, such as 2-(2-butoxyethoxy)ethanol and phenoxyethanol, may also be released to the air when an adhesive decomposes. Substances that are released upon decomposition are also absorbed into the concrete and can be released to the air at later point of time.

The corrosive potential of a specific concrete, i.e. how damaging it is to a material in contact with the concrete, depends on the relative humidity and the pore size distribution in the concrete. It is thought that concretes with smaller diameter capillaries are more damaging at lower relative humidities because the moisture condenses at lower relative humidity in small capillaries than in large capillaries. More work is needed to establish the relationship between moisture level, capillary size and corrosive potential of a concrete.

Several methods exist for testing alkali resistance. Choosing a test method requires knowledge about the conditions under which the product will be used, possible deterioration mechanisms in this environment as well as the limitations of the test method. Accelerated methods often give unrealistic results, underestimating the durability of a material, while non-accelerated methods are too slow. The GBR method is a useful real-time method for testing the alkali resistance of floor systems that can be extended to other parts of a building. Care must, however, be taken in the planning process.
Moisture in Concrete - Alkaline sensitive surface treatments. Literature study

References


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