CHALMERS





Performance of impregnation of concrete structures – Results from a 5-year field study at RV40 Borås

Master of Science Thesis in the Master's Programme Design and Construction Project Management

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Department of Civil and Environmental Engineering Division of Building Technology CHALMERS UNIVERSITY OF TECHNOLOGY Göteborg, Sweden 2015 Master's Thesis 2015:152

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Cover

Water drop on the impregnated concrete surface (photo by Oskar Malaga).

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ABSTRACT

Concrete is the most widely used construction material in the world. It is one of the most durable building materials and possesses properties such as high compressive strength and low permeability. Hydrophobic impregnations often referred to as water repellent agents, today mainly consisting of alkylalkoxysilanes, are often used on concrete to prolong the service life of the structure. This is accomplished by protecting the reinforcement bars from chlorides or by changing the moisture content inside. When the concrete is treated with a water repellent agent the properties of the surface layer becomes hydrophobic and thereby water droplets are stopped from entering, still allowing water vapour to pass through.

On behalf of the Swedish Transport Administration (TRV), the Swedish Cement and Concrete Research Institute (CBI) was consulted to investigate the effects of two commercial impregnation agents, applied on four different types of concrete and exposed during 5 years in the vicinity of Swedish national road RV40 between Gothenburg and Borås.

Results from the master thesis indicate reduced chloride content in concrete after hydrophobic surface treatment. No carbonation was detected on any sample. Moreover one of the two hydrophobic agents was consistently more efficient, most likely due to the gel based consistency.

Key words: hydrophobic impregnation, carbonation, chloride penetration, concrete, silane, surface treatment

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Preface

In this study, laboratory tests on 5-year-old field exposed concrete samples were carried out regarding hydrophobic impregnation penetration, chloride resistance, carbonation, ultrasonic velocity test, water absorption and microscopy analysis. The tests have been carried out from the fall of 2013 to the fall of 2014. The work is a follow-up of a research project investigating hydrophobic impregnation impact on frost and chloride resistance of field exposed concrete samples. The project was carried out at the Department of Construction Management, Chalmers University of Technology, Sweden and financed by the CBI Swedish Cement and Concrete Research Institute.

All tests have been carried out in the laboratory of the CBI Swedish Cement and Concrete Research Institute in Borås. I would like to thank my supervisors Nelson Da Silva and Elisabeth Helsing for guidance and planning throughout the work. From the academic side I would like to thank my supervisor Professor Tang Luping from the department of Civil and Environmental Engineering, Building Technology at Chalmers University of Technology. He has been very supportive throughout the thesis, with constructive input to help me achieve the best possible result.

Göteborg, December 2015

Oskar Malaga

1 Introduction

1.1 Background

Concrete is the most widely used construction material in the world. It is one of the most durable building materials and possesses properties such as high compressive strength and low permeability. Because of its low tensile strength, concrete is often reinforced with steel bars and fibres. The reinforcement in concrete is the most sensitive element resulting in corrosion.

Every year, the Swedish Transport Administration (TRV) is treating surfaces of both new and old concrete structures with hydrophobic agents, although scientific results concerning the impregnation agents' effectivity over a long time exposure are scarce.

The purpose of surface treatment of concrete structures such as bridges is to increase the resistance against ingress of water and chlorides, which may lead to concrete damage such as frost induced scalling and chloride induced corrosion of the reinforcement.

There are a few studies describing the effectivity of the concrete treatment, especially for modern concrete mixes with dense cover layer. Self-compacting concrete, concrete with low water cement ratio and concrete with fillers consisting of fine material provide a denser concrete structure and the question is if any hydrophobic treatment is necessary for dense concrete. Additionally, some research studies indicate that due to carbonation, natural densification of the pore structure at the surface layer occurs (Utgenannt 2005). On behalf of TRV, the Swedish Cement and Concrete Research Institute (CBI) was consulted to investigate the effects of two commercial impregnation agents, applied on four different types of concrete and exposed during 5 years in the vicinity of highway RV40 between Gothenburg and Borås, Sweden.

1.2 Aim

The aim of the study is to evaluate the long-term impact of two of the most frequently used hydrophobic impregnation agents for concrete in Scandinavia and to evaluate on which basis the agents are chosen by the industry and/or users.

1.3 Limitations

The evaluation of long-term efficiency of concrete impregnation will be performed on the basis of the test results of chloride penetration depth, frost resistance, carbonation, ultrasonic velocity and optical microscopy.

1.4 Method

The work will be based on the following steps:

- State-of-the-art a theoretical study will be performed and the latest level of knowledge will be presented.
- Laboratory tests preparation of samples, visual observations, physical tests, non-destructive and destructive testing, performed in the CBI laboratory.
- Analysis of data and results
- Discussion and conclusions

2 State-of-the-art

2.1 Hydrophobic impregnation agents

Hydrophobic impregnation agents are frequently applied on concrete structures. The primary goal of the impregnation in Sweden is to protect the reinforcement against chloride ingress from de-icing salts. Chloride transport into the concrete is prevented by giving the surface hydrophobic properties; in this way, the service life of a concrete structure can be prolonged.

2.2 Chemistry

Available on the market water repellent agents applied on concrete are primarily silane based. Silanes, properly named alkylalkoxysilanes, are small molecules and can be transported into concrete due to capillary suction. Silanes are created by reactions between silicon, alkyl groups and alkoxy groups. There are four possible reaction links for the silicon atom. Three of them react with alkoxy groups in present of water and bind other silanes nearby. What is unclear, is the degree of linkage between the pore walls of the concrete and the chemical endproduct. Furthermore the endproduct results in a fine network, (polymer siloxan (A) / silicon resin (B)) as illustrated in Figure 1. Moreover the forth link in the molecule consists of an alkyl group which is not reacting, but providing the hydrophobic (water repellent) effect of the impregnation (Selander A. , 2010).

In Sweden, concrete has been treated with silane coatings since the late 1980s. In the USA, research on silanes was already started in the 1940s, although the main objective was not to create a more durable concrete but for military applications. Besides the high interest about composite materials at this time, a need for strong and sustainable bonding arosed. Due to this need, silanes were analyzed in order to find out more about the ability to create persistent bonding between inorganic and organic materials (Plueddemann, 1991). Hydrophobicity is created when the inorganic surface of the concrete is bond with alkylgroups (paraffins), (Arkles, 1977). There are mainly three impregnants containing silanes, applied on concrete in Sweden. They only differentiate each other in size of the alkyl groups (Selander A., 2010).

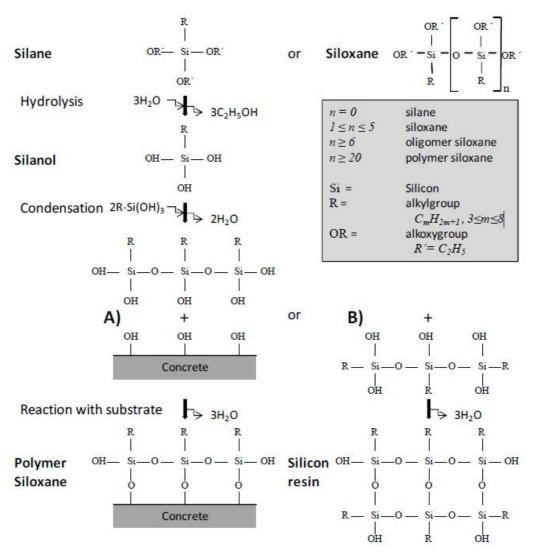


Figure 1 Reaction of an organofunctional triethoxysilane with the concrete matrix, based on. Ethanol is liberated during the hydrolysis. A) and B) represent two different scenarios whereas a fine network of polymer siloxane on the pore walls forms(A) or a silicon resin(B), (Selander A., 2010).

The size of alkyl group and alkoxy group is of significant matter; studies indicate differences in reaction kinetics, where methoxy groups react faster than ethoxy groups and that a big alkyl group slows down the reaction of the alkoxy group as well (De Clercq, 2001). These differences are created due to the variances of molecule properties such as energy and geometry of the molecular orbital (Gerdes, 2005).

2.3 Function

The water repellent surface of lotus leaf and flower is so called self-cleaning. Water drops completely roll of the leaf, carrying small particles off – also referred as the lotus effect (Neinhuis, 1997). The lotus effect is illustrated in figure 2 and it is an illustration of how water repellent agents function. It is depending on the contact angle between water drops and any given material. For concrete the contact angle is considered to be zero. In this case, a fine system of pores provides the right conditions for capillary suction when water is applied on the concrete surface. In addition, together with the transported water, ions are carried into the concrete. For instance chloride ions which are the focus of this report.

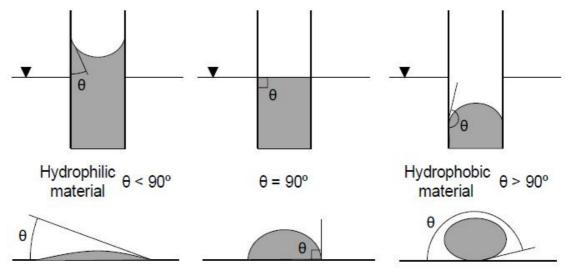


Figure 2 Difference between hydrophilic and hydrophobic material illustrated by contact angle.

By treating concrete with a hydrophobic agent, the surface layer becomes sealed against water. Meanwhile vapour diffusion becomes the only way for water to penetrate the concrete. In some cases this can function to lower the moisture content, for example when rain is unable to sink in and at the same time water exits through diffusion during hot temperatures (Johansson, 2008).

2.4 Environmental risks

The first generation of silanes did possess significant environmental and health risks, both in terms of the silanes and the organic solvents in the agent. Besides the environmental aspects, volatile organic compound (VOC) emissions contribute to potential health risks. The reaction mechanisms for methoxy silanes and ethoxy silanes produce methanol and ethanol, both of which are considered as environmental risks. First generation silanes are typically classified with: moderate skin irritation, severe eye irritation and respirotary irritation. Although intense toxicity is not most likely, not even after ingestion or dermal exposure.

Risks in terms of skin and eye irritation or damage to vegetation are potential risks grounded on solvent based or pure silanes. Further risk classifications are presented in table 1.

Safety recommendations are mostly similar between different manufacturers, such as the use of gloves, eye protection and avoidance of inhalation or ingestion of the materials (A Calder, 2009).

Table 1 Health and safety, and environmental risks (A Calder, 2009)

Product name	Identified risks
	Category 1: Solvent based silanes/siloxanes
1.1 Classed as irritant: skin	
1.2	Harmful to aquatic organisms
	Category 2: Water based silanes/siloxanes
2.1	No specific risk phases identified
2.2	Classed as irritant: skin; inhibitor is harmful by inhalation, ingestion or skin contact but is <5 percent of product
No specific risk phases identified; vegetation should be protection overspray	
2.4 No specific risk phases identified	
2.5 Classed as irritant: skin, may lead to sensitization	
2.6	No specific risk phases identified: vegetation should be protected from overspray
2.7	No specific risk phases identified; however does contain 1 percent methanol
	Category 3: Cream based silanes/siloxanes
3.1	Classed as irritant: skin
3.2	Harmful to aquatic organisms
3.3	Classed as irritant: skin, may lead to sensitization
	Category 4: Crystal growth pore-blockers
4.1	No specific risk phases identified
4.2	No specific risk phases identified; can damage vegetation

2.5 Penetration depth and efficiency

In order to be effective, the impregnation agent needs to penetrate the concrete pore structure to a certain depth. A badly impregnated surface suffers of a weak function and limited effect. The total protective effect is also decreasing faster. Moreover, there are three important factors in order to achieve effective penetration depth. These are: the concrete porosity, the contact time between impregnation agent and concrete (where capillary suction is active at the concrete surface) and the degree of water saturation in the pore system. High porosity, low degree of water saturation and long contact time

provide optimal conditions for higher penetration depth. The difference in porosity and moisture decides how much of the space is available for impregnation agent and contact time decides how deep the impregnation goes in to the concrete. The correlation between these three factors is illustrated in figure 3 (Selander A., 2009).

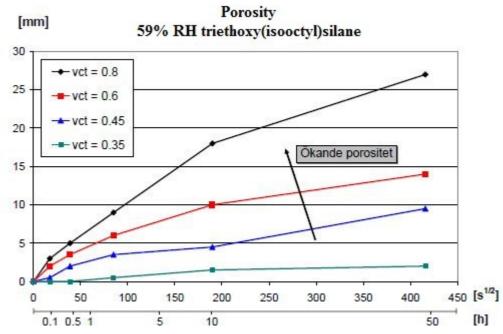


Figure 3 Influence of porosity and time on the effective penetration depth (Selander A., 2010)

There is no single recipe to achieve good effects; it varies depending on which quality of concrete is treated and what it has been exposed to, but also local varieties where some elements need higher concentrations of impregnation. The function of the surface is determined by the quality and concentration of the impregnation agent. Furthermore the quality is linked to the impregnation depth which is illustrated in figure 4. Moreover research has been made on relations between water-cement ratio and relative humidity. Results from these studies showed that the most efficient correlation is for the w/c = 0.70, RH = 65%) and least efficient for the w/c = 0.40, RH = 90% (Selander A. , 2010).

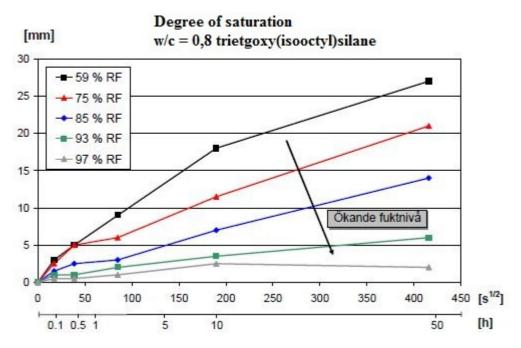


Figure 4 Influence of saturation and time on the effective penetration depth (Selander A., 2010)

2.6 Experiences

Surface treatment was studied since the 1970s in the USA. Studies have been made concerning bridge constructions treated with polymers, where the objective was to investigate corrosion of reinforcement bars in the bridge deck slab. Results were obtained from two bridges, Bethlehem 1975 and Boalsburg 1985, both located in Pennsylvania. The surface was treated with methyl methacrylate and applied at chosen areas. Results from Boalsburg point out a slower penetration of chlorides where the impregnation was applied. The highest differences after nine years were measured at a depth of 25-39 mm, with 40% lower chloride content. When looking at corrosion, results stated a 2.5 times faster corrosion rate in the reference concrete. The rate was even faster in Bethlehem, 10 times faster (Powell, 1993).

Promising results with impregnated concrete structures has been observed in Stockholm, where the main objective was to protect concrete against chlorides and water ingress. The penetration depth of the impregnation was assumed as a key factor for the function of impregnation (Karlsson, 1997).

Though many reports prove the fact of positive experience regarding concrete quality after surface treatment, there are many questions to be answered. Already at the CBI information day 2013, the question if impregnation of construction concrete make sense has been conferred (Olsson, 2002).

2.7 Regulations

According to (Helsing, 2014), until 2009 all edge beams in Sweden had to be impregnated. However, the current Swedish building legislation does not define any demand for impregnation of constructions. This does not mean that application of impregnations is excluded in new constructions, but rather that it is up to the project leader to specify its use. In addition, there are no public procurements concerning impregnation agents, meaning that the contractors are free to choose any agent suitable for the job (Helsing, 2014).

Regarding impregnation agents, the only requirements which are allowed to be given from the Swedish Transport Administration are in case the project should follow EN 1504-2. Since EN 1504-2 is a harmonized European standard all impregnating agents need to be CE certified. EN 1504-2 (table 2) is covering surface protection systems for concrete. Moreover there is nothing mentioned about ingress of chlorides in the standard. That is why the Swedish AMA 13 (a general description of material and works) is referring to EN 1504-2 and including an additional testing method regarding chloride ingress. The first AMA came out in 2007 and therefore projects started before 2007 applied regulations from Bro 2004 (SIS, 2004) (Vägverket, 2004) (Byggtjänst, 2013).

At the course "Water repellent impregnations", given by the CBI in August 2014, Kenth Jansson (Jansson, 2014) who was giving one lecture claimed that the most significant factor when choosing an impregnation agent is mainly dependent on the contractors experience with impregnation agents. While unexperienced contractors often went with the cheapest products without knowledge and routines for application, more experienced contractors could identify where different agents were most suitable. Not meaning that the cheapest products on the market gave lowest effects (Jansson, 2014).

One Swedish company selling impregnation agents which were attending the course had started to make official price lists of their agents to ease the process of ordering and selection for the contractors (CBI, 2014).

The majority of the course attendants did agree upon the lack of interest from the contractors' side regarding impregnations and claimed that the workmanship of impregnation is not prioritized in the field (CBI, 2014). This might have serious effects on the durability of concrete structures exposed to de-icing salts and on the total maintanance cost.

Table 3 — Performance requirements for hydrophobic impregnation

Loss of mass after freeze-thaw-salt stress This test is only necessary for structures which may come in contact with de-lcing salts. This test is only necessary for structures which may come in contact with de-lcing salts. Depth of penetration measured on 100 mm concrete test occurrence of 5.5 mm by breaking open the treated specimen and spraying the fracture surface with water (using the procedure given in EN 13579). The depth of photoic agent shall be stored according to the dry phenolophthalein) according to prevent and test test occurrence to alkali microsoft according to the dry occurrence occurrence occurrence or 13579. EN 13579 Drying rate coefficient EN 13579 EN 13580 EN 13579 EN 13579 EN 13579	No. of Table 1	1 Performance Characteristics	Test method	Requirements
Loss of mass after freeze-thaw-salt stress This test is only necessary for structures which may come in contact with de-icing salts. Depth of penetration measured on 100 mm concrete test cubes C (0,70) according to EN 1766 (not C (0,45) as given in EN 13579). After 28 days of curing according to procedure given in EN 13579). After 28 days of curing according to prevent in EN 13579. Drying rate coefficient EN 13579 EN 13579 EN 13579 EN 13579	1	2	3	4
Depth of penetration measured on 100 mm concrete test cubes C (0,70) according to EN 1766 (not C (0,45) as given in EN 13579). After 28 days of curing according to the dry phobic agent shall be stored according to the dry phobic agent shall be in accordance to EN 13579. The depth of penetration is measured with an accuracy of 0,5 mm by breaking open the treated specimen and spraying the fracture surface with water (using the EN 1766, the samples shall be stored according to the dry phobic agent shall be in accordance to EN 13579. Water absorption and resistance to alkali EN 13580 EN 13579	17	Loss of mass after freeze-thaw-salt stress This test is only necessary for structures which may come in contact with de-icing salts.	EN 13581	The loss of mass of the surfar of the impregnated specimen must occur at least 20 cycles later than that of the not impregnated specimen.
Water absorption and resistance to alkali EN 13580 Drying rate coefficient EN 13579	19	Depth of penetration measured on 100 mm concrete test cubes C (0,70) according to EN 1766 (not C (0,45) as given in EN 13579). After 28 days of curing according to EN 1766, the samples shall be stored according to the dry procedure given in EN 1766. The treatment with hydrophobic agent shall be in accordance to EN 13579.	The depth of penetration is measured with an accuracy of 0,5 mm by breaking open the treated specimen and spraying the fracture surface with water (using the phenolphthalein test method with water instead of phenolphthalein) according to prEN 14630. The depth of the dry zone is taken as the effective depth of hydrophobic impregnation.	class II: ≥ 10 mm
Drying rate coefficient EN 13579	23	Water absorption and resistance to alkali	EN 13580	Absorption ratio <7,5%, compared with the untreated specmen Absorption ratio (after immersion in alkali solution) <10 %.
Principle of the land of the second of the s	24	Drying rate coefficient	EN 13579	class I: > 30 %
Diffusion of chloride lons *	25	Diffusion of chloride ions a	subject to national standards and national regulations	

3 Description of materials and methods

3.1 Surface treatment agents

The following two silane based impregnation agents have been used:

- StoCryl HG 200, cream from Sto, application 500 g/m²
- DC 4341, liquid from Dow Corning, application 300 g/m²

These products where selected for this study due to their frequent use in Sweden. They will be referred to as HG and DC in this report.

3.2 Manufacturing of samples

Four different types of concrete were produced for laboratory and field exposure in 2008:

- Self-compacting concrete (w/c 0.45);
- Conventional concrete (w/c 0.45 without air entrainment agent);
- Conventional concrete (w/c 0.45 with air entrainment agent);
- Conventional concrete (w/c 0.70).

Plastic forms, type C232 were used for casting of approximately 200 specimens with dimensions 100 mm x 100 mm x 100 mm (figure 5).



Figure 5 Marked specimens in the conditioning room

In table 3 the concrete recipes are presented.

Table 3 Concrete recipes

		Self-con	pacting co	ncrete (w	/c 0.45)		
CEM 52.5 R	Aggregates	Aggregates	Limestone	Water	Superplast iciser		
(kg/m ³)	0-8	16-aug	Filler	(kg/m³)	(wt.% CEM)		
	(kg/m ³)	(kg/m ³)	(kg/m ³)				
430	789	827	110	194	1.0*		
		Conve	ntional con	crete (w/	c 0.45)		
CEM 52.5 R	Aggregates	Aggregates	Aggregates	Water	Superplast iciser		
(kg/m ³)	0-8	04-feb	16-aug	(kg/m³)	(wt.% CEM)		
	(kg/m ³)	(kg/m ³)	(kg/m ³)				
375	648	492	705	169	0.5*		
	Con	ventional	concrete w	ith added	air (w/c 0.4	5)	
CEM 52.5 R	Aggregates	Aggregates	Aggregates	Water	Superplast iciser	AEA	Air
(kg/m ³)	0-8	04-feb	16-aug	(kg/m ³)	(wt.% CEM)	(wt.% CEM)	(%vol.)
	(kg/m ³)	(kg/m ³)	(kg/m ³)			1.00	
375	526	561	677	169	0.7	0.015	4.5
		Conve	ntional con	crete (w/	c 0.70)		
CEM 52.5 R	Aggregates	Aggregates	Aggregates	Water			
(kg/m ³)	0-8	04-feb	16-aug	(kg/m ³)			
	(kg/m³)	(kg/m ³)	(kg/m³)				
275	655	499	713	193			
AEA = Air Er	ntraining A	gent					
*estimated							

3.3 Application of surface treatment

The cube shaped samples were conditioned for six weeks at 20° C and 50% RH. After that, the surface treatment was applied on all sides of the cubes according to the recommendations given by Sto (HG) and Down Corning (DC) and then conditioned for additional three weeks (figure 6).



Figure 6 Application of surface treatment

3.4 Field exposure

Samples were organized in steel frameworks, each carrying 66 cubes. The frameworks were placed in the field along highway 40 (RV 40) outside of Borås in December 2008 (figure 7).



Figure 7 Specimen at the field exposure site (left), and collecting of the samples (right) from RV 40, autumn 2013

3.5 Laboratory testing

All testing has been executed at the CBI:s laboratory in Borås, Sweden. After visual inspection, the following tests were performed: penetration depth of the impregnation agent, chloride content, carbonation depth, ultrasonic velocity measurements, water absorption and drying, contact angle and microscopy analysis.

3.5.1 Visual inspection

In September 2013, all field exposed samples were collected and brought to CBI for visual inspection, where the conditions of the samples after approximately five years of field exposure were assessed (figure 8).



Figure 8 Collected specimens from RV 40 after approximately five years of field exposure

3.5.2 Penetration depth of the impregnation agent

The impregnation depth was determined by splitting samples followed by spraying the split surfaces with water in accordance with EN 14630 (EN14630:2006). The analysis was performed by measuring the distance from each surface to the point where no hydrophobic effect was visible. The hydrophobic surface was distinguished by the dry surface with a lighter colour (figure 9).



Figure 9 Specimen after being water sprayed. A visible rim of dry surface surrounds the sample.

3.5.3 **Drop method**

Another method used for investigation of the penetration depth was the "drop method" in which powder samples were exposed to water by gradual application using a pipette (figure 10). The interaction between the water droplet and the powder was then ocularly

examined (figure 11), and the samples were classified as either hydrophobic or hydrophilic. This test method is typically used at the Statens vegvesen (Norwegian Public Road Administration) and recommended by Eva Rodum (Rodum, 2014).



Figure 10 Measuring hydrophobicity by the dropp method on the powder samples.



Figure 11 Detailed pictures of measurement of hydrophobicity Middle picture shows a hydrophilic effect where water is absorbed into the powder. Both pictures on the sides are showing a hydrophobic effect with clearly visible water bubbles.

3.5.4 Carbonation

Carbonation was determined using a similar procedure as that used to measure the penetration depth; the samples were split and the reactive chemical, phenolphthalein, was applied to the freshly split surface (figure 12). A comprehensive description of this method can be found in SS 137242, (SIS, 1987). The principle is that the fresh non-

carbonated surface is turning purple and the carbonated surface do not react with the phenolphthalein leaving the surface gray.



Figure 12 Split and phenolphthalein sprayed specimen. No carbnation was detected for the sample.

3.5.5 Chloride and calcium content

In order to determine the chloride and calcium content in the concrete, cubes were grinded on a lathe in order to obtain powder for chemical analysis by potentiometric titration. Each sample was ground to a depth of 40 mm on the following intervals: 0-2, 2-5, 5-10, 10-15, 15-20, 20-30, 30-40 (mm) (figure 13). After grinding, the powder was stored in paper bags and dried in an oven (at 105° C) until achieving constant mass. Finally, the samples were prepared and titrated according to the test method AASHTO T 260 (for chlorides and calcium) (American Association of State and Highway Transportation Officials, 2009).



Figure 13 Specimen after grinding. Different depths are marked by different diameters of the grindings levels.

3.5.6 Ultrasonic velocity measurement

Ultrasonic velocity measures were applied in order to analyse the exposed concrete for possible internal damage. The ultrasonic pulse transmission time is measured in µs and

the velocity is calculated to m/ μ s. Before the measurement, all cubes were conditioned for two days in 40° C/RH 65%. The ultrasonic instrument used in this work was an AU 2000 from CEBTP. Results were given in X, Y and Z coordinates, where the Z coordinate was set between the specimens top and bottom (casting direction).

3.5.7 Water absorption

Water absorption was studied by placing samples in a box with a wet surface so capillary suction could be enabled. At the bottom of the box 0/8 sand was filled, making an even layer and covered by a Wettex cloth. Moreover water was filled up to two millimetres above the Wettex cloth. Before weighing each cube, every cube needed to be swiped of with a humid Wettex cloth so there was no water dropplets at the surface. Weighing was made after 1, 2, 3, 4, 8, 24, 48 and 72 hours after placing the specimens into the water.

3.5.8 Microscopy

Light microscope for image analysis was used for analysing crack pattern in the sampleas (figure 14). A number of sampleas were selected for preparation of thin sections.



Figure 14 Leica DM4500 P, light microscope at the CBI (left) and analysed thin sections (right)

4 Results and discussion

4.1 Visual inspection

The following texts describe the specimens immediately after they were brought to the CBI laboratory, after being exposed in the field for approximately five years. The first observation was that the concrete type that kept its mechanial properties the best was the self-compacting concrete (SCC). These specimens included non-treated references, as well as surface treated with HG and DC. The outcome was such that all SCC samples were found to be undamaged. Furthermore, the samples made of concrete with w/c 0.45 remained generally undamaged with the exception of some isolated minor damages at samples' corners, thus suggesting that the corners were likely damaged during handling. However, slight surface scaling was noted in all types of w/c 0.45 concrete. Even for the w/c 0.45 concrete with added air. Still, this concrete had no damaged corners.

The following results are related to the concrete with w/c 0.70. Series called 0.70 HG and 0.70 DC proved to be completely intact with no detected damages. Each of the 0.70 R (i.e. R for reference) specimens had lost 10% of their volume and showed 100% active scaling at the bottom surface. All 0.70 R samples were considered damaged, wherein 40% of the top surface was scaled for R2 and R5. All samples were damaged on the sides, amounting to about 20% of the surface area being damaged. Major damage on the bottom of all samples was seen. Finally less than 10% of volume loss of all cubes was observed. All cubes pertaining to the 0.70 R series had slight scaling on the sides but major scaling on the bottom. Up to 40% of the total area had scaling and the total volume loss was lower than 5%.

4.2 Chloride content

The following four concrete powder types were titrated to establish their chloride concentrations, w/c 0.45, w/c 0.45 with added air, SCC and w/c 0.70. All types of concrete were divided in three impregnation groups: the none impregnated references R, the impregnated with HG and the impregnated with DC. The reference sample of w/c 0.70 was deteriorated too much to be ground and analysed. The results from the concrete with w/c 0.45 (figures 15 and16) indicate that the impregnated samples had lower chloride concentrations in comparison to the references. No major difference between the impregnations DC and HG was found in this study. Similar results were observed in the w/c 0.45 concrete with added air (figure 15). Although similar results were obtained, the references differed in the sense that the concrete with added air showed a 10 mm deeper penetration of chlorides. SCC did, however, show a different pattern (figure 17) regarding penetration depth, such that none of the impregnated surfaces resulted in improved conditions. Both DC and HG followed the reference chloride content curve with slightly lower values (HG lowest). These results correspond to the penetration depth of HG and DC which are presented further in the report.

The impregnated w/c 0.70-concrete specimens resisted the chloride ingression well, resulting in the highest level of chlorides being measured just below 0.5 % Cl of cement weight. Despite the poor concrete quality, the samples did not fail the test (figure 18).

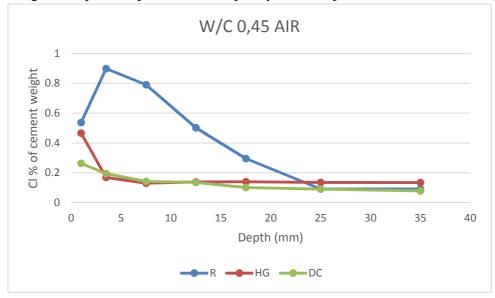


Figure 15 Chloride concentration of concrete with w/c 0.45 with added air

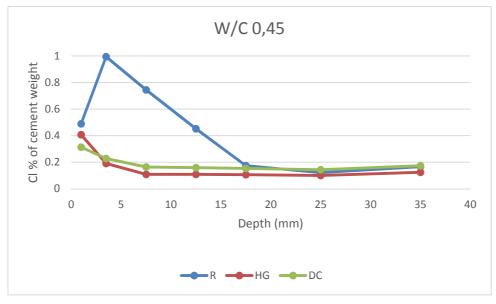


Figure 16 Chloride concentration of concrete with w/c 0.45

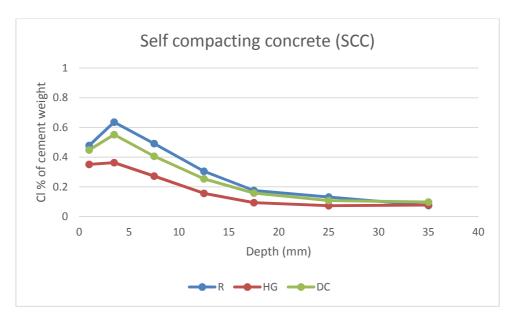


Figure 17 Chloride concentration in SCC, self compacting concrete.

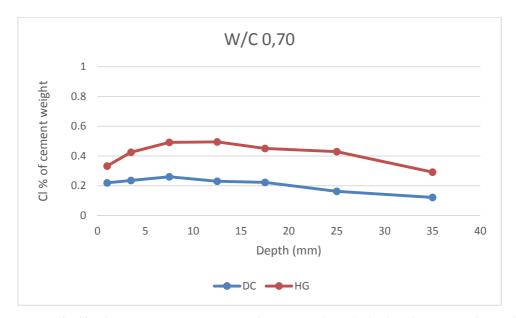


Figure 18 Chloride concentration in impregnated concrete with w/c 0.70. The reference sample was damaged due to the exposure field conditions.

4.3 Calcium content

Results after the calcium titration showed that the highest calcium concentration was found in the SCC, whilst concrete types w/c 0.45 and w/c 0.45 Air have similar calcium concentrations as presented in figure 19. The lowest concentration of calcium was found in concrete 0.70 HG (figure 19). The calcium profile is reflecting the recipes where SCC contained more cement and limestone filler which means therefore higher calcium content.

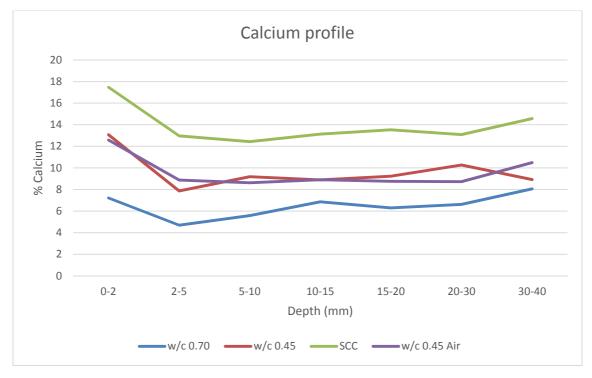


Figure 19 Calcium concentration of four concrete types.

4.4 Carbonation

Self compacting concrete (SCC) and concrete with w/c 0.45 were tested for carbonation depth. Results presented in table 4 indicate a significant resistance against carbonation. Out of the 18 analysed specimens, only two showed signs of carbonation as a corner effect. To ensure the certainty of these results the specimens were split again, but even then showed no signs of carbonation.

Table 4 Result of carbonation analysis

Specimens	Result	Second splitting	Result after second splitting
0.45 R1	No carbonation	X	No carbonation
0.45 R2	No carbonation		
0.45 R3	No carbonation		
0.45 DC 1	No carbonation	X	No carbonation
0.45 DC 2	No carbonation		
0.45 DC 3	No carbonation		
0.45 HG 1	No carbonation	X	No carbonation
0.45 HG 2	No carbonation		
0.45 HG 3	No carbonation		
SCC R1	No carbonation	X	No carbonation
SCC R2	Corner effect		
SCC R3	Corner effect		
SCC DC 1	No carbonation	X	No carbonation
SCC DC 2	No carbonation		
SCC DC 3	No carbonation		
SCC HG 1	No carbonation	X	No carbonation
SCC HG 2	No carbonation		
SCC HG 3	No carbonation		

4.5 Impregnation depth

The results obtained from the impregnation depth tests are presented in table 5. It can be observed that the impregnation agent HG provides a more effective penetration depth than DC. As earlier presented in the report, HG is gel-based, which means that the treatment has longer treatment time than the DC that is water-based and evaporates faster.

Table 5 Impregnation depth for alla samples and the calculated average impregnation depth.

Specimens	Impregnation depth (mm)
0.45 R1	0
0.45 R2	0
0.45 R3	0
0,45 DC 1	2
0.45 DC 2	1
0.45 DC 3	2
0.45 HG 1	5
0.45 HG 2	5
0.45 HG 3	5
SCC R1	0
SCC R2	0
SCC R3	0
SCC DC 1	0.5
SCC DC 2	0
SCC DC 3	0.5
SCC HG 1	3
SCC HG 2	4
SCC HG 3	2

Specimens	Average impregnation depth (mm)
0.45 R	0
0.45 DC	1.7
0.45 HG	5
SCC R	0
SCC DC	0.3
SCC HG	3

Also, the results from the drop test indicate that HG preforms better in terms of hydrophobic properties in comparison to DC. Nevertheless, both agents were classified as hydrophobic treatments in the top surface layer of concrete samples. Results are presented in table 6.

Table 6 Results of the Drop Test for measuring the hydrophobic effect on powder.

Depth (mm)	SCC DC4	SCC HG4	0.45 DC4	0.45 HG4
0-2	Hydrophobe	Hydrophobe	Hydrophobe	Hydrophobe
2-3	Hydrophobe	Hydrophobe	Hydrophile	Hydrophobe
3-4	Hydrophile	Hydrophobe	Hydrophile	Hydrophobe
4-5	Hydrophile	Hydrophile	Hydrophile	Hydrophobe

4.6 Ultrasonic velocity test

Results from the ultrasonic measurements are given in m/ μ s. Specimens with w/c 0.45; 0.45L, (where L stand for air) and self-compacting concrete SCC, were analysed. In table 7, results clearly indicate lower velocity through the 0.45L samples than for the 0.45 and SCC.

The lower velocity through 0.45L could be explained due to the lower density caused by the added air – creating air voids. When the specimens were produced, the top surface of casting direction was uneven in most cases, leading to lower accuracy of the Z coordinate measurements. Surface evenness has large impact on the velocity measurement due to possible air spaces between transducer and concrete surface.

Table 7 Results of ultrasonic velocity test (m/\mus)

0.45	HG 3	DC 3	R 3
Х	0,0043	0,0042	0,0041
Υ	0,0042	0,0042	0,0043
Z	0,0038	0,0037	0,0036

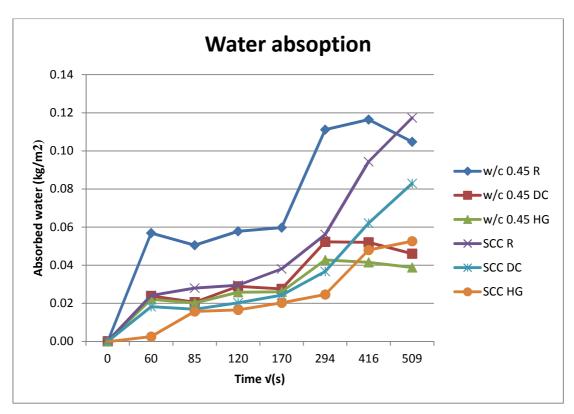
SCC	HG 3	DC 3	R 3
X	0,0041	0,0043	0,0041
Υ	0,0041	0,0039	0,0045
Z	0,0042	0,0037	0,0042

0.45L	R 4
Χ	0,0036
Υ	0,0038
Z	0,0036

4.7 Water absorption

Results from the 72 hour water absorption test are presented in figure 20. The analysis included 18 specimens divided into two concrete types, SCC and w/c 0.45. For every type of concrete, there were three types of surface treated samples, including HG, DC and non-treated references denoted by R. To summarize the table, very marginal changes were observed in the water absorption of all specimens. The maximal difference measured during the test was about 0.12 kg/m² (0.45 R3) and the lowest was about 0.04 kg/ m² (0.45 HG2). The reference specimens were clearly absorbing up to three times more water than the impregnated specimens. Among the impregnated specimens, HG showed lower water absorption than DC for both SCC and w/c 0.45 concrete.

Furthermore, different patterns were observed regarding the water absorption in HG and DC, whereby the DC samples tended to increase absorption from $170 \sqrt{s}$ (8 hours) until saturation. At $170 \sqrt{s}$ most of the specimens had similar water absorption. One explanation as to why the DC samples absorbed more water than HG samples could be due to the conditioning before the water absorption test. The specimens were collected from the field in August 2013 and stored for six months in a climate room (RH 65% 20° C). Since HG showed better resistance against water absorption, it could be that the diffusion rate was lower than in DC. Accordingly, the specimens treated with HG could have dried out less from the inside than specimens treated with DC. If that would be the case, the water absorption would increase significantly after passing the treated layer – as we see in figure 20.



Figur 20 Water absorption results

4.8 Microscopy

The microscopic results of thin sections were focused on detection of cracks/micro-cracks and observation of the crack propagation towards the inner parts of the sample. Only selected samples were prepared for the microscopic investigation. Some of the results are illustrated in the figres below.

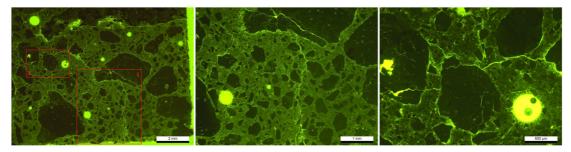


Figure 21 Microscopic analysis of thin section of the referens sample 0.45.

For the reference sample 0.45 R illustrated in figure 21, a number of cracks were observed. Cracks appeared mainly in the aggregates, around the aggregates and in the middle of the sample going into the paste.

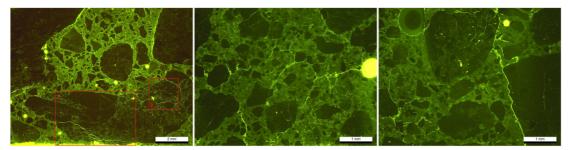


Figure 22 Microscopic analysis of thin section of the sample 0.45HG.

In figure 22 the 0.45 HG sample is presented. The cracks are more horisontal at the surface but not so pronouced as in the reference sample. At the top of the sample the spider cracks (collection of micro cracks) were also observed. At the middle of the thin section, cracking directions were more undefined and spider cracks were no longer spotted.

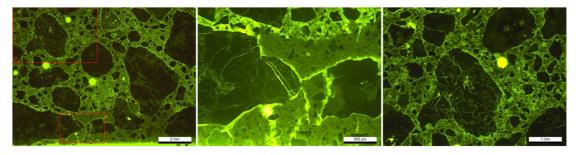


Figure 23 Microscopic analysis of thin section of the sample 0.45 DC.

In figure 23 the 0.45 DC sample is presented. The sample showed low degree of cracks, and those which are present are without prevailing direction. Some cracks appeared in aggregates. At the middle of the thin section a higher degree of cracks was seen comparing to the surface layer.

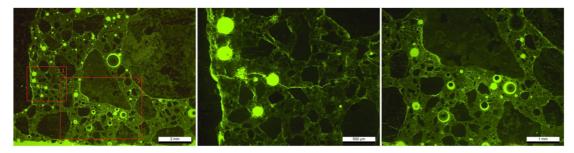


Figure 24 Microscopic analysis of thin section of the reference sample 0.45 L.

In figure 24 the reference sample 0.45 L (with added air) is presented. Mainly horizontal cracks however, at the average amount are seen at the top of the sample. Cracks appeared in aggregates and several spider cracks were identified.

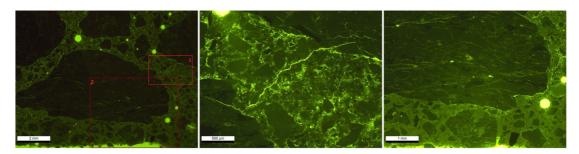


Figure 25 Microscopic analysis of thin section of the reference sample of SCC.

In figure 25 the reference sample of SCC is presented. Very few cracks were observed, although some cracks appeared in aggregates.

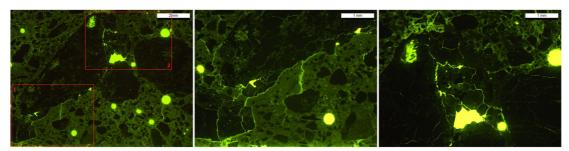


Figure 26 Microscopic analysis of thin section of the SCC HG.

In figure 26 the SCC HG is presented. Average rate of cracks is mainly in vertical direction. Cracks were visible in aggregates. Lower amount of cracks at mid depth of thin section.

The results from microscopic analysis of thin sections are summarized in table 8.

 $Table\ 8\ Results\ from\ microscopic\ analysis\ of\ thin\ sections.$

		Level of cracks	cks	Crack direction	tion	Cracks	Cracks in ballast	Middle of	Middle of thin section
Sample	Low	Medium High	High	Horizonta Vertical	Both	Yes	No	Increase of cracks	Increase of cracks No increase of cracks
F45 R3O		×			×	×		×	
F45 R3U		×			×	×			×
F45 HG30		×		×		×			×
F45 HG3U		×			×	×		×	
F45 DC30	×					×		×	
F45 DC3U	×				×	×		×	
F45L R40		×		×		×		×	
F45L R4U		×			×	×			×
F45L HG40	×				×	×			×
F45L HG4U		×		×		×			×
F45L DC40	×				×	×		×	
F45L DC4U	×				×	×		×	
FSKB R30	×				×	×		×	
FSKB R3U		×			×	×		×	
FSKB HG30	×				×	×		×	
FSKB HG3U		×		×		×			×
FSKB DC30		×			×	×		×	
FSKB DC3U		×		×		×			×

5 Conclusions

The main results from this thesis work can be summarized in the following subchapters.

5.1 Theoretical output

Most damage mechanisms in concrete are related to the moisture content in the material. A properly applied hydrophobic impregnation changes the conditions of moisture ingress into the concrete and consequently can prolong the service life of a concrete structure. It is of high importance to understand which damages are to be prevented and which type of impregnation agent is suitable for given object in the designated surrounding and climatic conditions.

In order to fully benefit from the impregnation the hydrophobic agent needs to sufficiently penetrate the concrete. If the impregnation agent stays on the surface the effects can be limited. Duration of the contact between the agent and the surface, degree of saturation and porosity of the substrate have all major influence on the penetration of the impregnation agent. Long duration of contact, large porosity and low degree of water saturation are the best conditions in order to achieve high penetration of the agent.

Swedish building legislation does not define any demand for impregnation of concrete structures. It is up to the project leader to decide whether impregnation is to be included in the project or not. Furthermore there is no demand in public procurement concerning impregnation agents. In case the Swedish Transport Administration requires application of EN 1504-2, all impregnation agents must be CE certified since EN 1504-2 is a harmonized standard.

Hydrophobic impregnation is not of the highest priority out of a contractor's perspective in Sweden. This may result in a lack of knowledge in workmanship which in turn lead to an impregnation with less hydrophobic effect. Since the procedure of selecting impregnations is rather unrestricted, the major factor for decision-making is the contractor's experience. Unexperienced contractors often go with the cheapest products without knowledge and routines for application. On the other hand, more experienced contractors tend to select suitable products for given damage preventions.

5.2 Analyses of exposed samples

The long-term impact of DC and HG-treated concrete samples demonstrated protection angainst chloride ingress after five years of field expousure. Both agents significantly reduced the chloride content comparing to the untreated samples. HG was however consistently more efficient than DC.

Penetration depth: Both tested products are silane based, however, with different penetration depth. Contact time for the impregnation agent on the surface of concrete is important for the penetration depth. DC is water based and applied with 300 g/m². Due to its liquid form the exposure time is short and the water is quickly evaporating from the surface. HG which is gel based and applied with 500 g/m² has a long exposure time on the surface and the higher penetration depth.

Chloride content: Both impregnation agents show efficiency against chloride penetration. The agents reduce the cloride content in the specimes, regardless of the concrete quality. The lowest impact of the hydrophobic agents is however observed in the self-compacting concrete.

Calcium content: The calcium content of the three concrete types is in alignment with the cement content in recipes of the mixes. SCC has the highest calcium content followed by the w/c 0.45 and w/c 0.70 concrete.

Ultrasonic velocity test: The highest velocity is measured through SCC and w/c 0.45 concrete, comparing to the w/c 0.45 concrete with added air which is less dense and results in lower velocity.

Water absorption: Both agents are efficient in reduction of water absorption.

Microscopy: All tested samples regardless of concrete type show micro-cracks in the samples and around the aggregates. Crack patterns do vary but no prevailed direction could be related to the type of concrete or impregnation. The reference samples had most of micro-cracks but the difference between the reference and impregnated sampleas should be investigated in more details.

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