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Technical report

Coatings for Concrete Protection against Aggressive Environments

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Abstract

The effect of polymeric coatings on concrete protection against chemically aggressive environments was evaluated. Two polymers – acrylic and epoxy – were applied on different concretes. The protection was measured by tests related with chemical resistance. The chlorides penetration, sulphates, acids and bases attack tests were used. Surface treatments act as a barrier between the environment and the concrete. This work intends to contribute to a better understanding of the performance of coated concrete in chemically aggressive environments, by presenting results of ion diffusion and resistance to aggressive solutions of several coatings used to protect concrete. The performance of the used coated concretes against chemically aggressive environments was generally better than the performance of the unprotected concretes. The results indicate that the overall performance of the used epoxy resin was better than that of other used coatings.

1. Introduction

Concrete can be a highly durable construction material as long as care and quality control are enforced at all stages of the design, production and construction processes. However, experience has demonstrated that its potential long-term durability is not always achieved; leading to early failure of reinforced concrete structures (Rodrigues et al. 2000). It should be recognized that concrete is intrinsically a porous material, despite the improvements on its formulation and quality control to the best possible extent, it is not possible to prevent completely the ingress of potentially harmful agents. Micro-cracks and macro-pores will always exist on the concrete surface, providing a path for the transportation of aggressive ions into the interior of concrete (Swamy et al. 1998).

It is now accepted that the durability of the reinforced concrete depends mainly on the composition and properties of the concrete surface layer (Kreijger 1984 and RILEM 1995). This layer, sometimes with a thickness close to the cover of the reinforcement, is most of the times the only responsible for the corrosion protection of the reinforcement. Surface treatments act as a barrier between the environment and the concrete. They prevent or retard the entry of harmful substances such as chlorides, sulphates, etc. (Pfeifer and Scali 1981). Surface coatings with appropriate "barrier" characteristics can cut off the transportation path into concrete. The standard EN 1504-2 (CEN 2004), establishes as a minimum requirement for the coated concrete ingress that the capillary absorption and the water permeability coefficient should not exceed $0.1 \ kgm^{-2}h^{-0.5}$ and the CO_2 permeability should at least correspond to a S_D (thickness of air layer with equivalent diffusion) value of 50 m.

Swamy and Tanikawa (1990) evaluated the effect of concrete coatings to preserve concrete durability and concluded that the application of an impervious surface coating to concrete is a very attractive solution to protect new and existing concrete structures. However, with a wide range of coatings available in the market, it becomes extremely difficult to choose the right type of coating, since similar generic types are known to possess considerably different characteristics. The performance of the available generic types under different service conditions needs to be studied. There is also a need to develop performance criteria for evaluation of concrete coatings and guidelines for the selection of coatings appropriate for various exposure conditions (Almusallam et al. 2002).

A low porosity, permeability and concrete penetration to moisture and gases are the first lines to defence against several deterioration mechanisms. The durability of concrete depends largely on how hard or easy fluids (water, carbon dioxide, oxygen) in liquid or gas form can migrate through the concrete hardened mass. They can move through the concrete in different ways, but all transport depends primarily on the structure of the hydrated paste (Neville 1995). One of the possible ways to protect the concrete is using coatings that act as a barrier against the environment. However, for some chemical environments the low porosity could not be sufficient to stop the degradation. If the chemical agents like sulphates, some acids and bases finally penetrate into the concrete, the low porosity could contribute to increase the degradation. The problem appears when the mechanism of degradation includes expansive reactions.

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2. Mechanisms of degradation

Reinforced concrete durability is determined by its resistance against various chemical and physical processes. For corrosion of steel to happen, it is necessary that carbonation damage or a presence of chlorides occur near the reinforcement rods. Also, the presence of moisture and the ingress of oxygen from the air are needed. The chlorides in the concrete either come from the components of the fresh mix (water, sand, aggregates, additives), or from external contamination agents (marine environment, de-icing salts). The chlorides, present in the mix, react in different ways (Kissel and Pourbaix 1996): a part ($\approx 5\%$) forms insoluble salts or is locked in a pore of the silicates that are insoluble in water; a part (≈ 85 till 90 %) forms soluble salts (Salt of Friedel: $C_3A.CaCl.10H_2O$) and a part ($\approx 5\%$) can be found in the concrete as free chlorides in solution, or easily soluble by adding water. The proportion of each status depends on the ratio of chloride ion to cement. Larger amount of chloride ion leads to smaller percentage of insoluble salts.

The chlorides coming from external contamination agents after the hardening of the concrete react very little with the solid phase of the concrete and can be found in the concrete as free chlorides (Schueremans et al. 2007). The water soluble salts (Salt of Friedel) act as a stock of free chlorides. In the presence of these salts, the water in the pores will enrich itself with chlorides until a final concentration is reached that equals the product of solubility. The chlorides that stand for the corrosion risk of the reinforced concrete structure are the chlorides in the pore water (the free chlorides) and a part of the soluble chlorides (Salt of Friedel). The risk of corrosion, with a given amount of chlorides in the pore water, will be much higher for a carbonated concrete structure.

The sulphates can appear into the concrete by two origins: inside or outside sources (Skalny et al. 2002). The Portland cement has calcium sulphate as constituent. The water present in concrete could also have sulphates. Regarding outside sources of sulphates, there are many possibilities: atmospheric pollution, soils, industrial solid wastes, water used by industries, water from rivers and lakes, and seawater. Sulphates carried into the inner sections of concrete by either ionic diffusion or capillary absorption of sulphate solutions may cause disruptive forces leading to cracking or scaling of the concrete.

The corrosive action of sulphates, however, depends on the presence of reactive hydration of products of the cement in sufficient concentration, e.g. Calcium hydroxide, CAH and CSH respectively. Penetrating sulphates may react with these phases, forming gypsum or ettringite. These expansive phases then may disrupt the structure of the concrete (RILEM 1995). Concrete of sufficiently low permeability is considered as non-susceptible to sulphate attack. Furthermore, sulphate-resisting cements with low concentrations of sulphate-reactive compounds may not undergo expansive phase transformations even upon access of sulphates, and the transport capacity of corresponding concrete mixes may then not reflect its resistance to sulphate attack in a correct manner.

The concept of ensuring protection from sulphate attack by specifying a minimum cement content has no scientific basis (Neville 1995). For example with 350 kg/m³ of ordinary Portland cement it is possible to obtain concretes ranging in cylinder strength from 14 to 41 MPa depending on the water/cement ratio. The durability of these concretes will clearly vary enormously. The use of compressive strength for specifying purposes is convenient but strength only reflects the water/cement ratio, cement type and, eventually, mineral additions added; it is this that is relevant to density and permeability. However, specifying the water/cement ratio regardless the nature of the cement used is inadequate: reference to the influence of the various blended cements on sulphate resistance.

The major constituents of the hydrated cement paste matrix and eventually also the aggregates, e.g. calcareous materials, are dissolved by mineral as well as by organic acids, the latter usually being less aggressive than strong mineral acids. The aggressiveness of acids is judged according to their pH, and a very severe attack is usually expected for acids with pH < 4.5 (RILEM 1995). Chemical attack of concrete occurs by way of decomposition of the products of hydration and formation of new compounds which may be leached out and, if not soluble, may be disruptive in situ. The attacking compounds must be in solution. The most vulnerable cement hydrate is $Ca(OH)_2$ but C-S-H can also be attacked.

The attack progresses at a rate approximately proportional to the square root of time because the attacking substance has to travel through the residual layer of the low-solubility products of reaction which remain after $Ca(OH)_2$ has been dissolved. Thus it is not only pH but also the ability of aggressive ions to be transported that influence the progress of the attack (Neville 1995). Sulphuric acid is particularly aggressive because, in addition to the sulphate attack of aluminate phase, acid attack on $Ca(OH)_2$ and C-S-H takes place. Reduction in the cement content of the concrete is therefore beneficial (Fattuhi and Hugges 1988), provided, of course, that the density of the concrete is unimpaired.

3. Experimental program

3.1 Materials
To evaluate the influence of cement, two types were used: a Portland cement (CEM I 42.5R) and a pozzolanic one (CEM IV/A (V) 32.5R), made with 35% of fly ash, according to EN 197-1 (CEN 2000a). **Table 1** shows the oxide composition of the cements. **Table 2** shows some physical characteristics of the selected cements. Crushed granite with a density of 2566 kg/m³, water absorption of 2.1%, fineness modulus of 5.89 and

a maximum size of 9.53 mm was used as coarse aggregate, while crushed sand with a density of 2477 kg/m³, water absorption of 1.36 %, fineness modulus of 3.16 and a maximum size of 4.76 mm was used as fine aggregate in the preparation of concrete specimens.

Three types of concretes were used. The composition of the concretes is presented in **Table 3**. The concretes I-A and IV had a water-cement ratio of 0.60 and a cement content of 320 kg/m³. The concrete I-B was made with CEM I 42.5R, water-cement ratio of 0.40 and a cement content of 500 kg/m³. The slump test achieved values of about 60 mm for concretes I-A and IV. The concrete I-B presented a slump of 180 mm. The average compressive strength of the concrete I-A attained 27.5 MPa at 28 days of age, the concrete I-B 55.6 MPa and the concrete IV 20.8 MPa. The experimental campaign was designed in order to test uncoated and coated concrete specimens.

Concrete coatings were selected to represent the following two generic types:
• Acrylic coatings (A);
• Epoxy resin coatings (E).

The two types were selected between the more used coatings for concrete protection. This study started with each generic type represented by two products from different producers. An aqueous based acrylic resin and a two component epoxy resin were selected because they are the most used coatings among the existing materials of each generic type. The selection that was made

has been presented in previous papers (Moreira *et al.* 2006 and Aguiar *et al.* 2007). **Table 4** shows the application properties of the selected concrete coatings.

The concrete specimens were moulded immediately after the production of the concrete. The coatings were applied when the concrete had 28 days. The application was made by brush following the recommendations of the supplier and after drying of the specimens under laboratory conditions for at least 7 days. Immediately before application of coatings the concrete surfaces were spurted with compressed air. The instructions of the supplier of the coatings were followed during all the process. The tests started 7 days after coatings were applied on concrete specimens.

3.2 Penetration of chlorides

The characterization of the resistance to chlorides penetration was made with tests based on a non-steady state, procedure known as CTH Rapid Method developed by Luping (1996). The depth of penetration of the chloride front is determined by a colorimetric method using sil-

Table 1 Oxide composition of the cements.

Chemical composition	CEM I 42.5R (%)	CEM IV/A (V) 32.5R (%)
SiO₂	19.64	39.24
Al₂O₃	4.34	4.80
Fe₂O₃	3.10	3.57
CaO	62.82	43.67
MgO	2.43	1.81
SO₃	3.33	2.81
Cl⁻	0.02	0.02
Loss on ignition	3.2	2.80
Insoluble residue	0.90	26.10

Table 2 Physical characteristics of the cements.

Physical characteristics	CEM I 42.5R	CEM IV/A (V) 32.5R
Density (kg/m³)	3110	2830
Blaine specific surface (m²/kg)	3873	4292
Fineness – 45 μm (%)	3.1	2.6
Water demand* (%)	28.6	31.4

* - water content of the standard consistency cement paste used for the determination of the initial and final setting times according to EN 196-3 (CEN 2005).

Table 3 Composition of the concretes.

Materials	Concrete I-A	Concrete I-B	Concrete IV
Cement CEM I (kg/m³)	320	500	-
Cement CEM IV (kg/m³)	-	-	320
Gravel 5 – 10 (kg/m³)	796	888	814
Sand 0 – 5 (kg/m³)	940	690	898
Water (kg/m³)	181	184	180
Superplasticizer (kg/m³)	-	5	-
w/c	0.60	0.40	0.60

Table 4 Description of the selected coatings.

Generic type	Acrylic	Epoxy
	acrylic resin aqueous based	two component epoxy resin
Consistency	dense liquid	dense liquid
Coverage rate (m²/dm³)	3.5	4.0
Density at 20 °C (kg/dm³)	1.40	1.30
Brookfield viscosity at 20 °C (mPa.s)	6000	1500
Surface drying time (min)	40	300
Interval between coats (h)	24	24

ver nitrate. The average penetration, measured with a precision of 0.5 mm, was considered to be the depth of penetration. The diffusion coefficient is obtained using the equation:

$$D = \frac{R \cdot T \cdot L}{z \cdot F \cdot U} \cdot \frac{x_d - \alpha \cdot \sqrt{x_d}}{t} \qquad (1)$$

with:

$$\alpha = 2 \cdot \sqrt{\frac{R \cdot T \cdot L}{z \cdot F \cdot U}} \cdot erf^{-1}\left(1 - \frac{2 \cdot c_d}{c_0}\right) \qquad (2)$$

where:

D: diffusion coefficient, m^2/s;
z: absolute value for ion valence, for chlorides, z = 1;
F: Faraday constant, F = 9.648 x 10^4 J/(V.mol);
U: absolute value of potential difference, V;
R: gas constant, R = 8.314 J/(K.mol);
T: solution temperature, K;
L: specimen thickness, m;
x_d: penetration depth, m;
t: test duration, seconds, t = t_{CTH} x 3600;
erf^{-1}: inverse of error function;
c_d: chloride concentration at which the colour changes, $c_d \approx 0.07$ N;
c_0: chloride concentration in the upstream cell, N.

After the production of the concretes, cylinders with Ø110x230 mm were moulded. These cylinders were cut in cylinders with Ø110x50 mm, in order to obtain the specimens used in the tests. The coatings were only applied in one face of the cylinders. **Figure 1** shows one test in development. After the time needed for the conclusion of the test, the specimens were taken-out from the equipment and broken in two halves by the mean of splitting tensile test. The solution of silver nitrate was then applied on the concrete surface and the colorimetric test was made in order to measure the depth of chlorides penetration (**Fig. 2**). Five specimens of each painting and compositions were tested.

Table 5 and **Fig. 3** present the results of penetration of chlorides in a non-steady regime. The use of coatings decreases the penetration of chlorides. In the case of the epoxy resin the diffusion coefficient was null. The penetration of chlorides depends on the porosity of the surface. The use of coatings contributes to decrease the porosity of the surface. The porosity depends on the solids volume, on the thickness and type of the product used. Epoxy resins with a solvent base have good resistance to chlorides penetration because they become strong and with low porosity (Almusallam et al. 2002).

The acrylic resin (ACR I-A and ACR I-B) decreases significantly the diffusion coefficient of the concretes I-A and I-B. The concrete I-B, with the highest cement content and with the lowest water-cement ratio presented without any painting a good resistance to chlorides penetration. The concrete ACR I-A presented only a diffusion coefficient 5.4% lower than the concrete I-B.

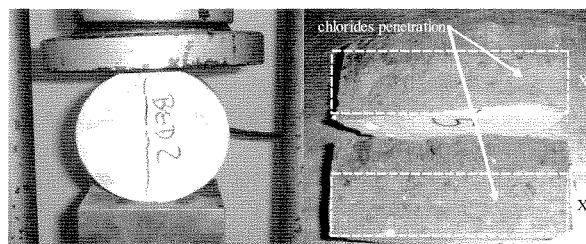Fig. 1 Test of penetration of chlorides.

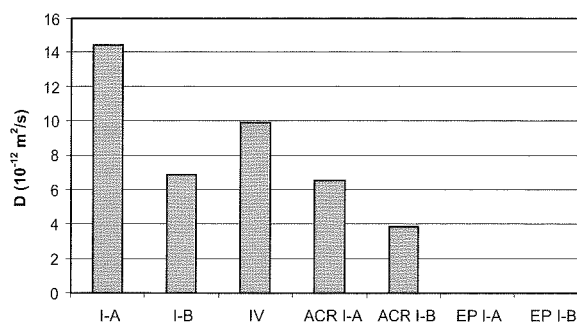Fig. 2 Colorimetric test of one specimen.

Fig. 3 Coefficients of diffusion in a non-steady regime for different concretes and different paintings.

The performance is similar. This shows that a good concrete could be a good solution to decrease significantly the penetration of chlorides.

The concrete IV presented a diffusion coefficient 31.8 % lower than the diffusion coefficient of concrete I-A, with the same water-cement ratio and the same cement content, 320 kg/m^3. This can only be explaining by the additions of the cement IV, like fly ash that can ameliorate the behaviour of the concrete in contact with chlorides. The study showed that even with low water-cement ratio and high cement content (concrete I-B), the use of coatings continue to decrease the diffusion coefficients of chlorides.

Table 5 Diffusion coefficients for different types of coatings and concretes.

References	Cement	Coating	C (kg/m^3)	w/c	D (10^{-12} m^2/s)
Concrete I-A	CEM I	Uncoated	320	0.60	14.40
Concrete I-B	CEM I	Uncoated	500	0.40	6.90
Concrete IV	CEM IV	Uncoated	320	0.60	9.87
Concrete ACR I-A	CEM I	Acrylic	320	0.60	6.53
Concrete ACR I-B	CEM I	Acrylic	500	0.40	3.86
Concrete EP I-A	CEM I	Epoxy	320	0.60	0.00
Concrete EP I-B	CEM I	Epoxy	500	0.40	0.00

3.3 Sulphates attack

The sulphates attack tests were made following C88 (ASTM 1999) with some adaptations. A sodium sulphate solution was used, prepared as recommended by C88 (ASTM 1999). The volume of the solution was at least five times the volume of the immersed specimens. The saturated solution of sodium sulphate covered the immersed specimens to a depth of at least 15 mm.

The procedure of the tests consists on cycles of immersion in the prepared solution of sodium sulphate for not less than 16 h and no more than 18 h. After the specimens were removed from the solution, permitted to drain for 15±5 min, and placed in the drying oven. According to C88 (ASTM 1999), the temperature of the oven shall have been brought previously to 110±5°C. The test of coated specimens did not recommend the use of a temperature such high. So, the temperature of the oven in our tests was changed to the maximum of 50±5°C. The specimens were dried at this temperature until constant weight. After, the specimens were immersed again in the prepared solution of sodium sulphate.

After the production of the concretes, cubes with 100x100x100 mm^3 were moulded. The sulphate attack tests were made with uncoated and coated specimens. A total of eight cycles was made. The evaluation of sulphates attack was made by the weight variation along the cycles. Each value presented is the average of the weight variation of five specimens.

Figure 4 presents the mass losses at the end of the sulphates attack tests. The used coats increased the protection against sulphate attack. For each type of concrete, the best performance was achieved with the epoxy coated concretes (EP I-A and EP I-B). However, the mass losses obtained for coated concrete I-A (ACR I-A and EP I-A) were similar. The uncoated concretes I-A and I-B presented similar mass losses. The use of higher cement content and lower water-cement ratio (concrete I-B compared with concrete I-A) did not contribute to increase the performance against sulphate attack. Less porosity is also less space to accommodate expansions caused by reactions between sulphates and cement constituents. High cement amount also increase the expansive reactions between sulphates and cement constituents. The concrete with cement IV did not solve the problem of sulphate attack.

3.4 Acids and bases attack

The resistance of coatings to severe chemical attack was measured by exposition of one face to the test liquid following EN 13529 (CEN 2003). The test apparatus consists of a metallic ring to take up the test piece and to clamp one or two bell-type caisson chambers (**Fig. 5**). The bell-type caisson chamber consists of a bell-type steel cup with an inner diameter > 100 mm (the tested area is a circle with a diameter of 100 mm inside of the glued tubes). It is connected to a compressed air vessel equipped with a pressure reducing valve via a compressed air hose. The pressure device is fixed on the coated concrete by a metal clamp. To ensure the tightness of the caisson chamber, a rubber cuff is placed between the coating and the caisson chamber.

The specimens used on the test had the dimensions of 750x400x50 mm made as mentioned in EN 1766 (CEN 2000b). The schema of the test consisted on putting eight PVC tubes Ø110x30 mm, on the specimen surface, when concrete is still fresh. Inside the tubes the solutions to be tested (acids and bases) were putted above the coated concrete, till a height of 10 mm. Two concretes were tested I-A and I-B, in order to verify how coatings can perform on different concretes. The test liquids used were the following: H$_2$SO$_4$ (pH:-0.19) and NH$_4$OH (pH:11.87), with a concentration of 20 % by volume. During the test the tubes were covered by a plastic film, in order to avoid the evaporation of test liquids.

The duration of the test was 28 days in accordance with EN 1504-2 (CEN, 2004). Once a week the degradation of the specimens was analysed comparing with no tested specimens and, if necessary, the level of the
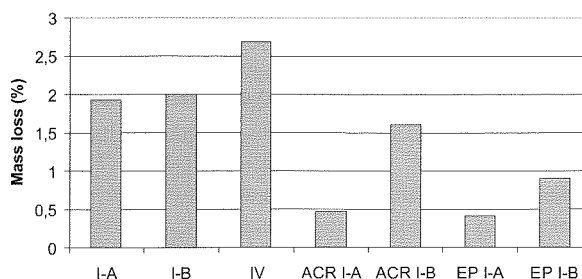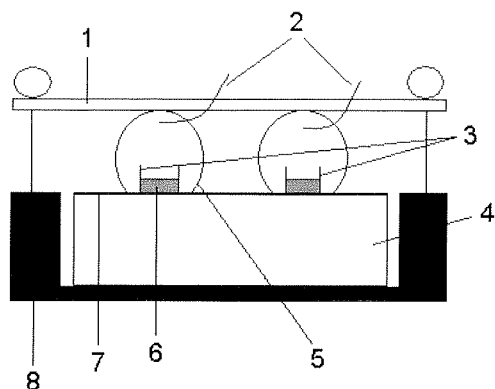
Fig. 4 Mass losses for different concretes and different coatings.

test liquid was reposed. The evaluation of the degradation of the coatings was made following an ISO standard (ISO 2003a) that establishes an uniform convention to the intensity of degradation, by the mean of degrees, in a numerical scale from 0 to 5: 0 means no deg-

1 : fixation base (used in pressure test);
2 : air compressed tube (pressure test);
3 : bonded tubes;
4 : concrete specimen;
5 : chamber (pressure test);
6 : test liquid;
7 : painting;
8 : metallic ring.

Fig. 5 Schema of acids and bases attack test.

radation and 5 means the most severe degradation. The results of blistering, cracking, flaking and chalking will be presented following ISO standards (ISO1990, ISO 2003b, ISO 2003c and ISO 2003d) that give methods for evaluation of the degrees of degradation of paintings (**Figs. 6 to 8**). At the end of the exposition to the test liquids, adhesion tests were made following D4541 (ASTM 2002) by pull-off method.

Tables 6 to 8 present the results of the visual inspections and of the adhesion tests made. In order to classify the degrees of degradation (**Tables 6 and 7**) the surface of the tested specimens were compared with the presented photos (**Figs. 6 to 8**). The ISO standard (ISO 2003a) was also taken into consideration. In general, the used acid (sulphuric acid) caused more degradation than the used base (ammonium hydroxide). For the acid, the degree of degradation 5 was caused in several parameters. For the base, 2 was the maximum degree of degradation caused, only for cracking (dimension) and for chalking (surface).

Considering the results of visual inspections (**Tables 6 and 7**), the concretes coated with acrylic resin (ACR I-A and ACR I-B) presented better behaviour than concretes coated with epoxy resin (EP I-A and EP I-B) for cracking and chalking parameters. The behaviour for flaking is similar and for blistering is favourable for the concretes coated with epoxy resin (EP I-A and EP I-B).

Considering the results of adhesion tests (**Table 8**), the loss in adhesion strength verified after exposition to the base was negligible, the maximum loss was only 5% for the concrete I-B coated with acrylic resin (ACR I-B).
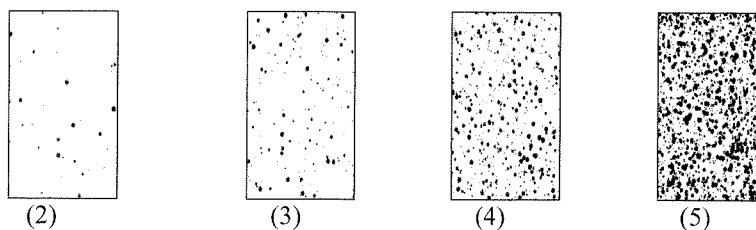
(2)　　　　(3)　　　　(4)　　　　(5)

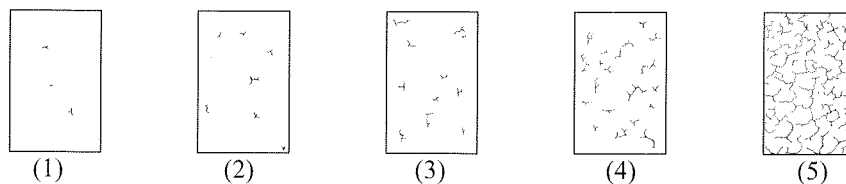Fig. 6 Photos to assessment of degree of blistering (ISO 2003b).

(1)　　　(2)　　　(3)　　　(4)　　　(5)

Fig. 7 Photos to assessment of degree of cracking, no preferential direction (ISO 2003c).
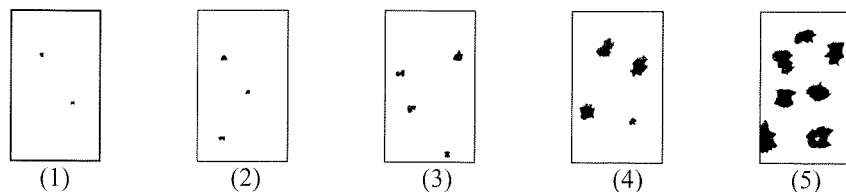
(1)　　　(2)　　　(3)　　　(4)　　　(5)

Fig. 8 Photos to assessment of degree of flaking, no preferential direction (ISO 2003d).

Table 6 Results of acid and base attack. Blistering and cracking.

References		Blistering		Cracking	
Test liquid	Concrete	Dimension (Degree)	Surface (Degree)	Dimension (Degree)	Surface (Degree)
H_2SO_4	ACR I-A	5	5	0	0
H_2SO_4	ACR I-B	5	5	0	0
H_2SO_4	EP I-A	3	3	4	2
H_2SO_4	EP I-B	5	2	5	4
NH_4OH	ACR I-A	1	1	0	0
NH_4OH	ACR I-B	0	0	0	0
NH_4OH	EP I-A	0	0	2	1
NH_4OH	EP I-B	0	0	2	1

Table 7 Results of acid and base attack. Flaking and chalking.

References		Flaking	Chalking
Test liquid	Concrete	Degree	Degree
H_2SO_4	ACR I-A	0	1
H_2SO_4	ACR I-B	0	*
H_2SO_4	EP I-A	0	2
H_2SO_4	EP I-B	0	4
NH_4OH	ACR I-A	0	2
NH_4OH	ACR I-B	0	0
NH_4OH	EP I-A	0	1
NH_4OH	EP I-B	0	2

* - the test was not possible due to 100 % of blistering.

Table 8 Results of loss in adhesion strength after acid and base attack.

References		Loss in adhesion strength (%)
Test liquid	Concrete	
H_2SO_4	ACR I-A	100
H_2SO_4	ACR I-B	100
H_2SO_4	EP I-A	8
H_2SO_4	EP I-B	10
NH_4OH	ACR I-A	0
NH_4OH	ACR I-B	5
NH_4OH	EP I-A	0
NH_4OH	EP I-B	1

However, the difference between acrylic and epoxy is important in adhesion after acid attack, with a better behaviour for epoxy coating.

Considering the results of the visual inspections and of the adhesion tests together, the degradation after sulphuric acid attack was important for all coated concretes.

4. Conclusions

The performance of the used coated concretes against chemically aggressive environments was generally better than the performance of the unprotected concretes.

The used epoxy coated concrete achieved the best results against penetration of chlorides. The composition of the concretes is an important factor affecting performance against chemically aggressive environments. Related to sulphates attack, uncoated and coated concretes that performed best were made with the lower cement content and the higher water-cement ratio. The high porosity is good to accommodate expansions caused by reactions that occur during this attack. The degradation caused by the used base and the used acid was measured by visual inspections and by adhesion tests. The ammonium hydroxide caused an insignificant degradation on the coated concretes. On the contrary, the degradation caused by the sulphuric acid was important.

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