

Service Life Prediction Model for Reinforced Concrete Constructions, Treated with Water-repellent Compounds

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1. Introduction

Chloride ingress, due to capillary suction or diffusion, is one of the most important causes of corrosion of the reinforcement in concrete structures. This is certainly the case for constructions that are highly exposed to chlorides such as on-shore constructions in marine environment. Those structures can be protected by means of a protective coating or by a water-repellent agent. The effectiveness of water-repellent agents is being studied in the Reyntjens Laboratory. Based on experimental results, it was decided in 1993 to apply this treatment on the quay-wall of the new container terminal at Zeebrugge harbour. In 1996, a research program was executed to evaluate the effectiveness of the treatment after 3 years of exposure in the real marine environment of the North Sea Coast.

This paper gives a brief overview of the durability problem of concrete structures in marine environment. The production, application, working mechanisms of hydrophobic agents and their behavior after 3 years of real marine exposure are discussed.

A probability method is proposed for interpretation of test results and prediction of the service life of the quay-wall. Once the chloride profiles are determined, a realization of the random diffusion coefficient is obtained at each point for which there is a measurement of the chloride content. These are used to estimate the probability density function of the diffusion coefficient. A reliability analysis is subsequently performed for different places on the quay-wall. A reinforced concrete element is considered to have failed in the analysis when corrosion initiates at the reinforcement, i.e. after a certain chloride concentration threshold is reached at the reinforcement. The probability of corrosion initiation in time is calculated for different locations at the quay-wall. The relevance of the mathematical model will be discussed.

This paper contributes firstly to the idea of increasing the durability of concrete by means of a preventive hydrophobic treatment against chloride ingress. Secondly it illustrates the methodology for life time prediction using a combination of an in-field testing program, the mathematical modeling of the degradation process due to the action of chloride ions in the environment of exposure and the reliability approach.

2. The durability problem of concrete structures in a marine environment

Chloride ingress is one of the most important actors in the corrosion process of concrete reinforcement rods. Especially for off-shore and marine constructions the exposition to chlorides from sea water and marine air play an important role. Also, the concrete of swimming-pools or sanitarian installations is heavily exposed to water rich at chlorides. The ingress of chloride ions into the pores of concrete is caused by diffusion through the pores if they are filled with water or by capillary suction if the pores are

empty. When the chloride ions reach the reinforcement bars, the passivating oxide layer may be depassivated, which initiates the local corrosion of the rods.

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 [3] : a part (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 : $C_3A \cdot CaCl_2 \cdot 10H_2O$) and a part (5%) can be found in the concrete as free chlorides, in solution, or easily soluble by adding water.

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. 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.

At the same time it is a fact that the risk for rust, with a given amount of chlorides in the pore water, will be much higher for a carbonated concrete structure.

For constructions in a marine environment or for constructions attacked by de-icing salts, the chlorides will penetrate through the concrete cover in a relatively short time, and when they reach the steel reinforcement surface, the corrosion process can start. Depending on the amount of chlorides, the corrosion process can take place in an alkaline environment.

3. Quay-wall of the container terminal at Zeebrugge

In order to increase the durability of the concrete, it was decided to apply an hydrophobic treatment on the quay-wall of the container terminal at Zeebrugge. The construction of the container terminal at Zeebrugge was ordered by the Ministry of the Flemish Community, Sea-Harbour Division and finished in 1993. Figure 1 gives a general view of the quay-wall. This quay-wall is constructed on top of cylindrical sunk down reinforced cells (caissons). The original concept of preventive corrosion protection provided in the use of epoxy coated steel reinforcements. For practical reasons of ease of application it was decided to apply an alternative solution, consisting of a hydrophobic treatment with highly concentrated solvent-free compounds based on isobutyl-triethoxy-silane in order to prevent it for damage caused by (1) chloride penetration, (2) pitting corrosion and (3) AAR.

By means of a preliminary research program [6] , the effectiveness of the hydrophobic agent was evaluated by the following criteria : 1) deep and active penetration to obtain a strongly improved long-term protection, 2) being applicable in combination with additional protective coatings in a multi-layer system, were the additional layers are applied to slow down the progress of the carbonation front, 3) easy application on the site : no special conditions should be required concerning : pH, moisture content and temperature of the concrete substrate (-15 till 45 °C) and 4) being environmental friendly. A solvent-free product should be preferred.

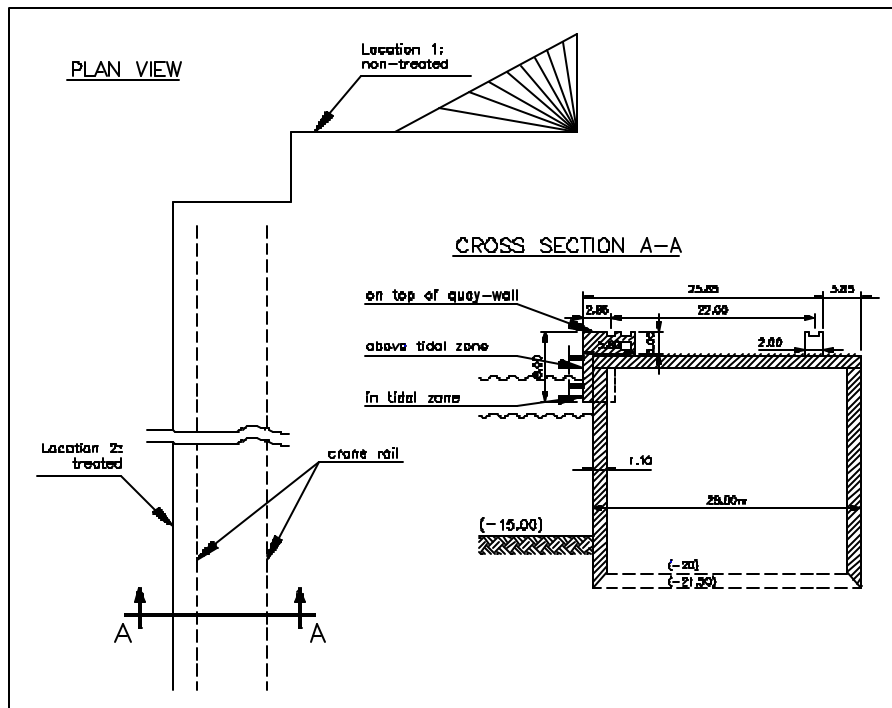


Figure 1 : Plan view and cross-section of the quay-wall and locations of the cores

4. Production, application and working mechanism of silanes

Various generations of protective products have been developed to counteract the aggressive actions of the environment against concrete. Good results have been obtained with barrier-penetrants : after penetration in the concrete they form a barrier against water and the salts dissolved in it. Different families of these hydrophobic agents or waterproofs are already being used for many years in construction industry : silicones, siloxanes, silanes, The silanes used for waterproofing are mostly alkyl-trialkoxo-silanes and thus monomer products. The siloxanes are oligomer or polymer alkyl-alkoxy-siloxanes.

The starting product for all silicon-organic compounds is alkyl-trichloro-silane, figure 2. In figure 2, the alkyl-group is represented by the symbol R. By transformation of this silane with alcohol ($R'-OH$) only, the corresponding alkyl-trialkoxo-silane is produced together with separation of hydrogen-chloride. The reaction with alcohol and water gives oligomer or polymer siloxanes, depending on the amount of water used. The last two products differ in their degree of polymerization, see figure 2.

The hydrophobic treatment reduces the absorption and transport of liquid water and salts dissolved in it. Whereas the penetration of water in liquid form should be entirely prevented, the waterproofing should hardly reduce the diffusion of water vapor. The water-repellents must penetrate as deep as possible into the concrete substrate to obtain a guaranteed long-term durability. At the same time a maximum penetration depth is an essential prerequisite for an effective protection against chloride ingress and chloride induced corrosion of the reinforcements. The penetration capacity of the waterproofing agent and its concentration gradient depend on system specific parameters such as molecular size of the active

organo-silicon compound and on the type of solvent used for dilution. Besides, the penetration and the concentration of the active ingredient also depend on the porosity and the permeability of the concrete substrate, on the amount of waterproofing material applied, on the water content of the substrate

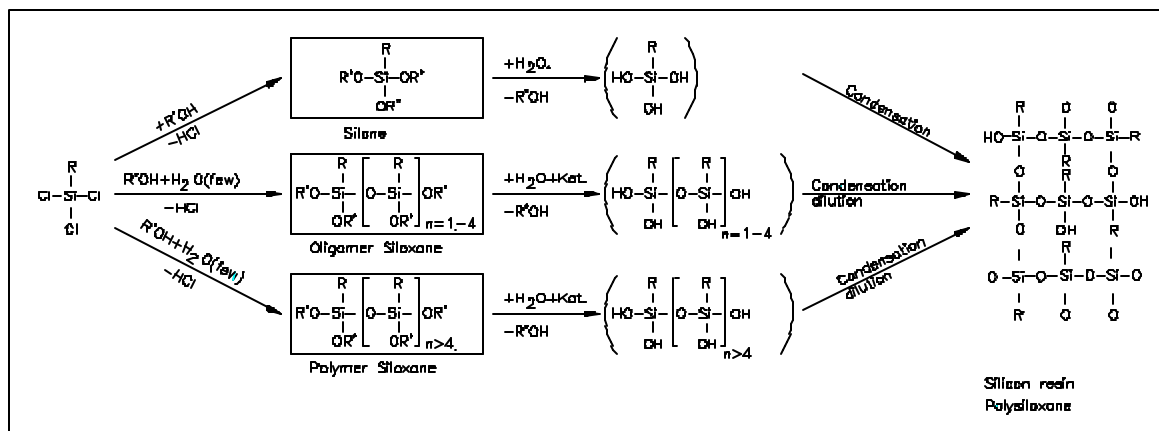


Figure 2 : Chemical steps in the production of silicon-organic compounds

Furthermore, a good water-repellent is characterized by a high alkali resistance and improves the resistance of the substrate against freeze-thaw action. Finally, there should be no undesirable side effects such as color changes of the surface or film formation. Waterproofs generally do not provide effective protection against carbonation caused by atmospheric CO_2 . A waterproofing can only affect the carbonation behavior through changes in the water content of the substrate [2].

Of the silanes, isobutyl-triethoxy-silane (IBTEO) based water-repellents have been found to be particularly suitable for waterproofing, especially if applied on low porosity construction materials [6]. Isobutyl-triethoxy-silane penetrates deeper in the concrete substrate than other silicon-organic compounds, in particular compared to siloxanes. As a consequence, a much longer durability is achieved. The good performances were confirmed in forced chloride penetration tests [7]. The action of hydrophobic agents stems from their dual character with both hydrophobic as well as hydrophilic properties, figure 2. The efficiency, stability and durability of the molecule are determined by its nature and magnitude. The hydrophilic components react with water. At this reaction, ethanol is separated. The produced silanol can be described as an alkyl-silica-compound. This highly reactive compound reacts with neighboring molecules to make polymers.

The silanol is also able to react with the inorganic surfaces in the concrete pores. The thus chemically bonded alkyl-groups form a protective coating for the concrete. Capillary suction is not only counteracted, but even reversed. The capillary forces prevent the penetration of liquid water, even if the water is pressurized. This effect can be compared with the working of a water-proof raincoat. Unlike siloxanes, and thanks to its dual character, the isobutyl-triethoxy-silane is also applicable on wet concrete substrates without remarkable influence on the penetration depth. The adhesion between the treated surface and an eventually additional coating is not affected as well. In the series of methyl-, ethyl-, propyl- and isobutyl-triethoxy-silane, the IBTEO waterproofing agent has the smallest hydrolysis

reaction speed, which enables its maximum penetration. Moreover, this penetration capacity is further improved by the lower surface tension of the solvent-free silane, compared to solvent containing, diluted silane mixes. The lower hydrolysis speed keeps the IBTEO hydrophilic during a longer time, and allows it to penetrate into wet concrete pores before the hydrophobic interfaces are formed.

5. Testing program after 3 years of in-service exposure

In 1996, the in-service performance of the IBTEO water-repellent was evaluated [8],[9] on cylindrical cores taken at different locations in the quay-wall (figure 1) : treated (location 2) and non-treated (location 1) locations, in the tidal zone, above water level and on top of the quay-wall.

To get a global view of the effectiveness of the hydrophobic agent, following characteristics were measured : penetration depth, porosity, pH at following depths : 0-9 mm, 11-20 mm, 40- 60 mm, carbonation depth, compressive strength, cement content and chloride content.

The characteristics relevant for this paper are summarized in table 1. A full report can be found in earlier papers [6], [10]. The pH-values are somewhat higher at the non-treated location. In the treated part, pH values from 12 to 12.5 were found, in the non-treated part values around 12.8 were measured. However, all pH-values are in the region of passivation.

Fully corresponding results are obtained by the measured carbonation depths. For the non-treated locations, the carbonation depth is limited to 1 mm, for the treated locations a carbonation depth up to 5 mm is measured, which declares the lower pH-values in the first zone which are mean values for a depth from 0 till 9 mm. The used water-repellent has no protective influence on the carbonation process of the concrete, as mentioned before. The measured penetration depth of the water-repellent was limited to 3.5 mm, which is lower than the results obtained in the laboratory tests.

The chloride content was measured at the following depths : 0-9 mm, 11-20 mm, 40-60 mm.

The content profiles of water soluble chlorides are shown in figure 3. The chloride contents were determined by means of wet chemical analysis, according to the Belgian Standard NBN B15-257. The chloride content, obtained by wet chemical analysis, equals the amount of free chlorides and a great deal of the chlorides, bound under the form of the Salt of Friedel which dissolves in the water during extraction. In fact, these water soluble chlorides mean the real danger for corrosion of the reinforcement.

The relation water soluble to acid soluble chloride content amounts 94 % in our tests [6]. Thus, the chloride content, present at the binding and transformed into insoluble chlorides is very restricted. The chloride loading is for the greater part caused by the external contamination, i.e. the marine environment. It can be observed that the chloride content in the non-treated zones is significantly higher than in the treated zones. The absence of an effective barrier, combined with the relatively high porosity, allowed the chlorides to penetrate very deep in the concrete. On the contrary, the chloride contents in the treated zones are still at acceptable level, and combined with the low carbonation depth, they present no danger for reinforcement corrosion.

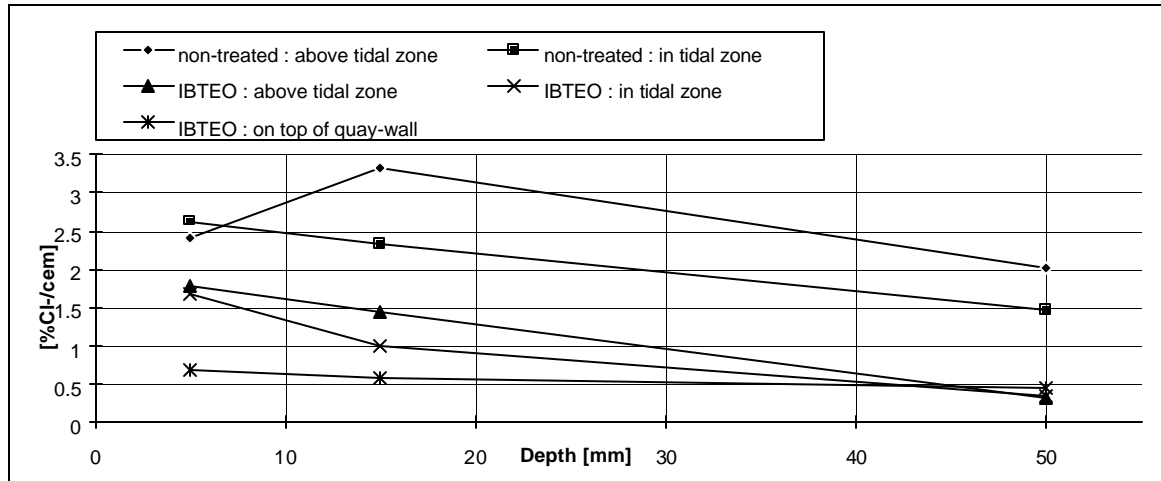


Figure 3 : Water-soluble chloride content by weight of cement at different depths in the cores

6. Service life prediction, a reliability analysis

Based on the measured material properties and chloride profiles, a service life prediction can be performed using reliability and stochastic concepts. It is becoming increasingly important to be able to predict the service life of concrete for new constructions and/or concrete in-service. For example, life predictions are a necessary input to life-cycle cost models, which consider both the durability and cost of concrete. While this approach is not often used, it is likely to have an increasingly important role in designing and evaluating concrete because of a) applications that require significantly increased service lives (as off-shore constructions), b) increased use of concrete in harsh environments (marine climate), c) the high cost of rebuilding and maintaining the nation's infrastructure, and d) the development of high-performance concrete for which a record of long-term performance is not available [1].

The reliability analysis presented here is applicable to concrete deterioration associated with steel corrosion initiated by the action of chloride ions. A similar analysis can be performed for other deterioration processes as long as the mathematical formulations governing the deterioration mechanisms are available.

With the present state of knowledge, it is virtually impossible to introduce a mathematical model taking into account all the variables involved in the corrosion process. To estimate the service life of a given concrete element, many assumptions have to be made. To model the chloride transport process in a porous material, it is assumed that Fick's second law applies, although it is a simplified representation of reality.

Fick's second law describes the transport of chlorides in the concrete due to diffusion. As seen before, the diffusion process is only valid in saturated conditions. When the pores are empty, capillary forces drag the outside solution into the concrete, bringing the salts along. Other assumptions are made that are not valid for concrete. In the derivation of Fick's law it is assumed that the porous material is homogeneous, that the medium is nonreactive and nonadsorptive, which are not valid for concrete [5].

Despite the differences between the assumptions on which Fick's law is based and reality for the application intended, Fick's law still provides the only way available to model chloride diffusion into concrete. Anyway, the same analysis as presented here, can be performed on more accurate models.

The diffusion law, Fick's second law for one-dimensional chloride diffusion into concrete, takes the form :

$$\frac{dC}{dt} = \frac{d}{dx} \left(D \frac{dC}{dx} \right), \quad (1)$$

in which concrete is assumed to be a homogeneous, isotropic material. When it is assumed that no reaction occurs between the concrete and chlorides, an explicit solution of this differential equation can be obtained, using the following boundary conditions :

- $C(x, t=0) = C_0$; $0 < x < \infty$ (the initial chloride concentration in the concrete mix) and
- $C(x=0, t) = C_s$; $0 < t < \infty$ (the chloride concentration loading from the marine environment)

$$C_i(x, t) = C_0 + (C_s - C_0) \cdot \operatorname{erfc} \left(\frac{x}{2\sqrt{Dt}} \right), \quad (2)$$

in which $C_i(x, t)$ is the amount of chlorides on time t at a distance x from the concrete surface.

A reliability analysis provides a means to evaluate the probability of failure of a component. The term component describes a structure or structural element whose limit state function is defined in terms of a single, continuous function known as the limit state function [4]. In the present problem, where only the diffusion coefficient D is considered to be random, the limit state function $g(D)$ can be written as :

$$g(D) = C_T - C(D), \quad (3)$$

where C_T is the threshold chloride concentration and $C(D)$ is the chloride concentration at a distance x from the exposed concrete surface at time t .

The function $g(D)$ - the limit state function - is positive only if the concrete element is in a "safe" state, i.e. the chloride concentration at the reinforcement (at a distance x from the concrete surface) is less than the threshold concentration. Having the probability density function of the diffusion coefficient D , the probability that the chloride concentration C_T

is exceeded can be expressed as :

$$P_f = P(C > C_T) = 1 - F_C(C_T), \quad (4)$$

where $F_C(C_T)$ is the cumulative distribution function of C . As a result of the one-by-one relationship between C and D , the exceedance failure probability can be rewritten as :

$$P_f = P(C > C_T) = P(D > D_T) = 1 - F_D(D_T), \quad (5)$$

where $F_D(D_T)$ is the cumulative distribution of D , and D_T is the threshold diffusion coefficient obtained by inversion of the solution of Fick's law : $D_T = F^{-1}(C_T)$. Assuming that the diffusion coefficient has a lognormal distribution the exceedance probability is consequently obtained as follows :

$$P_f = P(C > C_T) = 1 - \Phi \left(\frac{\ln(D_T) - \ln(D)}{\lambda_D} \right) = \Phi \left(- \frac{\ln(D_T) - \ln(D)}{\lambda_D} \right), \quad (6)$$

where λ_D and ξ_D are the parameters of the lognormal distribution and $\Phi(D)$ is the standard normal cumulative distribution function. Table 1 gives an overview of the material properties and the water-soluble chloride contents used in the reliability analysis [8], [9].

location	core identification number	n Vol%	Dry density [kg/m ³]	amount of cement [kg/m ³]	x = 4,5 mm			x = 15,5 mm			x = 50 mm		
					%Cl ⁻	%Cl ⁻ /cem	%Cl ⁻ /H ₂ O	%Cl ⁻	%Cl ⁻ /cem	%Cl ⁻ /H ₂ O	%Cl ⁻	%Cl ⁻ /cem	%Cl ⁻ /H ₂ O
non-treated (location 1 figure 1)	A1 above tidal zone	17.48	2157	284	0.359	2.77	4.43	0.444	3.443	5.48	0.282	2.18	2.74
	A2 above tidal zone				0.268	2.07	3.31	0.416	3.217	5.13	0.236	1.84	2.91
	A3 in tidal zone	14.57	2232		0.248	1.91	3.80	0.295	2.280	4.52	0.186	1.44	2.85
	A4 in tidal zone	14.76	2224		0.433	3.35	6.52	0.306	2.364	4.61	0.196	1.51	2.95
Treated (location 2, figure 1)	B5 above tidal zone	15.66	2234	260	0.263	2.24	3.75	0.145	1.233	2.07	0.045	0.38	0.64
	B6 above tidal zone				0.155	1.31	2.21	0.195	1.655	2.78	0.034	0.29	0.49
	B7 in tidal zone	17.38	2193		0.205	1.75	2.59	0.111	0.944	1.4	0.039	0.32	0.49
	B8 in tidal zone	16.70	2197		0.192	1.64	2.53	0.126	1.070	1.66	0.043	0.36	0.57
Treated on top of quay - wall	C9	14.86	2236		0.084	0.71	1.26	0.063	0.534	0.95	0.053	0.45	0.80
	C10				0.075	0.64	1.13	0.075	0.638	1.13	0.052	0.44	0.78

Table 1 : material characteristics and water-soluble chloride contents

7. Realization of the diffusion coefficient D

With the chloride profiles of the concrete samples exposed during a given period, the diffusion coefficient can be backcalculated using an inversion of Fick's second law (eq. 2). There is no explicit solution for the inverse of eq. 2 but a Newton-Raphson root-finding algorithm can be used to find the diffusion coefficient D associated with the measured concentration at given time t and distance x [5]. In the inversion of Fick's second law, following boundary conditions were used : $C_0 = 0.03$ % Cl and $C_s = 7$ % Cl/H₂O.

The initial chloride concentration in the concrete mix is taken from comparable concrete mixes from neighboring marine constructions at their erection time, as there were no test results available of the original chloride content from the quay-wall itself.

The chloride concentration loading at the concrete surface is taken higher than the chloride concentration of the salt sea-water, which equals 3,5 percent by weight of water. As can be seen from table 1, the water soluble chloride content as measured at depths of 0-9 mm and 10-20 mm exceed in many cases 3,5 percent by weight of water. Comparable results can be found in [5]. As the inversion of Fick's second is only soluble when $C_0 < C_i(x,t) < C_s$, a least square optimization proved that a value of $C_s = 7$ percent per weight of water results in chloride profiles that are nearest to the measured profiles. The higher value can be explained by the presence of alga at the concrete surface in the tidal zone, of which the chloride concentration can reach up to 9 percent by weight of water, and by salt crystallization.

Table 2 gives the mean value $\mu(D)$ and standard deviation $\sigma(D)$ of the diffusion coefficient D for the groups of cores, taken at different locations. Remarkable is the diffusion coefficient of the non-treated zone to be nearly an order of magnitude bigger than in the treated locations. Service life number of

years are given in the assumption that no erosion of the concrete surface takes place, which would remove the water-repellent, and thus also its beneficial effects on chloride ingress.

	$\mu(D)$ [cm ² /s]x10 ⁸	$\sigma(D)$ [cm ² /s]x10 ⁸	Service life [years] $C_T = 0.4 \text{ \% Cl/cem}$	Service life [years] $C_T = 0.7 \text{ \% Cl/cem}$
A (non-treated)	9.64	7.0	7.6	12.6
B (treated with IBTEO)	1.18	1.0	61	105.1
C (treated with IBTEO, on top of quay-wall)	1.32	1.7	70	123.3

Table 2 : diffusion coefficients and service life prediction ($P_f=0.5$) for the different locations

8. Service life prediction

With the computed parameters of the lognormal probability distribution for the diffusion coefficient, the service life can be predicted using the reliability analysis as outlined above.

A corrosion initiation mode (failure or limit state) can be characterized in a simplified way by specifying a threshold chloride concentration beyond or above which corrosion would start. Based on research, codes and standards have specified the threshold limit state for the chloride content. The reliability analysis is performed for threshold chloride concentrations that equal 0,4 percent per weight of cement and 0,7 per weight of cement. As the free chlorides mean the real danger for corrosion, only the water soluble chlorides (table 1) were taken into account (about 94 % of the total amount of chlorides).

Figure 4 shows the failure probability results for the different locations. The results reflect the assumption that the reinforcement located at 120 mm from the exposed surface, as could be measured from the taken cores.

For a probability of failure of 50 percent, table 2 shows the time it will take in years for the threshold chloride concentration to reach the reinforcement.

For the non-treated locations of the quay-wall, this is in accordance with the obtained test results. After 3 years of in-service exposure, the chloride content already exceeds 0,7 percent by weight of cement at a depth of 50 mm.

Table 3 shows the sensitivities of the probability of corrosion initiation with respect to the limit state function parameters : distance from the exposed concrete surface (x), time (t), initial chloride concentration (C_0), surface chloride concentration (C_s) and the threshold concentration (C_T). The sensitivities values shown in table 3 correspond to the point at which the probability of corrosion initiation is 50 percent. The sensitivities values give the variation of the probability of failure for one percent change in the limit state parameter [4].

The probability of failure is more sensitive to the reinforcement cover thickness than to time and has intermediate sensitivity to the chloride concentration. This conclusion also could be drawn in [5]. In contrary with the treated zones, the probability of failure in the non-treated locations is more sensitive to the surface concen

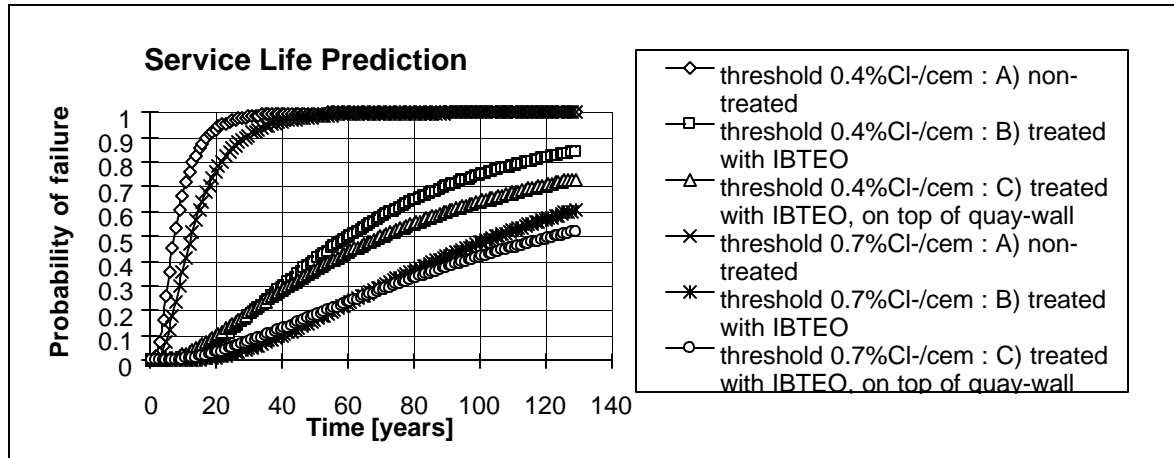


Figure 4 : probability of failure in function of time for two chloride content thresholds, caused by the much higher diffusion coefficient.

0.4%Cl-/cem/ 0.7%Cl-/cem	$x \cdot \frac{dP_f}{dx}$	$t \cdot \frac{dP_f}{dt}$	$C_0 \cdot \frac{dP_f}{dC_0}$	$C_s \cdot \frac{dP_f}{dC_s}$	$C_T \cdot \frac{dP_f}{dC_T}$
A: non-treated	-0.98// -1.23	0.62//0.62	0.40//0.06	-3.13// -3.19	-0.36// -0.6
B : treated	-1.05// -1.04	0.53//0.53	-0.03// -0.2	-0.19// -0.05	-0.55// -0.51
C : treated, on top of quay-wall	-0.81// -0.54	0.41//0.41	0.03//0.09	-0.018//0.09	-0.43// -0.42

Table 3 : sensitivity analysis

9. Conclusion

As could already be concluded from laboratory tests, an in-service test program and a service life prediction model show the effectiveness of the highly concentrated, solvent-free compound, based on isobutyl tri-ethoxy-silane as a water-repellent after 3 years of real marine exposure. Although the used reliability analysis only takes into account the diffusion process in the concrete, mathematically translated by Fick's second law, it proves the impact of this preventive protection method.

This positive result must stimulate engineers to stop considering a hydrophobic treatment as a last remedy for treating damage. On the contrary, they should consider the hydrophobic treatment as a full phase in the construction project. In this way it will be possible to improve durability of concrete constructions in an elegant and economic way.

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