

CHLORIDE PENETRATION IN RC-STRUCTURES IN MARINE CLIMATE: IN SITE SURVEY, ANALYSIS MODEL AND BENEFIT OF A PREVENTIVE HYDROPHOBIC TREATMENT

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ABSTRACT

Chloride ingress is one of the main causes of corrosion of the reinforcement in concrete structures that are highly exposed to chlorides, such as off-shore or on-shore constructions in marine environment. This paper deals with the quay-wall of the new container terminal at Zeebrugge Harbor, Belgium. This type of structure is subjected to chloride ingress, not only below sea-water level, but also in and above tidal zone, as well as on top of the quay-wall. Those structures can be protected by means of a water repellent agent. The effectiveness of water repellent agents is being studied in the Reyntjens Laboratory of KULeuven. Based on experimental results, it was decided in 1993 to apply Protectosil ® BH N (former Dynasytan® BH N: alkyltriethoxysilane) on the quay-wall. Three subsequent in site surveys have been conducted to evaluate the effectiveness of the treatment: in 1996, 1998 and recently in 2005, respectively after 3, 5 and 12 years of exposure in the real marine environment of the North Sea Coast. The chloride ingress has been determined as a function of time, both in a non-treated and treated location. Because of the long-term data sequence, the long-term effectiveness of the treatment can be assessed in an objective way. The obtained data are used to model the chloride ingress into the structure, for which Ficks' diffusion law is used. A probabilistic analysis method is applied. The methodology allows estimating the remaining service life of the structure, both for the treated and non-treated location.

Keywords: chloride ingress, preventive treatment, service life, hydrophobic agent.

1 QUAY-WALL AT ZEEBRUGGE HARBOUR – IN SITE SURVEY

In order to increase the durability of the concrete, it was decided to apply an hydrophobic treatment on the new quay-wall of the container terminal at Zeebrugge [1,2]. The construction of the container terminal at Zeebrugge was ordered by the Ministry of the Flemish Community, Sea-Harbor Division and finished in 1993. Figure 1 gives a general view of the quay-wall, constructed on top of cylindrical sunk down reinforced cells (caissons). For practical reasons of ease of application it was decided to apply an hydrophobic treatment with highly concentrated solvent-free compounds based on alkyltriethoxysilane, in order to prevent the wall for damage caused by chloride penetration, pitting corrosion and alkali-aggregate-reaction [3-5]. The in site consumption was 300 g/m².

By means of a preliminary research program [6,

7], the effectiveness of the hydrophobic agent was evaluated using 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, where 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 environmentally friendly. A solvent-free product should be preferred.

The mid-long term effectiveness of the hydrophobic agent has been evaluated based on two subsequent in site surveys: 1996 and 1998, after 3 and 5 years of in site exposure respectively [8,9]. Based on these surveys, the mid-long term effectiveness could be demonstrated [10-15]. Also, the deep and active penetration was determined from in site samples [16, 17].

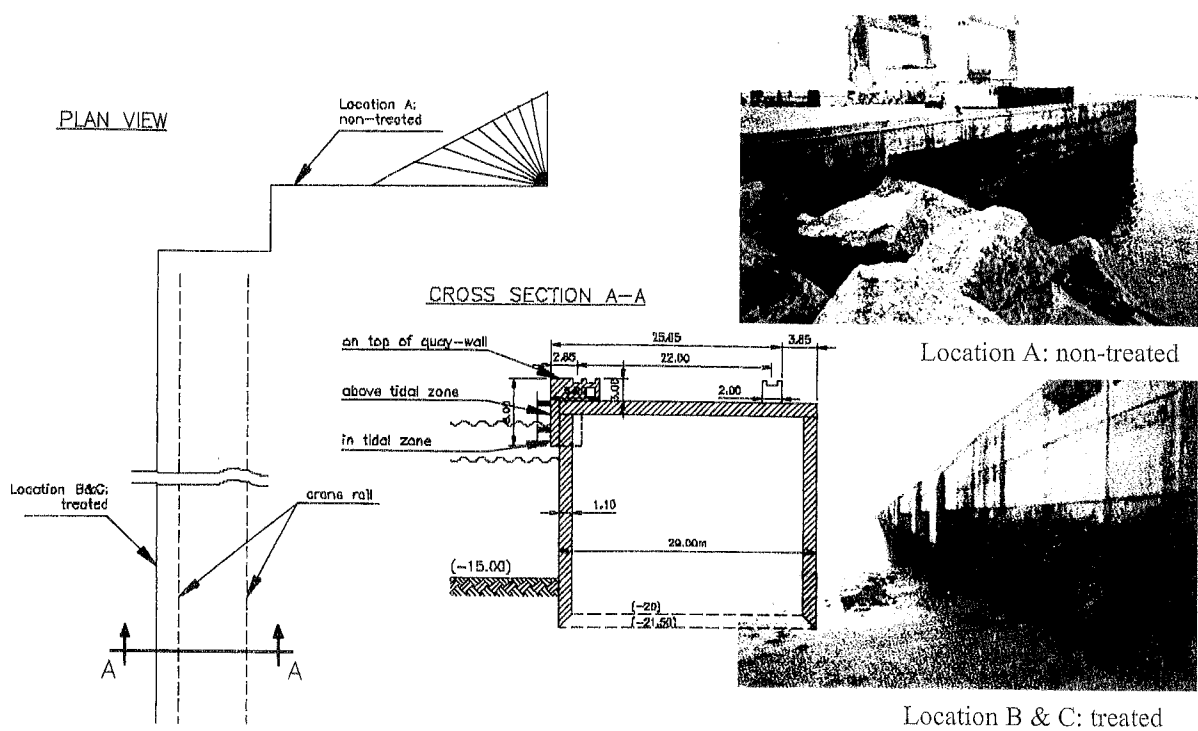


Figure 1: Plan view and cross section of the quay-wall at Zeebrugge Harbor

On January 18, 2005, a third in site survey on the quay-wall of Zeebrugge Harbour has been performed after almost 12 years of in site exposure [18]. Goal of this in site survey is to update the progress of chloride penetration into the concrete caused by the salty sea-water and to compare the results obtained at different locations, built in the same period, with comparable parameters, see also Figure 1:

- a location not treated with an hydrophobic agent in the tidal zone (location A);
- a location not treated with an hydrophobic agent above the tidal zone (location A);
- a location treated with the hydrophobic agent - Protectosil® BH N - in tidal zone (location B);
- a location treated with the hydrophobic agent - Protectosil® BH N - above tidal zone (location B);
- a location treated with the hydrophobic agent - Protectosil® BH N - on top of the quay wall (location C).

These results allow assessing the effectiveness of this type of preventive treatment on a long term. Therefore, the values obtained after 12 years of in site exposure are compared with the data from former in site surveys. Following key dates apply:

- (treatment of quay-wall with Protectosil® BH N from Degussa AG: 1993);
- survey 1: april 2, 1996 – after 3 years of in site exposure;
- survey 2: april 8, 1998–after 5 years of in site exposure;

- survey 3: january 18, 2005 – after 12 years of in site exposure.

2 LABORATORY TESTS AND RESULTS

2.1 Test Campaign

During the in site campaign, concrete cores are drilled at neighbouring positions. In general, 3 types of tests are performed on the cores drilled (diameter = 50mm) [18]:

- on all cores the penetration depth of the hydrophobic agent is determined based on a visual interpretation, by wetting a fresh cracked surface;
- on all cores the carbonation depth is measured before the cores are sawn into slices, on the site directly after drilling as well as in the laboratory the day after drilling for comparison;
- water soluble and acid soluble chloride contents are measured at several depths, Table 1 and Figure 2, to be able to estimate a chloride penetration profile at different locations

2.2 Penetration Test - Hydrophobic/Hydrophilic Action

The hydrophobic action of the hydrophobic agent is still active. Based on visual inspection by wetting a fresh cracked surface, the penetration depth of the hydrophobic agent is of the same order of magnitude as the values reported based on the in site survey in 1998 [9]. Thus, based on this visual inspection, the

hydrophobic action is still present after 12 years on in site service. The hydrophobic agent - Protectosil® BH N - has been applied to the concrete surface in 1993.

In general, the interface between hydrophilic and hydrophobic behavior was rather clear and easy to determine visually. Absorption in the hydrophilic part gave a clear discoloration of the surface, Figure 3.

2.3 Carbonation Depth

The carbonation depths are determined by treating the fresh sawn surfaces with a phenolphthaleine solution (dissolved in 1% ethyl alcohol). The values are listed in table 3.

Table 1: Summary of test program

Location	Core no.	Analysis
A: non-treated location		
above tidal zone	A1	3
	A2	3
In tidal zone	A3	3
	A4	6
B & C: Treated with Protectosil® BH N		
above tidal zone	B1	3
	B2	3
In tidal zone	B3	3
	B4	6
on top of quay wall	C1	3
	C2	3

Legend:
 3 = Water soluble and acid soluble chloride content at 3 different depths, figure 2
 6 = Water soluble and acid soluble chloride content at 6 different depths, figure 2

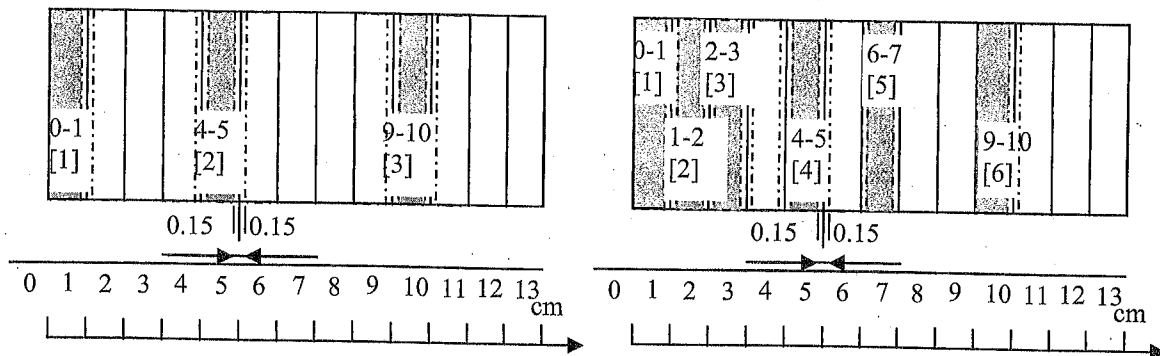


Figure 2: Determining the chloride content at 3 different depths (left) or at 6 different depths (right)

Table 2: Hydrophobic action and visual inspection by wetting a fresh cracked surface

Location	Core no.	Penetration depth [mm] - hydrophobic/hydrophilic action
B : Above tidal zone	B1	hydrophobic - 4-5 mm (2-6)*
	B2	hydrophobic - 6 mm
B : In tidal zone	B3	hydrophobic - 5 mm
	B4	hydrophobic - 6 mm
C : On top of quay-wall	C1	hydrophobic - 1-2 mm (1-3)
	C2	hydrophobic - 1-2 mm

Legend: * the values between brackets are the values reported earlier, dd. April 1998 [9]

The carbonation depth remains approximately zero in the non-treated location. In the treated location the carbonation depth varies from 4-6 mm in tidal zone, towards 8-12 mm above tidal zone and up to 12-16 mm on top of the quay-wall. Only the values on top of the quay-wall seem to have increased compared to the former campaign in April 1998 [9]. Comparing the different locations, it is clear that the diffusion of CO₂ into the concrete is improved causing carbonation since the entrance of water is prevented by means of the hydrophobic agent. This is in line with the increase of carbonation depths found in tidal zone (4-6mm), above tidal (8-12mm) zone and on top of the quay wall (12-16mm) and the fact that for the non-treated locations, the carbonation depth remains negligible.

2.4 Water soluble and acid soluble chloride content – chloride profiles

Two different values of chloride concentration

have been determined:

- the water soluble chloride content;
- the acid soluble chloride content.

To determine the chloride penetration profiles, the cores were sawn in slices and both types of chloride content have been determined at several depths on slices of average thickness of 7 mm, see Figure 2.

The chloride contents were determined by means of wet chemical analysis, according to the Belgian Standard NBN B15-250 (1990). 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 Salt of Friedel which dissolves in the water during extraction. Since the water soluble chlorides represent the chloride ions endangering the steel to corrode, these are reported here solely.



Figure 3: Example of hydrophobic action by means of visual inspection by wetting a fresh cracked surface (slice thickness equals 7 mm) – core B2 (left) and core C1 (right)

Table 3: Carbonation depth

Location	Core no.	Carbonation Depth [mm]
A: non-treated		
Above tidal zone	A1	0 (0-0.5)*
	A2	0
in tidal zone	A3	0 (0)*
	A4	0
B and C: treated with Protectosil® BH N		
Above tidal zone	B1	8-12 (6-12)*
	B2	8-12
in tidal zone	B3	4-6 (5-10)*
	B4	4-5
On top of quay-wall	C1	12-16 (5-10)*
	C2	12-16
Legend: * the values between brackets are the values reported earlier, dd. April 1998 [9]		

The experimental results $-\%Cl^-/cem$ - are presented in Figures 4 up to 8. Additionally, the chloride concentration of the former surveys - 1996 and 1998 - is added as well. Each of the values, plotted in the figures, is the average of 3 comparative chemical analyses:

- Figure 4: water soluble chloride content - location A - non-treated - above tidal zone (1996-1998-2005);
- Figure 5: water soluble chloride content - location A - non-treated - in tidal zone (1996-1998-2005);
- Figure 6: water soluble chloride content - Location B - treated with Protectosil® BH N- above tidal zone (1996-1998-2005);

- Figure 7: water soluble chloride content - Location B - treated with Protectosil® BH N - in tidal zone (1996-1998-2005);
- Figure 8: water soluble chloride content - Location C - treated with Protectosil® BH N - on top of quay-wall (1996-1998-2005);

With respect to the presented chloride penetration profiles, some remarks have to be made regarding the accuracy of the experimental results. The data are subject to scatter due to the heterogeneous composition of concrete - aggregates and cement paste as well as the relatively small size of the samples analysed (cores with diameter of 50 mm and

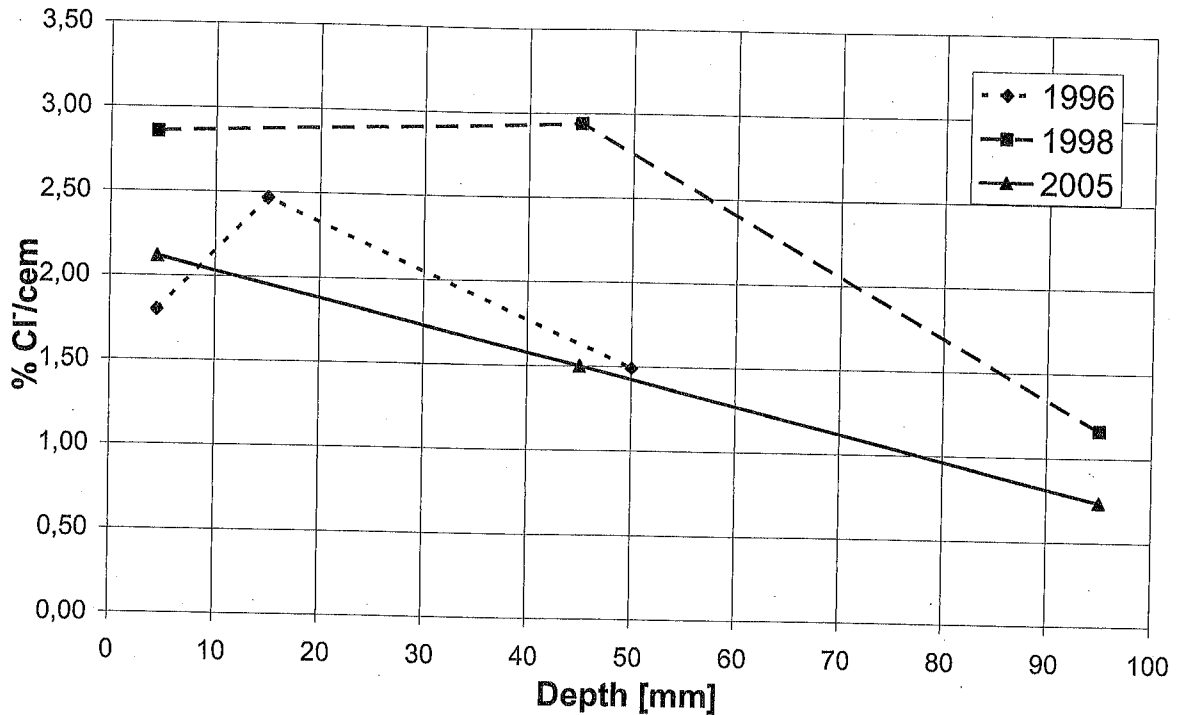


Figure 4: water soluble chloride content - location A - non-treated - above tidal zone (1996-1998-2005)

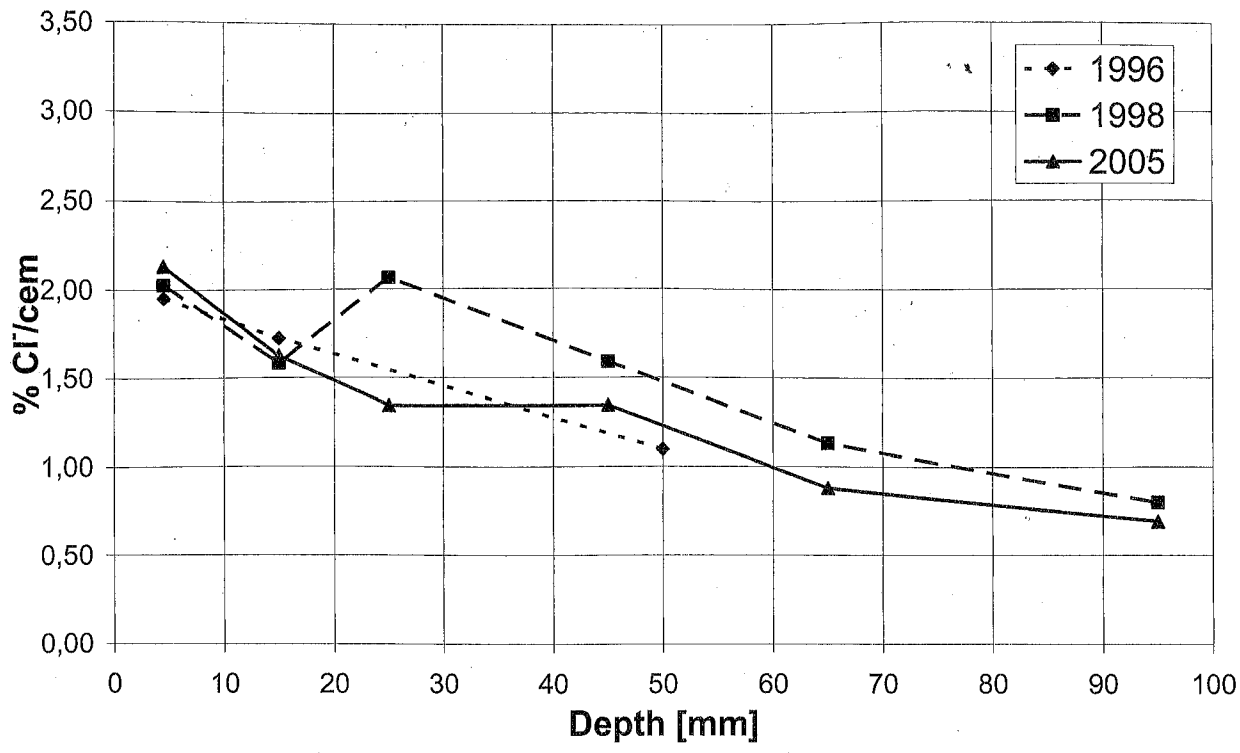


Figure 5: water soluble chloride content - location A - non-treated - in tidal zone (1996-1998-2005)

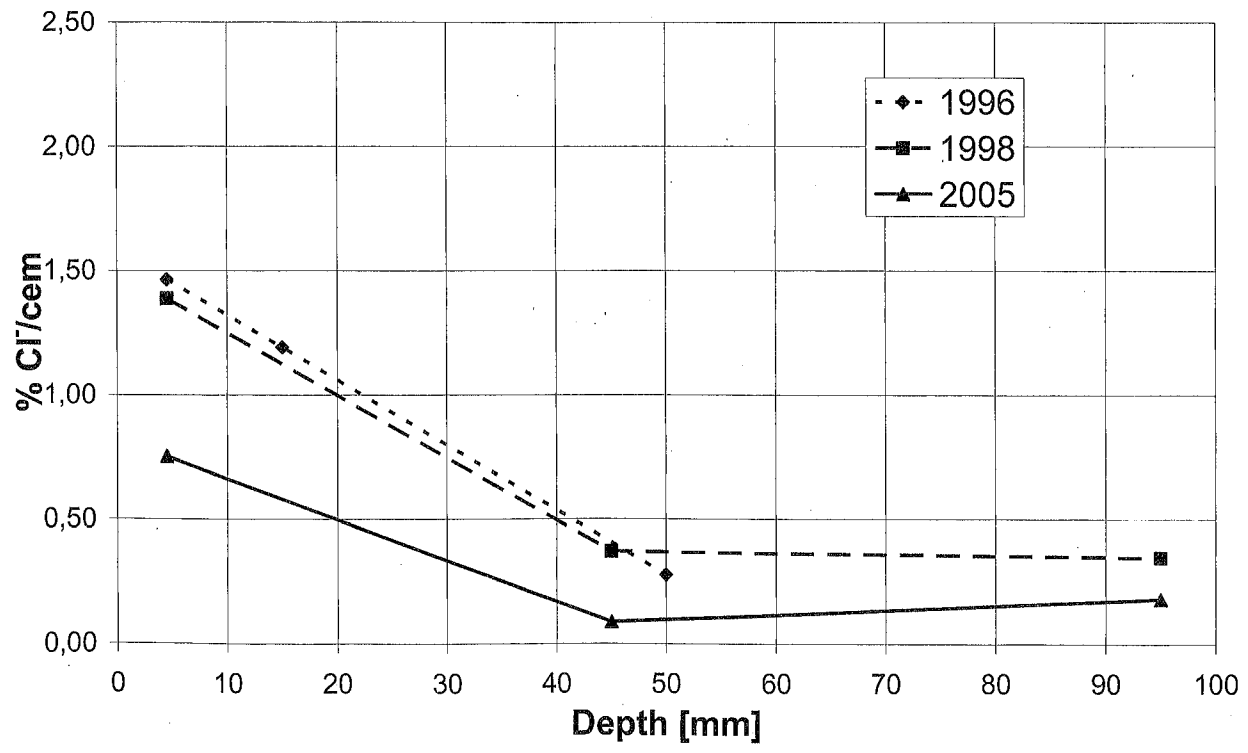


Figure 6: water soluble chloride content - Location B - treated with Protectosil® BH N- - above tidal zone (1996-1998-2005)

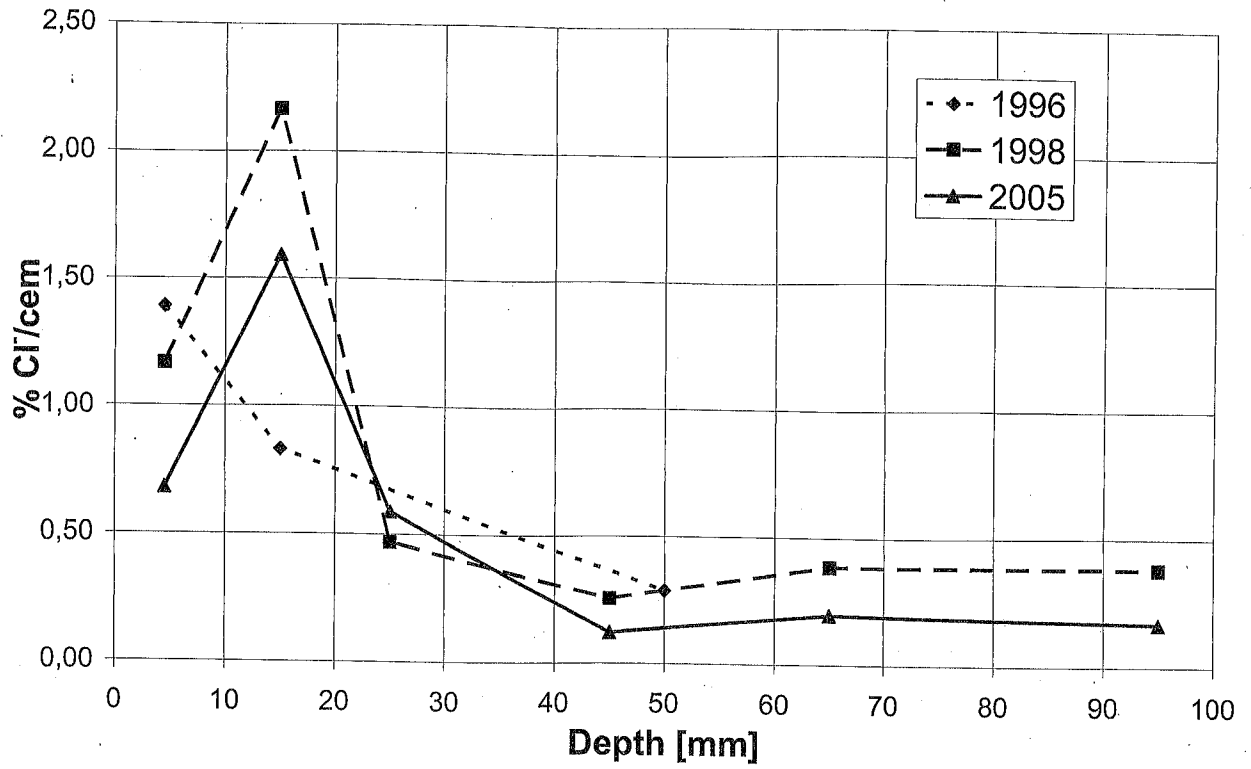


Figure 7: water soluble chloride content - Location B - treated with Protectosil® BH N - in tidal zone (1996-1998-2005)

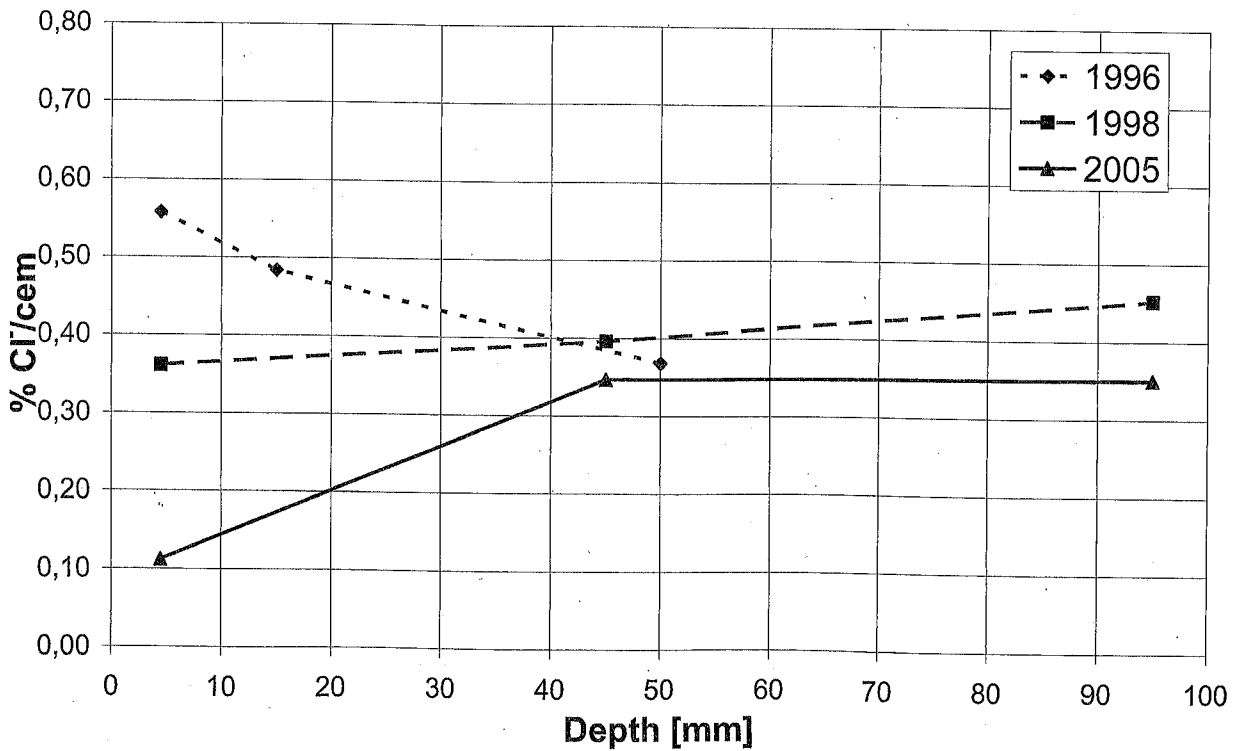


Figure 8: water soluble chloride content - Location C - treated with Protectosil® BH N - on top of quay-wall (1996-1998-2005)

slices of concrete with an average thickness of 7 mm). A variation in results might originate from the number and size of aggregates within the tested sample. When the difference between the values obtained from different cores, taken at the same depth and similar location (so: $i = \%Cl_{1,i} - \%Cl_{2,i}$ at $i=15$ mm for example) are calculated, (these cover the heterogeneous nature of the test samples analysed as well as inaccuracy of the chemical analysis, which is considered to be small compared to the heterogeneous nature of the samples), following results are obtained:

- difference in case of water soluble chloride content: average (i) = -0.010%Cl; standard deviation (i) = 0.026 Cl;
- difference in case of acid soluble chloride content: average (i) = 0.022%Cl; standard deviation (i) = 0.061 Cl;

These values are a measure of the error that one might encounter in the results and that might be attributed mainly to the heterogeneous nature of the samples analysed.

In general, following conclusions can be drawn:

- the scatter is largest for the data of chloride concentration above the tidal zone. In the tidal zone, the scatter is significantly smaller, that can be related to the higher level of uncertainty related with the exposure conditions above tidal zone (algae, splash water, increased carbonation depth,...);
- the water- and acid soluble chloride concentration in the non-treated area (A) in the tidal zone, remain within the same high levels. The values vary at the surface from 2.20%Cl/cem (water soluble) to 0.90%Cl/cem at a depth of 95 mm. For the acid soluble chloride content the respective values are: 2.30%Cl/cem at the surface and 1.00% Cl-/cem at a depth of 95 mm;
- the corresponding water- and acid soluble chloride concentration in the treated area (B) in the tidal zone, remain significantly lower. The values vary at the surface from 1.20%Cl/cem (water soluble) to 0.28%Cl/cem at a depth of 95 mm. For the acid soluble chloride content the respective values are: 1.25%Cl/cem at the surface and 0.21% Cl/cem at a depth of 95 mm;
- Surprising however is the apparent storage of chloride ions which is found at the treated location (B) in tidal zone at a depth of 15 mm, Figure 7. Initially, 1996 - after 3 years in service - this was not observed. Subsequently, in 1998 and again to a similar extent in 2005, a peak value in the water and acid soluble chloride concentration can be noticed around 15 mm depth, Figure 7. The value of the chloride concentration at the peak for the treated area is of the same order of magnitude of the chloride concentration at the surface for the corresponding non-treated areas. In the non-treated areas, this buffering or storage of chloride concentrations in the

tidal zone is not present, see Figure 5. Nevertheless, this peak value at 15 mm beyond the surface does not seem to affect the overall chloride concentration profile at larger depth from the surface. At a depth of 25 mm chloride concentrations are obtained which are expected without the intermediate peak-value.

3 SERVICE LIFE PREDICTION – RANDOM DIFFUSION COEFFICIENT

Based on the measured material properties and chloride profiles, a service life prediction can be performed using a time dependent reliability analysis [10, 13-14, 19-21]. The reliability analysis used in this paper is applicable to concrete deterioration associated with steel corrosion initiated by the action of chloride ions. Similar analyses can be performed for other deterioration processes as long as the mathematical formulation governing the failure mechanisms is available.

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, some assumptions need to be made. To model the chloride transport process in a porous material, it is assumed that Fick's 2nd law applies, although it is a simplified representation of reality [20, 21-25]. Fick's second law describes the transport of chlorides in the concrete due to diffusion. In reality, 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 non-reactive and non-adsorptive, which are not completely valid for concrete. Despite of the differences between the assumptions on which Fick's law is based and reality for the application intended, it is still accepted as the common model to describe chloride transport into concrete [21-25]. Of course, a similar analysis could be performed on more accurate models, as they become available. The transport model and its limitations also count for a classical deterministic analysis, and certainly is not a limitation of the probabilistic analysis.

The diffusion law, Fick's 2nd law for one-dimensional chloride diffusion into concrete, takes the form :

$$\frac{\delta C}{\delta t} = \frac{\delta}{\delta x} \left(D \frac{\delta C}{\delta x} \right) \quad (1)$$

in which concrete is assumed to be a homogeneous, isotropic material. When assumed that no reaction occurs between 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 at the surface of the concrete due to the marine environment):

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

in which $C_i(x,t)$ is the amount of chlorides at time t , at a distance x from the concrete surface and $\operatorname{erfc}()$ is the error function.

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 function known as the limit state function [17]. In the present problem, where only the diffusion coefficient D is considered to be a random variable, 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 surface at time t .

With the aid of the cumulative density function (CDF), the failure probability can be written as:

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

In which, $F_C(C_T)$ is the cumulative density function of C . Due to the one-to-one relation in between C and D , the failure probability can be rewritten into:

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

In which $F_D(D_T)$ is the cumulative density function of D and D_T is the limit value for the diffusion coefficient found by the inverse relation of Fick's 2nd Law:

$$D_T = f^{-1}(C_T) \quad (6)$$

Assuming the diffusion coefficient has a lognormal distribution, the exceedence probability is consequently obtained explicitly as [20]:

$$P_f = P(C > C_T) = 1 - \Phi\left(\frac{\ln(D_T) - \lambda_D}{\xi_D}\right) \quad (7)$$

where λ_D and ξ_D are the parameters of the lognormal distribution and $\Phi(D)$ is the standard normal cumulative distribution function.

With the chloride profiles of the concrete samples exposed during a given period, the diffusion coefficient can be back-calculated using an inversion of Fick's 2nd law (eq.2). In the inversion of Fick's second law, the following boundary conditions were used:

- $C_0 = 0.03\% \text{Cl}^-$;
- $C_S = 7\% \text{Cl}^-/\text{H}_2\text{O}$ [13,14];
- $C_T = 0.7\% \text{Cl}^-/\text{cem}$.

Table 4 gives the mean value $\mu(D)$ and the standard deviation $\sigma(D)$ as well as the coefficient of variation $\operatorname{cov}(D) = \sigma(D)/\mu(D)$ for the groups of cores taken at different locations during the 3 subsequent surveys in 1996, 1998 and 2005. The first columns reflect the obtained values of the diffusion coefficient and predicted service life after 5 years in site exposure; the subsequent columns present the updated values after 12 years of in site exposure, also accounting for the survey performed in 2005.

The predicted service life reflects exceeding the threshold value C_T with a failure probability that equals $P_f=0.5$ at a depth of $x=12$ cm from the concrete exposure surface, starting from the initial exposure at completion of the structure in 1993. The main concrete reinforcement bars are located at this depth from the exposed surface. Remark that after this period, the structure will not collapse. The predicted service life represents the probability for the chloride ions to reach the reinforcement bars with sufficient concentration of $0.7\% \text{Cl}^-/\text{cem}$ and has to be interpreted as the possible start of the corrosion process or the end of the initiation period of the concrete deterioration process.

It is remarkable that the diffusion coefficient in the non-treated locations is nearly an order of magnitude higher than in the treated locations. Further more, the mean value of the diffusion coefficient ($\mu(D)$) remains relatively constant, which is also reflected in the calculated service life. On the other hand, the standard deviation seems to increase ($\sigma(D)$). This is mainly related to the complexity of gathering the experimental chloride concentration data. Overall, this indicates that for the moment, after 12 years of in site exposure, possible degradation of the applied protection system is not significantly observed from the obtained results.

Table 4: Diffusion coefficient D – parameters of lognormal distribution and predicted service life after 5 and 12 years of exposure respectively

Location	Based on 1996 and 1998 survey data			Based on 1996, 1998 and 2005 survey data		
	$\mu(D)$ [cm ² /s]x10 ⁻⁸	$\sigma(D)$ [cm ² /s]x10 ⁻⁸	Service life [y] C _T =0.7% Cl/cem	$\mu(D)$ [cm ² /s]x10 ⁻⁸	$\sigma(D)$ [cm ² /s]x10 ⁻⁸	Service life [y] C _T =0.7% Cl/cem
A (non-treated)	9.64	7.0	12.6	9.58	10.55	16.5
B (treated)	1.18	1.0	105	1.61	2.49	107
C (treated-on top of quay-wall)	1.32	1.7	123	2.13	3.46	91

4 CONCLUSIONS

An in-service test program and a service life prediction method, based on a time-dependent reliability method, are presented. The in-service test program - executed after 3, 5 and 12 years of in-service exposure, results in an important long-term data-set of experimental values on in site chloride concentration in a reinforced concrete structure. This paper contributes to the idea of increasing the durability of concrete by means of a preventive hydrophobic treatment against chloride ingress. Based on long-term in site data of chloride concentration in the reinforced concrete structure, an objective judgment of the effectiveness could be established. The comparative chloride profiles of a treated and non-treated location, demonstrate the effectiveness of the highly concentrated, solvent-free compound based on alkyltriethoxysilane as a water-repellent agent. Although the used time dependent 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.

ACKNOWLEDGEMENT

The financial support for the post-doctoral research position from Research Fund KULeuven is gratefully acknowledged.

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