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Concrete durability improvement in the presence of chlorides using silane based hydrophobic impregnating agents

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SUMMARY: The use of hydrophobic impregnating agent is a well-known and proven technique to prevent chloride ingress into concrete. The aim of this study is to show the effect of a silane based hydrophobic impregnating agent with regards to chloride penetration when different concrete mixes (XS1, XS2 and XS3 of EN 206-1) are subjected to chloride attack. Methods using forced current are compared to a ponding method. The results show that using forced current, the performance improvement scatter is larger than the fluctuations of the ponding test results. Nevertheless, they clearly show the beneficial effect brought by the use of hydrophobic impregnation in order to protect concrete structures against chloride attack.

KEY-WORDS: hydrophobic impregnation, chlorides,

INTRODUCTION

Applying hydrophobic impregnating agents to the surface of a concrete structure is a well-documented and accepted method to delay the migration of chloride into concrete.

Numerous papers, such as [1-3] to mention only a few, have been published with regard to the efficiency of hydrophobic impregnating agents in reducing chloride migration into concrete.

In Europe, the concrete mix shall be designed according to the exposure class - defined in EN 206 part 1. The standard provides relevant information for a typical structure design life of 50 years.

The aim of this study is to compare different concrete mix designs, both treated and untreated with hydrophobic impregnating agents, using various chloride test methods, such as induced current and traditional ponding, in order to evaluate the influence of hydrophobic treatments with regard to the durability of concrete.

DEFINITIONS ACCORDING TO EN 206-1 STANDARD

EN 206-1 [4] defines the different classes of exposure according to the environment of the structure, as shown in Table 1.

Table 1: Exposures classes (extract from EN 206-1:2000) for corrosion induced by chlorides from sea water

Class Designation	**Description of the environment**	**Informative examples where exposure classes may occur**
XS 1	Exposed to airborne salt but not in direct contact with sea water	Structures near to or on the coast
XS 2	Permanently submerged	Parts of marine structures
XS 3	Tidal, splash and spray zones	Parts of marine structures
Where concrete containing reinforcement or other embedded metal is subject to contact with chlorides from sea water or air carrying salt originating from sea water, the exposure shall be classified as above:		

This standard also defines the concrete mix design according to the different classes of exposure, as shown in Table 2.

Table 2: Basic requirements of the mix design based on EN 206-1, Table F1

	Chloride-induced corrosion (from sea water)		
Exposure class	**XS1**	**XS2**	**XS3**
Maximum w/c ratio	0.50	0.50	0.45
Minimum strength class	C30/37	C35/45	C35/45
Minimum cement content (kg/m^3)	300	320	340

CONCRETE MIX DESIGNS AND CHARACTERISATION

Table 3 shows the various concrete mixes used in the study.

Table 3: Concrete mix design and characterisation.

	Control	**XS1**	**XS2**	**XS3**
Cement type	CEM II/A-LL 42.5 N	CEM III/B 42.5 N (ENCl)	CEM III/B 42.5 N (ENCl)	CEM III/B 42.5 N (ENCl)
Cement content	320	300	320	340
Aggregates (mm)	0-32	0-32	0-32	0-32
Superplasticizer (carboxylate type) (% w/w)	0.25	0.4	0.3	0.52
W/C ratio	0.53	0.50	0.50	0.45
Density (kg/m^3)	2421	2434	2415	2440
Air content (%)	1.2	1.2	1.2	1.2
Workability test				
Flow table spread (mm)	400	420	410	420
Slump (mm)	70	150	150	180
Compactability	1.05	1.07	1.07	1.05
28-day compressive strength (N/mm^2)	41.1	50.0	43.6	54.5
Dry density (kg/m^3)	2420	2430	2430	2420
Water conductivity (g/m^2)	14.1	11.1	11.9	9.2
LP (%)	1.2	1.1	1.3	0.50
Porosity (%)	4.8	3.5	4.1	3.0

SAMPLE PREPARATION AND HYDROPHOBIC TREATMENT

For all experiments, cubes of 150 mm side length were produced. The cubes were prepared according to EN 12390-2, and stored for 28 days after casting at 20°C / 95% relative humidity.

After humid storage, the cubes were stored at 20°C / 60 % RH until they are older than 90 days from their preparation. Then cores with the required dimensions for the various tests were taken and left to dry at 20°C / 60% RH.

Application of the hydrophobic agent (pure triethoxy(2,4,4-trimethylpentyl) silane with 99% active content) was carried out by total immersion for 60 seconds.

Preliminary tests were conducted to determine the number of necessary immersion per concrete mix design to reach an application of 180-200 g/m^2 of the hydrophobic impregnation agent. With two to three immersions per sample, this amount could be achieved in each case and the penetration depth obtained ranged from 2.5 to 4.5 mm.

After application, the core samples were stored again at 20°C / 60 % RH for at least three weeks. Subsequently, the tests were carried out on both untreated reference and the treated (hydrophobic) samples for comparison using the four different procedures described below.

TEST METHODS

SIA 262/1

The application of a voltage in concrete specimens saturated with water activates the diffusion of chloride ions. The measure of the penetration depth of the chloride ions allows calculating the chloride migration coefficient on the basis of penetration depth, applied voltage - as well as other parameters (Figure 1).

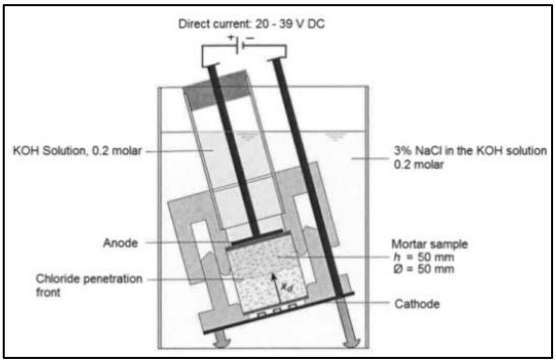


Figure 1. Schematic drawing of the SIA 262/1 migration cell

Catholyte solution:	3% by mass of NaCl in 0.2 M KOH solution (30 g NaCl in 970 g of 0.2 M KOH solution)
Anolyte solution:	0.2 M KOH solution (11.22 KOH per liter of deionised water)
Applied voltage:	20 Volts
Duration:	24 hours

ASTM C1202-12

"This test method consists of monitoring the amount of electrical current passed through 50 mm thick slices of 100 mm nominal diameter cores or cylinders during a 6-h period. A potential difference of 60 V DC is maintained across the ends of the specimen. One specimen is immersed in a sodium chloride solution, the other in a sodium hydroxide solution. The total charge passed, in coulombs, has been found to be related to the resistance of the specimen to chloride ion penetration."

Figure 2. The ASTM C 1202-12 setup

Chloride solution: 3% by mass of NaCl solution (30 g NaCl per 970 g of deionised water)

Sodium hydroxide solution: 0.3 M in distilled water (12 g of NaOH/liter deionised water)

Applied voltage: 60 Volts

Duration: 6 hours

NT Build 492

"An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration, the specimen is axially split and a 0.1 M silver nitrate solution is sprayed onto one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation. The chloride migration coefficient can be calculated from the obtained value of penetration depth."

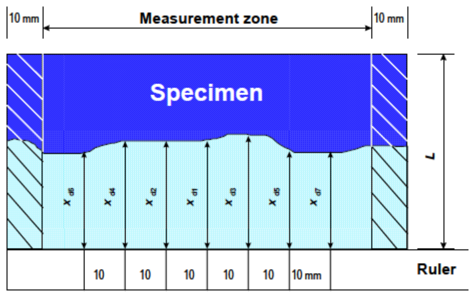


Figure 3: Illustration of the set-up for the measurement of chloride penetration depth.

Catholyte solution: 10% by mass of NaCl solution (100 g of NaCl in 900 g of water)

Anolyte solution: 0.3 M NaOH in distilled water (~12 g NaOH in 1 litre water)

Applied voltage: 60 Volts

Duration: 30 hours

ASTM C1543-10a

"A sodium chloride solution is ponded on the surface of concrete specimens. Samples from specified depths are periodically extracted and chemically analysed to determine the chloride content of the concrete at those depths."

Chloride solution: 3% by mass of NaCl solution

Ponding head: 15 mm ± 5 mm

Ponding period: Initially 3 months; subsequent after 6, 12 months and interval of 12 months thereafter

RESULTS

Table 4 presents the results obtained for the four test methods.

Table 4. Results obtained for the four tests.

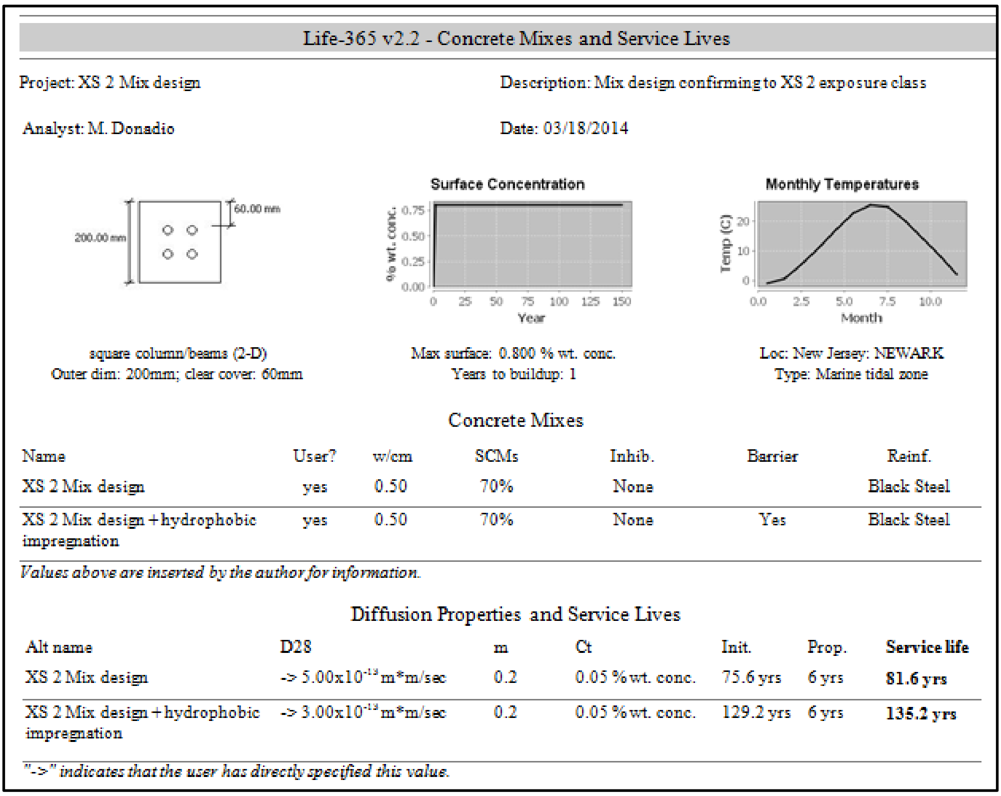
	Control	XS1	XS2	XS3
Chloride resistance as per SIA 262/1 (chloride migration coefficient)				
Untreated	13.1 x10^{-12}	2.8 x10^{-12}	0.5 x10^{-12}	0.9 x10^{-12}
Treated with hydrophobic agent	1.2 x10^{-12}	1.7 x10^{-12}	0.3 x10^{-12}	0.6 x10^{-12}
Reduction of chloride penetration	**91%**	**39%**	**40%**	**33%**
Chloride ion permeability as per ASTM C 1202 (charge passed in Coulomb)				
Untreated	2158	432	500	334
Treated with hydrophobic agent	355	154	455	120
Reduction of chloride penetration	**83%**	**64%**	**10%**	**64%**
Chloride ion permeability as per NT Build 492 (chloride migration coefficient)				
Untreated	8.8 x10^{-12}	2.9 x10^{-12}	0.5 x10^{-12}	3.0 x10^{-12}
Treated with hydrophobic agent	1.5 x10^{-12}	1.6 x10^{-12}	0.3 x10^{-12}	1.6 x10^{-12}
Reduction of chloride penetration	**88%**	**45%**	**40%**	**28%**
Chloride ion penetration as per ASTM C 1543-10a (chloride profile)				
After 180 days of ponding:				
Untreated (in % w/w of cement) 0 - 10 mm 10 - 20 mm 25 - 35 mm 40 - 50 mm 55 - 65 mm	 0.80 0.54 0.33 0.17 0.14	 1.15 0.12 0.12 0.09	 0.84 0.09 0.08 0.07	 1.02 0.09 0.12 0.10 0.09
Treated (in % cement) 0 - 10 mm 10 - 20 mm 25 - 35 mm 40 - 50 mm	 0.14 0.04 0.06 0.08	 0.22 0.07 0.07	 0.24 0.06 0.10 0.08	 0.20 0.05 0.04
Reduction of chloride penetration				
0 - 10 mm	**82%**	**81%**	**71%**	**80%**

SERVICE LIFE ESTIMATION – LIFE-365™ V2.2

The **Life-365™ V2.2** program is a software designed to estimate service life of concrete structure in different typical environments. The program was written by M. A. Ehlen under contract to the Life-365 Consortium II which consist of various chemical, reinforcement bars and raw material suppliers. The program can be downloaded at the following link: www.life-365.org/.

We used this software and input the results of the NT Build 492 analysis above in order to estimate the service life of a structure using the different mix designs presented in this paper.

The software used the US coast line for the environmental conditions – as an example of durability improvement, the inserted data for the State of New Jersey and the city of Newark were used. A 60 mm concrete cover was selected and the concrete element chosen for the analysis is a square column (200 mm). Depending on the mix design (Control, XS1, XS2 or XS3), the software allows calculating the life expectation according to the location of the structure (in the tidal zone, near the sea or away from it…).An example of a report using the Life-365™ program is shown in the Figure 4 below for the XS2 mix design.



Life-365 v2.2 - Concrete Mixes and Service Lives

Project: XS 2 Mix design

Description: Mix design confirming to XS 2 exposure class

Analyst: M. Donadio

Date: 03/18/2014

square column/beams (2-D)
Outer dim: 200mm; clear cover: 60mm

Max surface: 0.800 % wt. conc.
Years to buildup: 1

Loc: New Jersey: NEWARK
Type: Marine tidal zone

Concrete Mixes

Name	User?	w/cm	SCMs	Inhib.	Barrier	Reinf.
XS 2 Mix design	yes	0.50	70%	None		Black Steel
XS 2 Mix design + hydrophobic impregnation	yes	0.50	70%	None	Yes	Black Steel

Values above are inserted by the author for information.

Diffusion Properties and Service Lives

Alt name	D28	m	Ct	Init.	Prop.	Service life
XS 2 Mix design	-> 5.00×10^{-13} m*m/sec	0.2	0.05 % wt. conc.	75.6 yrs	6 yrs	**81.6 yrs**
XS 2 Mix design + hydrophobic impregnation	-> 3.00×10^{-13} m*m/sec	0.2	0.05 % wt. conc.	129.2 yrs	6 yrs	**135.2 yrs**

"->" indicates that the user has directly specified this value.

Figure 4. Example of Life-365 program

DISCUSSION

The results of the XS3 mix appear abnormal; despite higher cement content and a lower water to cement ratio compared to the mix design XS2, the results from all migration tests, except the ASTM C 1202, are slightly higher.

Nonetheless, these results are relevant as apart from the XS3 mix, the values of chloride migration or permeability are in relation to the mix design used.

Using a normal OPC with limestone additive (CEM II/ALL) in the control mixes gives systematically a high chloride migration and permeability.

The use of CEM III/B with high slag content confirms the good behaviour of this cement in marine environment as even without further treatment, a relative low chloride migration or permeability is achieved for the 3 mixes tested (XS1, XS2 and XS3) containing this cement.

We noticed that a surface treatment —such as a silane impregnation— further reduces the permeability as well as the migration of chloride.

We can clearly see that the results obtained from the tests using induced current show a rather large scatter (reduction of chloride migration between treated and untreated concrete in the range from 10 to 91%). When using the ponding method, the reduction of chloride penetration is more consistent (ranging from 71 to 82%).

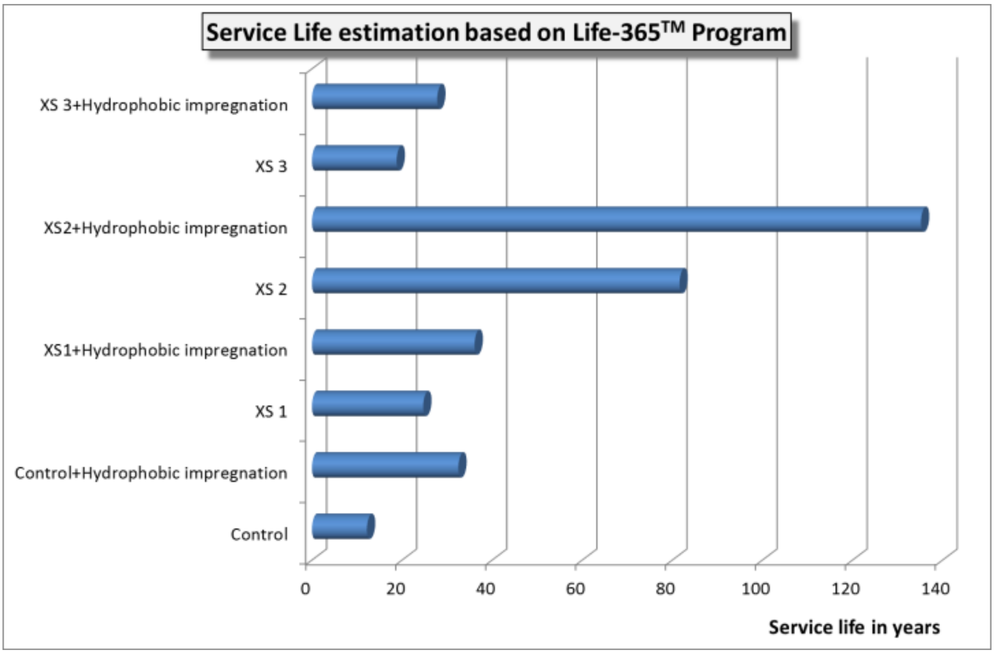


Figure 4. Service life improvement using hydrophobic impregnation

Chlorides migrate into concrete via two main mechanisms: in solution with the ingress of water due to capillary action and as ion diffusion.

Hydrophobic impregnations act by preventing water absorption by capillary action (modification of surface tension). This is reflected in the ponding method and could explain the consistency of the reduction of the chloride ingress. On the other hand, induced current activates the migration of chloride by diffusion thus explaining the larger scatter of results.

Using the **Life-365^{TM}** program to estimate the design life of a structure clearly demonstrates that the use of a surface treatment significantly increases the life expectation (even if the results achieved with the surface treatment do not entirely reflect their effectiveness). The improvement of the service life using hydrophobic impregnation ranges from 46 to 166%.

Apart from the benefits brought by the initial reduction of the chloride migration, hydrophobic impregnations will further improve the protection of the structure IF they are applied after surface shrinkage cracks develop by preventing chloride migration via these new cracks paths.

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