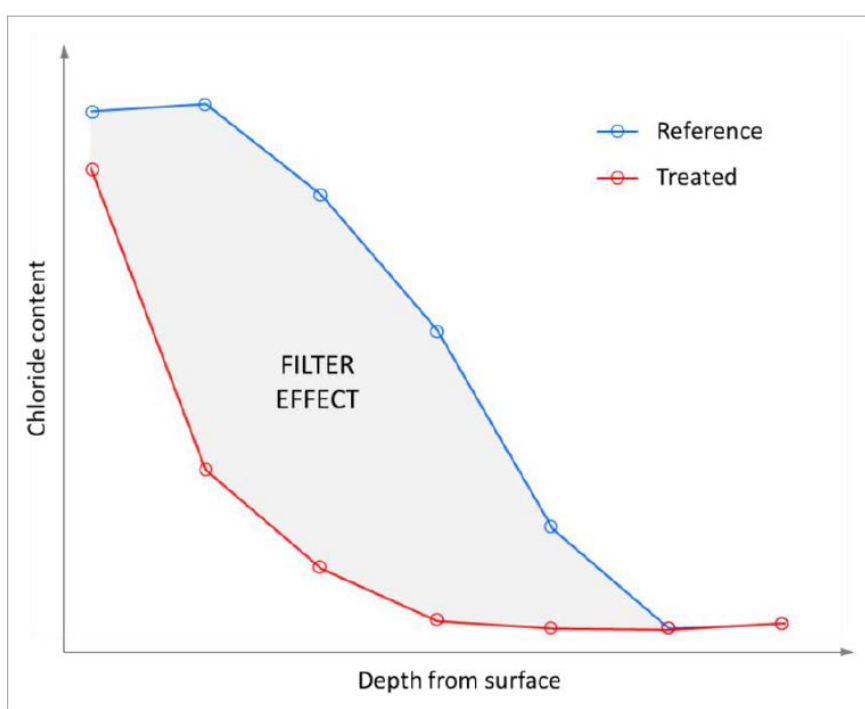# Nordic method for testing hydrophobic impregnations for concrete, with regard to prevention of chloride ingress

Gemensam nordisk metod för provning av vattenavvisande impregneringsmedel för betong med avseende på skydd mot kloridinträngning

CBI Swedish Cement and Concrete Research Institute

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NORDIC METHOD FOR TESTING HYDROPHOBIC IMPREGNATIONS FOR CONCRETE, WITH REGARD TO PREVENTION OF CHLORIDE INGRESS

Gemensam nordisk metod för provning av vattenavvisande impregneringsmedel för betong med avseende på skydd mot kloridinträngning

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Contents

1 SUMMARY

Chloride induced corrosion of the reinforcement is the major cause of degradation of reinforced concrete structures. In particular in the Nordic countries, the use of de-icing salts during winter, leads to severe degradation of edge beams in road bridges. Thus, in order to extend the service life and decrease maintenance costs, hydrophobic impregnations are commonly used to prevent or slow down chloride ingress into concrete.

The transport and roads administrations of Sweden, Norway and Finland use different national methods to evaluate the performance of this type of products meaning that the same CE-marked product needs to meet different requirements. Therefore, the transport and road administrations of Sweden, Norway and Finland initiated a project with the aim to establish a common Nordic method for classification of hydrophobic impregnations with regard to their capability to protect concrete from chloride ingress.

The project was divided into three phases consisting of the analysis of existing test standards (national and international), a pre-study to evaluate the influence of different tests parameters and a round robin test involving three laboratories (one in each country).

A thorough comparison of the existing methods and review of relevant literature allowed to define which test parameters could be used in the formulation of the new method and which ones required further studies. It was found that the type of surface to be treated, the length of the preconditioning period, the length of the curing period and whether the surface to be impregnated should be soaked with $Ca(OH)_2$-solution or not should be further investigated.

The results of the pre-study showed that the application of the impregnation to form surfaces led to somewhat better chloride blocking effect. Despite this, it was decided to use sawn surfaces in the new method, since it is much easier to obtain reproducible surface characteristics that way. The characteristics of a form surface depend, among other factors, on the form material, use of release agents and curing conditions.

Saturation of the surface with calcium hydroxide solution before impregnation was found slightly beneficial on the chloride blocking effect compared to when such a treatment was omitted. However, since this did not contribute to the robustness of the test results, it increased the number of experimental steps and it is not representative of practice in real structures, it was decided not to incorporate such treatment in the new method.

The chloride protection slightly increases with the impregnation curing time. It was not clear which factor was most dominant; the continuous polymerization of the hydrophobic impregnation or the continuous cement hydration. Since enough curing time is necessary for the treatment to be efficient, it was decided that the curing period before exposure to chlorides should be 28 days.

With the primary objective of determining the reliability and reproducibility of the new method, a round robin exercise was carried out. Three laboratories were involved in this phase; CBI-Borås in Sweden, SINTEF in Norway and VTT in Finland. The results show that despite some differences in both materials and methods, such as the type of cement or preconditioning and curing

environments, highly reproducible results were obtained. In addition, a detailed discussion on the influence of the details of the method on the chloride profiles and on the filter effect is presented.

Within the round robin test, the relative humidity before and after impregnation and the moisture content of the powder samples for chloride analysis were found to be the major parameters leading to the discrepancy of the results.

In addition, handling of the wet concrete surfaces after exposure to chlorides and the time period (and temperature) between the end of the chloride exposure and powder sampling for chloride analysis were found to have surprisingly large effects on the form of the chloride profiles in the samples. Therefore, in the final method this part of the procedures were made much more precise in order to increase its reproducibility.

The method can be briefly described as follows:

Concrete specimens are prepared by sawing 100 mm cubes into two halves, three cubes per test series. The sawn surfaces are defined as exposure faces. Three halves are treated with the hydrophobic impregnation to be tested and the other three halves are kept untreated as reference. The specimens are submerged in 15% NaCl-solution for 56 days. After exposure, the chloride ingress is determined by profile grinding and the total amount of penetrated chlorides is calculated. The chloride blocking effect of the hydrophobic impregnations, is expressed as the Filter Effect, FE, which is determined as 1 minus the ratio between the amount of penetrated chlorides in treated and in reference specimens.

The results obtained in both the pre-study and round robin exercise were compared to those obtained with the existing national methods in order to establish proper requirement levels with the new method. Despite the many differences between the methods, it was found that a filter effect of approximately 0.65 correlates well with the existing requirement in the Swedish method and in the Norwegian method. However, given limited data available and also considering data from field investigations, a level of 0.60 is proposed as appropriate for a really well performing hydrophobic impregnation.

2 INTRODUCTION

2.1 Background and aim of the project

The road authorities of the Nordic countries allocate, yearly, vast sums on the hydrophobic surface protection of reinforced concrete structures (e.g. bridges) in order to reduce the risk for chloride induced corrosion, caused by the extensive use of de-icing salts during harsh winters or sea water ingress. The harmonised standard EN 1504-2 (CEN, 2004) is the basis for CE-marking and declaration of performance of hydrophobic impregnation systems, but the property "diffusion of chloride ions" is, according to the standard, "subject to national standards and national regulations". This means that the same CE-marked product needs to meet different requirements regarding this property, which are imposed by the local authorities. Such requirements are based on different local test methods. Thus in Sweden, Finland and Norway the requirements are today based on three different test methods, one for each country. Therefore, the Swedish Transport Administration (Trafikverket), the Finnish Transport Agency (Liikennevirasto) and the Norwegian Public Roads Administration (Statens Vegvesen), have started a project with the following main objectives:

1. To develop a common Nordic test method to measure the ability of hydrophobic impregnation agents to prevent chloride ingress in concrete. The new method shall be based on the existing Finnish, Norwegian and Swedish test methods;
2. To define relevant acceptance criteria for the new method, by testing different hydrophobic agents with both new and the three existing national test methods;
3. To validate the new test method against long term field data.

2.2 Basic outline of the project

The work was divided into three main phases:

Phase 1: Review and comparison of the details of the three existing national methods and other similar methods, in order to decide on which test parameters a mutual acceptance could be reached and which test parameters require further studies before formulating a new method.

Phase 2: A pre-study in order to clarify the influence of the test parameters unsettled after phase 1 and leading to a preliminary formulation of the new test method.

Phase 3: A round robin exercise involving three laboratories (one from each country) with the preliminary new test method and several impregnation products, leading to the final formulation of the new test method and a proposal for definition of acceptance criteria. Long-term field data should be taken into account.

In the first phase only representatives from the three national transport administrations were involved. The pre-study (phase 2) was carried out by CBI Betonginstitutet AB. The work in phase 3 was coordinated and lead by CBI Betonginstitutet AB, and involved three laboratories: CBI-Borås in Sweden, SINTEF in Norway and VTT in Finland.

This report focus on the work carried out and coordinated by CBI, i.e. the outcome of the pre-study and the round-robin exercise. However, since the decisions taken during the first phase are vital for the design and details of the new method, an overview of phase 1 is also given.

Throughout the report the terms hydrophobic agents, impregnation agents, hydrophobic impregnations, surface treatments or simply products are used with the same meaning, i.e. hydrophobic impregnation according to Principle 1 in EN 1504-2 (CEN, 2004). The terminology used in the final method description (Annex A) is in accordance with EN 1504-2 (CEN, 2004). In addition, chloride blocking effect, chloride protection and filter effect are used with the same meaning and expressed either as a value (between 0 and 1) or in percentage; for the final method description, the term "filter effect" was selected, expressed as a value between 0 and 1.

3 REVIEW AND COMPARISON OF EXISTING METHODS

3.1 Existing methods

The following methods were reviewed:

- The Swedish method as described in section LFB.311 of (Svensk byggtjänst, 2010);
- The old Finnish method described in (VTT, 1994);
- The new Finnish method described in (VTT, 2009);
- The Norwegian method described in (SINTEF, 1998);
- NT Build 489 (Nordtest, 1999) which is intended for paint systems;
- EN 13396 (CEN, 2004) which is intended for concrete repair products.

The methods were scrutinised and compared by putting all details into a table where each row represented one essential test item (e.g. number of specimens, preconditioning, duration of exposure, etc.). Each item was then discussed and it was decided whether a unanimous decision on how the item should be treated in the new method could be reached without further studies or if further studies or tests were needed. The table with all the details can be found in Annex B, Table B.1.

The outcome of the discussions, with main motivation, on each item is given in the following:

- *Specimens to cast:* It was decided to use 100 x 100 × 100 mm cubes, which were used in most methods.
- *Type of concrete:* The importance of a well defined concrete was recognised and it was decided to use a concrete according to EN 1766 with w/c = 0.45, with a CEM I and a maximum aggregate size of 10 mm.
- *Curing of cast specimens:* The importance of a well defined curing period was recognised and it was decided to let the cast specimens be water cured for 28 days before further treatment.
- *Test specimens:* It was decide to use 3 half cubes per impregnation agent and 3 half cubes for reference. Half a cube is large enough to encompass the entire chloride profile.
- *Preconditioning of test specimens:* This is one of the points where the methods diverge most. In some cases the surfaces are first soaked in lime-water. In several of the existing methods exposure to saturated $Ca(OH)_2$-solution before the actual preconditioning in lower relative humidity (RH) is used in order to have a well defined moisture condition as starting point and to prevent leaching. In other cases, specimens are either covered with a plastic foil or directly exposed to a RH of the environment varying between 50% and 65%. The length of this period varies from 1 hour to 14 days or longer. It was decided to study this parameter more thoroughly.
- *Test surface:* It was decided to use sawn surfaces for both impregnated and reference samples, and to protect the other surfaces by coating with epoxy. Sawn surfaces were preferred before form surfaces because it is easier to control the condition of a sawn surface, and there is less risk that special surface phenomena like the wall-effect on the structure, presence of laitance, release agents, leaching and air voids influence the results. However, it was decided to also include this parameter into a pre-study.
- *Application of the impregnation product*: It was decided to apply the quantity according to the recommendations of the producer. The application should be carried out in a way that ensures control of the quantity used.

- *Curing of the impregnation product:* As with the preconditioning this is another point where the methods differ. The RH varies between 50 and 70%, and the period between 2 days and up to 28 days. Several methods use 14 days. It was decided to study this parameter more thoroughly.
- *Chloride exposure method:* Exposure to chlorides is, in the reviewed methods, carried out in three principal ways; permanent submersion in a chloride solution, by submersion in chloride solution and exposure to air cyclically or by salt-spraying and drying cyclically. The concentration of the salt solution also varies between 3% and 16.5% NaCl. The method that most resembles what happens in real structures is intermittent salt-spraying as in the Norwegian method. However this method gives large variations between results obtained at different laboratories. A method giving better reproducibility was therefore preferred, like permanent submersion in 15% NaCl-solution which would be used preliminarily. The influence of the exposure method was studied in another project carried out by the Norwegian Public Road Administration (Rodum, 2013) and was therefore not included in the further studies to be carried out within this project.
- *Exposure time:* It was decided to use 56 days of exposure.
- *Concrete sampling for chloride measurement:* The procedure described in the Norwegian method was preferred and decided upon, i.e. analysing the chloride content in layers (profile grinding) down to a depth where no external chlorides exist.
- *Type of chloride measured:* It was decided to use the acid-soluble chloride (total chloride content).
- *Chloride measurement method:* Several methods are possible. This was not seen as a controversial issue, as long as the precision is acceptable.
- *Evaluation criteria (requirement):* It was decided to use "*the filter effect*" as in the Norwegian method. The division into several classes as in the Finnish method should also be considered.

3.2 Implications of the review on the continuation of the project

With the review as a basis, it was decided to carry out a pre-study where the influence of two of the three items having the largest deviations should be studied more thoroughly; the preconditioning before treatment and the length of curing of the impregnation. For the third item where large deviations exist, the chloride exposure method, it was decided to rely upon the study carried out in Norway (see 3.3).

3.3 Norwegian study on different exposure methods

In (Rodum, 2013) four different chloride exposure methods were investigated:

A. Permanent submersion in 16.5% NaCl-solution;
B. Alternating submersion in 16.5% NaCl-solution and drying in 50% RH (1 day cycles, prolonged to 3 days at weekends);
C. Exposure by spraying 3% NaCl-solution alternating with drying in windy laboratory air (4 hour cycles);
D. Permanent submersion in 3% NaCl-solution.

All samples to be impregnated were conditioned for 3 days at 20°C and 50% RH. The impregnation curing period was 7 days at 20°C and 50% RH. While the impregnated specimens were preconditioned, impregnated and cured (10 days) the reference samples were kept with the sawed surface sealed with plastic. All specimens were exposed to chlorides for 56 days.

The chloride levels in the samples after exposure were totally different with the four exposure methods. Permanent submersion in 16.5% NaCl-solution (A) yielded the highest chloride levels of all the methods tested whereas permanent submersion in 3% NaCl-solution (D) yielded the lowest. On the other hand, cyclic exposure yielded higher levels with 3% NaCl-solution (C) than with 16.5% NaCl-solution (B). Worth noting is also the fact that the type of exposure method has a greater influence on the reference specimens than on impregnated samples; no significant differences were found between A and D or between B and C.

Even though there is a big difference with regards to the amount of chloride in the samples, the calculated effect with regards to the prevention of chloride ingress, the filter effect (see 4.3.2) was surprisingly constant. For methods A, B and D the filter effect was in the range 85.9% to 81.5%. Method C resulted in a somewhat lower value, 72.4%. The standard deviation was lowest for methods A and B.

Based on this study it was then decided to use the most convenient method giving the smallest standard deviation, i.e. permanent submersion in a solution with a high NaCl-concentration.

4 PRE-STUDY

4.1 Objective of the pre-study

The pre-study was carried out to clarify the influence of the following parameters on the efficiency of the impregnation products with regard to prevention of chloride ingress into concrete, i.e. their chloride blocking effect:

- The length of the pre-conditioning period;
- Saturation of the surface to be impregnated by $Ca(OH)_2$-solution;
- The length of the impregnation curing period.

The penetration depth of the impregnations should also be recorded.

4.2 Experimental

The following sections summarize the materials and methods used in this study. The pre-study was carried out at CBI.

4.2.1 Impregnation agents

Two different silane-based surface treatments were selected for this study:

- A liquid, applied with a concentration of 300 g/m^2 (active silane = 297 g/m^2) . This product is further on referred to as P1;
- A gel with bentonite clay, applied with a concentration of 500 g/m^2 (active silane = 450 g/m^2). This product is further on referred to as P2.

Reference specimens (without treatment) are further on referred to as R. More data on the products can be found in Annex E.

4.2.2 Specimen preparation

The concrete used in this study was of the type MC (0.45) according to EN 1766 (CEN, 2000); the mixture proportions are given in Table 1.

Concrete cubes, with the dimensions of 100 x 100 x 100 mm^3, were cast in plastic moulds, demoulded after one day and stored in water for 27 days. No release agent was used. At the age of 28 days, the cubes were sawn in halves and all the surfaces, except the sawn surface, were coated with epoxy resin and allowed to dry for one day before pre-conditioning. For some of the specimens, the sawn surface was coated and instead, one of the lateral form surfaces was left uncoated. In total, 96 specimens (test surfaces) were prepared, corresponding to 3 replicates per impregnation product and per reference for each parameter tested:

- 81 specimens (sawn surfaces) were used for studying the influence of the pre-conditioning environment before application (of the impregnation) and curing time after application of the impregnation, on the chloride blocking effect;

- 9 specimens (form surfaces) were used for studying the influence on the chloride blocking effect of using a form surface instead of a sawn surface;
- 6 specimens (sawn surfaces) were used for measurements of the penetration depth of the impregnation agent.

Table 1. Concrete mixture proportions.

CEM I 42.5 R (kg/m^3)	Aggregates 0-10 (kg/m^3)	Water (kg/m^3)	Superplasticizer (weight % CEM)	Air (%)
395	1800	178	0.5	1.5

The specimens to be treated with the hydrophobic agent were wrapped using a band (30 mm wide) of adhesive neoprene, protruding 5-10 mm from the cut surface, in order to guarantee a homogeneous distribution of the hydrophobic agent and preventing it from running off the test surface. The inner edges were sealed with moisture resistant contact glue. This is shown in Figure 1.
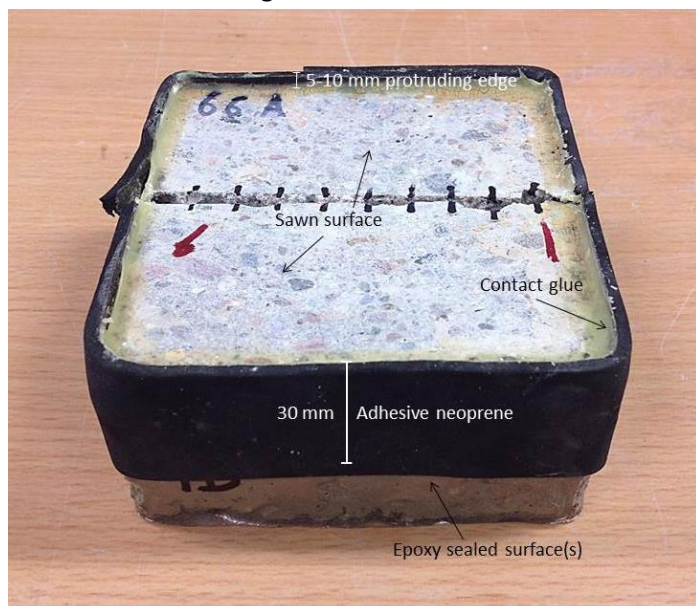
Figure 1: Illustration of impregnated test specimen. (This specimen has been cut for determination of penetration depth).

4.2.3 Specimen preconditioning

The influence of three different preconditioning procedures, i.e. conditioning before application of the impregnation, on the chloride blocking effect was evaluated:

- 7 days at 20±2 ^{o}C and 60±10 % RH, further on referred to as S;
- 3 days in saturated Ca(OH)$_2$ solution followed by 3 days at 20±2 ^{o}C and 60±10 % RH, further on referred to as C3;
- 3 days in saturated Ca(OH)$_2$ solution followed by 7 days at 20±2 ^{o}C and 60±10 % RH, further on referred to as C7;

It is well known that the moisture condition of the surface when impregnated influences the penetration of the hydrophobic impregnation which probably also affects the resulting chloride

blocking effect. The chosen procedures are mainly aimed to determine the influence of the length of the pre-conditioning before impregnation in lower RH:s (3 days and 7 days) and whether pre-wetting of the surface with a lime solution is necessary in order to obtain a reproducible state of the surface and reproducible results. Neither extremely short periods, which could be relevant for marine structures, nor very long periods, which would not be realistic to use in an accelerated test, were included in the study. In this study priority is given to characterisation of the performance of hydrophobic impregnations in road environments with de-icing salts. For determining if the results are relevant for their performance in a marine environment, other studies will be consulted.

Specimens aimed at studying the influence of type of concrete surface used and measurement of the penetration depth, were preconditioned for 7 days at 20±2 ^{o}C and 60±10 % RH (type S).

4.2.4   Application and curing of the impregnation agent

The hydrophobic agents, P1 and P2, were applied following the recommendations given by the producers, in the concentrations of 300 g/m^2 and 500 g/m^2, respectively. After application of the impregnation the specimens were cured during 7, 14 and 28 days at 20±2 ^{o}C and 60±10 % RH and are further on referred to as 7, 14 and 28. Treated form surfaces and sawn surfaces for measurements of penetration depth were cured in the same conditions 20±2 ^{o}C and 60±10 % RH during 28 days, as required for determination of penetration depth according o EN 1504-2 (CEN, 2004). (According to EN 1504-2 determination of penetration depth for the declaration of product properties shall however be carried out on a concrete with w/c = 0.70). The application method is shown in Figure 2.
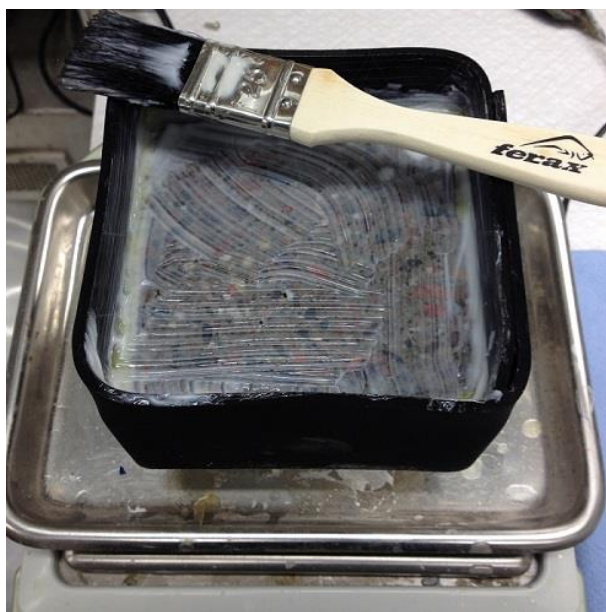
Figure 2: Impregnation of a specimen.

4.2.5   Penetration depth

The penetration depth of the surface treatment was measured by splitting the test specimens, spraying the fresh split surface with water and measuring the distance from the treated surface down to the border line between dry and wet concrete. The method is described in EN 1504-2 (CEN, 2004) and EN 14630 (CEN, 2006) in which phenolphthalein is replaced by water.

4.2.6   Exposure and chloride analysis

After curing, both references and impregnated specimens were immersed in a 15 % (by weight) NaCl-solution, in separate containers (Figure 3), during 56 days.

Figure 3: Specimens during exposure.

At the end of the exposure period, the samples were removed from the solution, surface dried with absorbent paper, sealed in plastic bags and stored at 20±2 $^{\circ}$C before dry grinding on a lathe with a diamond tool. The length of the storage period was not specified, neither the dates and times when the different specimens were ground was recorded. From the notes on recorded dates for the withdrawal from the NaCl-solution and the chloride analysis it can be deduced that all specimens with the impregnation curing period equal to 7 days were stored for less than one week, and that all specimens with the impregnation curing period of 28 days were stored for 30 days or more, prior to grinding. Some of the specimens which had experienced 14 days of impregnation curing may have been stored for less than 7 days and some for more than 30 days.

The following layers were ground: 0-2; 2-4; 4-6; 6-10; 10-15 and 15-20 mm, with an accuracy of 0.5 mm. After grinding the powder samples were dried at 105°C and stored in a desiccator prior to the chloride analysis. The acid soluble chloride content in each layer was determined by potentiometric titration according to AASHTO-T260 (1997) and expressed in % of the dry sample weight. For some selected specimens, the calcium content was also determined by potentiometric titration using a calcium selective electrode and 0.1 N EDTA solution as titrant (Tang, 2003). The results were used to

estimate the cement content in the concrete, since the aggregates used do not contain soluble calcium.

4.3   Results and discussion

This section summarizes the experimental results from this study. The complete set of results can be found in Annex C.

4.3.1   Penetration depth

The average penetration depth measured 28 days after application of the impregnation is shown in Figure 4. Figure 5 shows a fracture surface after spraying with water. As can be seen in Figure 4, P2 has a slightly larger penetration depth compared to P1; however the figure also shows the large scatter, i.e. standard deviation, for this type of measurement (the individual test results are given in Annex C.1). These values cannot be compared to values declared according to EN 1504-2 (CEN, 2004) since those are obtained on a concrete with w/c = 0.70.
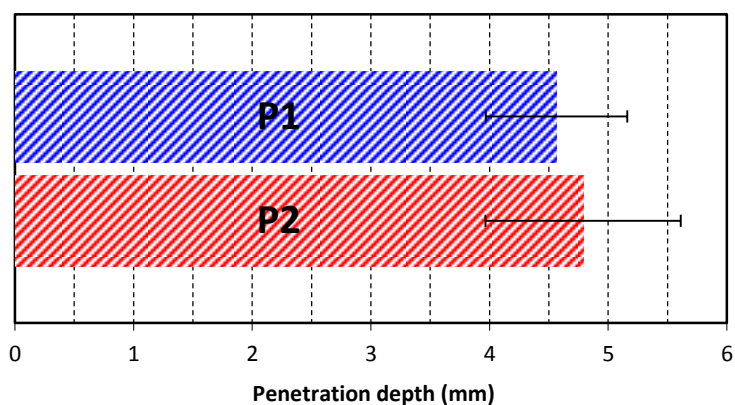
Figure 4. Average penetration depth of the two products after an impregnation curing period of 28 days. (Pre-conditioning type S). The error bars represent 2 standard deviations.

Figure 5. Fracture surface before measurement of the penetration depth.

Additional measurements were made on specimens on which preconditioning type S and C3 were used and 7 days of curing after impregnation in order to study the influence of the preconditioning on the penetration depth of the hydrophobic agent. The results in Figure 6 show that after an impregnation curing period of 7 days, the penetration depth of both products tested is comparable to that obtained after a curing period of 28 days (Figure 4) when the same preconditioning is used (type S). A similar tendency is obtained when comparing both products under precondition type C3. The results in Figure 4 also show a clear reduction of the penetration depth (for both products) when preconditioning type C3 is used instead. In all cases (in both figures) the product P2 has a somewhat larger penetration depth than product P1.
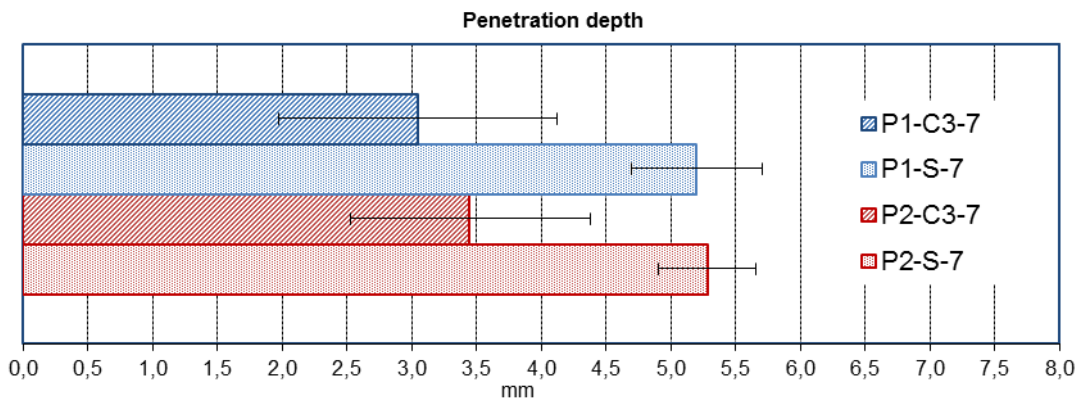
Figure 6. Average penetration depth of the two products after an impregnation curing period of 7 days, for 2 of the pre-conditioning types tested. The error bars represent 2 standard deviations.

4.3.2 Chloride ingress

This section summarizes the results from the chloride measurements under the influence of various experimental parameters, after exposure (by immersion) of the specimens in 15% NaCl-solution during 56 days. The chloride content is expressed as % of the oven-dry concrete weight and the total chloride ingress, in g/m^2, is calculated assuming a concrete density of 2370 kg/m^3 and a background chloride level of 0.005 % of the concrete weight.

4.3.2.1 FILTER EFFECT - DEFINITION

In order to characterize the chloride blocking effect of the hydrophobic impregnations, the Filter Effect (*FE*) as defined below, was used.

$$Filter\ Effect = FE = [1 - \frac{Cl_{treated}}{Cl_{reference}}]$$

where

$Cl_{treated}$ is the amount of chloride that has penetrated the treated specimens during the exposure
$Cl_{treated}$ is the amount of chloride that has penetrated the refernce specimens during the exposure

4.3.2.2 INFLUENCE OF THE PRECONDITIONING BEFORE APPLICATION AND THE CURING TIME

Figure 7 to Figure 9 show all the chloride profiles, taken as the average of three parallel specimens, for the different preconditioning environments and grouped as function of the impregnation curing period. The total chloride ingress and the filter effect are shown in Figure 10 and Figure 11, respectively. The complete set of results is given in Annex C.2.
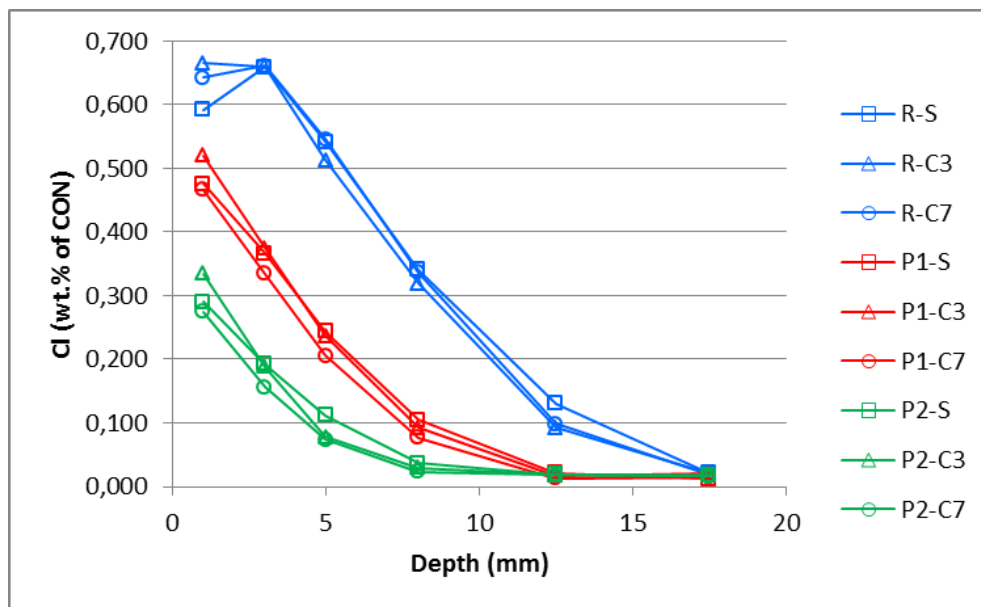
Figure 7. Chloride profiles measured for all series after an impregnation curing period of 7 days.
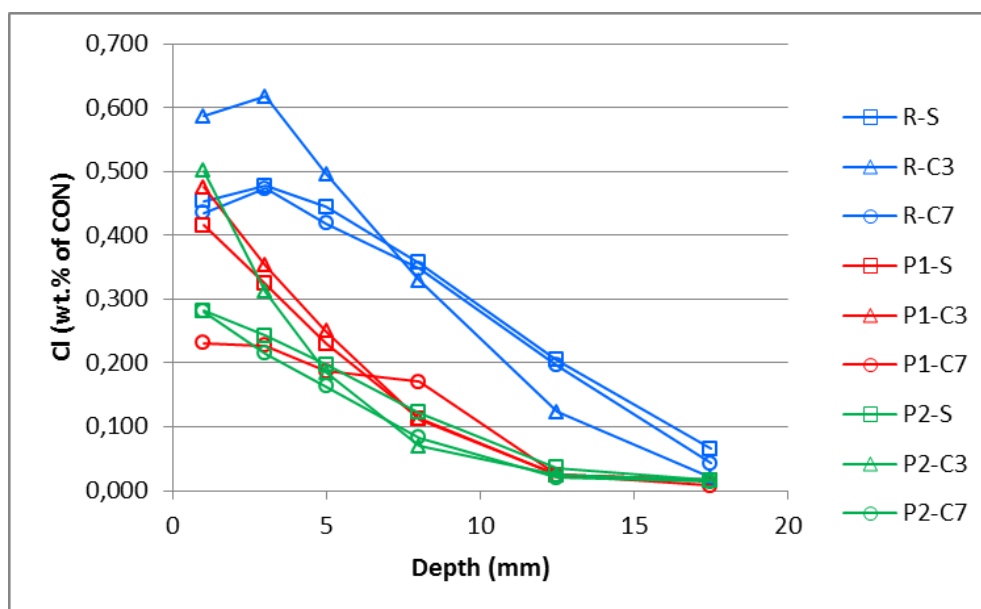
Figure 8. Chloride profiles measured for all series after an impregnation curing period of 14 days.
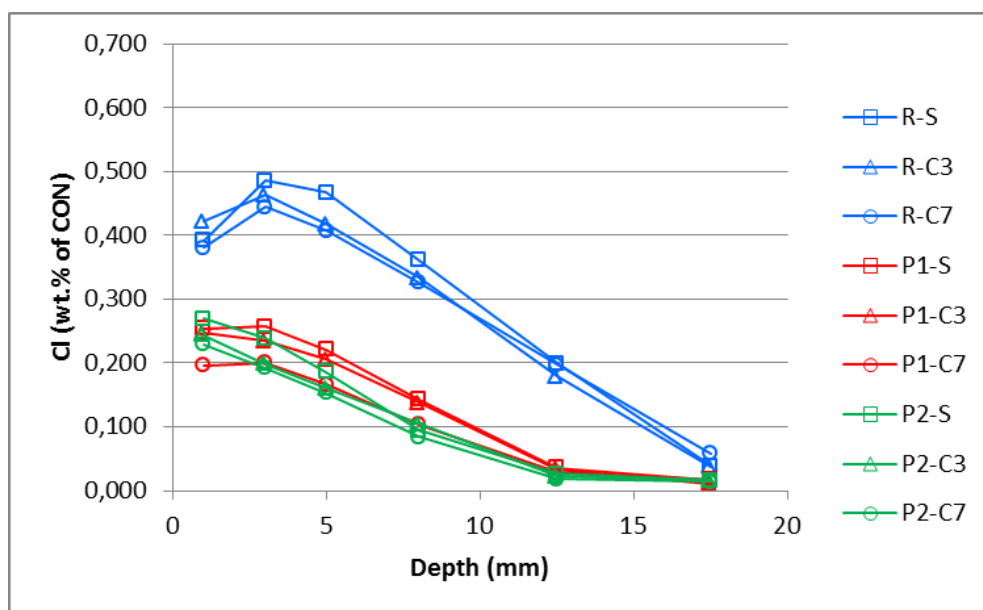
Figure 9. Chloride profiles measured for all series after an impregnation curing period of 28 days.
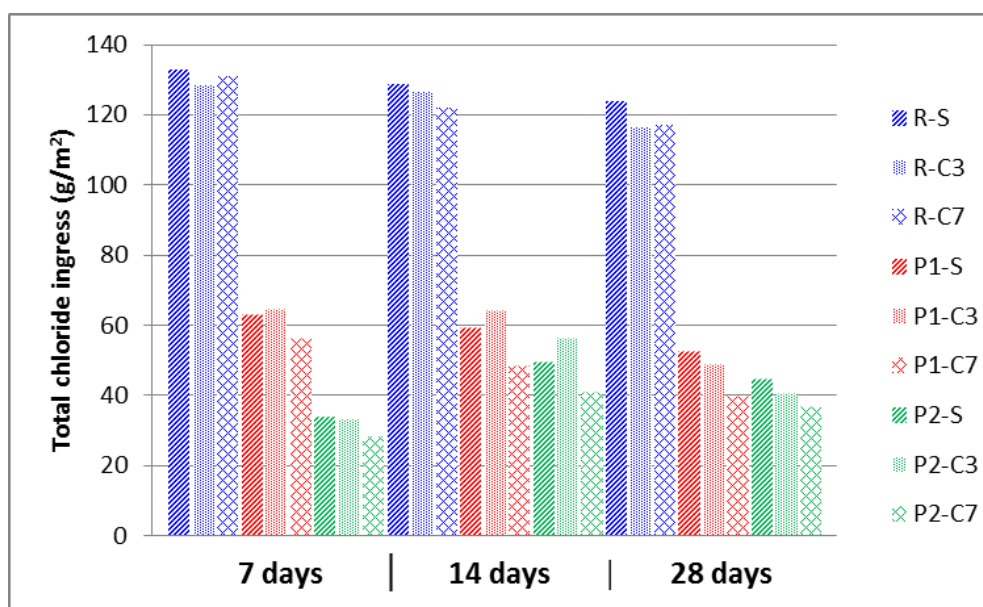
Figure 10. Total chloride ingress as a function of the impregnation curing period and the preconditioning.
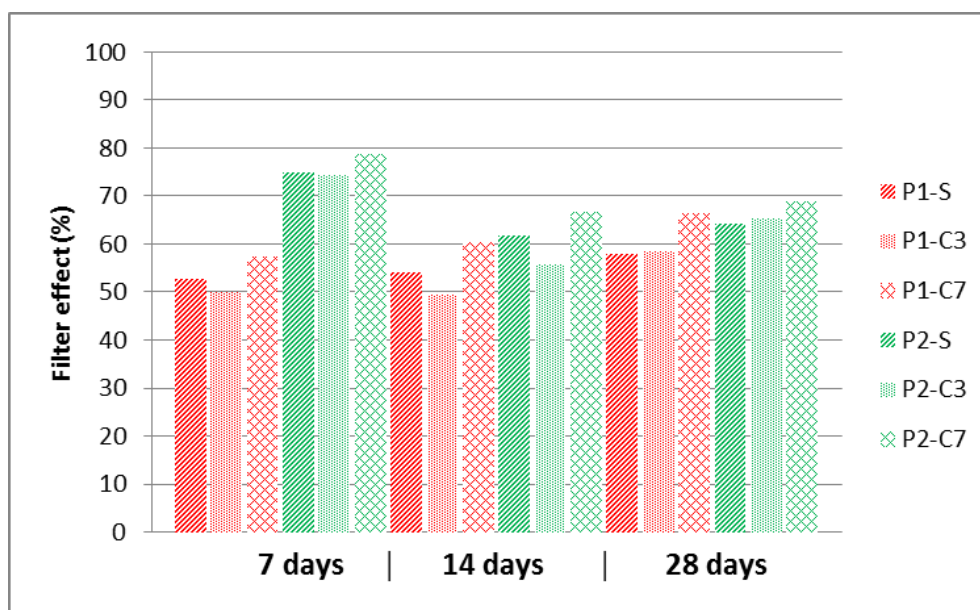
Figure 11. Filter effect for each product as a function of the impregnation curing period and the preconditioning.

Some general observations can be made:

- With the exception of product P2 with and an impregnation curing period of 7 days, the total chloride ingress in the impregnated specimens is generally larger the shorter the impregnation curing period, irrespective of the type of pre-conditioning. Correspondingly, the total chloride ingress in the references is larger the shorter the period they are exposed to 65% RH.

- With the exception of product P2 with an impregnation curing period of 7 days, the filter effect of the impregnation products is larger the longer the impregnation curing period, irrespective of the type of pre-conditioning.

- The chloride profiles after an impregnation curing period of 7 days and some of those with 14 days curing resemble profiles obtained in a diffusion governed process from the depth of 3 mm for the references and 1 mm for the impregnated samples inwards. The profiles in specimens with 28 days of curing and some of those with 14 days curing exhibits a more levelled out appearance, a lower chloride content close to the surface and a higher chloride content further inwards. This peculiarity appears both in references and in the impregnated samples.

4.3.2.3 Measured CaO-content in samples

The CaO-content was determined on all samples in the main series (sawn surfaces) of pre-conditioning type S and C3 and an impregnation curing period of 7 days. As can be seen in Figure 12 there seem to be no systematic variation with the depth which implies that the composition of the ground samples are fairly representative for the overall concrete composition over the whole depth investigated. There is however a small tendency that type S samples have a little bit lower CaO content in the first two millimetres than in the rest and the contrary for the C7 specimens, which have their highest value at that point. A higher value for C7 could be explained by an enrichment due

to the lime soaking and a lower value for the S specimens could be explained by carbonation. However, these tendencies are very weak and could also be due to the measuring error and variations in the composition. The conclusion is however that it does not seem to be necessary to determine the CaO-content to obtain representative results for this kind of concrete when sawn surfaces are used for the test.
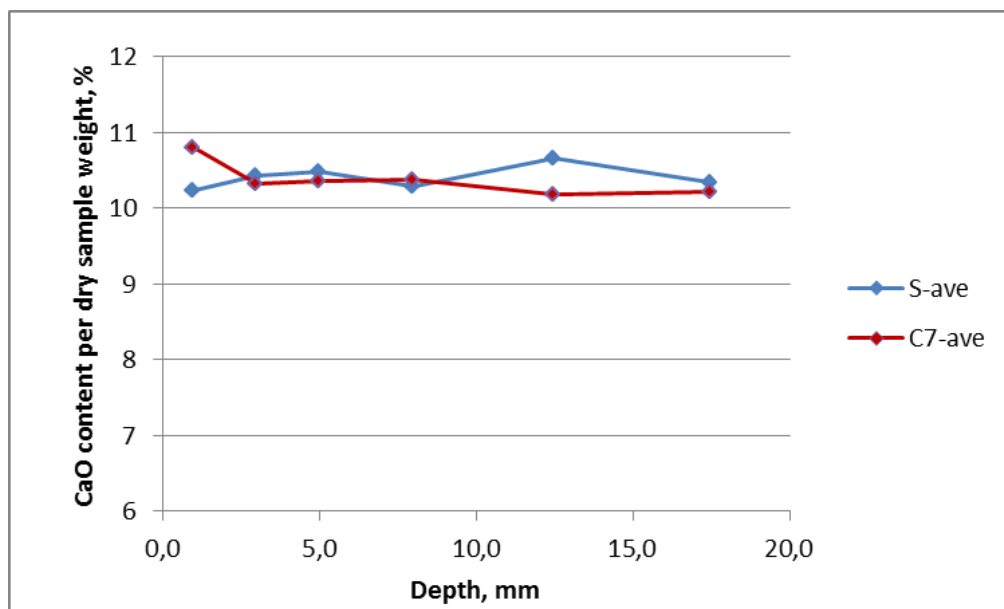
Figure 12: Average CaO content for 9 samples of series S and C7 (R, P1, P2) after an impregnation curing period of 7 days.

4.3.2.4 INFLUENCE OF THE TYPE OF SURFACE USED

The influence of the type of surface that was impregnated and exposed to chlorides on the chloride blocking effect was also studied. All specimens were preconditioned during 7 days at 20±2 ^{o}C and 60±10 % RH (type S) and cured after application of the impregnation for 28 days at 20±2 ^{o}C and 60±10 % RH. In a similar manner as in 4.3.1, the results for sawn and form surfaces are expressed in the form of chloride profiles in Figure 13. Figure 14 and Figure 15 show the total chloride ingress and the filter effect for the form surfaces, compared to those obtained for the sawn surfaces. Individual measurements and calculations are given in Annex C.3.
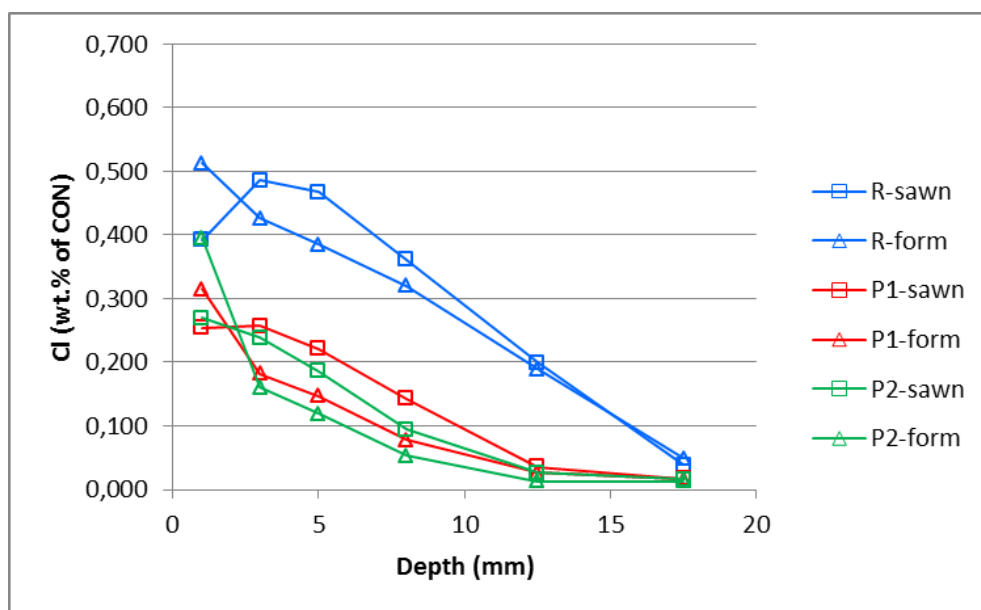
Figure 13. Chloride profiles as a function of the type of surface treated/exposed.
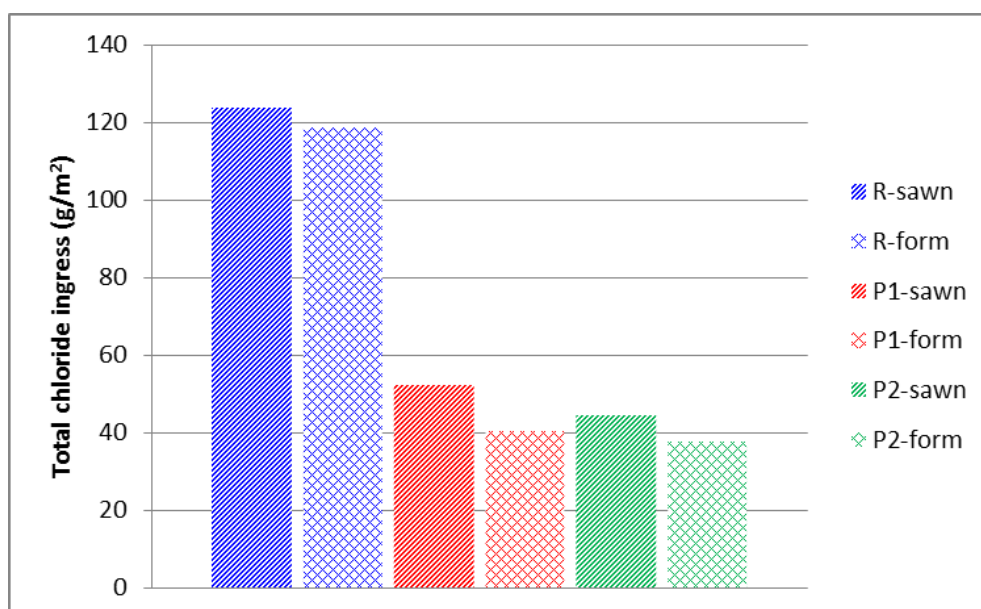
Figure 14. Total chloride ingress as a function of the type of surface treated/exposed.
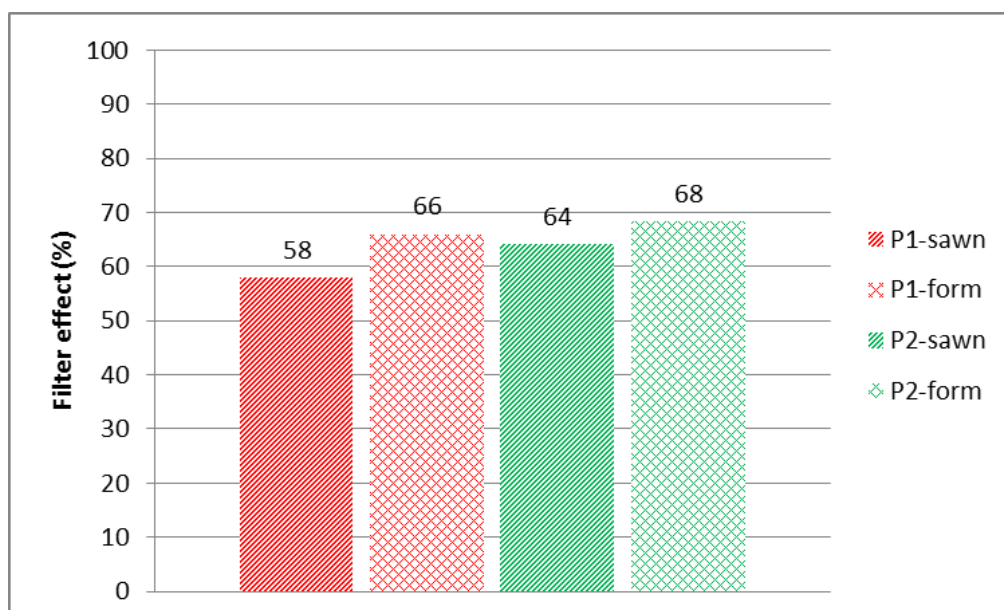
Figure 15. Filter effect for both products tested, as a function of the type of surface treated/exposed.

4.3.3 Discussion

4.3.3.1 PENETRATION DEPTH

As can be seen in Figure 4 there is no significant difference between the impregnation depths of both products tested. Although P2 shows a slightly better/deeper penetration, it also shows somewhat higher uncertainty in the measurement. The most important parameters influencing the efficiency of the treatment are the porosity of the concrete (mainly controlled by the w/c-ratio and curing conditions), the moisture content of the concrete at the time of the treatment and the contact time between the impregnation agent and the concrete surface (Selander, 2010). In the present study, both products tested are of the silane-type; however, while P1 is a liquid, P2 is a gel containing a thickening agent (bentonite clay) which can contribute to an increased contact time between the impregnation agent and the concrete surface and lead to a deeper penetration. This is important when hydrophobic impregnations are used in the field, especially in the case of denser surfaces (low w/c-ratios, e.g. 0.45) and wet/moist concrete. It is worth noticing that P2 is also applied in higher concentration (500 g/m^2) than P1 (300 g/m^2). However, the difference in the test will not be as pronounced as it would be in a real case since the side of the specimens were wrapped in order to prevent the liquid from running off.

The better/deeper penetration measured for specimens for which preconditioning type S is used (Figure 4) may be explained by the length of the drying period (7 days without pre-wetting for S and 3 days after 3 days pre-wetting for C3), since the moisture content of the concrete at the time of the treatment is one of the most important parameters influencing the penetration depth.

With regards to the development of a test method, it is recommended that preconditioning type S is adopted; as it is apparent from Figure 4, this not only leads to deeper penetration of the impregnation, but also to lower absolute standard deviation (at least when compared to series type C3).

4.3.3.2 CHLORIDE INGRESS

4.3.3.2.1 Influence of preconditioning before application

The chloride profiles in Figure 7 to Figure 9 show that there is no significant influence of the pre-conditioning environment before surface treatment on the chloride blocking effect, except for the specimens on of type C3-14, which show higher chloride contents up to a depth of about 6-7 mm and similar or lower chloride concentrations at deeper levels (Figure 8). The total chloride ingress (Figure 10) is somewhat higher for reference specimens of type S. Treated samples of type C7 always gives the lowest value. Consequently, the filter effect (Figure 15) is always slightly higher for specimens type C7, i.e. it is slightly increased when the surface is soaked with saturated $Ca(OH)_2$-solution before being exposed to lower RH:s and subsequently exposed to chlorides. If this is due to chemical or physical reasons cannot be deduced from this study. In addition, specimens of the type C3 were pre-wetted and only dried for 3 days before impregnation, which, as discussed in 4.3.1, significantly limits the penetration depth of the impregnation agent.

With regard to the development of a test method, none of the preconditioning environments gave a clearly better robustness in the test results, as can be seen from the absolute standard deviations (Figure 16 to Figure 18). Thus, pre-wetting of the surface which is added in order to provide a well defined moisture condition at the start of the pre-conditioning does significantly increase the reproducibility and can be omitted. Moreover, from a practical point of view, preconditioning types C3 and C7 increases the number of experimental steps. Additionally, it should be pointed out that such a procedure (immersion in or soaking with calcium hydroxide solution) is not something that is used in practice when real structures are impregnated.
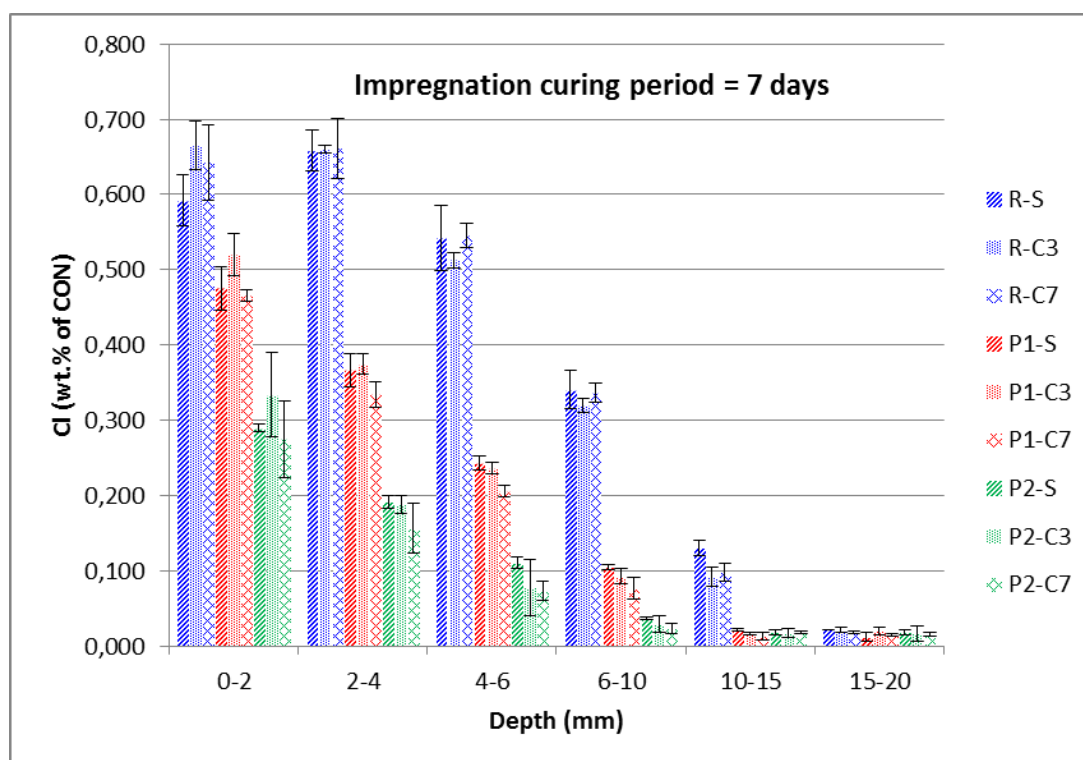
Figure 16. Chloride profiles measured for all series after an impregnation curing period of 7 days. The error bars represent 2 standard deviations.
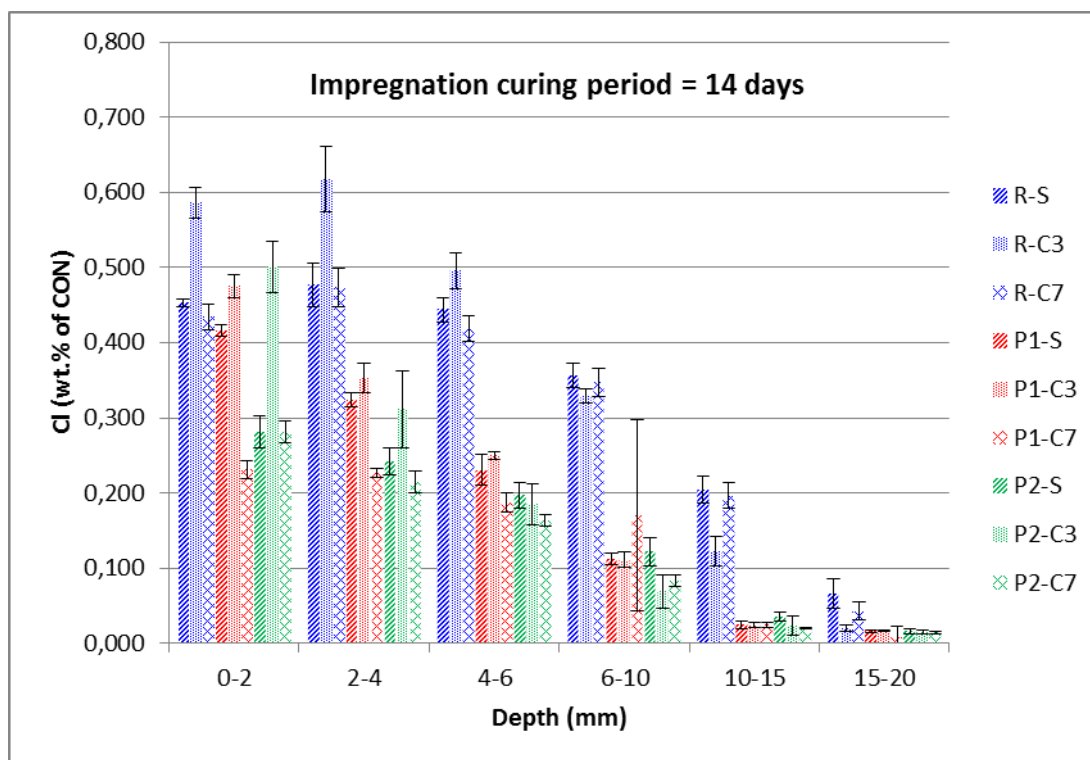
Figure 17. Chloride profiles measured for all series after an impregnation curing period of 14 days. The error bars represent 2 standard deviations.
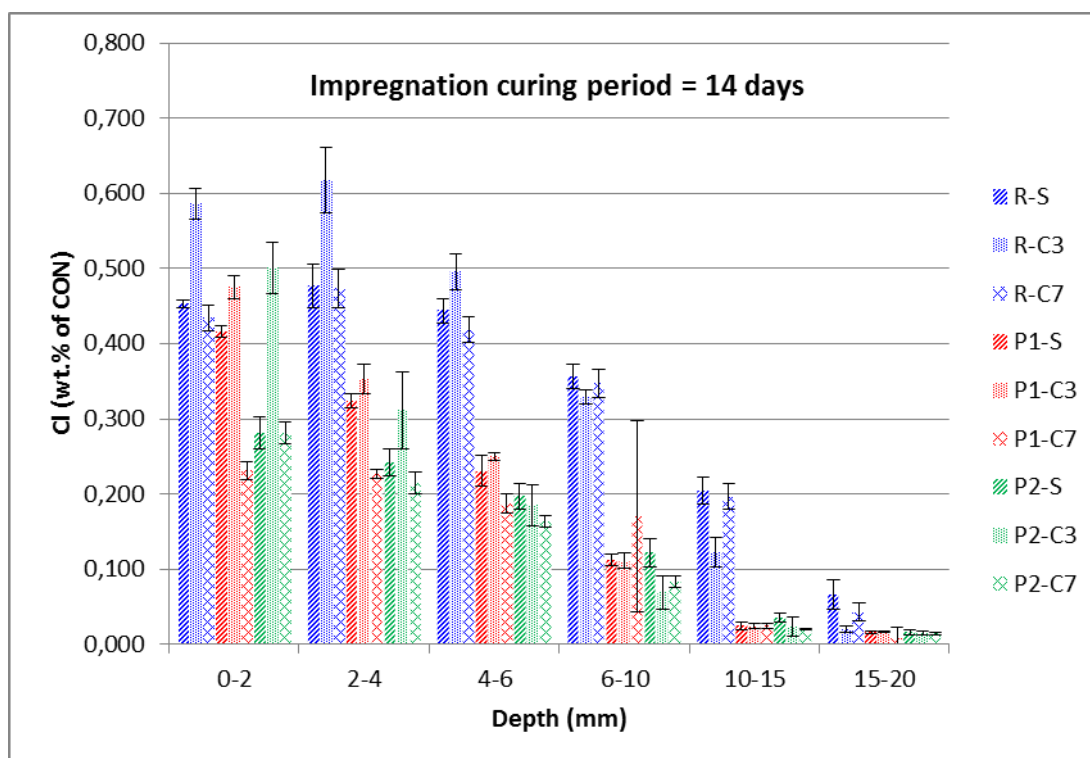
Figure 18. Chloride profiles measured for all series after an impregnation curing period of 28 days. The error bars represent 2 standard deviations.

4.3.3.2.2   Influence of the impregnation curing time

In order to achieve a durable impregnation, it is very important that the impregnation agent is given enough time to penetrate the concrete and polymerize within its pore structure (Selander, 2010). In this regard an effective contact (sufficient contact time and adequate moisture level in the specimen) and a sufficient impregnation curing period play significant roles. The results in Figure 10 show that the total chloride ingress slightly decreases with the curing time for both reference samples and specimens treated with product P1, while for specimens treated with product P2, there is a minimum after an impregnation curing period of 7 days curing and maximum for the specimens cured during 14 days. A decrease in total chloride content with increasing length of the impregnation curing period may be explained by the continuous cement hydration (leading to a lower porosity and a refinement of the concrete pore structure) and increasing polymerization of the silane agent leading to a larger degree of hydrophobicity in the pore structure. The combined effect of this is that the filter effect will increase with increasing impregnation curing period. This is also the case except for the values obtained with product P2 after an impregnation curing period of 7 days, where the filter effect is surprisingly high compared to the values after 14 and 28 days curing. Why does then this gel product seem to be extremely effective after only 7 days of curing? The answer may be due to the nature of this gel. Bentonite clay is added to the silane in this gel, in order to prolong the time the concrete surface is exposed to the silane by decreasing the evaporation rate of the solvent. In (Rodum, 2013) this particular product was used, and when applied to a concrete with w/c = 0.45 the product still formed a continuous wet gel on most of the concrete surface after 7 days of curing. After 28 days of curing the product had dried completely and the concrete surface shows a flaky structure consisting of deposited bentonite clay. When applied on a w/c = 0.70 concrete the product has dried already after 7 days curing. It may be that this continuous gelatinous surface layer provides an extra physical barrier as long as it persists, slowing down the ingress of chlorides. Thus, the filter effect obtained with specimens with such a vulnerable surface layer will not give results that are representative for the effect of the product on long term.

Another peculiarity of the chloride profiles are the difference in shape observed between those specimens cured 7 days and 28 days and the instability of those cured 14 days, pointed out in 4.3.2.2.

When specimens with a moisture profile with very high RH:s at the surface and a dryer interior are sealed in plastics for a period of time a certain redistribution of the moisture profile will take place. The diffusion and levelling out of chlorides may also continue in a sealed specimen at high RH:s.

The specimens were sealed in plastics and stored in 21±2 °C until grinding was taking place. No time limits were given for this period and as pointed out in 4.2.6 the length of this period varied substantially between different specimens and series. A maximum time of one week was used for those cured 7 days while those cured 28 days were stored for a minimum of 30 days. For those cured 14 days it was not possible to draw any decisive conclusions. Looking at the chloride profiles it is highly probable that redistribution of chlorides within the specimens when the storage time was extended, plays an important role in the difference between the appearance of the chloride profiles.

With regard to the development of a test method it is important to realize that:

- the performance of impregnation agents is influenced by the length of the impregnation curing period;

- it is not certain that full polymerization will be achieved even after 14 days;
- short-lived phenomenon like the one experienced with product P2 may exist
- extended storage times between exposure to chlorides and analysis must be avoided.

It is therefore recommended that the storage time should be kept as short as possible and an impregnation curing period of 28 days is used before the specimens are exposed to chloride solution. For structures in road environment, the impregnation is often carried out during the summer, months before they are exposed to chlorides during the winter season. For structures in a marine, exposed to sea-water, 28 days may seam as an unrealistically long period. However, one must bear in mind that in the method constant immersion at a rather young age is used while the surface of real marine structures may be exposed to the first shower very soon after the impregnation, but will have the possibility to dry and harden between showers.

4.3.3.2.3 Influence of the type of surface

One debated feature of the proposed test is that sawn surfaces are used instead of form or cast top surfaces. In the existing Finnish method form surfaces are used but in the Swedish and Norwegian methods, sawn surfaces are treated and exposed to chlorides. Although form surfaces or cast top-surfaces would be more realistic with regard to surfaces on which impregnation agents are normally used, it was decided to use sawn surfaces at a rather early state of the project. The motivation for this is that reproducible surface characteristics are more easily obtained that way. The surface characteristics on a form surface depend among other things on the form material, possible use of release agents and subsequent curing conditions. On a cast top-surface laitance may increase the variability even further.

Close to the form surface the composition of the concrete deviates from the composition in the interior of a specimen due to the *wall effect*. In a container packed with mono-sized particles only the wall effect causes the fraction of voids to attain one maximum value at the distance equal to the particle diameter from the wall and a minimum value at a distance equal to half the particle diameter from the wall then increasing asymptotically towards 1 at the interface, se for instance (Mueller, 1997). Fresh concrete is composed of particles ranging in sizes from D_{max} of the aggregate to the grain size of the cement or additions; in this study from 10 mm down to μm. Even though this smoothens out the curves of the fraction of voids, there will still be a certain influence of the wall effect close to a form surface. This will manifest itself as a larger proportion of paste in this zone and possibly also a somewhat higher w/c-ratio leading to a higher porosity per concrete volume close to the surface than in the interior of the specimen. This layer will therefore have a larger proportion of pores, which may contain free chlorides and more hydration products. This may increase the chloride binding capacity of the concrete. Using form surfaces for the test would thus lead to higher maximum chloride levels in the surface layer compared to when sawn surfaces are used.

How large may the influence of the higher porosity caused by the wall effect be on the maximum chloride levels? From data presented in (Boubitsas, et al., 2014) it was found that the binder content in the 2 mm closest to the surface is in average 15% higher than in the interior. Logically, the calculated chloride levels also rises by 15% due to the wall effect. In data made available by VTT the cement content in the surface layer of a concrete was about 35% higher than in the bulk.

Thus the wall effect when form surfaces are used may for the concrete used in these experiments cause an increase in chloride content (per weight of concrete) with around 0.09-0.10% in the

outermost surface layer compared to sawn surfaces, when all pores are filled with 15% NaCl-solution.

In addition to the wall-effect the curing of the concrete will directly influence the state of the surface to be treated and exposed. A curing regime that is as similar as possible to what a concrete structure (with the exception of under-water cast structures and structures in the splash zone), that is going to be impregnated is subjected to in reality would be a short period of wet curing or curing under cover and thereafter curing in air. Even though the RH-level for the air curing may be specified, it is difficult to control the other parameters influencing the hydration process of the concrete and the state of the surface; for instance movement of air around the specimen which influences the drying rate and the CO_2-content of the air causing carbonation. The conditions during water curing are more easily controlled than during air curing and is therefore normally used for the first 28 days after casting, when tests are carried out on form surfaces. However, water curing may also induce unwanted variations on the surface conditions.

One un-disputable effect of applying different curing regimes to the surfaces is that it will lead to a difference in *degree of hydration* between the surface and the bulk concrete. The interior of the specimen will have reached a relative humidity (RH) close to what is obtained by self-desiccation, i.e. around 90% (Johansson, 2015) and this level will prevail during the experimental period, irrespective of if the specimen is water cured or cured at lower RH. An RH-level of 100% will only be reached close to a water exposed surface. For a specimen that is moist cured during only the first days and then air cured the RH-level will be close to the ambient RH in the outermost surface layer. Thus, a water cured specimen will reach a higher degree of hydration at 28 days in the surface layer (form surface or top-surface) than in the interior (sawn surface). An air cured will have a lower degree of hydration in the surface than in the interior. Higher degrees of hydration lead to lower porosity and consequently lower maximum chloride level. If the degree of hydration is assumed to be increased with 0.10 in the concrete used in these experiments, the pore volume is decreased with about 6%, which will lead to a decrease of the chloride content of the samples with 0.035%-units when all pores are filled with 15% NaCl-solution.

Another effect that may occur when specimens are water cured is *leaching* of $Ca(OH)_2$ from the specimen surface to the water bath. Leaching of $Ca(OH)_2$ lead to an increase of the pore volume in the concrete, a volume that can be filled with NaCl-solution, thus increasing the maximum chloride level in the concrete.

Assuming a degree of hydration of 0.7 and that all $Ca(OH)_2$ present in the surface of the hydrated concrete has leached out there is a potential to increase the chloride level with about 0.16%-units in the concrete used in the pre-study.

During air curing the sample surfaces will inevitably carbonate unless special measures to exclude CO_2 from the surrounding air are taken. Carbonation also leads to a change in the pore structure. As regards to the total pore volume it will be decreased since the carbonation product, $CaCO_3$, has a slightly larger volume than $Ca(OH)_2$. However, this volume change is only marginal.

All these phenomena will influence the porosity, the moisture condition and the degree of hydration in the surface layer and especially if they are varied during the period when the pore-structure and properties of concrete are built up, i.e. during the first 28 days. Surfaces created by sawing specimens at a stage when the specimens have reached a relatively high and uniform degree of hydration are not as sensitive to variations in ambient climate as "young" surfaces.

How are then these differences manifested in the chloride profiles and the functioning of the impregnation agents?

Chloride profiles of water cured form surfaces and sawn surfaces from the same concrete were obtained in the pre-study (Figure 13) in a study for Wacker Kemi AB[1] and by combining data from the round robin exercise with data obtained with the existing Finish method, both sets from VTT and within this project. The results from these studies are shown in Figure 19. The results from Wacker are obtained on a concrete with a w/c = 0.70.
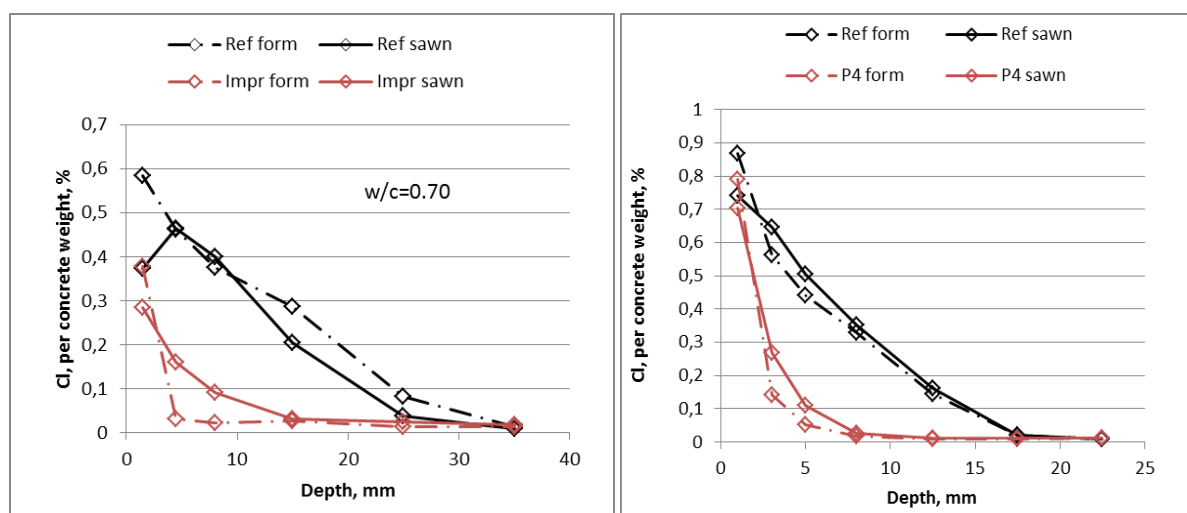
Figure 19: *Measured chloride levels in reference samples with form and sawn surface From Wacker to the left and from data obtained at VTT to the right.*

In all these cases, the measured maximum chloride content is always higher when form surfaces are used compared to when sawn surfaces are used, both in references and in impregnated samples. The profiles in the form references always have their maximum at the surface with a smoothly sloping curve towards the interior. When sawn surfaces are used the chloride level close to the surface seems to be lower than expected, and in most cases the highest chloride level is obtained in the next layer. This peculiarity is probably not directly caused by the wall-effect or the other possible causes put forward here and will be more thoroughly discussed in 5.4.3.

With form surfaces a more smooth curve continuously decreasing from the surface as one goes deeper into the specimen is obtained in the references.

In all the results, the chloride content is lower in the impregnated specimens in which the form surface was treated and exposed, with the exception of the first surface layer. This is also valid for all references except those obtained by Wacker.

In all these cases, the chloride content is expressed in weight % of concrete. It is reasonable to assume that when expressed in weight % of binder, the chloride content in this first layer (2 mm thick) is substantially decreased and possibly similar to what would be obtained if sawn surfaces were used.

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[1] Personal communication, Susanne Stubb-Eliasson, Sales manager, Wacker Kemi AB, Stockholm, Internal report, 2010

Additionally, the results in Figure 14 show a slightly lower total chloride ingress in all specimens, both references and treated ones, when form surfaces are used. The same tendency can also be seen in the results shown in Figure 19.

Figure 15 shows that the filter effect becomes somewhat larger when form surfaces are used. When the filter effect is calculated based on data shown in Figure 19 the same observation can be made. With the VTT results the filter effect becomes 0.60 with a sawn surface and 0.63 with a form surface. When it comes to the Wacker results, obtained on a less dense concrete, the difference is large; the filter effect obtained on a form surface becomes 0.78 while on a sawn surface only 0.63.

One must though bear in mind that none of these two options represent how a concrete surface that is to be impregnated is treated, and can not be regarded as giving "true" values directly comparable to what will be obtained in the field.

The aim here is to find a method to classify hydrophobic impregnations with regard to their chloride blocking effect. For that the surface they are applied on, in the test, must be as well defined as possible, to avoid measuring uncertainties. The easiest way to obtain a test surface that will give reproducible results is to use surfaces sawn out of mature water cured concrete specimens, and keep the period in 65% RH before application of the impregnations short enough to avoid significant carbonation. As has been shown in 4.3.2.3 it is then not necessary to determine the CaO-content in the samples.

4.3.3.2.4   Performance of the impregnation agent

The results in Figure 11 show that both products behave in a similar way with regard to the chloride blocking effect in the long run; this is not surprising, since both product P1 and product P2 are agents of the silane-type. Regardless of the conditions tested, both products reduce the total chloride ingress with more than 50% when compared to non-treated samples.

4.4   Concluding remarks on the pre-study

In the pre-study, the influence of the precondition environment before treatment and the length of the impregnation curing period on the chloride blocking effect was studied. From the results obtained, the following major conclusions can be drawn:

- None of the three preconditioning environments tested (S, C3 and C7), resulted in significantly different chloride blocking effects or better repeatability of the method. The duration of the exposure to 65% RH was found to be the most influential factor;
- The chloride protection slightly increases with the impregnation curing time. However, it is not clear which factor is most dominant for this increase; if the continuous polymerization of the impregnation agent or the continuous cement hydration. Nevertheless, enough curing time is necessary for the treatment to be efficient, which may not be achieved at the end of 14 days. therefore it is recommended that a curing period of 28 days is given before exposure to chlorides;
- Impregnation applied to form surfaces exhibit somewhat better chloride blocking effect compared to when the product is applied to sawn surfaces. However, if this is due to an

easier and better penetration of the impregnation product in the outermost surface layer which is richer in paste and more porous or some other reason is not clear;

- Of the two products tested, product P2 generally shows a higher chloride blocking effect. However, this difference decreases with the impregnation curing period. Both products reduce the total chloride ingress by more than 50%.

Additionally, the penetration depth was also studied and no significant differences were found between the two products tested. However, for both products tested, preconditioning for 7 days in 60% RH (type S) was found to lead to deeper penetration and lower standard deviation when compared to 3 days in 65% RH (type C3), which also included pre-wetting with $Ca(OH)_2$-solution.

4.5   Implications and recommendations for the further development of the method

From the results obtained in this pre-study, the following main experimental test conditions are recommended in the design of a test method:

- Surfaces sawn out of water cured concrete cubes (w/c = 0.45, D_{max} = 10 mm) at the age of 28 days shall be used;
- Preconditioning of the test surfaces before treatment: 7 days at 20±2 ^{o}C and 60±10 % RH;
- Curing of the hydrophobic agent: 28 days at 20±2 ^{o}C and 60±10 % RH;
- Acid soluble chloride content (total chloride content) determined by potentiometric titration or another method with the same or better precision;
- Determination of the CaO-content is not necessary for this type of concrete when sawn surfaces are used.

5 ROUND ROBIN TEST

5.1 Objective and general set-up of the round robin exercise

The round robin exercise was carried out with the preliminary formulation of the test method based on the two previous phases of the project. The primary objective was to determine the reliability and reproducibility of the method with regards to the chloride blocking effect of hydrophobic impregnations.

Three laboratories were involved in this phase, CBI-Borås in Sweden, VTT in Finland and SINTEF in Norway.

The basis for the round robin exercise was as follows:

- All parts of the tests should be carried out by the separate laboratories according to given specifications;
- The concrete specimens should be manufactured at each laboratory using local materials;
- The same three impregnation agents should be used in all laboratories;
- The chloride profiles and the chloride blocking effect of the impregnation agents should be determined;
- The concrete compressive strength and density and the penetration depth of the impregnations should also be determined.

5.2 Experimental

Sections 5.2.1 to 5.2.6 summarize the specification of materials and methods for the round robin exercise. Section 5.2.7 points out some details that are diverging in the manner the three different laboratories have carried out the testing. The description of the test method for the chloride blocking effect given for the round robin exercise can be found in Annex D.1.

5.2.1 Impregnation agents

Three different silane-based surface treatments were selected:

- The same gel with bentonite clay, applied with a concentration of 500 g/m^2, of which 90% is silane, as was used in the pre-study referred to as P2.
- A liquid, applied with a concentration of 200 g/m^2 (99% silane). This product is further on referred to as P3 (This product is essentially the same as product P1 used in the pre-study, marketed under a different trade name. Since the concentration differs from in the pre-study it is given a separate designation).
- A thixotropic cream with 80% silane, applied with a concentration of 200 g/m^2. This product is further on referred to as P4.

More data on the products can be found in Annex E.

5.2.2   Specimen preparation

Concrete specification:

- Type MC (0,45) according EN 1766 (CEN, 2000);
- Cement: 395 kg/m^3 of concrete of CEM I 52.5 N or R;
- Maximum aggregate size: 10 mm;
- w/c ratio: 0.45.

Specimens were prepared and cured according to the same procedures as described in section 4.2.2. 5 specimens (half-cubes) per impregnation product and another 5 half-cubes for reference (referred to as R) were used for chloride exposure.

The penetration depth was determined on three half-cubes with the "split method" (see 4.2.5) and on one half-cube with the "drop method" for each impregnation product (see 5.2.5). Additionally the compressive strength and the density at the age of 28 days were determined as the average of three specimens.

5.2.3   Specimen preconditioning

The specimens to be used in the chloride ingress test and penetration depth tests should be preconditioned 7 days at 20±2 °C and 60±10 % RH.

5.2.4   Application and curing of the impregnation agent

The hydrophobic agents, P2, P3 and P4 were applied following the recommendations given by the producers, in the concentrations of 500 g/m^2, 200 g/m^2 and 200 g/m^2 respectively. After application of the impregnation the specimens should be cured for 28 days at 20±2 ^{o}C and 60±10 % RH.

5.2.5   Penetration depth

The penetration depth of the surface treatment shall be determined after 28 days of impregnation curing with two methods:

- "Split method", same as in the pre-study (4.2.5):
- "Drop method": Grinding the concrete in intervals down to a depth of 7 mm from the exposed surface. The following layers were ground: 0-2; 2-3; 3-4; 4-5; 5-6 and 6-7 mm. The powder from the different layers was then exposed to water drops and the hydrophobic properties (wetting behaviour) of the powder shall be observed and evaluated as hydrophobic or hydrophilic.

5.2.6   Exposure, chloride and CaO-analysis

After curing, both references and impregnated specimens were immersed in a 15% NaCl solution (by weight), in separate containers, during 56 days. At the end of the exposure period, the samples were removed from the solution, surface dried with absorbing paper, sealed in plastic bags and stored at 5±2 ^{o}C before profile grinding. The period between end of exposure and grinding was maximised to 7 days.

The following layers were ground: 0-2; 2-4; 4-6; 6-10; 10-15, 15-20 and 20-25 mm.

The acid soluble chloride content in each layer was determined by potentiometric titration according to AASHTO-T260 (AASHTO, 1997) (or another method with the same or better precision) and expressed in % of the dry sample weight.

For selected specimens, the calcium content was determined by potentiometric titration (see Annex D.1.8) as an estimate of the cement content.

5.2.7 Used materials and methods - some diverging details

It was attempted to make the specification of the experimentals in the round robin exercise as clear and unambiguous as possible. However there was some degree of freedom and also some details in the specification that gave room for interpretations; consequently there has been some divergence in the execution of the tests in the different laboratories. The differences we have become aware of are listed in the following.

5.2.7.1 CEMENT TYPE

The cement in the concrete specimens was specified as a CEM I 52.5. CBI used a CEM I 52.5 R (Cementa Snabbhårdnande) and VTT used a similar cement from Finnsementti (CEM I 52.5 R, Pika-sementti). Both of these rapid hardening cements had a grain size between 500 and 600 m^2/g. However, SINTEF used a CEM I 52.5 N intended for infrastructures with a limited equivalent alkali content (max = 0.6 %) and a grain size around 400 m^2/kg.

5.2.7.2 CLIMATES DURING PRE-CONDITIONING AND IMPREGNATION CURING

The climate during pre-conditioning and impregnation curing was specified as 21±2 °C and 60±10 % RH for both references and impregnated specimens. CBI and VTT used climate rooms regulated at 65±5 % RH for both these periods. SINTEF, however, used a climate room regulated at 60±5 % RH for the pre-conditioning and a climate room regulated at 50±5 % RH during the impregnation curing period. In the climate rooms at SINTEF and VTT there were no registration of the carbon dioxide, but in both cases with little personnel movement. The climate room used at CBI is one frequently used also for other purposes, with many passages in and out. In this climate room the carbon dioxide content is registered and it lies normally around 0.4% CO_2.

5.2.7.3 OVEN DRY OR SURFACE DRY?

The specification said that the dry density should be recorded and that the chloride analysis should be carried out on dry specimens. However, when it comes to concrete dry may signify several different conditions, anything between surface dry and oven-dry. In this case VTT interpreted dry as surface dry while SINTEF and CBI interpreted it as oven-dry.

The final method specification must be totally clear on this point.

5.3 Results and observations

5.3.1 Compressive strength and density

Although there was no specification with regard to the compressive strength of the concrete in the project and different cements were used (5.2.7), it can be seen from Figure 20 that the deviation

between the compressive strength after 28 days of the concrete cubes fabricated in the three laboratories is small.
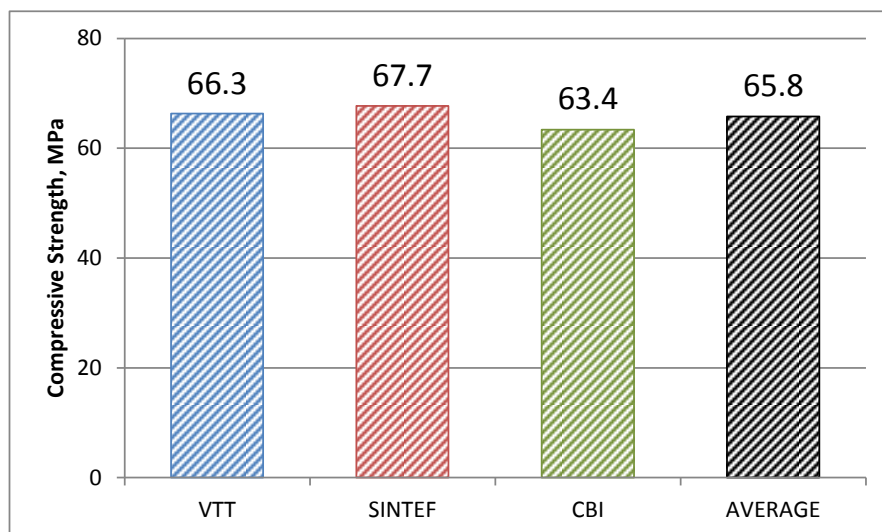
Figure 20: 28 day compressive strength of the concretes.

When it comes to recorded dry density the deviation is somewhat larger as can be seen Figure 21, where the value obtained at VTT is surprisingly high. However, this was found to be due to different interpretations of the term dry density. CBI and SINTEF have measured the oven-dry density while VTT determined the surface dry density. If the degree of hydration is supposed to be 0.7 at 28 days the oven dry density of the concrete at VTT becomes approximately 2260 kg/m^3 which is very close to the values obtained at CBI and SINTEF.
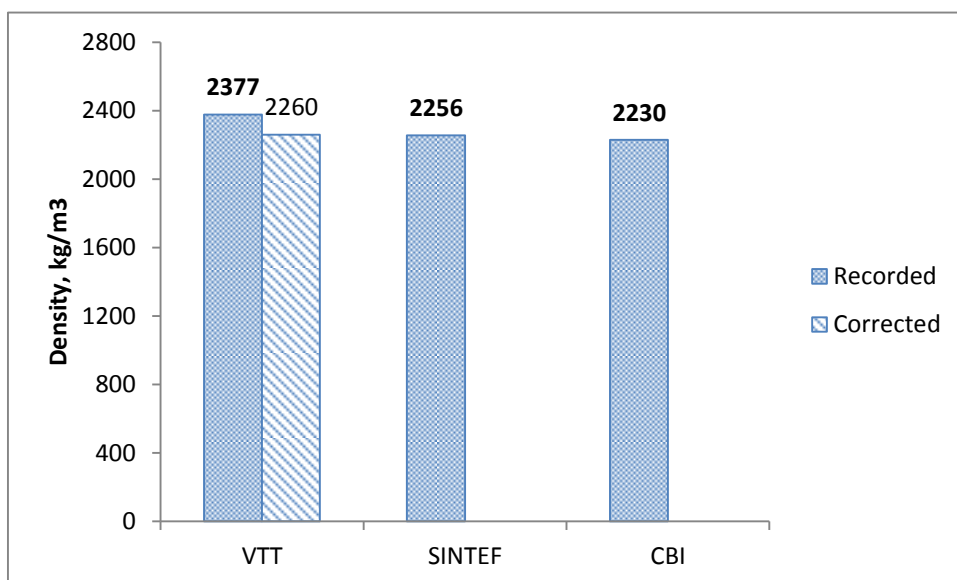
Figure 21: Recorded and corrected oven dry density of the concretes.

5.3.2 Penetration depth

Determination of the penetration depth is not the primary subject of this project, and not a part of the proposed method. However the penetration depth is a crucial parameter for obtaining an impregnation that withstands weathering for longer periods and it is important to ensure that the chloride blocking effect determined with the proposed method is obtained on an impregnation that has penetrated sufficiently into the concrete surface. The penetration depth was therefore tested on impregnated specimens.

The penetration depths determined with the method referred to in EN 1504-2, the "split method" are shown in Figure 22. It is well known that this method is very subjective and the results vary substantially when it is used by different persons, especially in the case of dense concrete as in this project. This is also manifested in the results, where the difference between laboratories is substantial.
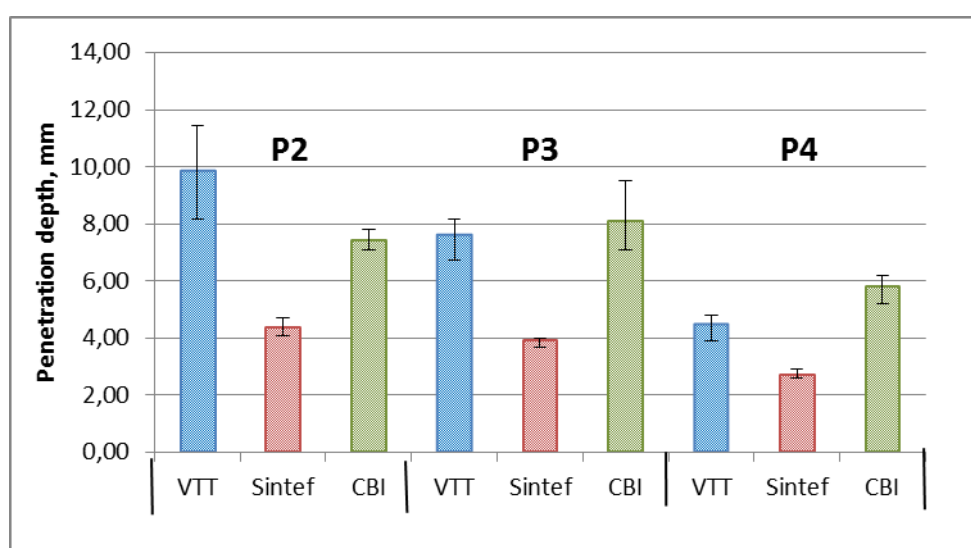
Figure 22: Penetration depths obtained with the split method (EN 1504-2). Average + max and min values.

The penetration depth was therefore also estimated by exposing powder ground from the surface of the impregnated specimens in layers down to 7 mm to a water drop, followed by evaluation of the hydrophobic appearance of the powder samples.

The results from the "drop method" were recorded somewhat differently by the three laboratories. SINTEF and CBI made a graded evaluation where the hydrophobicity in the layers was graded from 6 (full hydrophobicity) to 0 (no hydrophobicity). VTT graded the layers into only two categories, hydrophobic or not. The graded values were transformed into a single penetration depth in the following way: the sum of the thickness of each layer multiplied by the evaluated hydrophobicity of the layer (value between 0 and 6), divided by 6 and multiplied by 7 (= total evaluated thickness).

The results obtained with the "drop method" are given in Figure 23 together with the results obtained with the "split method". As can be seen the coherence between the "drop-method" results is higher than between the "split-method" results.
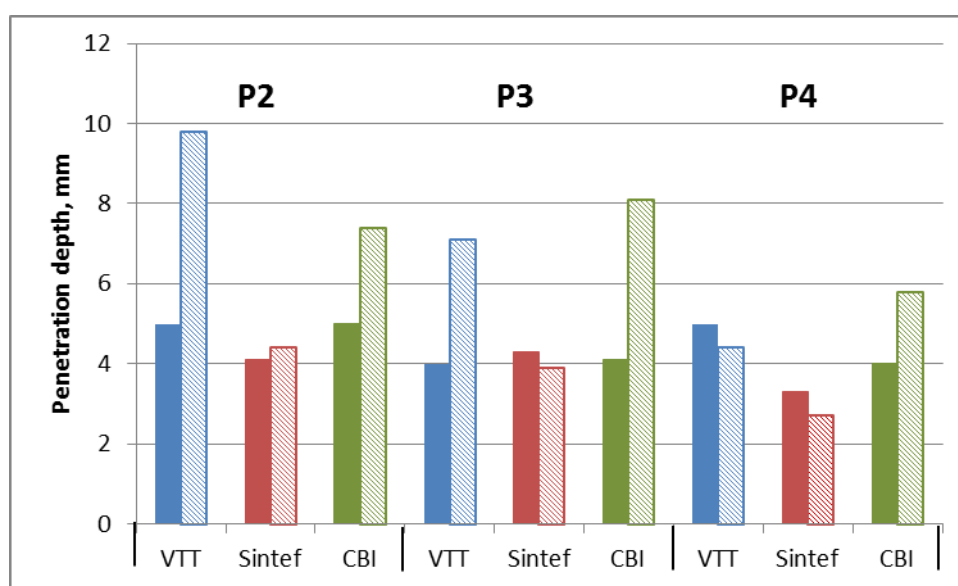
Figure 23: Penetration depth evaluated with the "drop method" (darker bars) and with the "split method" (lighter bars).

5.3.3 Chloride ingress

5.3.3.1 CHLORIDE PROFILES PER DRY SAMPLE WEIGHT

Chloride profiles obtained at the three laboratories, as % per oven-dry sample weight, where the initial chloride content has been deducted are given in Figure 27 to Figure 27 for the references and the impregnated samples. As can be seen in these figures highly reproducible results were obtained, especially in the inner parts of the samples. Yet, some differences are noticeable.

In the references the chloride profiles of the three laboratories are very similar from the layer representing 6-10 mm (value at 8 mm) and beyond, but diverges closer to the surface (Figure 24). CBI shows a profile where the chloride level in the two first layers (0-2 and 2-4 mm) is approximately the same. The VTT results show a higher chloride level in the first 0-2 mm and a rather even slope down to the layers beyond 6 mm. The chloride profile obtained on the references at SINTEF has the same chloride level in the first layer as CBI, but thereafter an even slope down to the layers beyond 6 mm.

When it comes to the chloride profiles of the impregnated samples, VTT shows a higher chloride level in the first layer (0-2 mm) than CBI and SINTEF, which show similar chloride for all three products. Chloride profiles obtained at the three laboratories for products P2 and P3 are otherwise more or less similar. For product P4 there is a larger divergence between the chloride profiles obtained at the three laboratories also between 4 and 10 mm (Figure 27).
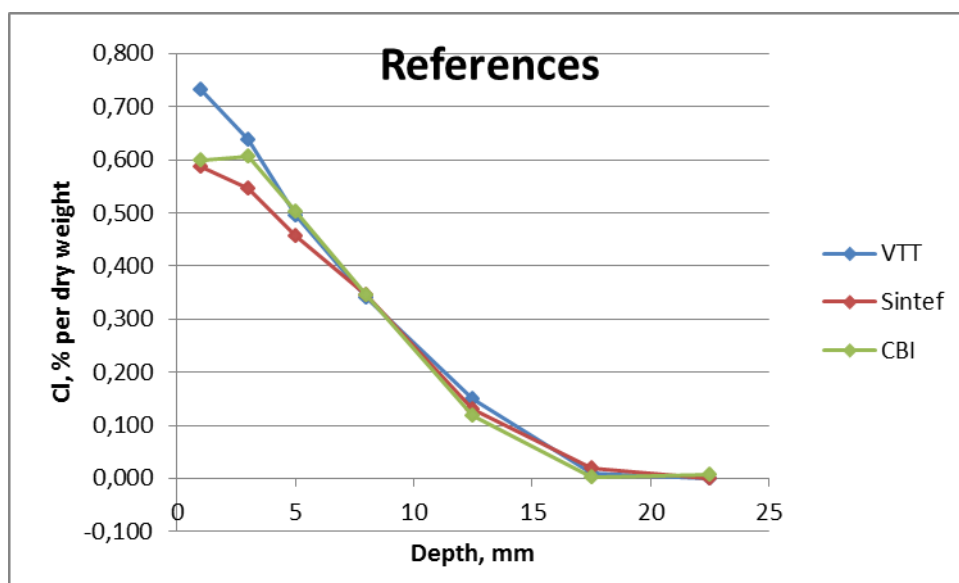
Figure 24: Chloride profiles in references (average of 5 samples).
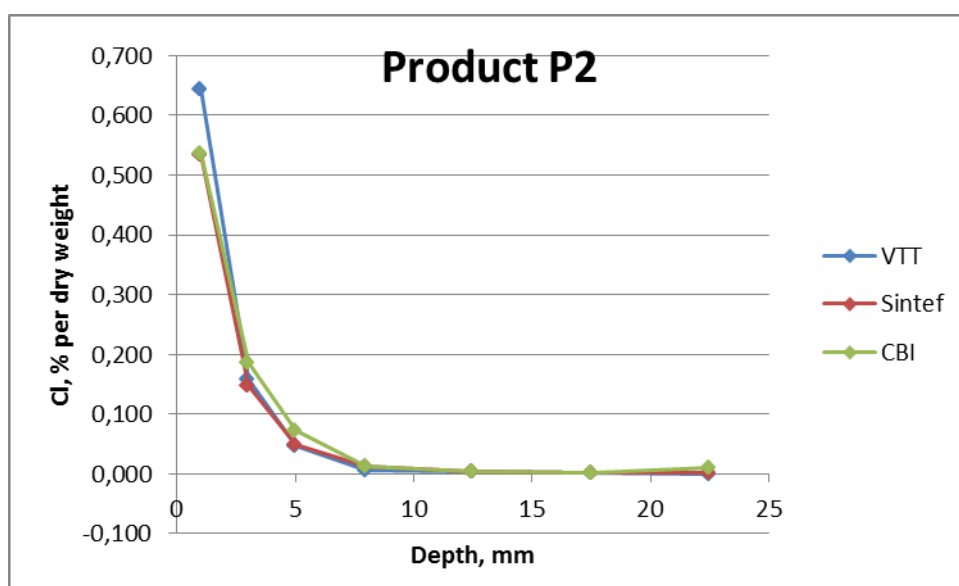
Figure 25: Chloride profiles in the samples treated with product P2 (average of 5 samples).
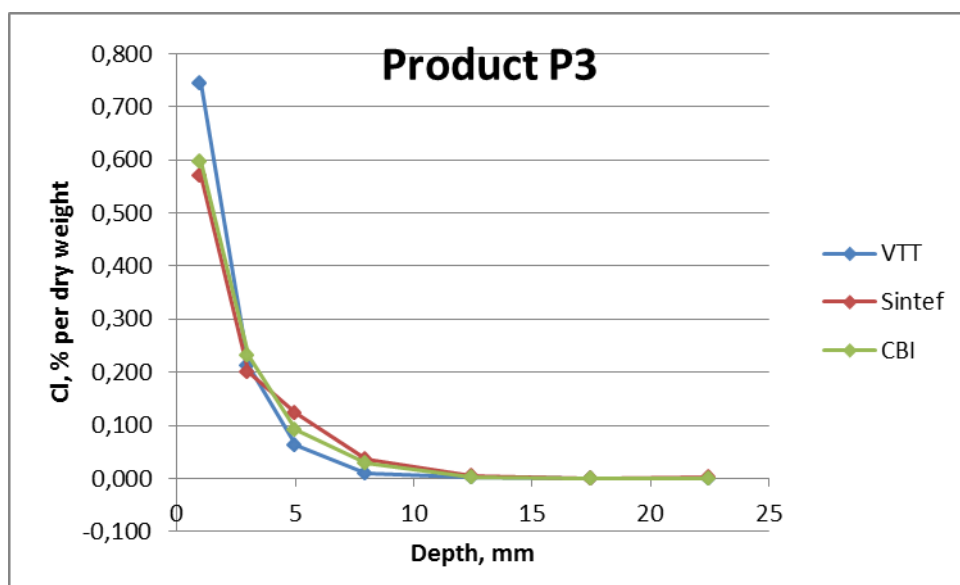
Figure 26: Chloride profiles in the samples treated with product P3 (average of 5 samples).
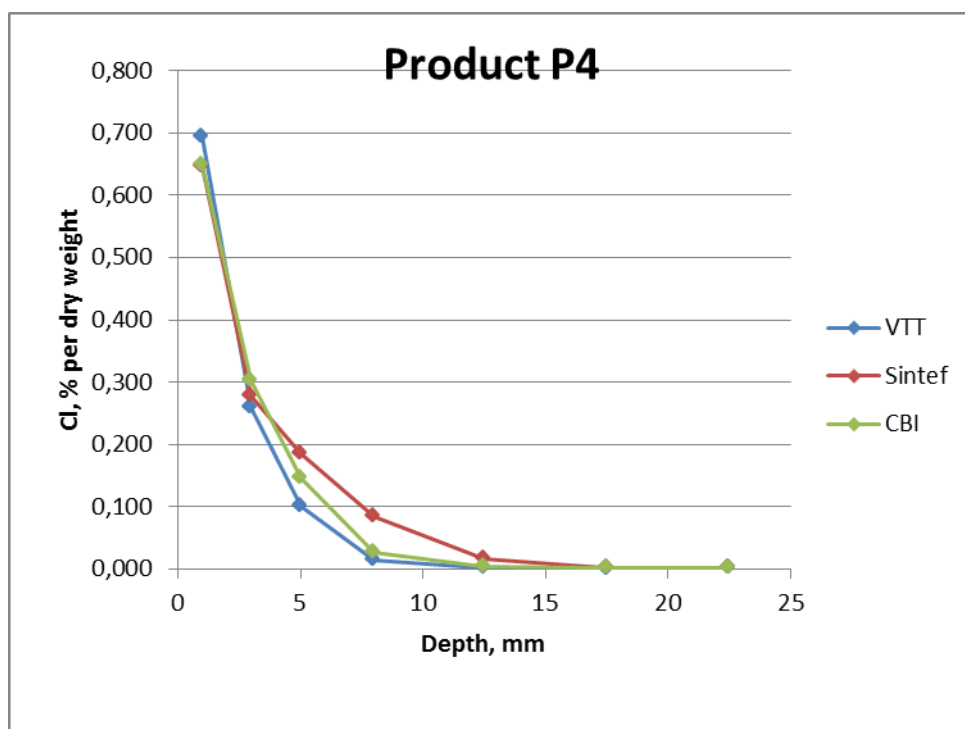
Figure 27: Chloride profiles in the samples treated with product P4 (average of 5 samples).

The standard deviation between samples in the different series was studied, and the only grouping of data that showed a systematic behaviour was between the different laboratories, see Figure 28. Data from the specimens with an impregnation curing period of 28 days in the pre-study is also included in this figure. VTT has a higher standard deviation up to 4 mm depth, probably due to the fact that they used surface-dry samples, not oven-dry. Beyond 4 mm for VTT and 2 mm for CBI (round robin and pre-study) and SINTEF the standard deviation is low and comparable for all laboratories.
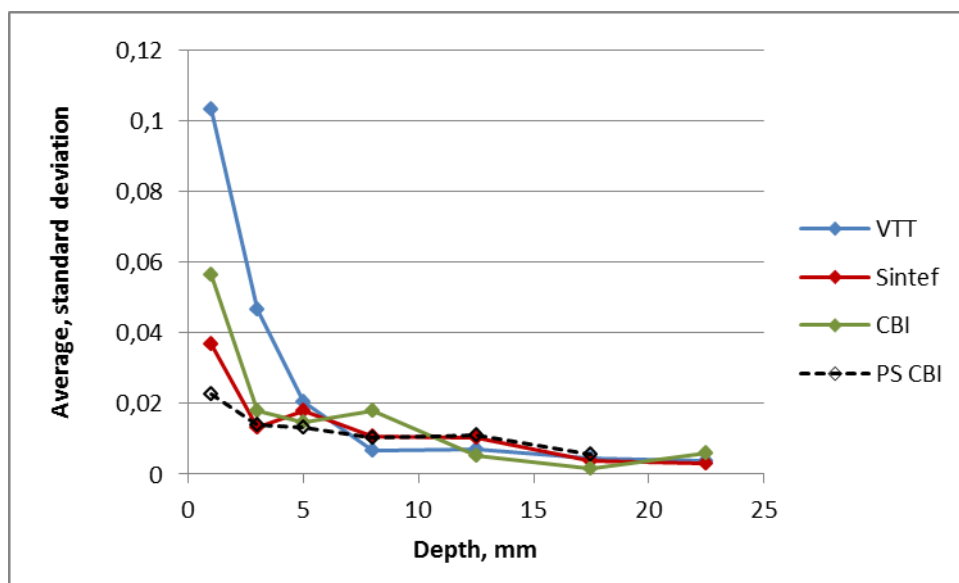
Figure 28: Average standard deviation of chloride levels in the different layers obtained at the three laboratories and in the pre-study.

5.3.3.2 TOTAL CHLORIDE CONTENT PER DRY SAMPLE WEIGHT

The total chloride content in the samples is shown in Figure 29. The differences in chloride profiles showed in 5.3.3.1 are to a certain degree reflected in the total chloride content. However, with regard to the total chloride content in impregnated samples the deviation between laboratories is not large and the three laboratories range the products in exactly the same way; P2 has the lowest, P3 the intermediate and P4 the highest total chloride level.
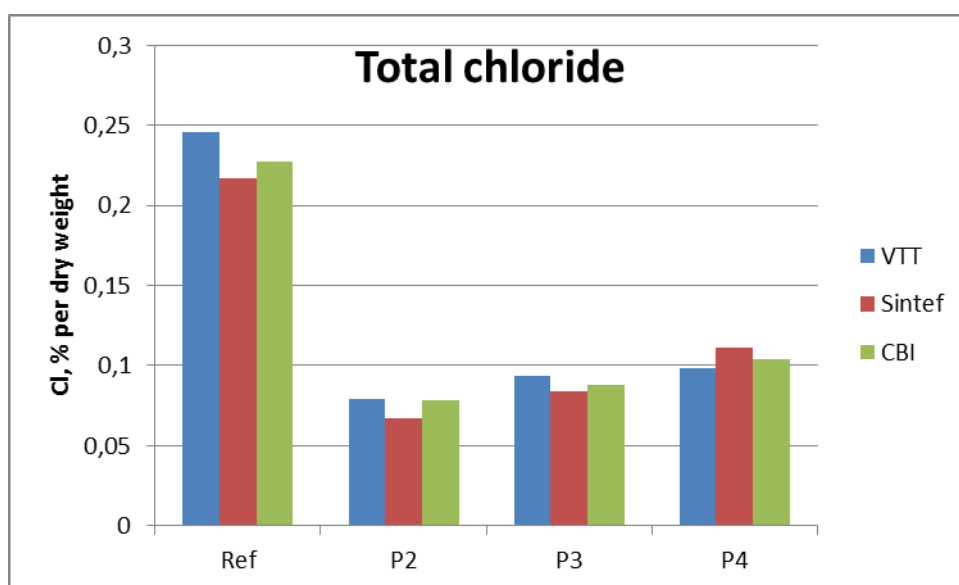
Figure 29: Total chloride content in the samples, average of 5 samples.

5.3.3.3 FILTER EFFECT BASED ON VALUES PER DRY SAMPLE WEIGHT

The filter effect for the three products is shown in Figure 30. The values are based on the chloride content per dry sample weight and with deduction of the initial chloride content.
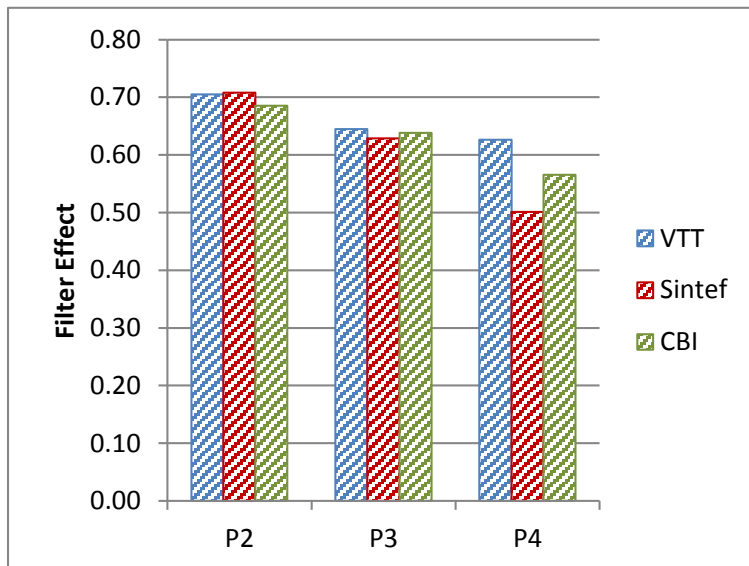
Figure 30: Filter effect for the 3 products.

As can be seen the filter effects obtained at the three laboratories are for the two products P2 and P3 more or less at the same level. For product P4 the deviation is larger. However, when ranging the products from best to worse all three labs get the same order; P2 is best (highest *FE*) and P4 is worse (lowest *FE*) and P3 stays in the middle.

5.3.3.4 CALCIUM OXIDE CONTENT

In order to further clarify whether there is an advantage with regard to representativeness to measure the CaO-content in the ground samples, as an indirect measure of the cement content, the CaO-content was measured on ground samples from one specimen of the references and one specimen per impregnation product. The CaO-content in the layers is shown in Figure 31 to Figure 33 for the three laboratories.
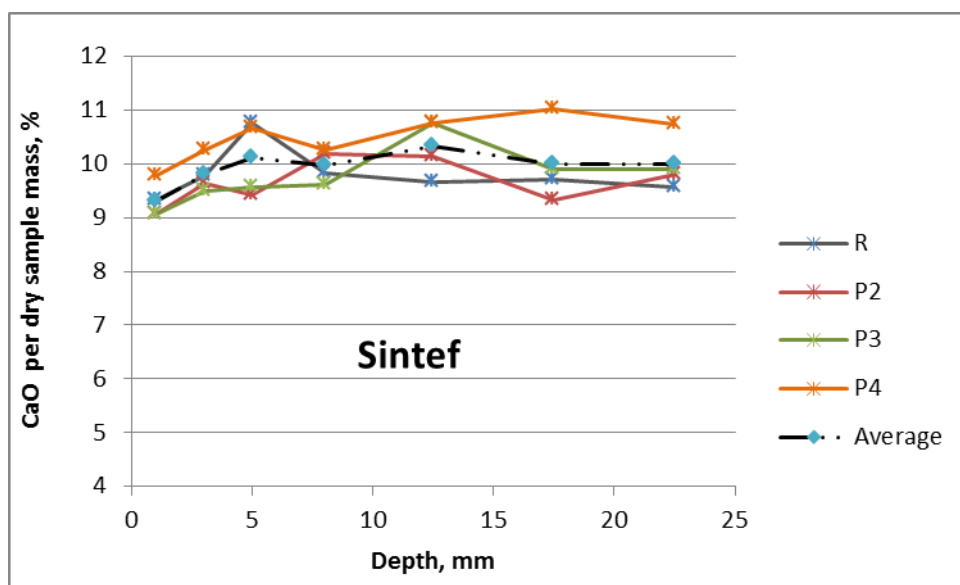
Figure 31: CaO-content in samples obtained at SINTEF.
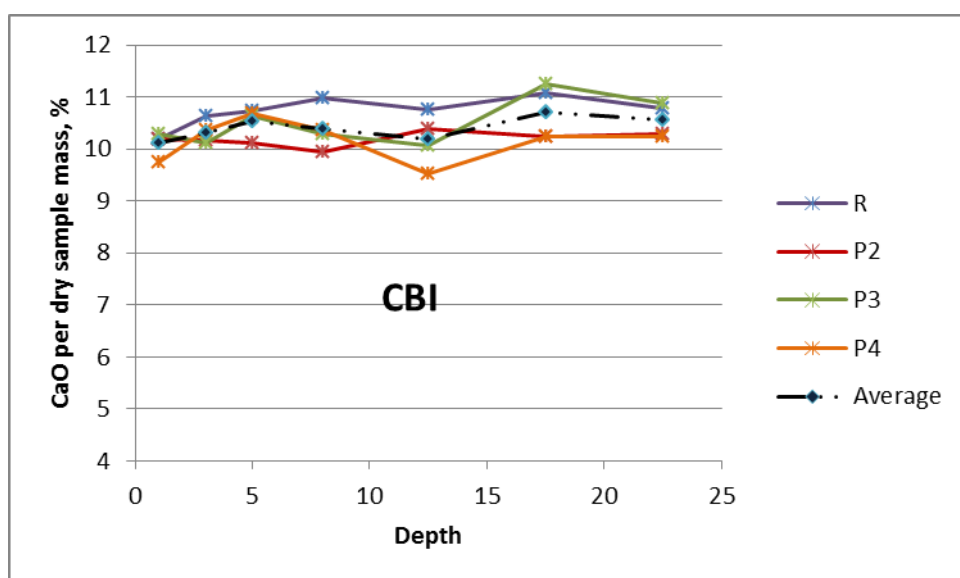
Figure 32: CaO content in samples obtained at CBI.
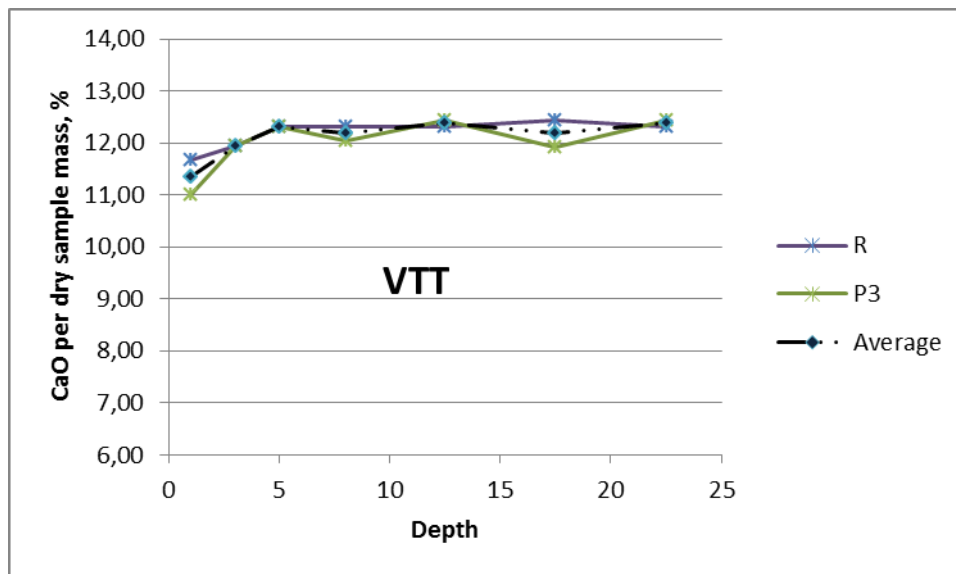
Figure 33: CaO-content in samples obtained at VTT.

As can be seen in these figures there is a certain decrease in CaO-content in the two outermost layers (up to 4mm) from the concrete surface. This may have several causes; leaching of CaO during the preparation or exposure of specimens or an increase of the dry sample weight due to carbonation of the concrete. A vague tendency in the same direction could be distinguished in the samples which were not lime-soaked (S) when the CaO-content was measured in the pre-study (4.3.2.3) where it was tested on samples with an impregnation curing period of only 7 days.

However, the variation in CaO-content throughout the investigated depth is surprisingly small, considering that the analysis is made on concrete, not paste or mortar. Apart from the systematic decrease at the surface, it can be questioned whether the variation in the results reflects a variation in the composition of the powder samples, or if it is due to the testing and measuring accuracy.

5.3.3.5 CHLORIDE PROFILES PER CEMENT WEIGHT

If the recorded variation in CaO-content reflects a true variation in the composition and cement content of the samples, chloride profiles determined per cement weight would result in a smoother curve than chloride profiles determined per dry concrete weight, since the chloride penetrates through the pore system which is dependent on the paste content. However, no such difference can be distinguished in the data. As an example the chloride profiles of the samples on which the CaO-content was determined obtained at SINTEF are shown in Figure 34 per cement weight and in Figure 35 per dry concrete weight.
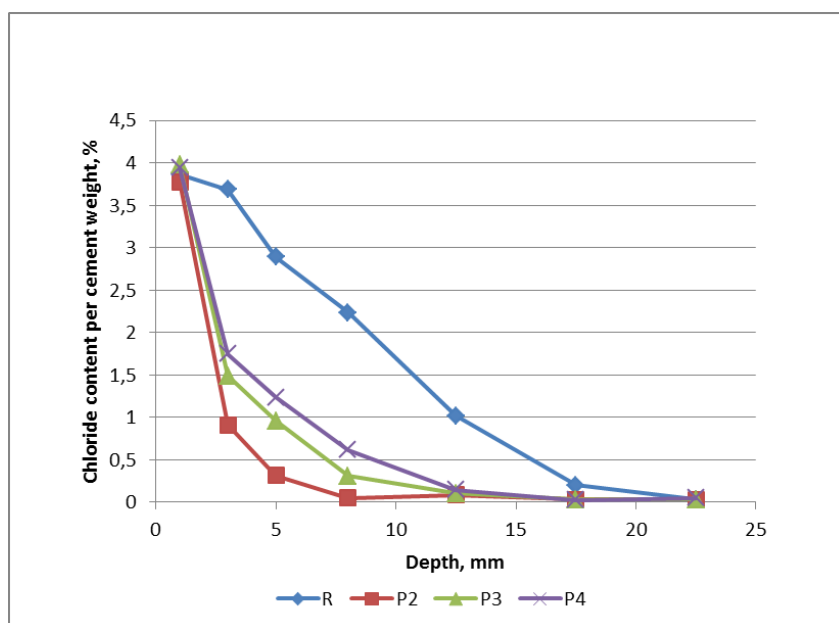
Figure 34: Chloride profiles per cement weight in the samples on which the CaO-content was determined (SINTEF).
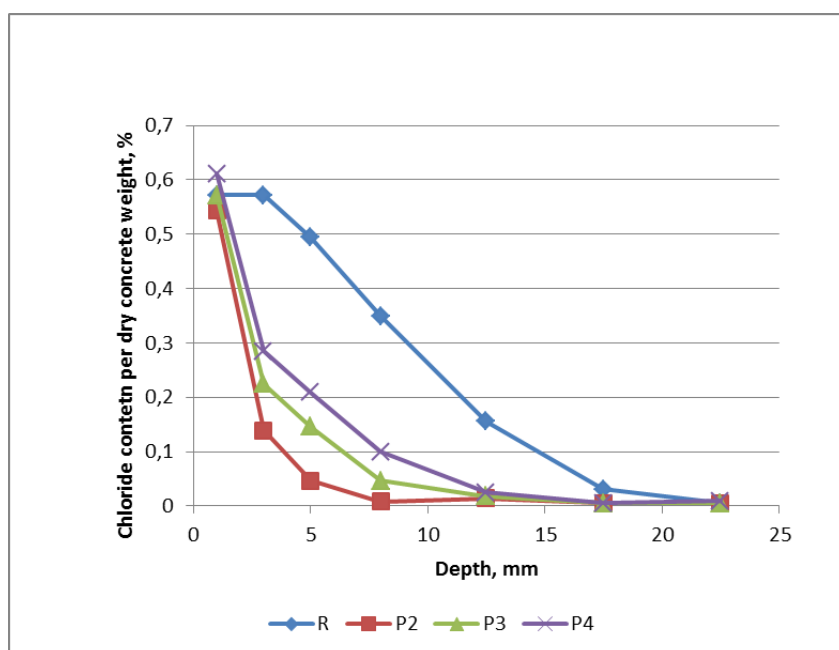
Figure 35: Chloride content per dry concrete weight in the samples on which the CaO-content was determined (SINTEF).

5.3.3.6   FILTER EFFECT BASED ON CHLORIDE CONTENT PER CEMENT WEIGHT

Figure 36 shows the filter effect of the samples (one per impregnation product and laboratory) on which the CaO-content was determined, both when the chloride content is recorded per cement weight and per dry concrete weight. For simplicity reasons no deduction for initial chloride contents was carried out in these calculations. As can be seen the difference between the values are never larger than 0.027 %-units for any product at any laboratory. In this figure the corresponding average

filter effect calculated per dry concrete weight is also shown (based on five samples). Only in one case in Figure 36 there is a large deviation between the values obtained on one sample and the average value, and that is for product P3 obtained at CBI. Unfortunately, here an impregnated specimen (P3-1) in which the Cl-level in two of the layers were unproportionally large (outliers) was used for the CaO-analysis (see Annex D.4.3).
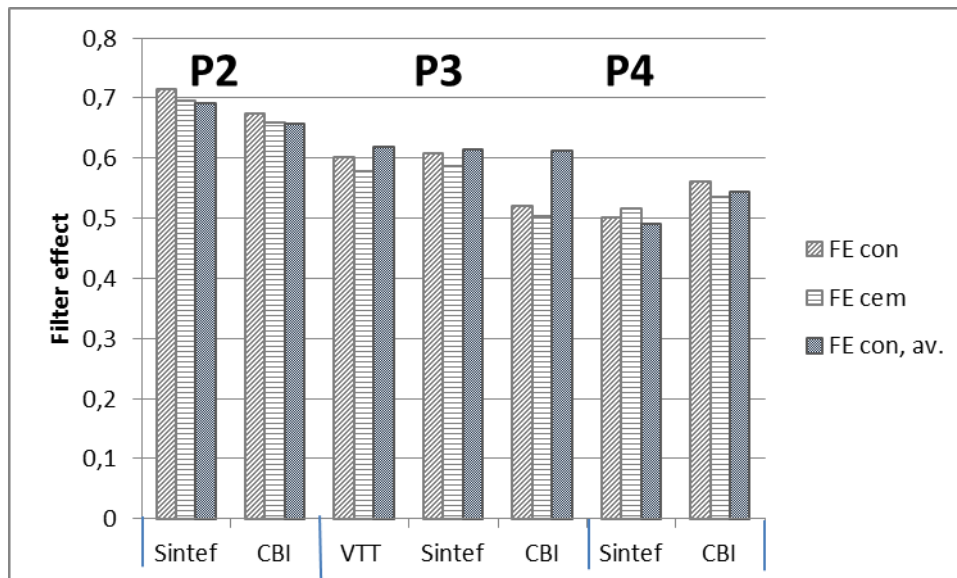
Figure 36: Filter effect calculated based on chloride content per cement and per dry concrete weight of one sample, and average of five samples based on dry concrete weight.

5.4 Discussion

5.4.1 Determination of penetration depth

Since the method to measure the penetration is not the main subject of this study it will be only briefly discussed here. As have been discussed already in the pre-study and with the results of the round robin exercise, the method referred to in EN 1504-2 (CEN, 2004), the "split method" is a very subjective method, and the level of the results depends on who does the evaluation. However different persons normally range the different results in the same order. With the used "drop method" the results obtained at the different laboratories shows much less variation, although only one specimen per product was studied.

5.4.2 Chloride ingress and chloride binding in concrete

5.4.2.1 BOUND VERSUS FREE CHLORIDES IN CONCRETE

Chlorides in concrete may be kept in the structure in three ways. Either they can be chemically bound to hydrated cement phases or they can be bound physically in the pore structure (absorbed) or they can be dissolved in the pore liquid (free chlorides).

Pure chemical binding occurs between chlorides and C_3A and C_4AF phases with the formation of Friedel's salt. If chlorides are present at the start of the hydration, the chemical binding of chlorides take place simultaneously with the other chemical reactions and a substantial part of the chlorides become bound already at early stages of hydration (Sumranwanich & Tangtermisirikul, 2002).

However if the chlorides are introduced at a much later stage, when most of the C_3A and C_4AF phases already have hydrated, the chemically binding process becomes quite different and is governed by more parameters than only the availability of these minerals and chlorides (Arya, et al., 1990). This is a slow process, which becomes important when concrete structures are exposed to chlorides for long periods. Chemically bound chlorides dominate the chloride content in concrete samples after long exposure periods (years). In this study the concrete has been exposed to chloride at the age of 42 days or later. At this age almost all C_3A and C_4AF have already reacted and hardly any reaction between chlorides and unhydrated C_3A and C_4AF can take place. Since the exposure period is as short as 56 days the secondary reaction (ion exchange) between the hydration products of C_3A and C_4AF and the chlorides in the pore solution has hardly started. The presence of chemically bound chloride has therefore been ignored in this study.

The relationship between (physically) bound and free chlorides has been the topic of several studies. Tang and Nilsson (1993) concluded that the chloride binding capacity of concrete is strongly dependent on the content of CSH gel, regardless of w/c ratio and that the relationship between bound chloride and free chloride can be well described by chloride binding isotherms which also show a certain amount of hysteresis, i.e. some chlorides become trapped in the gel structure. The main difference between physically bound and free chlorides is how difficult it is to remove the chlorides once they have penetrated into the concrete structure. As shown in several studies, e.g. (Tang & Nilsson, 1993) filling of the gel pores or the inter-laminar space between hydration products with the chloride solution occurs in a question of days when the chloride solution is present in the larger pores.

The method which is the topic of this project only aims to measure the ability of impregnations agents to delay the chloride ingress into concrete, a process that always starts with chlorides entering the pore system, either by diffusion through the pore solution or by capillary suction of chloride solution into empty pores. How readily the chlorides penetrate the different pores is of importance for the study, while the way they are bound to the concrete structure is only of secondary importance for this study.

5.4.2.2 PENETRATION OF CHLORIDES INTO A MATURE CONCRETE

When a concrete specimen surface is exposed to a chloride solution, the chlorides penetrate into the concrete by diffusion if the capillary pore system of the concrete is water saturated at the start of the exposure. If the concrete surface layer has dried out beforehand, the chloride solution first saturates the pore system by capillary suction and when the pore system is saturated or an obstacle for the capillary suction occurs, the further ingress will take place through diffusion. I.e. the maximum amount of chlorides penetrating the pore system should be related to the amount of pores in the concrete. In a concrete permanently exposed to a chloride solution at one surface, the maximum concentration of chlorides in the pore solution should be registered at the surface layer and it should after enough time of exposure reach equilibrium. The chloride level in the surface layer of the specimen should then be about equal to what is obtained if the pore structure accessible to chloride penetration is completely filled with a solution with the same chloride concentration as the surrounding solution.

Is there then a correspondence between the chloride level registered in the concrete and the pore volume and Cl⁻ concentration of the solution in concretes permanently exposed to a chloride solution?

In order to clarify this, the registered Cl⁻ levels close to the surface in the CBI reference samples in the round robin test have been used. The total pore volume per dry concrete weight was calculated based on traditional relations for pure portland cement mixes, e.g. (Powers & Brownyard, 1947) and the composition of the concrete. As a basis the degree of hydration of the cement (α) was assumed to be 0.70, which is quite normal for a well-hardened concrete with w/c=0.45. The solution contained 15% NaCl, which at this concentration has a density of 1.112 kg/l.

In Figure 37 the measured values obtained in the round robin tests at CBI are given together with the results of the calculations (as Cl⁻ per oven dry concrete by weight) obtained for α =0.6; 0.7 and 0.8 when all capillary and gel pores are assumed to be filled with NaCl-solution, and also for α= 0.7 if in addition to this all the air volume (no air-entraining agents were used) are assumed to be filled with the solution. The air volume was estimated based on density measurements. Since the air volume in the concrete is constant the displacement of the level (around 0.06 %- units) will be the same for all degrees of hydration.
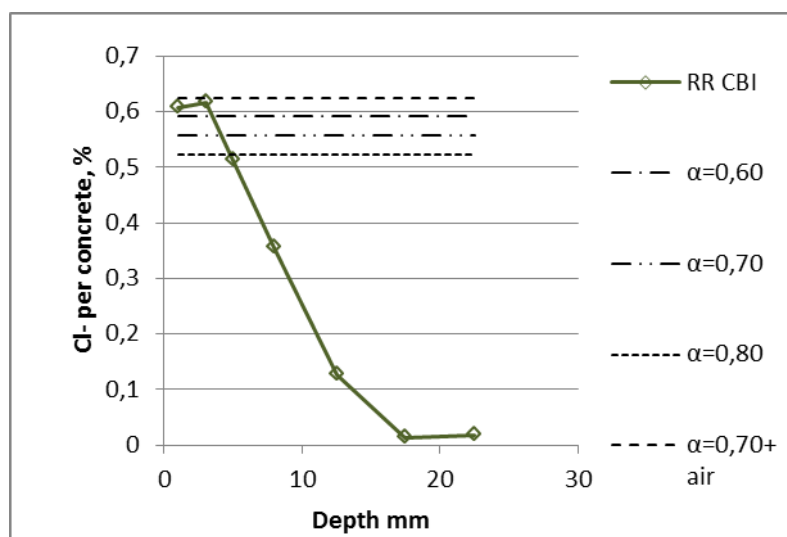
Figure 37: Cl- levels in the reference samples from the round-robin test from CBI compared to anticipated Cl- levels when the pore system is filled with 15% NaCl-solution.

As can be seen from the figure, the measured chloride level for the two measuring points closest to the surface corresponds rather well to when the total pore system is assumed to be filled with a solution with the same Cl⁻ concentration as the one in the bath used in the tests (15% by weight).

Looking at the chloride levels in the reference samples from all laboratories in the round-robin exercise and also including the references from the pre-study carried out at CBI where the pre-conditioning and curing of the samples were somewhat different and varied, it is clear that the reference curves vary substantially close to the exposed surface although similar concrete compositions were used in all these samples (Figure 38). This will be discussed in detail in the following section.
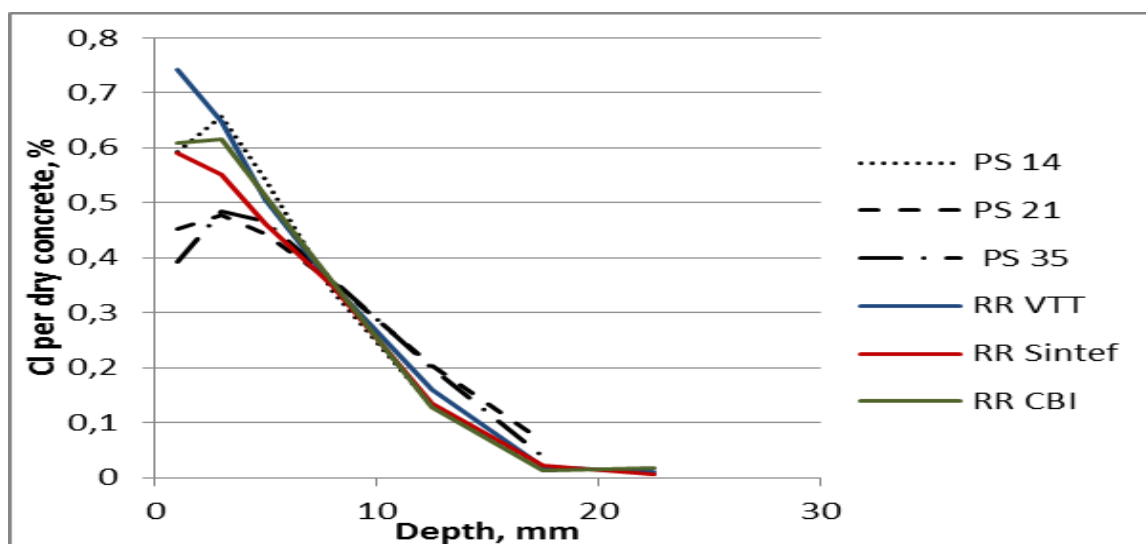
Figure 38: Measured chloride level in the reference samples in the pre-study (PS, number signifies time in 65±5% RH before exposure) and the round-robin tests (RR) which were all in 60 ± 10 %RH 35 days before exposure.

5.4.3 Influence of the details of the method on chloride profiles and the chloride blocking effect

5.4.3.1 WHAT MAY BE THE CAUSES FOR THE OBSERVED DEVIATIONS IN CHLORIDE PROFILE OF THE REFERENCE SAMPLES?

An attempt to explain these deviations shown in Figure 38 and some other deviation visible in other tests will be made in the following. When possible the influence will be quantified. The possible causes will be treated in the order they may appear in the testing procedure.

5.4.3.1.1 Concrete composition and choice of materials

In the method the composition of the concrete was specified by some key values: In principle MC (0.45) according to SS-EN 1766 (CEN, 2000) with w/c = 0.45, amount of cement 395 kg/m³, CEM I 52.5, aggregate size = 0/10 and target value of slump = 180 mm.

For the pre-study a CEM I 42.5 R from Norcem with a grain size around 370-380 m^2/kg was used. With regards to the cement for the method and the round robin exercise it was decided that a plain CEM I 52.5 should be used. In the round robin tests CBI and VTT used CEM I 52.5 R cements. Both these rapid hardening cements had a grain size between 500 and 600 m^2/g. However, SINTEF used a CEM I 52.5 N intended for infrastructures which has a limited equivalent alkali content (max 0.6 %) and a grain size around 400 m^2/kg. Since the chloride exposure in the tests does not start until the concrete has reached the age of at least 42 days, and the average compressive strength at 28 days of the concretes are at very much the same level for the three laboratories in the round robin exercise (see 5.3.1) the analysed concretes will probably have obtained the same degree of hydration. Thus, there will be no or only a marginal influence on the total pore volume caused by varying degree of hydration due to a difference in fineness between the cements. The main difference between the pore structure at that age would be a somewhat finer pore-size distribution in the capillary region (water recorded between 40 and 95% RH of the desorption isotherm) when cements with higher

specific surfaces are used and a somewhat coarser pore structure in the capillary region when a low-alkali cement is used (Atlassi, 1991).

5.4.3.1.2   Curing of concrete specimens before sawing.

Since sawn surfaces are used in the test method and no grinding takes place near the surfaces exposed to the curing environment the actions during this stage of the procedure should not cause any of the deviations in the chloride profiles or the chloride blocking effect.

5.4.3.1.3   Sawing

Sawing of concrete is carried out under sprinkling of water and the sawed specimens are brushed and rinsed under tap water afterwards to remove the cutting slurry, which may cause some leaching of $Ca(OH)_2$.

The mechanical sawing may in itself cause micro-cracks in the concrete close to the sawn surface. Micro-cracks may function as channels for capillary transport of the chloride solution past the cracked surface layer into the subsequent layers, serving more or less as a shortcut to the interior. This would lead to a faster initial ingress of chlorides than with an un-tooled surface. As can be seen from Figure 19, the chloride ingress just after the outermost 2 mm always seems to be somewhat higher for a sawn surface than for a form surface.

A first hypothesis when looking at the results was, that micro-cracks formed during sawing, was the main cause for the big scatter in chloride level in the surface layer showed in Figure 38, and the dip in chloride level showed at the surface evident in Figure 19. The difference between tests at different laboratories could then be caused by varying "brutality" of the sawing equipment.

However, if micro-cracks are formed during sawing this could be counteracted by healing the surface by exposing it to saturated $Ca(OH)_2$-water after the sawing process before preconditioning and exposure to chlorides. This would also counteract any leaching experienced during sawing.

In the pre-study the effect of exposure to lime water (saturated $Ca(OH)_2$-water) after sawing was studied. One set of reference specimens were placed in lime water for 3 days immediately after sawing and then in 65% RH for 14, 21 and 35 days before being exposed to chlorides. Another set was kept in 65% RH from sawing until exposure. The resulting chloride curves are shown in Figure 39. As can be seen from this figure the influence of the lime water is only marginal and not unambiguous with regard to the deviations close to the surface. It is therefore unlikely that micro-cracks or leaching during sawing are the main reason for the deviations in the chloride curves close to the surface.

Since the effect of lime-soaking was only marginal in the pre-study it was decided to omit this from the test method. This decision was further motivated by the fact that lime-soaking is not a preparation procedure normally used when impregnations are carried out on real structures.
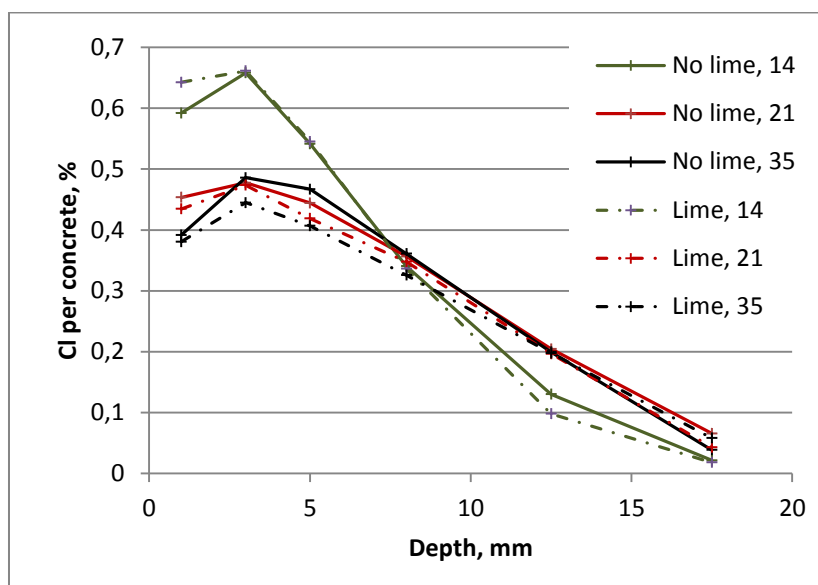
Figure 39: Chloride levels in reference samples soaked in lime water after sawing and not. The number designates the number of days in 65 % RH before exposure to chlorides.

5.4.3.1.4 Pre-conditioning of sawn samples

The preconditioning is always carried out by exposing the specimens to lower RHs during a certain period. The influence of the length of the exposure period for a fixed RH-level was studied in the pre-study. The RH-level was controlled at 65±5 % RH. The reference samples were preconditioned during 14, 21 and 35 days.

According to the preliminary formulation of the test method used for the round robin test, the reference samples should be preconditioned in 60±10 % RH and 20±2 °C for 7+28=35 days before the exposure to chlorides. Following up the round robin tests it was however found that SINTEF had used a climate room regulated to 60±5 % RH for the first seven days and then another regulated at 50±5 % RH for the remaining 28 days. CBI and VTT both used rooms regulated at 65±5 % RH for the whole period. What effects may these differences in RH-level and the length of the preconditioning period have on the pore structure and on the measured chloride levels of the references after the exposure?

Moisture profiles

The obvious difference is that the RH profile and consequently the proportion of pores with water will be different at the start of the exposure to chlorides. If a totally water saturated specimen is exposed to a chloride solution the transport of chlorides will take place by diffusion into the specimen. If the specimen has dried for a certain period the pore structure at the start will only be partially filled with water. When exposed to a chloride solution, at first the empty pores in the surface will be filled with the solution through capillary suction. When the pore structure is saturated with liquid, the transport of chlorides continues by diffusion into the specimen.

Although the amount of larger capillary pores influences the transport rate, capillary suction is always a faster process than diffusion. The amount of chloride solution that enters by capillary suction in the beginning of the exposure period depends among other things on the percentage of the pore volume that is empty at the start, i.e. on the moisture profile at the end of the pre-conditioning period. This profile depends on the concrete composition, the ambient RH-level during the pre-conditioning and the length of the pre-conditioning period.

An estimation of the moisture profile, shown in Figure 40, expressed as evaporable water per cement weight, has been carried out with the help of TorkaS (Fuktcentrum, 2015) (a computer program for estimation of moisture in concrete floors) and the desorption isotherms in (Nilsson, 1986). A line representing a completely filled gel and capillary pore system is also included in this figure. TorkaS is however based on moisture characteristics for another cement type than any of those used in this study, a CEM II/A-LL according to (CEN, 2011), i.e. a portland-limestone cement. Although the moisture sorption characteristics of this cement are not extremely different from those of a CEM I, the curves in Figure 40 should only be interpreted as an indication of the actual moisture profile at the end of pre-conditioning period.
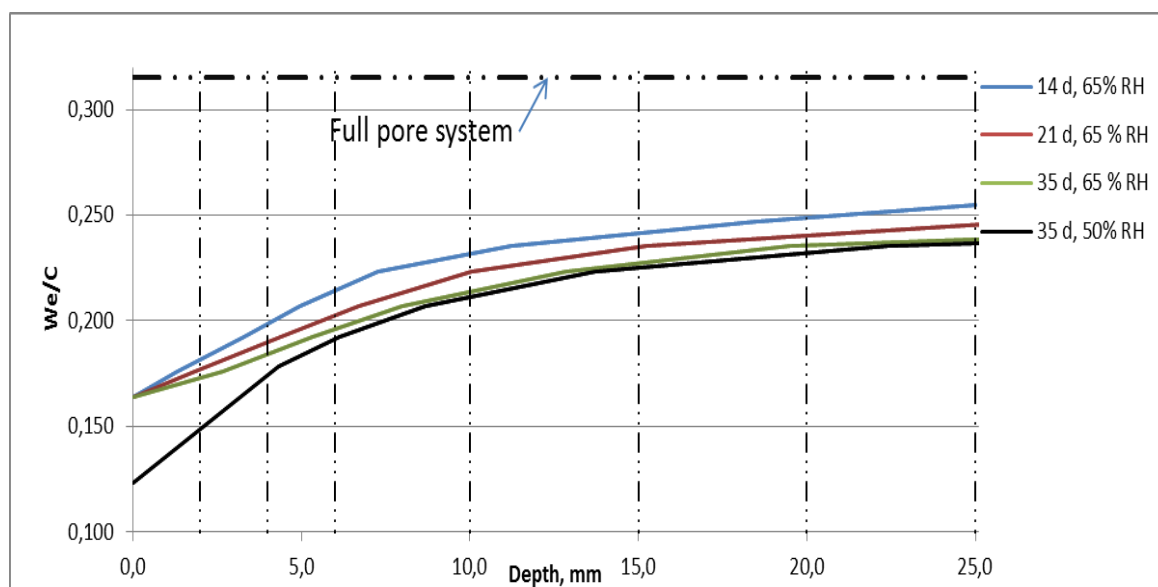
Figure 40: Approximate moisture profiles at the end of the preconditioning period.

However, undisturbed capillary suction into a specimen can only occur if the air in the empty pores can move through the porous system to a free surface. If a partially dried specimen is put into a basin with water so that water penetrates the specimen from all sides, the air inside the specimen will be trapped. When equilibrium between the capillary forces pushing inwards and the compressed air pushing outwards is achieved, the further ingress of water will be blocked (Atlassi, 1993) . The only way air can be transported to the surface is then by dissolution into the pore liquid, a very lengthy process. Thus, the capillary suction of the saline solution into the specimens when submerged into the NaCl-bath, will only fill the total pore volume in a superficial layer, moving the chloride front inwards. Although there are empty pore spaces also in the inner parts, these spaces will not be reached by capillary transported chloride ions.

To reach the inner parts the chloride ions has to be transported by diffusion from the saturated surface layer. Diffusion of ions can only take place in a liquid phase, thus the water content in the inner parts will limit the maximum chloride content there. A RH-level in the interior of 90% corresponds to that approximately 80% of the gel and capillary pores are filled (Nilsson, 1986) (Atlassi, 1991) will in our case represent a maximum chloride level in the interior parts of the specimens which is about 0.45% per dry weight.

Despite the obstruction for free capillary suction into the specimens, Figure 39 indicates that the chloride levels beyond a depth of about 8 mm become higher when the specimens have been preconditioned in 65% RH for 21 days or longer periods.

As can be seen from Figure 40, the evaporable water content will reach about 55% of the total capillary and gel pore volume at 65% RH (the value for 65% RH at the surface/the value for a full pore system) and at 50% RH about 40% of the pore volume will be filled In the inner parts 78% of the pore system is water filled. If the capillary transport of the solution into the specimen was not obstructed, the chloride level that would result from total capillary filling of the pore system would in the interior reach about 0.11% per dry sample weight [=(1-0,78)*0.15*0.6*2250/395].

Judged from the chloride profiles in Figure 38, we have in the CBI specimens after 56 days exposure a capillary pore structure completely filled with 15 % NaCl-solution in the 4 mm closest to the surface. One part of the chlorides in this layer comes from capillary suction of the solution at an early stage, which according to calculations above can represent a maximum of about 45% of the amount of chlorides in the samples preconditioned at 65% RH. The rest, 55%, in this layer must have been transported by diffusion of chloride ions. When pre-conditioned in 50% the proportions will be about 60% by capillary suction and 40% by diffusion.

Coarsening of the pore structure

Apart from diminishing the water content in the concrete, pre-conditioning at lower RH:s will be accompanied by a coarsening of the capillary pore structure. When the RH-level in a specimen decreases the pores are gradually emptying, starting with the largest pores. In a saturated pore structure there are no capillary forces influencing the distribution of the hydration products which leads to that certain hydration particles will be present in a meta-stable position. Drying of concrete induces capillary tension in the water surface which is transmitted to the surface of the hydrate particles, forcing the particles to find a position which is in equilibrium in the structure with regard to the tension. This is the background for the difference between the first desorption isotherms of concrete and desorption isotherms obtained after fist desorption and absorption (Helsing Atlassi, 1996) where the first desorption isotherm in the capillary (above 40% RH) region always is situated above the subsequent desorption isotherms. In Figure 41 based on data from (Atlassi, 1992) the first and the second desorption isotherms of a mortar with w/c = 0.50, are presented as evaporable water content (W_e) per cement content for the whole isotherm and as degree of pore filling ($W_e/W_{e, saturated}$) for the region above 50% RH.

The coarsening effect is larger the lower the minimum RH-level attained (as long as it is above 40%) in the pore structure since the capillary tension increases with diminishing RH and the area of water surface under tension becomes larger the more the pore system is emptied. At the start of the chloride exposure period there will exist a RH-profile in the proximity of the surface. If the drying

time is short the profile will be very steep and the longer the drying period is, the RH-profile becomes less steep and reaches further into the specimen. Close to the surface the RH is in equilibrium with the RH of the ambient atmosphere (e.g. 65% or 50%) but in the interior the RH level remain at the level reached by self-desiccation. For the concrete in question this will be around 90% RH.
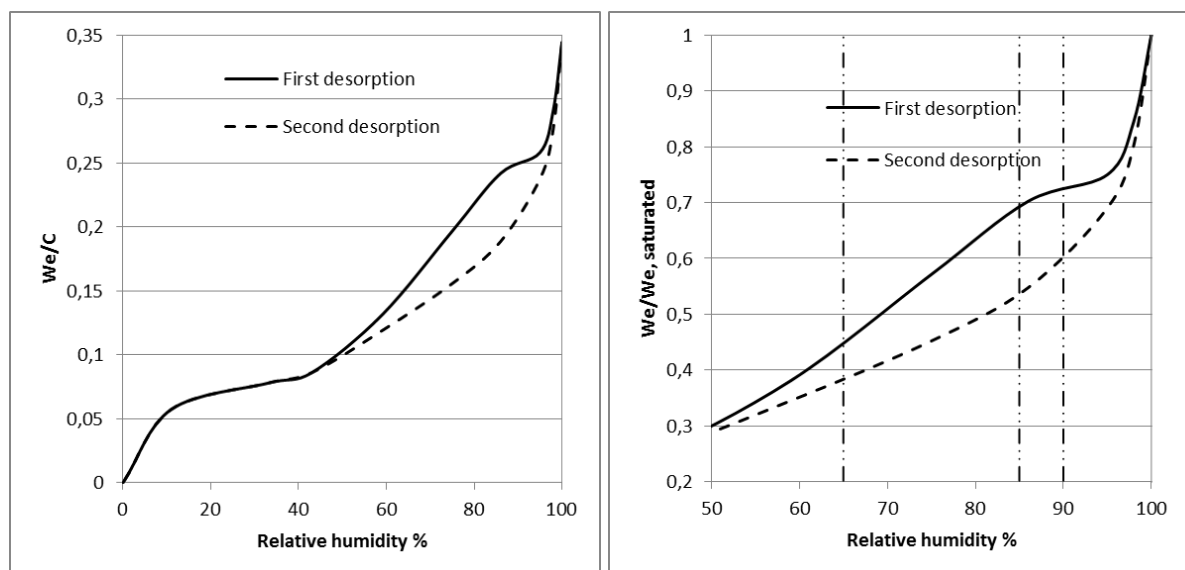
Figure 41: First and second desorption isotherms of a cement mortar with w/c = 0.5 presented as W_e/C and $W_e/W_{e, saturated}$.

To what degree and how deep into the specimen the degree of coarsening of the pore structure has reached in a specimen when the chloride exposure period starts should depend on the RH-profile obtained at that moment.

In the pre-study differing preconditioning periods in 65% were used, with exactly the same methodology for the rest of the procedures. The chloride curves of the references are shown in Figure 39. It is evident that there is a surprisingly large difference between the curves obtained after 14 days and those obtained after 21 and 35 days. In the first 6 mm the chloride level is much higher (around 0.2%-units) when exposed earlier which can be caused by a difference in degree of hydration, c.f. Figure 37, and beyond 10 mm the curves of those exposed at later stages are higher.

However, when the experiments were repeated in the round robin exercise (with similar but not exactly the same materials and procedures, and with other persons carrying out the tests) significantly higher maximum chloride values were achieved after 35 days in 65% RH, as can be seen in Figure 38 (PS 35 and RR CBI).

Apart from the pure geometric changes to the pore structure that drying imposes, this influences the hygroscopic behaviour of the concrete. The first desorption isotherm lies above the second desorption isotherm, i.e. at a certain RH-level more water is retained in the pores when the concrete has never dried before than when it has. The slope of the second desorption isotherm is steeper at very high RH- levels than in the first desorption isotherm. In large pores as at the highest RH-levels the water is very loosely bound by capillary forces, and a small change in surrounding RH causes a large change in water content. The coarsening of the pore structure due to drying amplifies this influence even further. The water held at the highest RH-levels is then very sensitive to even a slight

change in RH. As can be seen in Figure 41, when decreasing the RH-level from 100% to for example 90% the loss of water is about 50% larger at second desorption than at first desorption.

Even applying an absorbing tissue to a saturated concrete surface may suck out the water held in the largest pores close to the surface, especially if the pore size distribution is coarsened by previous drying. This will be further discussed when the stage of removing the specimens from the NaCl-bath for the chloride analysis is treated (5.4.3.1.6).

In addition to the coarsening of the pore structure caused by drying, carbonation also leads to a change in the pore size distribution towards a coarser structure, even though it diminishes the total pore volume (see 4.3.2.4).

5.4.3.1.5   Exposure to the chloride solution

The influences occurring during water curing may also occur during exposure to the NaCl-solution, i.e. increasing degree of hydration in the surface layer and leaching of $Ca(OH)_2$.

The most obvious cause of differences during this period may though be deviations in the concentration of chlorides in the exposure solution. Either a difference between the different containers due to the manner the solution is obtained, or a difference in each container over time. The chloride level in the solution in one container will decrease as chlorides are penetrating the specimens. According to the procedures distributed for the round robin exercise (see Annex D.1), the chloride level in the containers should be checked and adjusted at least once a week. The risk for more than marginal deviations in chloride concentration between containers during one test series carried out by the same persons at the same laboratory are negligible. And under condition that the same checking procedures are used for all containers in one test series, the variation over time will be similar in different containers. This may explain the relatively small difference in the surface chloride content for the reference specimens obtained at different laboratories during the round robin exercise.

In the pre-study, however, the reference specimens were sequentially exposed to the same NaCl-bath without adjusting the concentration. This may be one cause for the decrease in chloride content at the surface with increasing pre-conditioning time (14 to 35) that can be seen in Figure 38.

In order to enhance the reproducibility of the method, this stage in the procedures was further specified for the round robin exercise by measuring (and if necessary adjusting) the chloride concentration of the exposure solution after 14 and 28 days exposure. In addition, for the final method descriptions, it was specified that, in order to prevent evaporation, the containers should be kept closed and a certain volume of exposure liquid in relation to the area of the exposed concrete should be kept, as in NT Build 443.

5.4.3.1.6   The period from withdrawal of the specimens from the solution to grinding

The specimens shall during this operation be transferred from a soaked immersed condition to an oven-dry condition. It is important that this is carried out so that the chloride levels that exist in the specimens at the end of the exposure are maintained until the specimens are analysed. How this is

carried out in practice may have a crucial influence on the final chloride levels in the surface layer of the specimens. When taken from the chloride solution the pore system in the surface of the specimen is totally filled with NaCl-solution and the RH is 100%. As mentioned earlier the liquid filling the largest pores is very loosely bound in the structure and may easily be removed by evaporation when exposed to lower RHs or by capillary action by an absorbing tissue. The amount of larger pores is higher the longer the preconditioning period and the lower the RH the specimen has been exposed to.

When the liquid is a chloride solution the effect of the removal of some of the liquid will result in different chloride levels in the sample depending on the manner by which the liquid is removed. If the removal is carried out through evaporation of water, the salt in the liquid will stay in the sample and increase the chloride concentration of the pore solution close to the surface and some part of the chlorides may also crystallize in the pore structure. Some of it may be transported to the evaporation surface. If however an absorbing tissue is used to wipe the specimen, the tissue will suck out some of the liquid with its salt, diminishing the salt remaining in the surface layer of the sample for analysis. How much and how deep into the specimen the effect will reach will depend on the absorptivity of the tissue, the time used for wiping and the sensitivity of the specimen surface, i.e. the pore size distribution in the outermost layer.

In the pre-study and in the round robin exercise wiping with an absorbing tissue was indicated, without any further instructions, which presumably is the main cause for the large deviation in the chloride profiles close to the surface of the references.

A handling of the specimen at this crucial point of the procedures that preserves the chloride profile existing in the specimen just before it is taken out from the solution and which leads to more reproducible chloride profiles would be preferred.

Another effect that may occur during storage is a redistribution of chlorides within the specimens. As discussed in 4.3.2.2, based on the observed differences in the chloride profiles of the pre-study, the length of the storage period plays an important role in this phenomenon. Another important factor is the temperature during storage. The chloride diffusion coefficient is much lower at lower temperatures than at higher temperatures (Yuan, et al., 2009). Thus, the longer the storage period and the higher the temperature during this period the more internal diffusion and re-distribution will take place.

In the pre-study the specimens were sealed in plastics in 21±2 °C and the storage period was not well controlled and varying from a few days to more than one month. In the round robin exercise the storage temperature was set to 5±2 °C and the storage period limited to 7 days. Figure 38 supports this hypothesis. As can be seen from Figure 43 this is also the case.

The PS14 profiles which were ground within a week are very similar to those obtained in the round-robin exercise also within a week, while the PS21 and PS35 profiles show signs of substantial re-distribution of chlorides. (PS14 = 7 days impregnation curing, PS21 = 14 days impregnations curing and PS35 = 28 days impregnation curing).

In order to prevent too much redistribution of chlorides inside the specimens the storage temperature should be kept as low as possible and the storage period as short as possible.

Sucking out pore-liquid from the surface layer will decrease the total chloride content in the 25 mm used for calculating the effectiveness of the impregnations. However a redistribution of chlorides will not decrease the total chloride content as long as the redistribution is kept within these 25 mm. Assuming that only re-distribution takes place the total chloride level in the specimens should be about equal despite the fact that the form of the chloride profile varies substantially. As can be seen in Figure 42, this is more or less the case. The total chloride content in the pre-study is somewhat lower which may be due to a more vigorous use of the absorbing tissue or a re-distribution beyond the analysed 20 mm.
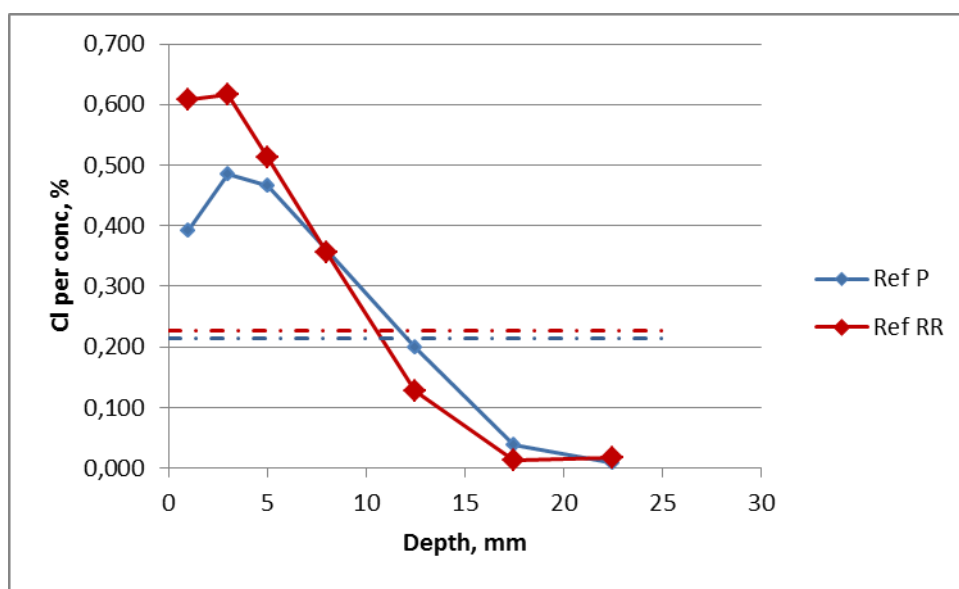
Figure 42: Chloride profiles and total chloride level (broken lines) obtained at CBI in the references in the pre-study and the round robin exercise.

The way to handle the wet specimens and redistribution of chlorides during storage is also likely to be one of the main causes for the differences in the chloride profile and total chloride content between the different laboratories in the round robin exercise and the redistribution of chlorides the main cause for the difference between the results obtained at the same laboratory (CBI) in the pre-study and the round robin exercise. The total chloride level in the references in the round robin exercise and the pre-study is shown in Figure 43.
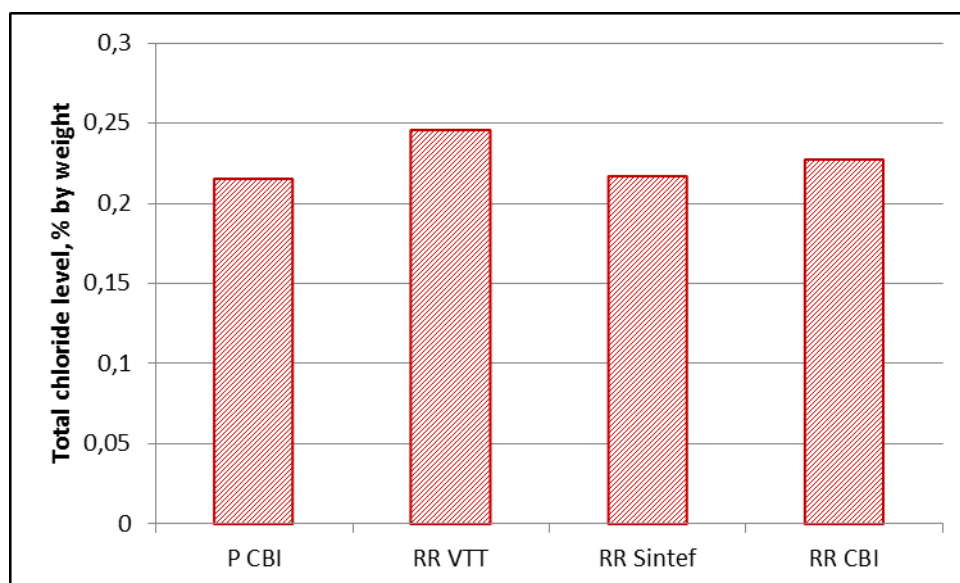
Figure 43: Total chloride level in references obtained in the pre-study and the round robin exercise.

5.4.3.1.7 Grinding

During the actual grinding, deviations may occur due to the size of the ground area and the accuracy of the grinding with regard to the depth measurement. Variations in depth may influence the chloride profile, but will only have a marginal effect on the total chloride content.

5.4.3.1.8 Handling of ground samples

After grinding the samples should as soon as possible be dried to a very low RH, preferably by drying at 105 °C to constant mass, and kept in a dry CO_2-free atmosphere until the chloride analysis is carried out in order to avoid moisture absorption and carbonation that otherwise will influence the dry weight of the sample. If all $Ca(OH)_2$ in the concrete hardened to a degree of hydration equal to 0.7 the increase in dry weight of the concrete due to carbonation will in this case only be about 1.5%. Absorption of moisture to 40 % RH increases the weight with about the same amount.

In the procedures it was stated that the analysis should be carried out on dried samples. However, "dry" is not a well specified condition, which demonstrated itself in that one of the laboratories interpreted this as surface-dry and the other two as oven-dry. There is also a possibility that dry weight is interpreted as the weight of the dry materials (non-evaporable water excluded). The laboratory using the surface dry condition shows a larger scatter between parallel samples in the samples taken close to the concrete surface, than the two labs using oven-dried samples (see Figure 28). This is logical since it is much more difficult to control the moisture content in a surface-dry sample throughout the test than in an oven-dry sample, especially when the sample is ground.

5.4.3.1.9 Chloride analysis

In the procedures distributed to the three laboratories it was stated that the acid soluble chloride content in each layer should be determined by potentiometric titration according to AASHTO-T260

(AASHTO, 1997) (or another method with the same or better precision) and expressed in % of the dry sample weight. All laboratories followed AASHTO-T260.

To understand if the chloride analysis has an influence on the obtained chloride profiles and the resulting filter effect, we plotted the standard deviation of the chloride content at each level, for each product and laboratory (Figure 44 to Figure 47). The results show that the variability within the reference specimens is much less than that of the treated samples, in particular in the outermost layers (0-2 and 2-4 mm); such difference may be associated to the treatment and penetration depth of the impregnation agent, possibly implying that during sample preparation for analysis, not all the chlorides are released into solution. One can also see that the variation within these two layers of treated samples (especially with products P2 and P3) is higher at VTT. In addition, it is also shown that the variability obtained at Sintef is, in general, smaller when compared to the other laboratories. Does this variation affect the filter effect? In Figure 48, the filter effect obtained in the round robin exercise is plotted for different depths, i.e. up to 5 mm (FE_{0-5}) up to 15 mm ($FE\ 0_{-15}$), and compared to the total filter effect considering the whole chloride profile down to 25 mm (FE_{0-25}). The results clearly show that even though the variability is large at depths below 5 mm, there is only a marginal effect on the chloride blocking effect of the impregnation. Moreover, looking only at the results in Figure 47 and Figure 48, one can see that the variability between samples of the same series is not the cause for the differences (and strange behaviour) on the blocking effect obtained at VTT with product P4. The individual chloride profiles can be found in Annex D.4.
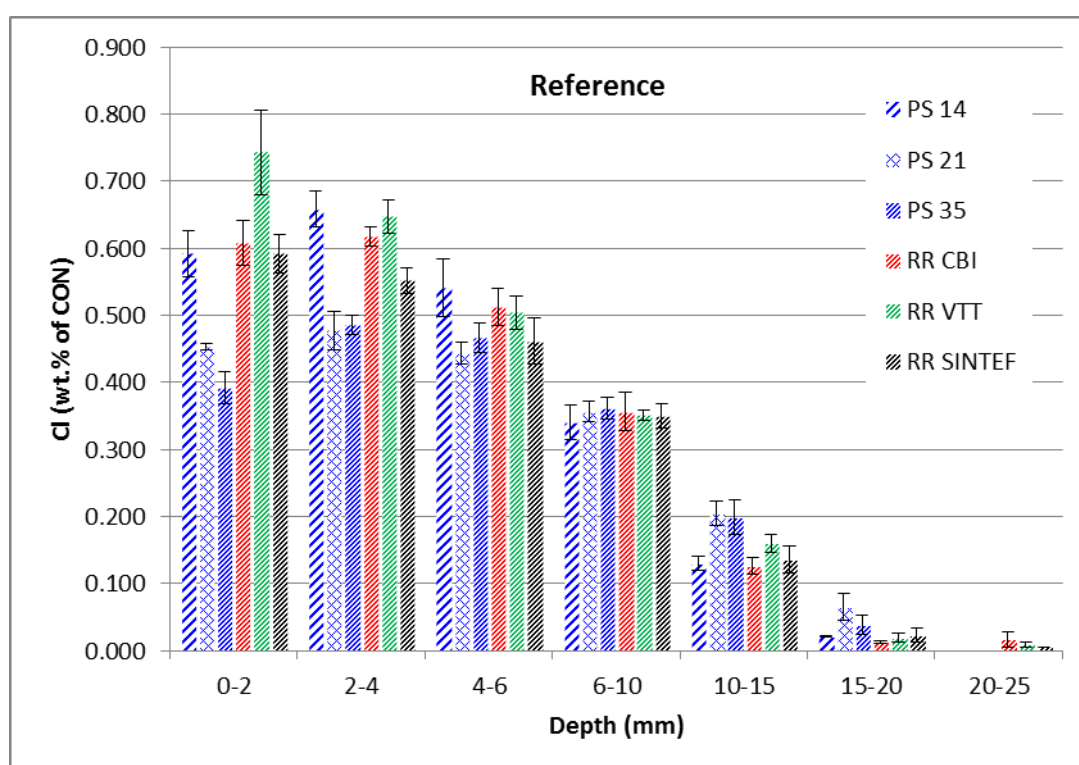
Figure 44: Chloride profiles for the reference samples obtained in the pre-study and the round robin exercise. The error bars represent 2 standard deviations.
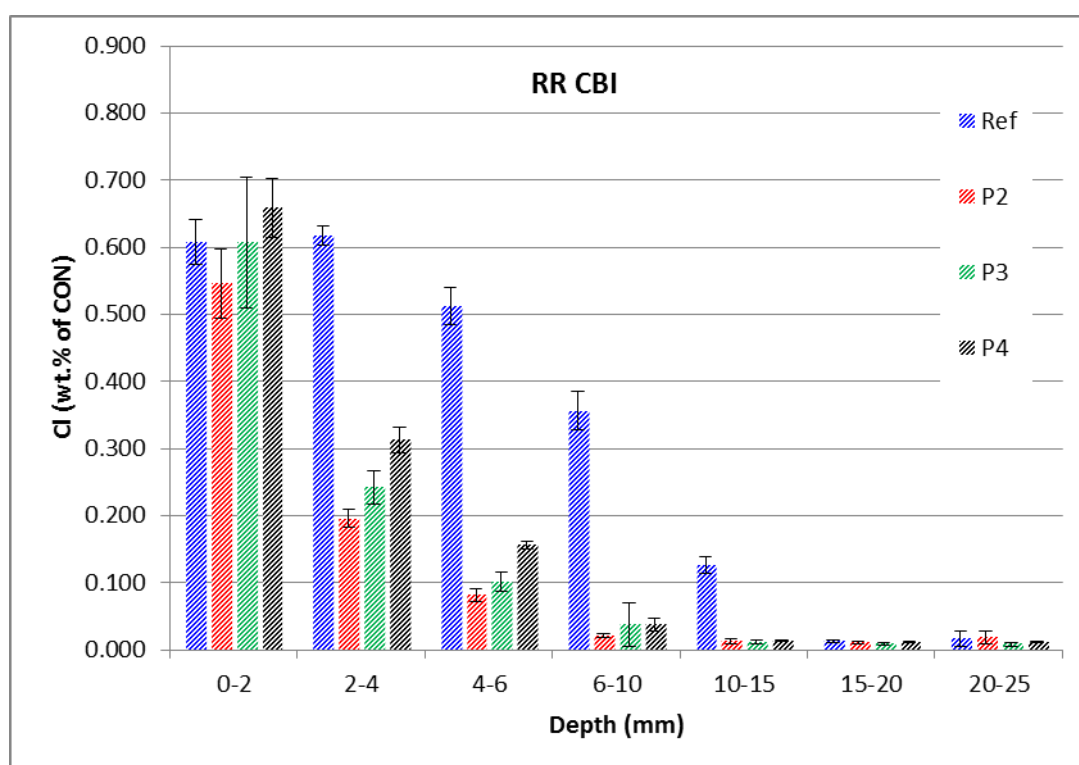
Figure 45: Chloride profiles obtained at CBI in the round robin exercise. The error bars represent 2 standard deviations.
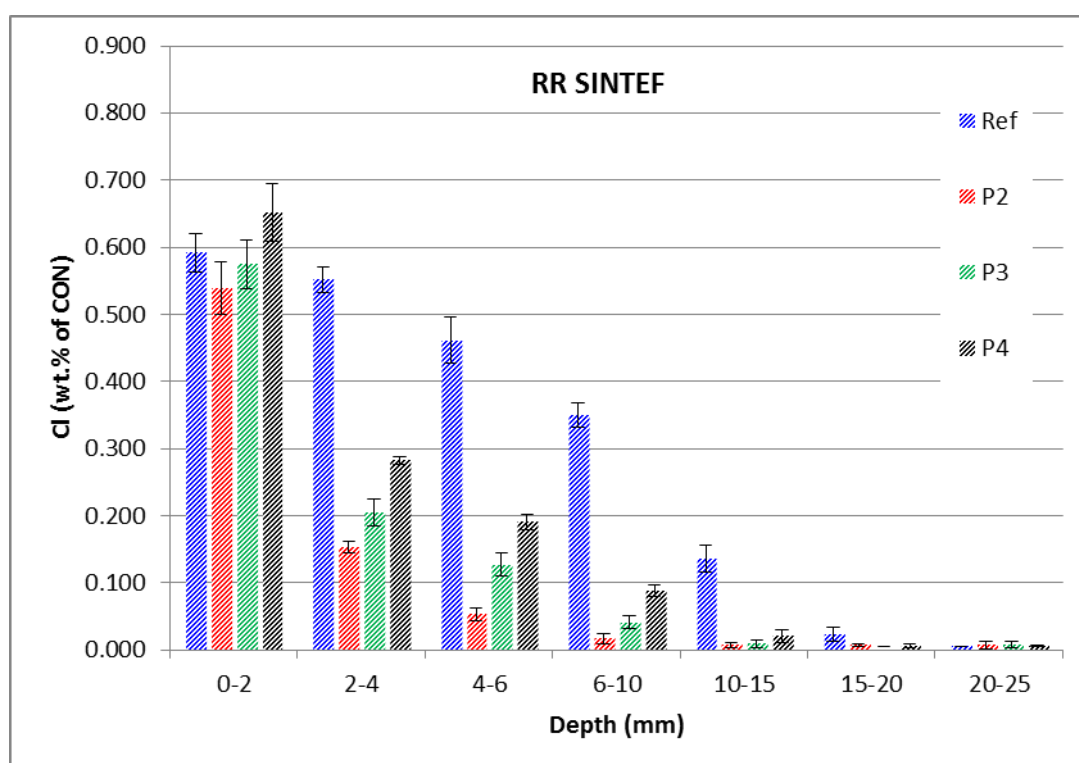
Figure 46: Chloride profiles obtained at Sintef in the round robin exercise. The error bars represent 2 standard deviations.
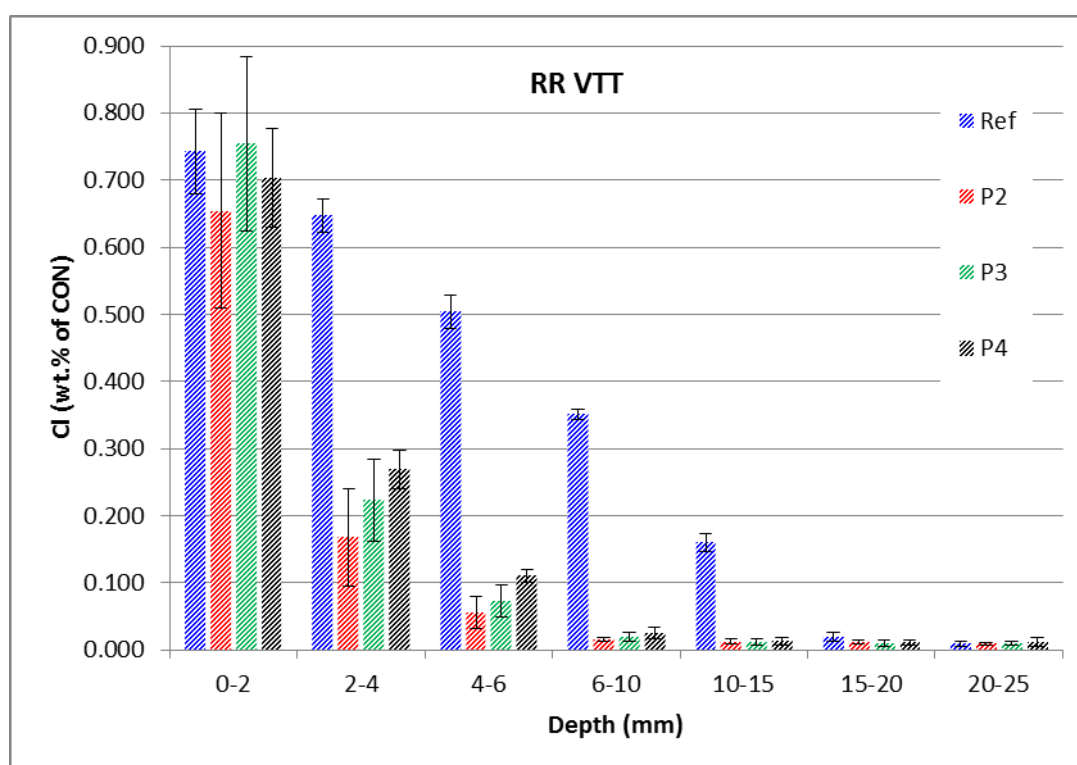
Figure 47: Chloride profiles obtained at VTT in the round robin exercise. The error bars represent 2 standard deviations.
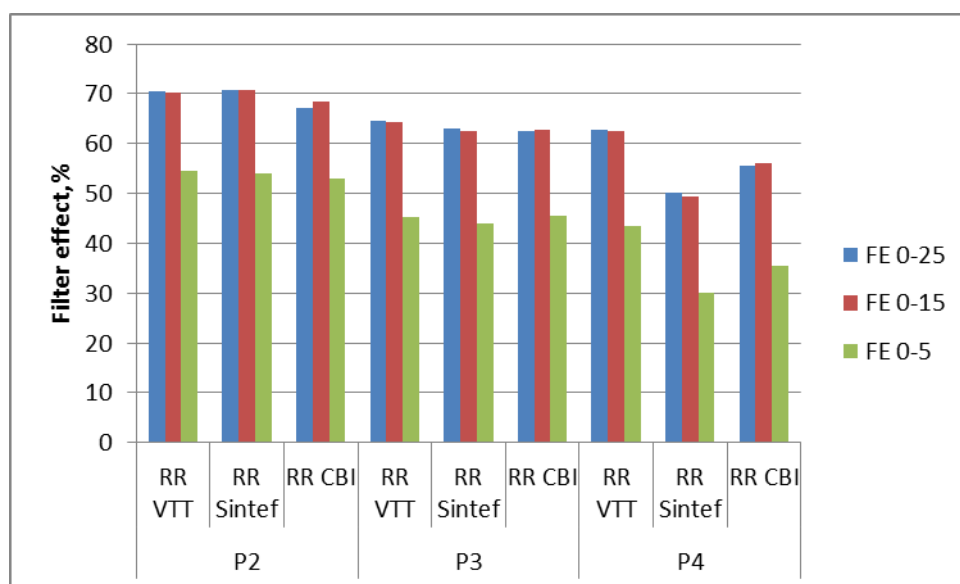
Figure 48: Filter effect at different depths, for the different products and laboratories obtained in the round robin exercise.

5.4.3.2 INFLUENCES OF VARYING TEST PROCEDURES ON THE IMPREGNATION AND ITS CHLORIDE BLOCKING EFFECT

One important observation in the results from both the pre-study and the round-robin exercise is that even though the chloride profile of the reference specimens varies substantially close to the exposed surface in the different test series, the calculated filter effect varies to a much lower degree.

This indicates that, as long as the reference samples and the impregnated samples are treated in the same way within a test series, the factors influencing the results discussed for the reference samples in 5.4.3.1 influence the chloride ingress of the impregnated specimens to approximately the same degree or proportionally.

5.4.3.2.1 Influences of the chemical nature of the concrete surface

Potentially, the chemical properties of the concrete surface the impregnations are applied to, may influence the functioning of the impregnating agent by influencing the bond between the cement hydration products and the impregnating agent. Variation in chemical properties may occur due to for instance different cement types, leaching of $Ca(OH)_2$ during curing or carbonation of the surface. However, the general view among references is that it is not the chemical but the physical differences of the concrete surface, i.e. variations in pore structure and the moisture condition, that affect the efficiency of the impregnating agent as a barrier against moisture and chlorides.

The chemical composition influences the relation between bound and free chlorides, but as concluded in section 3.3.1.1, this is not of importance for the test, since the specimens are not exposed to chlorides at a young age and the exposure period is short. Moreover, only pure portland cements are used in the test and no blended cements, which makes the variations in chemical composition of the concrete small. Thus, the influence of the chemical composition of the concrete substrate will not be discussed further in this report.

Another item in the procedures that may cause a difference of chemical nature on the efficiency of the impregnating agents is whether the surface is exposed to a saturated lime-water solution or not. The reaction between the silanes and the concrete takes place between OH-groups in both the silane and the concrete. Soaking in lime-water may increase the number of OH-groups present in the concrete and preserve the pH-value at the surface, which may increase both the binding capacity per volume of the concrete and the polymerisation rate (Selander, 2010). This would enhance the efficiency of the impregnating agent. In the pre-study the effect of lime-soaking was studied, and there was a small but clear increased efficiency of the impregnation agents when lime-soaking was used as can be seen from Figure 49. Irrespective of the curing time after impregnation (7, 14 and 28 days) the filter effect is for both products approximately 5 %-units higher when the surface is lime-soaked after sawing. Soaking with lime solution also entails wetting of the surface, such that the RH in the surface of the lime-soaked specimens is probably higher than in those where no lime-soaking took place. A higher RH would result in a smaller penetration depth and possibly a lower efficiency of the hydrophobic impregnations. If this effect is taken into consideration the pure effect of addition of $Ca(OH)_2$ would be even more pronounced than what can be seen in Figure 49. More clear-cut conclusions on the influence of $Ca(OH)_2$ could have been drawn if pre-wetting for three days with pure water had been included in the study. However, all surfaces were cut with a diamond saw under the presence of water, so all surfaces experienced a certain amount of pre-wetting.
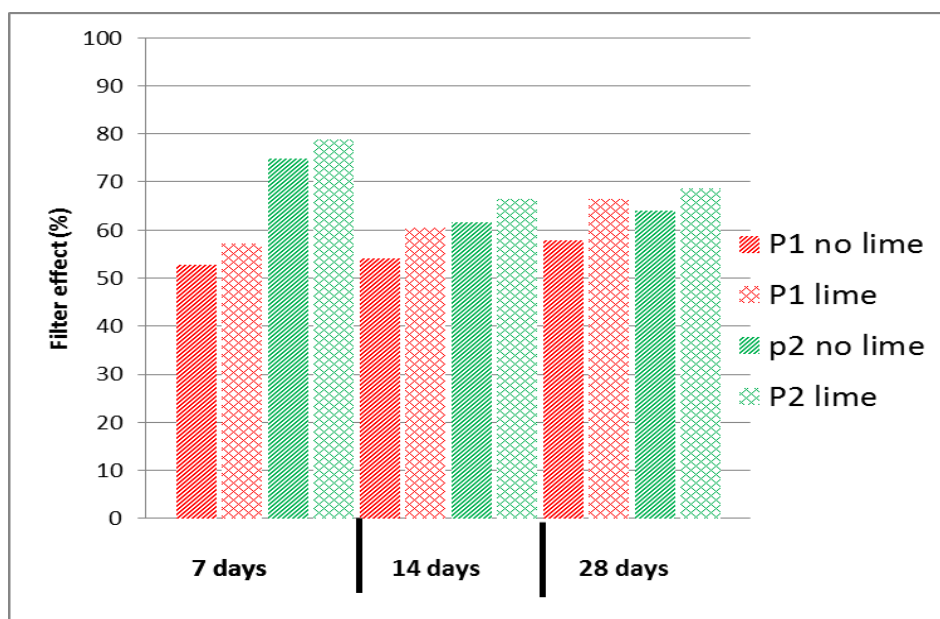
Figure 49: Filter effect with and without treatment with lime-water (3 days) after sawing with two different impregnating agents. Filter effect after 7, 14 and 28 days curing after impregnation.

However, pre-wetting with or without lime is normally not used in practice.

Enrichment of $Ca(OH)_2$ in the surface layer may also occur in form surfaces due to the wall effect. This enrichment may also contribute to the higher filter effect of impregnating agents applied on form surfaces compared to on sawn surfaces, in addition to the physical causes which will be discussed in 5.4.3.2.2.

5.4.3.2.2 Influences of the physical nature of the concrete surface when impregnated

On the penetration of the impregnating agent

Impregnation does not rely upon pore-filling for its efficiency but on the reaction between the silane and the OH-groups of the hydration products to form a water-repellent layer on the surfaces of the hydration products. In this process ethanol is released.

When discussing penetration depths one must also point out that the standardized method to measure the penetration depth (splitting, moistening and measuring the visual limit between wet and dry surface) is a method with bad reproducibility, partly due to that the evaluation is very subjective. According to paper II in (Selander, 2010) the observed limit corresponds to a concentration of the product equal to about 1.5 mg of silane per g of concrete measured with FTIR. At that level the reaction product between the silane and the concrete occupies a very small part of the available pore-volume (less than 2-3 %). When the release of ethanol during the polymerization process is taken into account it becomes even smaller. The highest concentration level recorded in paper II in (Selander, 2010) in a concrete with a w/c-ratio = 0.45 was about 6.5 mg/g of concrete.

Moreover, the standard method only gives one limit, not the concentration profile and no hint about the maximum level. Thus, the actual amount of silane within the zone detected as hydrophobic with the standardized method may vary substantially (Selander, 2010).

In order to use the products optimally a situation where as much silane as possible gets the possibility to react with the cement hydration products should be created. For the effect to be durable the impregnation should penetrate as far as possible into the treated specimen, in order to withstand weathering and abrasion, i.e. the optimal surface conditions should be extended as far as possible into the concrete to be treated. The polymerization of the product also needs some time to form the water repellent surface, so measures to provide sufficient time and optimal conditions for the polymerization process to take place should also be taken.

In (Selander, 2010) it is shown that the moisture sorption properties of an impregnated sample depends on the relative humidity in the concrete when the impregnation is carried out. The lower the RH, the larger is the amount of empty pore space that exists when the impregnation is carried out and the more the moisture sorption properties of the concrete are influenced. The influence is restricted to the capillary pore region, i.e. at higher RHs (above 40%). Selander has also shown that the hygroscopicity of the gel porosity is not influenced.

The total amount of pores in the specimens at a certain time is determined by the w/c-ratio, the paste content, the degree of hydration and to some degree the cement type, especially if blended cements are used. As discussed in 5.4.2.2 and 5.4.3.1 the amount of pores that at a certain time will be empty depends in addition on the ambient RH and the pore-size distribution. The pore-size distribution will be influenced by the grain size of the cement to a certain degree, and it will be affected by the moisture sorption history of the surface, i.e. if it has previously undergone desorption or not.

In the present study the same w/c-ratio, the same composition and approximately the same cement was used. Most tests are carried out on sawn surfaces of water cured mature concrete (minimum 35 days of age) which gives only small variations in degree of hydration. Thus the total pore volume in the surface will be approximately the same in all cases. One exception is the comparative study with form surfaces, where the wall-effect leads to a higher pore volume close to the surface. For the rest of the study, variations in empty pore volume at the time of impregnation are mainly caused by differences in the preconditioning climate and period. Three different pre-conditionings were used in the pre-study. Unfortunately the penetration depth of the impregnation was not measured for all these cases, only for preconditioning types S (65% RH for 7 days) and C3 (3 days in saturated lime-water and 3 days in 65% RH). The impregnation depths were measured 7 days after impregnation. For both products the penetration depth measured for type C3 is only about 60% of the penetration depth for the longer period (type S), see Figure 6.

When an impregnating agent is applied on a surface with a high amount of pores that are not water filled there will exist pore surfaces that are directly exposed to the impregnating agent with which they may react. Transport of the impregnating agent towards the interior will also be facilitated by a lower moisture content.

When the impregnating agent is applied on a surface with few empty pores the agent will not penetrate as easily and will not be transported further into the specimen as easily. With less exposed pore surface, the reaction will probably be slower.

Since the specimens normally are exposed to lower RHs after application of the product the drying of the surface will proceed even after application, continuously decreasing the water content in the pore space giving opportunity for further ingress of the agent during the polymerization period. Therefore, the agents appear to have some effect even when applied on a saturated surface. If, however, the saturated situation prevails also after the application and during the polymerization period, the effect, if any, would probably be strictly superficial on the specimen.

Under real conditions where the amount of agent is not limited, the effect of varying empty pore space on the penetration will primarily be manifested in the way that a surface with more empty pores appears to be more absorbing than one with less. It will consume more impregnating agent, which will penetrate further into the concrete. A surface with few empty pores will appear saturated when much less agent is used, perhaps too little to create a durable moisture and chloride barrier.

The product P1 in the pre-study and product P3 in the round robin exercise were essentially the same product but the amounts applied differed, 300 g/m^2 and 200 g/m^2 respectively. However, the measured penetration depths were about the same, compare Figure 4 and Figure 23.

Unfortunately, the impregnation depths were not measured in the series in the pre-study with form surfaces and sawn surfaces. The penetration depth of two different impregnation agents on dry and saturated sawn and form surfaces was measured by Wacker[2] They demonstrated generally a higher penetration depth on form surfaces than on sawn surfaces. However, the experimental details (for example how the specimens were cured, how the surfaces were obtained and conditioned) were not well described which make the interpretation of the results difficult.

In (Rodum, 2013) the penetration depth on sawn surfaces and form surfaces after varied preconditioning periods in 50% RH were compared, and in that case the penetration depths were larger on the sawn surfaces than on the form surfaces.

On the chloride blocking effect of the impregnating agent

The influence of the pre-conditioning period on the chloride blocking effect is not as large as on the penetration depth as can be seen in Figure 50 where the filter effect of the two products in the pre-study for which the penetration depth was measured (Figure 6). However, not only the length of the pre-conditioning period differed. Lime-soaking was used on those with shorter periods and not on those with longer. As mentioned earlier, lime-soaking has a positive effect on the chloride blocking effect of the impregnation agents (see Figure 49). This may have diminished the effect of the pre-conditioning period recorded.

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[2] Personal communication; Dr. Hartmut Ackermann, Wacker Chemie AS, Extract from internal report, 2010.
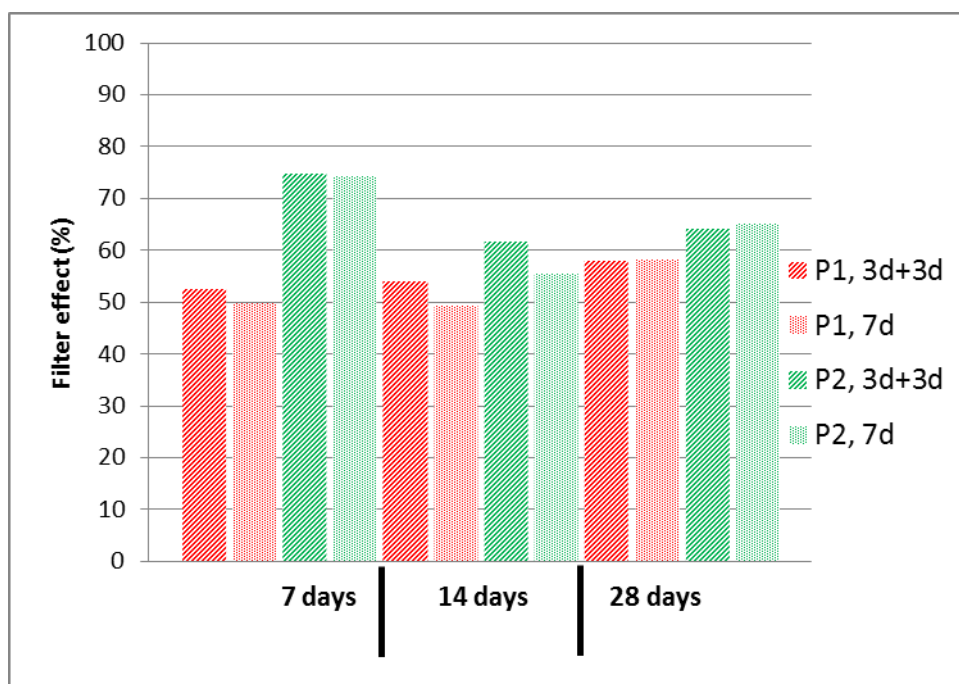
Figure 50: Filter effect for two impregnation products after the same pre-conditioning procedures as in Figure 6 and after 7, 14 and 28 days of curing after impregnation.

In Figure 51 the filter effect after the two preconditioning procedures, where the only difference is that 3 or 7 days in 65% RH after 3 days of pre-wetting in $Ca(OH)_2$-solution is used is shown. In all cases the filter effect is larger after 7 days of drying than after 3 days. The difference varies between 5 and 11 %-units.
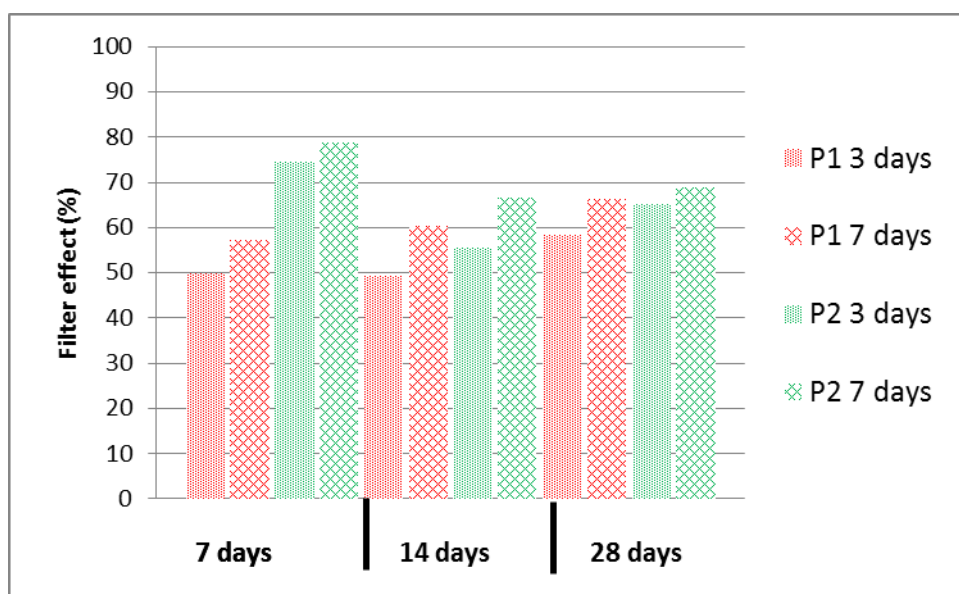
Figure 51: Filter effect for the two products in the pre-study after pre-conditioning for 3 or 7 days in 65 % RH after 7,14 and 28 days curing. All specimens were previously submitted to saturated lime-water for 3 days.

Thus, the conclusions that can be drawn regarding the influence on the conditions (RH-level, soaking in lime-water) during pre-conditioning and its length is that longer exposure to lower RHs before application of the impregnation agent has a large positive effect on the penetration depth. The influence on the measured chloride blocking effect is also positive, but not as large as on the

penetration depth. It seems as if a penetration depth that is around 2-3 mm is achieved, this will give a clear effect on the short run. However, a larger penetration depth provides robustness and durability to the effect.

5.4.3.2.3 The manner to impregnate - is it of importance for the chloride blocking effect?

Another important factor influencing the penetration depth is the time period the concrete surface is exposed to the impregnating agent. In the method a certain amount of the agent is applied on a horizontal upper surface and hindered to flow off through the sides of the specimen. This may be more favourable conditions for the penetration of the product than what can normally be obtained in reality. However, in order to obtain good reproducibility with the method the conditions have to be as well specified as possible, which would not be the case with more realistic impregnation conditions. Even though the measured effect may become somewhat better with this method than in reality, the interrelation between the products as regards their effect (i.e. the classification) should not be affected.

The length of the impregnation curing period

Hydrophobic impregnation products need a certain curing period under relatively favourable conditions, so that it can penetrate the concrete surface, react with the cement hydration products, and polymerize in order to provide the concrete with a hydrophobic surface. How long this period needs to be depends on the nature of the hydrophobic agent, and probably also on the moisture condition in the concrete surface. In the pre-study the chloride blocking effect of two different products, one gel and one liquid, after three different impregnation curing periods (7, 14 and 28 days) was investigated, and it was found that the most stable results were obtained when the impregnation curing period was 28 days. One product showed a better filter effect at 7 days than at 14 and 28 days.

A possible reason for the surprisingly large filter effect after an impregnation curing period of 7 days was put forward in 4.3.3.2.2, i.e. that a vulnerable continuous gelatinous surface layer gives an extra protection against chloride after short impregnation curing periods. Since the impregnation curing time in the test has to be sufficiently long to avoid the influence of such layers and provide a result that is stable and representative for the effect on long term, it was decided to use 28 days for the curing of the impregnation.

The moisture conditions during preconditioning

The impregnation curing in the round robin test should be carried out in 60±10 % RH and 20±2 °C for 28 days. However, SINTEF had used a climate room regulated at 50±5 % RH while at CBI and VTT rooms regulated at 65±5 %RH were used. (The pre-conditioning was carried out in 65% RH in all three laboratories). Can any variations in the results as regards the impregnation depth and chloride blocking effect in the samples be contributed to this difference? There are a lot of studies, see for example (Selander, 2010), where the influence of the RH level of the surface when the sample is impregnated is studied, but there are not many references dealing with the influence of the RH level during the penetration and polymerization phase. Silanes are highly volatile and it would be expected that the lower the RH-level during this phase, the larger the amount of silane that will evaporate into

the surrounding air will be. The volume and the movement of the air surrounding the specimens during curing may also be a factor influencing the amount of silane that is actually left on the concrete surface.

A lower RH level during this period than during the pre-conditioning will also increase the evaporation of water from the concrete surface immediately after the start of the exposure, a movement that may pull along the impregnating agent and obstruct the penetration.

One observation that may indicate that the conditions during the curing of the impregnation may have an influence on the penetration depth is that the penetration depths measured at SINTEF were surprisingly small compared to those obtained at VTT and CBI in the round-robin exercise. Especially when measured with the standardized method, see Figure 23, even when the subjectivity of this method is taken into consideration.

In addition to a different RH-level SINTEF used a coarser cement with a low alkali content, which gives a more coarse pore structure. This may also have influenced the penetration depth.

Does the difference in RH-level during the impregnation curing period influence the measured chloride ingress in the impregnated samples and the calculated filter effect? The filter effect obtained for the three products at the three laboratories involved in the round robin exercise is shown in Figure 52. Looking at the average of the filter effects obtained at the three laboratories and the penetration depths one can see that best filter effect is achieved with the product which has the highest average penetration depth. Within each laboratory you can also draw the same conclusion for VTT and SINTEF, but not for CBI.
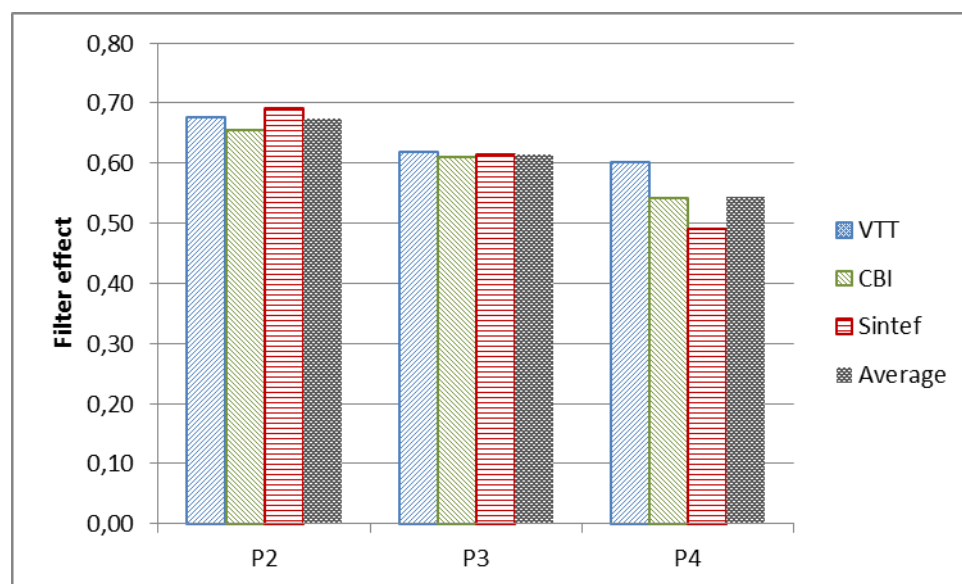
Figure 52: Filter effect for the three products in the round robin exercise obtained at the three laboratories.

If, however, the penetration depths acquired by the drop method is used, also the results from CBI follows the hypothesis that larger penetration depths gives better protection against chloride ingress. However, relating the chloride protection directly to the value of the penetration depth obtained with the standard method, irrespective of if it is obtained by different persons and on

differing substrates, is impossible. For example P3 shows the same filter effect at all laboratories but the penetration depths measured varies between 2.0 and 5.8 mm, i.e. almost by a factor 3.

There is a need for a less subjective method than the one used in EN 1504-2 (CEN, 2004) to determine the penetration depth both in laboratory conditions and on real structures, which is not too complicated to use. Preferably, such a method should also give the concentration profile. Although also somewhat subjective, the drop method used in this study gives more reproducible results.

Product P4 is the one with the lowest filter effect and also the largest variations between the values obtained at the different laboratories. It is a thixotropic cream with 80% silane. There is a possibility that this cream is more sensitive to low RH-levels (50% for the case of SINTEF) during the impregnation curing phase, than the gel (P2) and the liquid (P3). However, the causes for the variations obtained for product P4 need to be studied more thoroughly.

Based on the results, one may conclude that for most products the method is not sensitive to a difference in RH-level within the range 60±10 % during the impregnation curing period, as long as the same RH-level and the same procedures throughout the rest of the test is used for both the references and the impregnated samples. However, since there are products that seem to be more sensitive than others, the range of the RH-level would preferably be narrowed down to 65±5 %, for both the pre-conditioning period and the impregnation curing period, in order to increase the reproducibility of the method.

The possibility that products behave differently with regard to the chloride blocking effect at different RH:s and the implication of this need further studies.

5.4.3.2.4 Chloride exposure period, withdrawal from the NaCl-solution and grinding

The same potential influences discussed for the reference samples may also influence the impregnated samples. After 56 days of exposure, the pore volume in the outermost surface layer of the impregnated samples is also filled with chloride solution. In Figure 53 the chloride levels (maximum, minimum and average) in the surface layer (2 mm) for all products and the references obtained at the different laboratories are given. In this figure a line marking the average of all samples from each laboratory is added. Some striking systematic observations may be made from this figure.

1. The values, both the overall level and the interrelations between average values for the reference and the three products, obtained at CBI and SINTEF are similar.

2. Variations between samples analysed at VTT are generally larger than at both CBI and SINTEF.

3. In specimens impregnated with product P2 (gel) the chloride level in all cases lies slightly under the level in the references.

4. In specimens with product P3 (liquid) the chloride level is in all cases at approximately the same level as in the references.

5. In specimens with product P4 (thixotropic cream) the chloride level in the impregnated specimens lies clearly above the level in the references in the results from SINTEF and CBI, but not VTT.
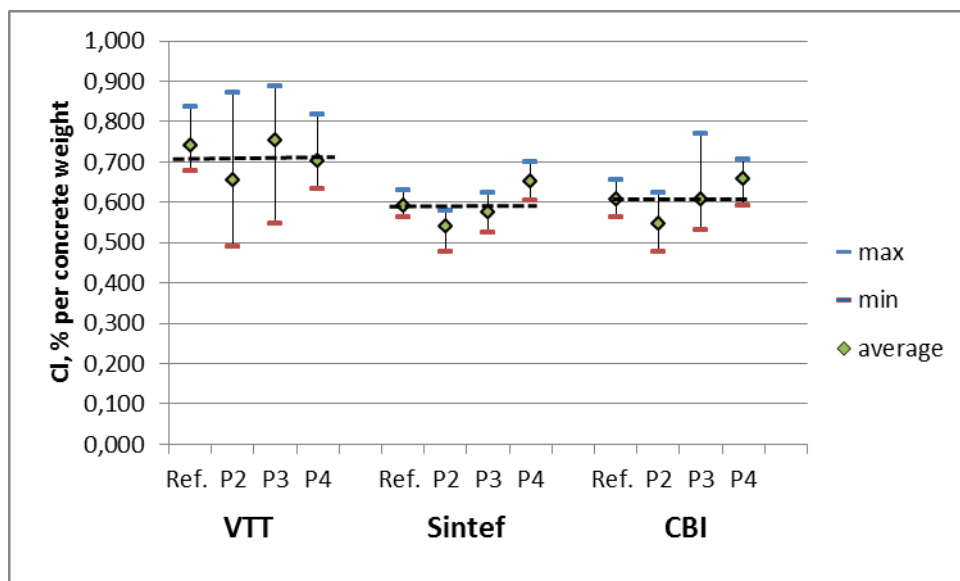
Figure 53: Chloride level in the outermost 2 mm for both impregnated specimens and references, for the three laboratories in the round robin exercise. The broken line is the average level for each laboratory, respectively.

In 5.4.3 it was concluded that the maximum chloride level in the references in this layer could be calculated on the assumption that the entire pore volume in this layer was filled with the 15% NaCl-solution. A lower value in the impregnated samples may be caused by different factors.

- The pore volume is the same but it is not completely filled with the 15% solution.

- The pore volume is the same and it is completely filled with the 15% solution but not up to the whole thickness of the layer.

Irrespective of the cause, a more detailed distribution of the chlorides close to the surface may look as in Figure 54.
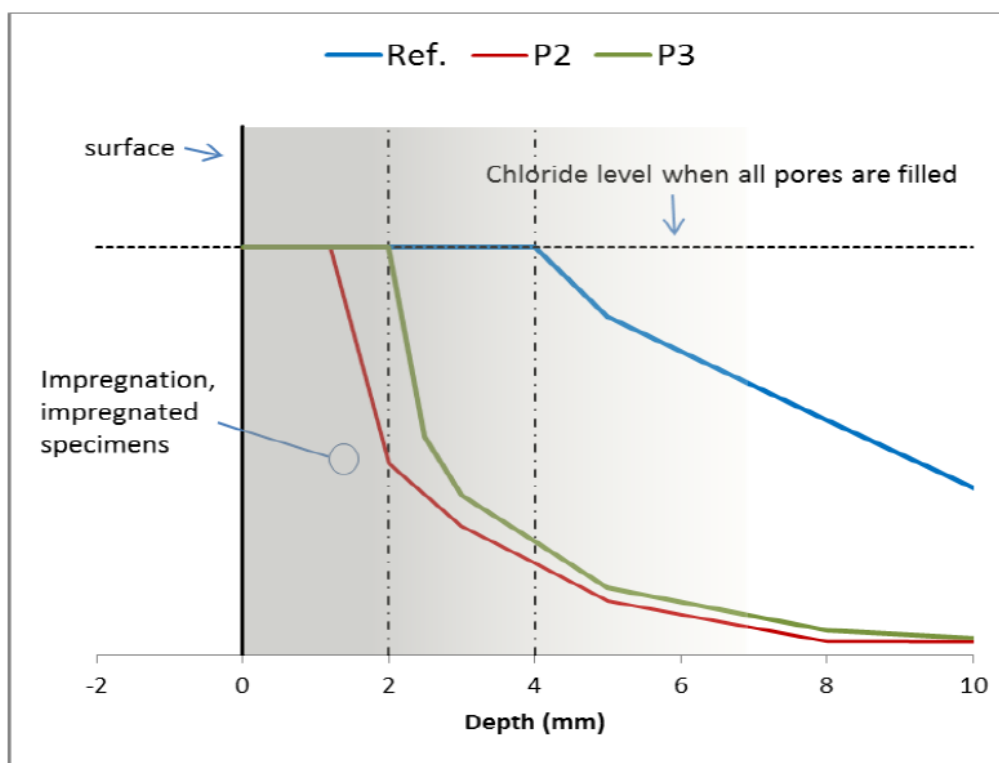
Figure 54: Possible detailed chloride distribution close to the surface for specimens impregnated with products P2 and P3 and the references.

The fact that the chloride value obtained with product P4 is higher than what is obtained with the references, is more intriguing. The difference, about 5%-units, is too high to be caused by a lower degree of hydration only (5.4.3.1.2). This product must in some way affect the specimens or its pore structure so that more chlorides penetrate the surface layer when impregnated than when not. This may be caused by increased leaching, increased degree of filling of air pores, chemical binding of chlorides to the substrate or to the impregnating agent or some other mechanism. It is possible that the compound in this impregnating agent that provides the thixotropy also binds chlorides.

It should however be noted that the increased chloride level only concerns the outermost surface layer (2 mm). In the inner parts the chloride levels in specimens impregnated with product P4 are well below the levels in the references. When looking at the chloride profiles for the three products (see CBI results in Figure 55) one can however see that the vertical distance between the curves of the three products that appears in the surface layer is preserved down to a depth below 5 mm.
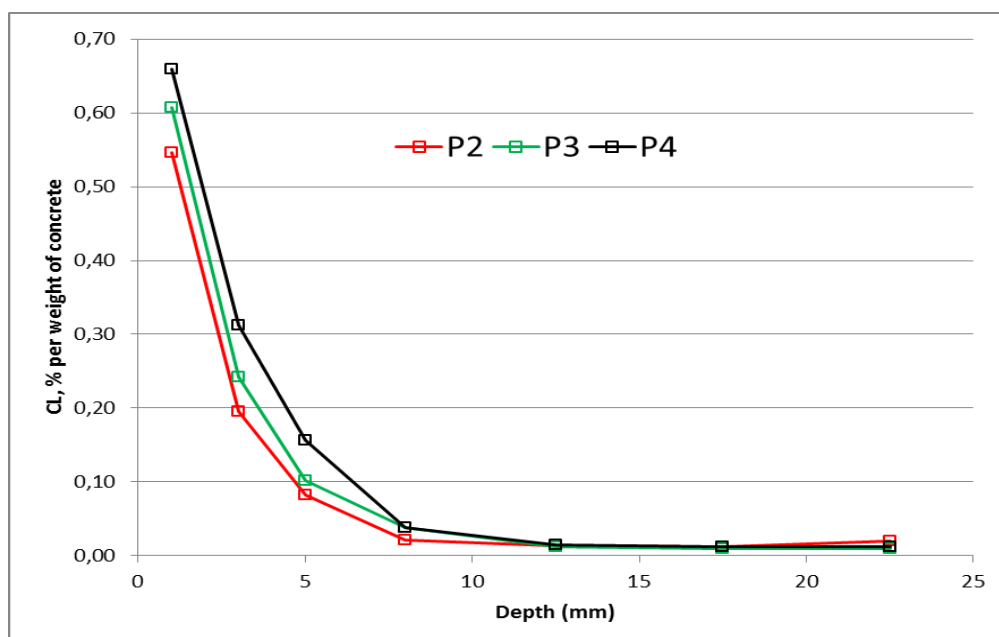
Figure 55: Chloride profiles for the three products in the round-robin exercise obtained at CBI.

In the references from CBI the 4 outermost mm have obtained the maximum chloride level. Since at the beginning of the chloride exposure 55% of the pore volume in this layer is already filled with water (moisture profile according to Figure 40) maximum 45% of the chlorides in this layer has entered the specimens through capillary transport, the rest is transported by diffusion from the solution bath to the already present pore water. In the impregnated samples this combined capillary transport and diffusion has only filled the 2 mm (approx.) which has a chloride level corresponding to the 15% NaCl-solution.

One may wonder what would happen with the chloride blocking effect if this totally solution filled layer would stretch beyond the impregnation depth in the impregnated samples?

Handling of the wet surface at withdrawal from the chloride solution and redistribution of chlorides during storage

As mentioned in 5.4.3.1.6 the handling of the specimens when taken from the chloride bath has a large influence on the chloride level in the outermost surface layer. The chloride solution in the largest pores may be sucked out if it is wiped with an absorbing tissue. However, this may lower the chloride content in the outermost surface layer in both the references and the impregnated samples. How much this influences the total chloride level depends on the thickness of the layer which is totally filled just before the specimens are taken from the chloride solution, the sensitivity of the pore-liquid to the exposure to an absorbing tissue (i.e. the pore-size distribution of the specimen at high RH:s). Redistribution of chlorides during storage may also occur in the impregnated samples.

As shown in Figure 56, the same observation as for the references (Figure 42 and Figure 43) can be made for the specimens impregnated with product P2; i.e. even though the form of the chloride profile varies substantially the total chloride content is approximately constant. The change in the profile in the impregnated samples seems to be more dramatic than for the references (Figure 42) probably due the very steep form of the chloride profile in this layer so close to the surface and to the hydrophobicity of this layer.
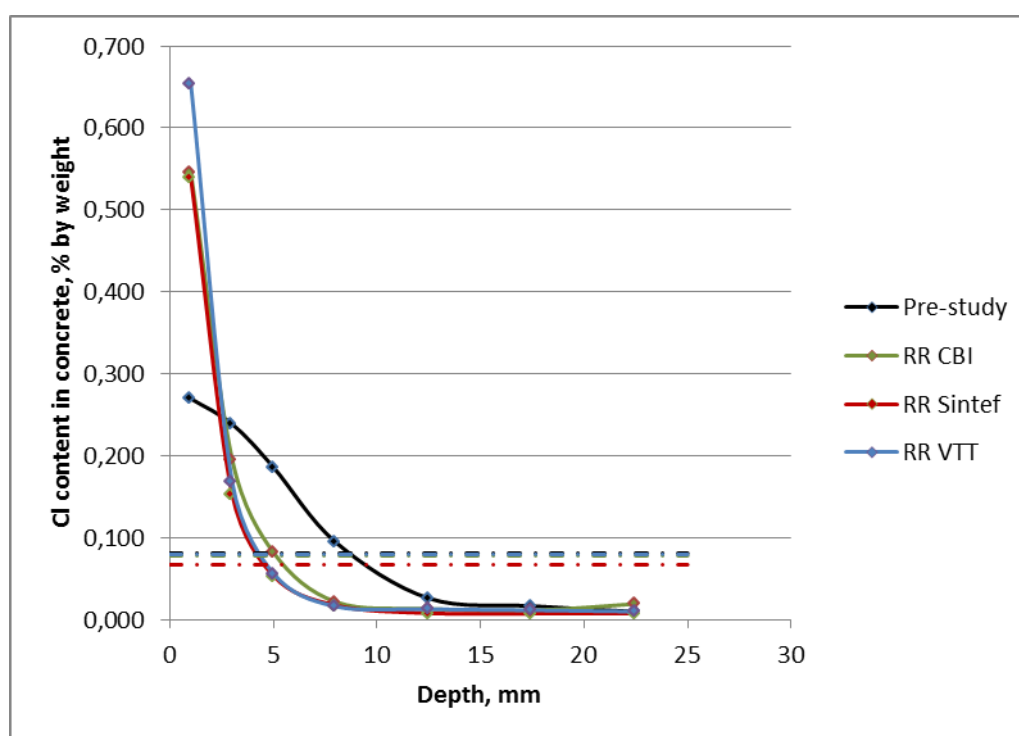
Figure 56: The chloride profile and the total chloride content (broken line) for the specimens impregnated with product P2 in the pre-study and the round robin exercise.

If the influence of these factors on the total chloride value obtained on impregnated samples and references is equal or proportional, the calculated filter effect should not be affected. In Figure 52 the filter effect for all products and laboratories in the round robin exercise is shown, and it can be seen that the filter effect for products P2 and P3 do not vary more than marginally. As noted before the variations for product P4 are much larger, and the reasons for this is not quite clear.

5.4.3.3 POSSIBLE IMPROVEMENTS OF THE PROCEDURE TO OBTAIN A BETTER REPRODUCIBILITY

Based on all the findings in 5.4.2.1 to 5.4.3.2 it can be concluded that the main improvements of the procedures that will give a better reproducibility of the method concerns:

- The specification of the handling of the specimens when taken from the chloride solution.

- The specification of the handling of the specimens after grinding and until the chloride analysis is carried out.

5.4.3.3.1 Suggestions for clarification of the procedure when taken out from the chloride solution

Changes and additions to the procedures used in the round-robin exercise:

- Up to 3 products can be tested simultaneously with the same reference series.

- When the specimens are taken up from the NaCl bath the excess solution on the surface is carefully wiped off with a cloth moistened in the NaCl-solution and squeezed in order to remove excess liquid.

- If it is not possible to grind the specimens directly thereafter, the specimens shall be tightly sealed in plastics and stored at 5±2 °C.

- The specimens shall be ground within 7 days from the moment they are taken up from the NaCl solution. All parallel specimens (same product/reference) shall be ground the same day. The grinding of the impregnated samples and their corresponding references shall be carried out within 48 hours.

5.4.3.3.2 Suggestion for clarification of the handling of the material between grinding and chloride analysis.

Changes and additions to the procedures used in the round-robin exercise:

- After grinding the ground samples shall be oven-dried in 105±5 °C until constant mass is achieved.

- The ground samples shall, before and after oven-drying, be kept protected from humidity and CO_2 until the chloride analysis is carried out.

5.5 Expression of results

5.5.1 The filter effect

Rather early in the project it was decided to formulate the requirement as a filter effect, as in the existing Norwegian method (see4.3.2.1) :

$$Filter\ Effect = \left[1 - \frac{Cl_{\,treated}}{Cl_{\,reference}}\right]$$

I.e. how much of the chlorides penetrating a non-treated specimen is filtered away when an impregnating agent is applied to the surface.

However, there is a to need provide some further specifications on how the filter effect shall be determined; for example:

- Which depth shall be considered?
- Shall the basic chloride level in the specimens be subtracted from the values or not?
- Is it ok to use the chloride content per dry sample or should the chloride content per cement weight be used?
- Is it necessary to determine the chloride profile, or could the analysis be made on the whole depth in one step?

5.5.1.1 TO WHICH DEPTH?

The aim should be to try to encompass the entire chloride profile in both the references and the impregnated samples. In the pre-study a depth of 20 mm were used, however the chloride level in the references were in the level 15-20 mm higher than the initial chloride level of the specimens in

most cases after 56 days exposure. In the round robin exercise, the profile was ground so that the deepest layer was from the depth 20-25 mm. At this level generally no external chlorides were detected. Thus when a concrete with w/c = 0.45 is used as substrate, it could actually be sufficient to use the outermost 20 mm for the chloride profile, assuming a zero level of external chlorides at 20-25 mm. This is however not recommended, due to shortness of available experimental results.

5.5.1.2 DEDUCTION OF THE INITIAL CHLORIDE CONTENT OR NOT?

Another item that needs to be settled is whether the initial chloride level in the specimens should be excluded when calculating the filter effect or not. This level may either be estimated based on the composition and the chloride content of the constituents or determined by analysis on a piece taken at a depth where no external chlorides exist.

How much does then the grinding depth and the inclusion/exclusion of the initial chloride content influence the calculated filter effect? Grinding down to 25 mm as in the round -robin exercise gives the advantage of a rather good estimation of the initial chloride level. Based on the chloride values at this depth from all specimens the initial chloride content in specimens cast and analysed at VTT and CBI is approximately 0.010% and for the SINTEF specimens the value is about 0.005%.

The filter effect calculated on values from 0-25 mm (FE_{25}) and 0-20 mm (FE_{20}) respectively for products P2 and P3 at the three laboratories involved in the round robin exercise are shown in Figure 57. When calculating the filter effect two alternatives have been used, either the initial chloride level is ignored (Ign) or the initial chloride level is deduced (Ded).
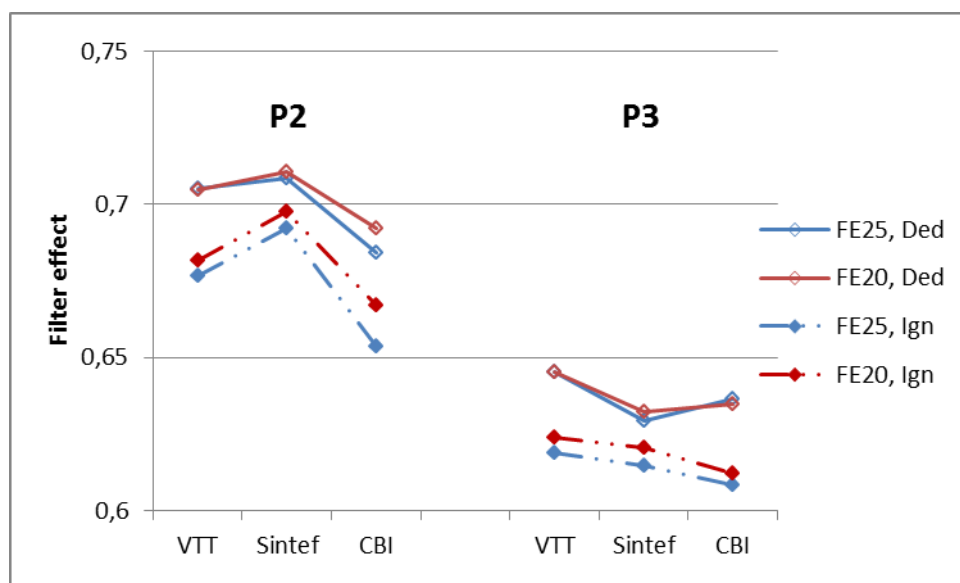
Figure 57: Filter effects calculated for two products based on values from three laboratories, using 20 or 25 mm depth and with and without deduction of initial chloride level.

Theoretically, the most correct results should be obtained with the larger depth and when the initial chloride content is deduced. As can be seen the difference between using 20 or 25 mm depth when the initial chloride level is deduced is very marginal, which is anticipated in this case (when w/c = 0.45) since no external chlorides have penetrated below 20 mm depth. If a higher w/c-ratio is used the chloride would probably have penetrated further and using another depth would have been more appropriate.

Ignoring the initial chloride level leads generally to lower filter effects. How much lower depends naturally on the initial chloride level (normally 0.005 to 0.010%) and also on the depth considered. The decrease in the calculated filter effect, if no deduction for the initial chloride level is carried out, becomes about $0.11*d*Icl$ where d is the used analysed depth in mm and Icl is the initial chloride level in % of dry sample mass. When the initial chloride level is 0.010% and the depth 25 mm is used as for the specimens from VTT and CBI the decrease becomes $0.11*25*0.01 = 0.0275$, i.e. around 2.8 %-units. When the initial chloride level is halved as in the specimens from SINTEF the decrease is also halved and becomes about 1.4 %-units.

5.5.1.3 CHLORIDE CONTENT PER DRY SAMPLE MASS OR PER CEMENT WEIGHT?

Will the precision of the method be better if the cement content in each layer is determined and the chloride is expressed per cement weight? In the pre-study the CaO content was measured and the cement content estimated based on this value for two different pre-conditionings (pc1: 7 days in 65% RH and pc2: 3 days in lime water+3 days in 65% RH) for the two products and the impregnation curing period equal to 7 days. The filter effects were calculated based on chloride per dry sample weight and chloride per cement weight over the 20 mm analysed. The results are shown in Figure 58, and as can be seen the influence on the filter effect is hardly noticeable. Similar observations were made in the round robin exercise, see 5.3.3.6. Thus, the extra operation of analysing the CaO content of the samples will not enhance the reproducibility of the method and is therefore not motivated.
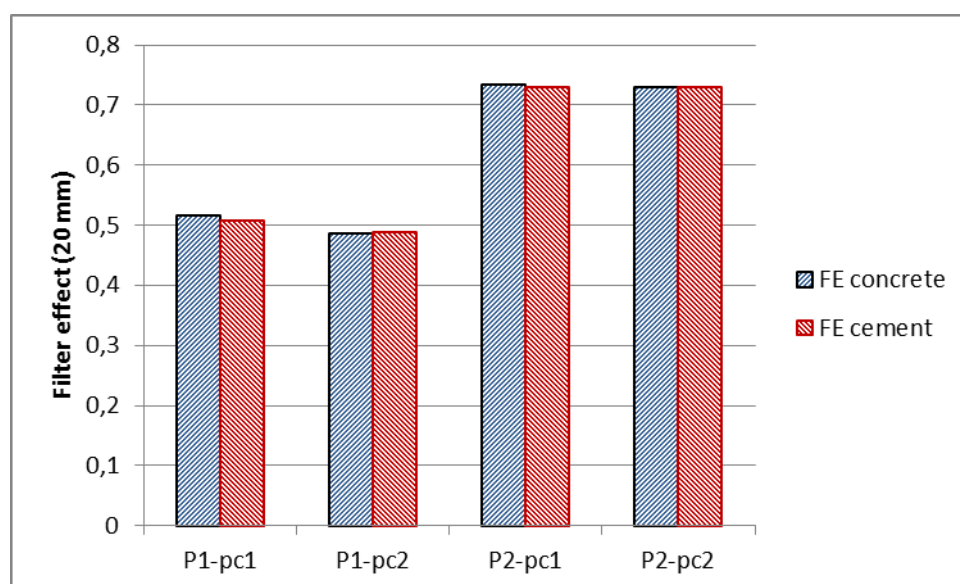
Figure 58: Filter effect (20 mm) based on chloride per dry sample weight and chloride per cement weight for two impregnations products in the pre-study and two different pre-conditionings. Impregnation curing period = 7 days.

5.5.1.4 CHLORIDE PROFILE VERSUS CHLORIDE CONTENT OF THE TOTAL DEPTH?

Since the filter effect, as formulated, uses the total chloride content in the analysed depth it could be argued that it is not necessary to obtain the total chloride profile by analysing the chloride content in 7 separate steps, one could simply analyse the entire level (20 or 25 mm), which would reduce the costs for the analysis. However, one would then never be sure if really all the penetrated chlorides are included in the analysis or not. Obtaining the whole curve assures that it can be controlled that

all penetrated chlorides are included. Using 25 mm as a depth also leads to that by analysing the last step normally gives a good estimate of the initial chloride level. For research reasons, it has been very useful to have small steps (2 mm) close to the surface. This is perhaps not necessary when the method is used for classifying impregnation products only, where larger steps could be used throughout the analysed depth.

5.5.1.5 CONCLUDING REMARKS - FILTER EFFECT

From these observations the following conclusions regarding the method can be drawn:

- The filter effect should, for a concrete with w/c = 0.45, be based on results from chloride profiles obtained by analysing the chloride content in continuous layers maximum 5 mm thick down to a depth of 25 mm from the exposed surface.
- The chloride content should be calculated and given as chloride per oven dry sample weight.
- The initial chloride content shall be deduced from the total chloride content of the layers for the calculation of the filter effect.
- The initial chloride content may normally be estimated based on the chloride level of the layer 20-25 mm of both references and impregnated samples.

6   COMPARISION WITH OTHER METHODS

The new method shall replace the three existing, different Nordic test methods used for classifying the chloride blocking effect of impregnating agents. It is therefore necessary to have a clear view of the differences between the old and the new method and also to have an estimation of how these differences influences the value selected as criterion for classification, i.e. the filter effect. In 6.1 to 6.3 the comparison with the three existing methods will be carried out, one at a time.

6.1   Comparison with the existing Swedish method.

The existing Swedish method is given in section LFB.311 of (Svensk byggtjänst, 2010) and can be briefly described as follows (see also Annex B):

- The concrete is not unambiguously specified. 100 mm cubes of any of the concretes in EN 1776 can be used. In tests, both w/c-ratios of 0.45 and 0.70 have been used. (This comparison will focus on results obtained with a concrete with w/c = 0.45).

- 20 mm thick slices sawn at 28 days age are used (3 references and 3 for impregnation).

- The slices are pre-conditioned in 60±10 % RH and 21±2 °C for 14 days before impregnation.

- Impregnation is carried out on both sides of the slices.

- All slices are placed in 60±10 % RH and 21±2 °C for a further 14 days.

- The slices are stored in 15% NaCl-solution (in separate containers for references and impregnated specimens) during 56 days.

- After exposure a 50 mm cylinder is cored from the slice.

- 2.5 mm of the outermost surface layers is ground away and the chloride content per cement weight is determined on the rest of the cylinder.

- The requirement for approval is that the chloride level in the impregnated cylinders should not exceed 15% of the level in the references.

The main differences between the Swedish method and the new method are the different preconditioning and curing periods and the form of the exposed and analysed samples. No deduction of initial chloride level is carried out, and the evaluated value is expressed in another way.

6.1.1   *Influence of longer pre-conditioning time and shorter impregnation curing time on the filter effect*

In the pre-study 3 and 7 days of preconditioning were used and there was a difference in resulting filter effect as can be seen from Figure 51 which is around 6% when the impregnation curing period is 28 days. 7 days longer pre-conditioning leads to an even dryer surface, and better conditions for the penetration of the impregnation product. In Figure 59 the approximate RH-distribution in the surface layer (calculated with TorkaS (Fuktcentrum, 2015)) after 3, 7 and 14 days are shown. If the effect of

the impregnation is related to the available empty pore volume, the increase of the effect of the agent caused by a further 7 days of pre-conditioning in 65 % RH would be approximately the same as the increase recorded between 3 and 7 days, since about the same amount of pores will be emptied in the impregnation zone (approximately 5 mm) during the further 7 days as between 3 and 7 days.
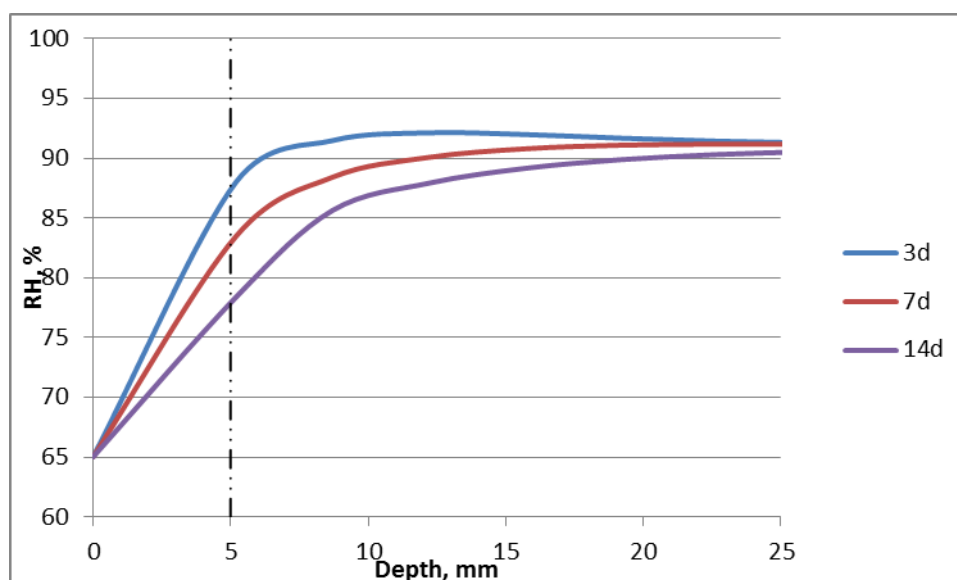
Figure 59: approximate RH-level after 3, 7 and 14 days of preconditioning in 65 % RH for a specimen with w/c=0,45. (Calculated with TorkaS **(Fuktcentrum, 2015)**)

The existing Swedish method uses 14 days in 65% RH for the impregnation curing period whereas the new method uses the same RH-level but 28 days. Both 14 and 28 days curing times were also used in the pre-study, and the filter effect decreases when the curing time becomes shorter. The average of the difference in filter effect (decrease from 28 days to 14 days) for the two products in the pre-study for all pre-conditioning types is a decrease with around 6 %-units.

Thus, the effects of the longer pre-conditioning period and the shorter impregnation curing period of the existing Swedish method on the filter effect compared to what is obtained with the new method balance out each other; an increase with about 6 %- units and a decrease with about 6 %-units respectively.

6.1.2 Influence of the exposed specimen form and the form of the analysed parts on the filter effect

In the existing Swedish method both surfaces of the 20 mm thick slice are exposed to the NaCl-solution. Chlorides will thus penetrate from both sides. Since chlorides in the references normally penetrates down to 15-20 mm, the chloride profiles from the two ends will overlap each other and the total chloride level in the 20 mm slice will be doubled compared to when the chlorides penetrate from one side only. The chlorides do not penetrate as deeply into the impregnated samples, but the chloride content will also in this case be doubled compared to one-sided penetration. Since both values in the relation forming the evaluated result of the effectiveness are doubled this does not affect the level.

Moreover, in the Swedish method the outermost 2.5 mm at both surfaces are not used in the determination of the effectiveness. In order to compare results obtained with the new method to results obtained with the Swedish method (w/c = 0.45) , the total chloride content of the part from the depth of 2.5 mm to the depth of 17.5 mm of the specimens should be used. A schematic picture of the chloride levels in a specimen used with the Swedish method is shown in Figure 60. These exact depths were neither used in the pre-study nor the round robin exercise, the closest depths being 2 mm depth and 20 mm. In 5.5.1.1 and 5.5.1.2 it was concluded that there was hardly any difference between filter effect calculated based on data from 20 mm thickness and 25 mm thickness, because the amount of external chlorides between 20 and 25 mm depth is almost 0. Using 20 mm as instead of 17.5 mm will therefore not have more than a very marginal effect on the filter effect. In the pre-study and the round-robin exercise 0-2 mm was used as the first analysed level. The chloride curve is very steep at this level, both in impregnated samples and references, and a difference of 0.5 mm could give a rather big difference in chloride content. However, the form of the curve in this part is unstable and influenced by how the specimens are treated after exposure (see 5.4.3.1.6 and 5.4.3.2.4). Since this part of the methodology is not very well treated neither in the Swedish method description nor in the method description used in the pre-study and the round-robin exercise, stability of the values obtained at this level is probably not very high and 2 and 2.5 mm can be regarded as giving approximately the same results.

Therefore, the most adequate value deduced from the results of the pre-study and the round-robin exercise for comparison with values obtained with the Swedish method is the filter effect calculated using the part from 2 to 20 mm of the specimens with no deduction of initial chloride content; here called $FE_{2\text{-}20N}$.
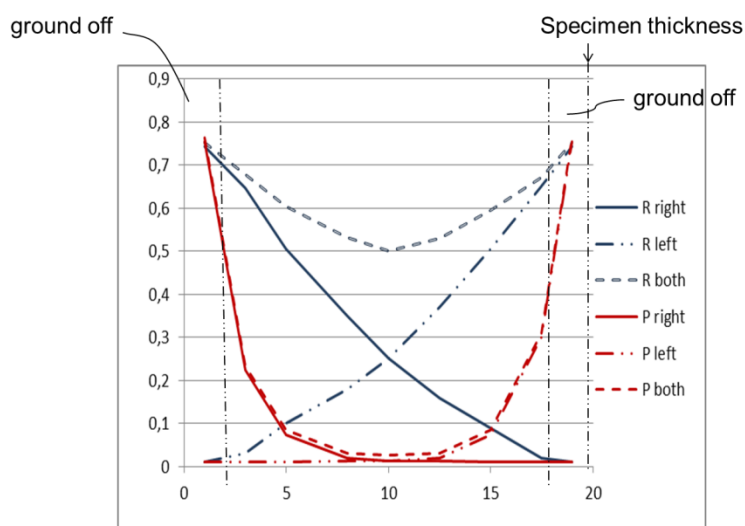
Figure 60: Schematic chloride distribution in specimens tested according to the Swedish method. P= in impregnated samples and R= in references.

The requirement of the Swedish method will then be equivalent to $FE_{2\text{-}20N}$ = 1-0.15 = 0.85. Results obtained with the Swedish method transformed to filter effects will be called FE_{Sw}. The filter effect, chosen for the new method, FE_{25} (FE calculated on 25 mm with deduction of initial chloride content) is in Figure 61 compared to the $FE_{2\text{-}20N}$ for the three products used in the round-robin exercise. As can be seen the $FE_{2\text{-}20N}$ values are substantially higher than the FE_{25} values calculated on exactly the same

data. The ratio between the values varies somewhat (between 0.83 and 0.76) for the three products. The higher the FE_{25} value the higher the ratio. For all the values the average ratio is about 0.79.
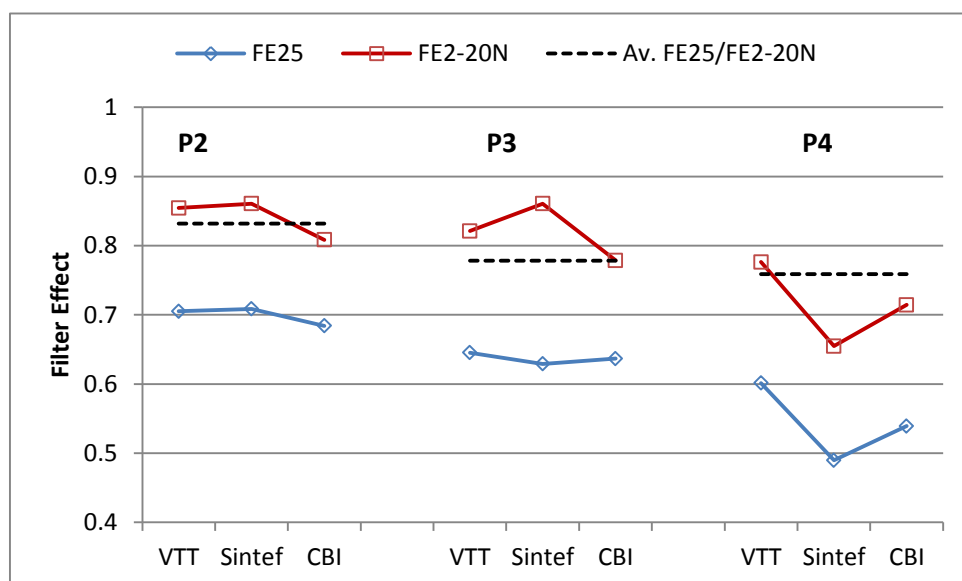
Figure 61: FE_{25}, $FE_{2\text{-}20N}$ and the average ratio between these two calculated based on data from the three products in the round robin exercise.

In order to maintain the same requirement level as with the existing method which could be valid for all type of products, the requirement on FE_{25} should be somewhere around 0.79*0.85=0.67.

Comparison between data obtained with the Swedish method (FE_{Sw}) and $FE_{2\text{-}20N}$ from the pre-study and the round robin exercise for products P2 and P3 are given in Figure 62.
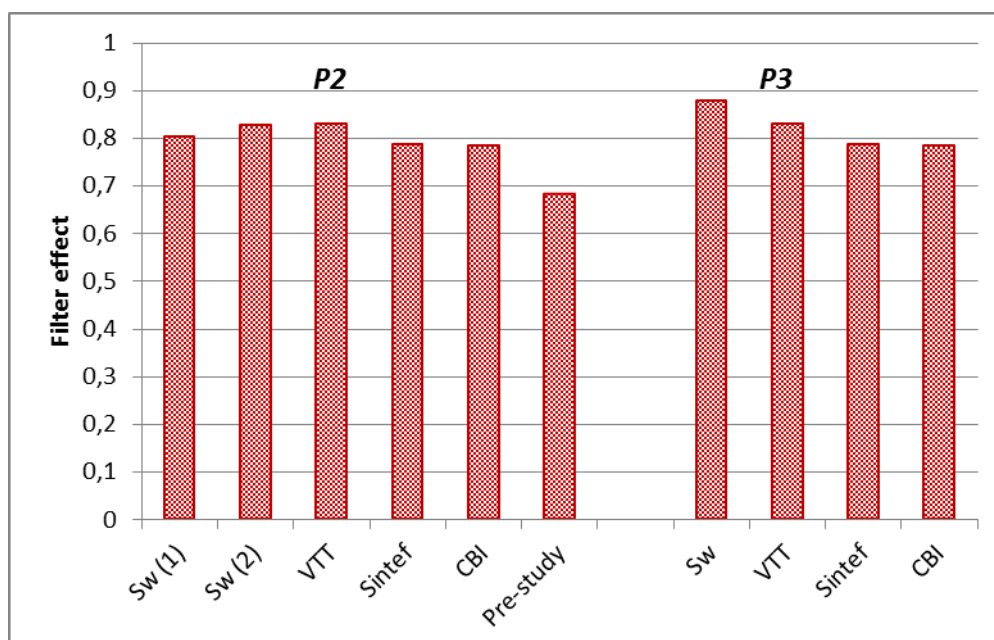
Figure 62: FE_{Sw} and $FE_{2\text{-}20N}$ from the round robin and the pre-study for two of the products.

There are some variations, but the overall level is about the same for both products. The value for P2 from the pre-study is substantially lower than the other values for this product, which is probably

due to a large redistribution of the chloride profile during storage, which also is evidenced in Figure 42 and Figure 56. In that case the ratio between FE_{20} and $FE_{2\text{-}20N}$ is also much higher, i.e. the value obtained when 2 mm are omitted is much closer to the value obtained over the whole thickness than in the other cases.

6.2 The existing Norwegian method

The existing Norwegian method is described in (SINTEF, 1998) and can be briefly described as follows (see also Annex B):

- The age of the concrete specimens (100 mm cubes) used for the tests is not specified (though ≥ 28 days).

- Cubes are sawn into halves, the sawn surface is used for the analysis

- The specimen surface is Ca(OH)$_2$-saturated after sawing and after epoxy-coating of the side and the bottom faces.

- The references are kept Ca(OH)$_2$-saturated until the exposure starts.

- Pre-conditioning of specimens to be impregnated is only one hour in 50±5 % RH and 20±2 °C.

- Impregnation curing time is two days in 50±5 % RH and 20±2 °C.

- Exposure to chlorides is carried out in a salt-spray chamber, 4 hours exposure to spray of 3% NaCl solution, 4 hours drying in windy laboratory air. Duration 42 days.

- The chlorides are analysed in layers down to a level where no initial chlorides are present

The main differences between the existing Norwegian method and the new method are the lime-water saturation, differing pre-conditioning and impregnation curing periods, and of course the totally different type of exposure to chlorides.

The requirement with the Norwegian method is expressed as the filter effect with the initial chloride content deduced. The thickness included is 15-20 mm depending on how far the external chlorides have penetrated. This filter effect is here called $FE_{No.}$

The main problem with the Norwegian method is that the deviation between results obtained both within one exposure chamber and at the two laboratories performing these tests is large. The reason mainly has to do with the reproducibility of the exposure conditions which are difficult to control.

6.2.1 Influence of lime-saturation, period and RH level for preconditioning period and impregnation curing.

In (Rodum, 2013) results with a modified Norwegian method are compared to results from the existing Norwegian. This modified method has more in common with the new method and the results from the modified method are therefore more easily compared to the results from the new method. In the modified method no lime-soaking takes place, pre-conditioning is extended to 3 days in 50% RH, the impregnation curing period is extended to 7 days in 50% RH and the exposure period

is prolonged to 56 days. The filter effect obtained for Product P2 with this modified method is about 8 %-units lower than the filter effect obtained with the ordinary Norwegian method.

As discussed in section 5.4.3.2.1 (Figure 49) lime-soaking leads to about 5 %-units higher filter effect irrespective of the length of the pre-conditioning period, but this effect is already included in the effect of the modification of the Norwegian method.

The modified Norwegian method will in the following be used as a new base for the quantification of influences of differences. In both the modified and the existing Norwegian method the reference samples are treated (from sawing to exposure) in a way that differs from the way the impregnated samples are treated; they are either kept in lime-water or kept sealed in plastic until exposure. This may influence the total chloride content of the references compared to if the references are exposed to the same climate (from sawing to exposure) as the impregnated samples. Better curing conditions may result in less pore volume, and higher RH at the surface may result in less empty pores so this influence is not easily quantified and will be ignored here.

The influence of prolonging the pre-conditioning period from 3 to 7 days was treated in 4.3.2.2 and it caused an increase in filter effect with between 5 and 10 %-units. However this was in the case where exposure was carried out by submersion, and with a higher RH during this period than in the Norwegian method.

The influence of prolonging the impregnation curing period (for both the impregnated and references) from 7 to 28 days is difficult to quantify, especially since the Norwegian comparative experiments were carried out with product P2, which has a surprisingly high filter effect at 7 days which decreases at 14 days and then increases again (see Figure 11). With the other product (P1) a steadily increasing filter effect is experienced. Judging from product P2 the effect decreases with about 10 %-units when going from 7 to 28 days and judging from product P1 it will increase with about 7 %-units. Whether the proposed reason for this phenomenon in 5.4.3.2.3 is also experienced when the specimens are exposed by means of salt-spraying is hard to say. In addition, it may be that the lower RH during the drying cycles of the exposure period in the Norwegian method compensates for the influence of the shorter impregnation curing period. Especially since the RH-level used during the impregnation curing period and probably also during the exposure (windy normal laboratory air) is lower than the one used during the impregnation curing period with the new method.

Thus, the only clear influence of the differing pre-conditioning and impregnation curing conditions is the one shown in the comparison between the existing Norwegian method and the modified Norwegian method.

6.2.2 *Influence of type of chloride exposure on the filter effect.*

The influence of the exposure method and the concentration of the salt-solution used has been studied in (Rodum, 2013) chapter 5. Permanent submersion in 16.5% NaCl-solution, cyclic submersion-drying in 16.5% solution (1 day submersion + 1 day drying), permanent submersion in 3% salt solution and cyclic salt-spraying with a 3% salt solution as in the Norwegian method (4 hours spraying + 4 hours drying). However, the salt-spraying method did not follow the existing Norwegian method but the modified one described in 6.2.1. All procedures lasted 56 days. Although the final chloride levels in the specimens varied substantially between the different exposure methods, the calculated filter effect (FE_{20}) for the three submersion methods were surprisingly constant (between

81.5 and 85.6%). The filter effect obtained with the specimens subjected to intermittent salt-spraying (modified Norwegian method) was somewhat lower (FE_{MoNo} = 72.4%). The difference in filter effect between permanent submersion and the salt-spraying used in the test is thus an increase with around 11%-units.

6.2.3   Comparison between test data for the same product

Since there are so many differences between the Norwegian method and the new method for which there are no data on which they can be quantified the only available method to relate the results from the two methods is to compare test results obtained with the two methods with the same products. Unfortunately there is only one product (P2) that has been tested with both methods, see Figure 63. As can be seen in this figure the results with the Norwegian method lies substantially above the results obtained with the new method. Using the averages the value with the new method is about 0.85 times the value obtained with the existing Norwegian method. The value obtained with the modified Norwegian method is only slightly above the value obtained with the new method. It is not ascertained that the same observations would be experienced with other product ascertained.

However, if this relation is used the requirement level with the Norwegian method FE_{No} = 0.75 will correspond to FE_{25} = 0.85*0.75 = 0.64.

This FE_{25} value is almost the same as the FE_{25} value corresponding to the requirement of the existing Swedish method (0.65), see 6.1.2.
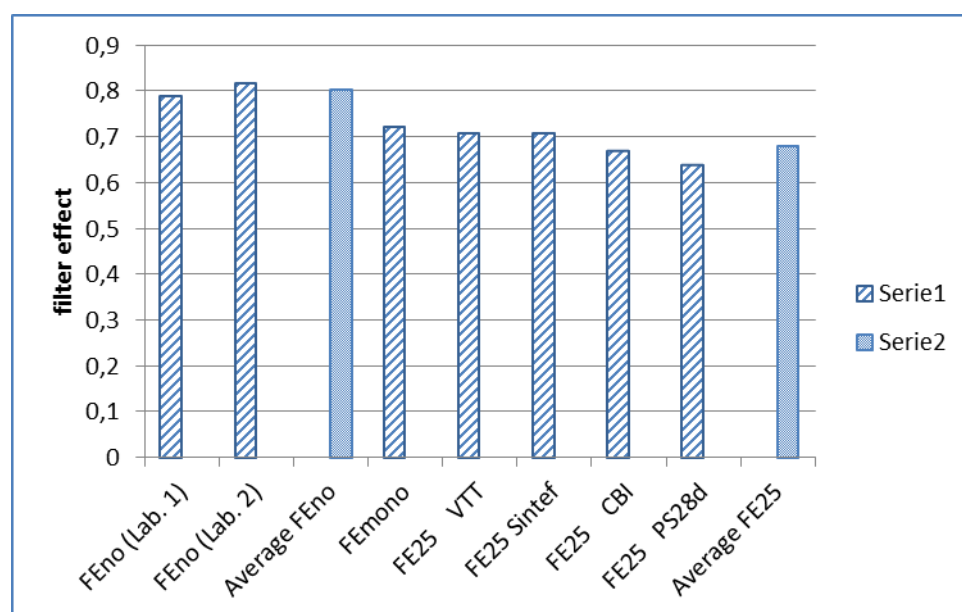
Figure 63: Filter effects obtained with the Norwegian method and the new method for product P2.

6.3   The existing Finnish method

The Finnish method resembles the Swedish method and the new method with permanent submersion in 15% NaCl-solution for 56 days. The current version is described in (VTT, 2009). The main differences between the Finnish method and the new method are (see also Annex B):

1. Entire concrete cubes are used.

2. The cubes are cured in 95% RH until 28 days age and then grit blasted.

3. The cubes are pre-conditioned in 65% RH for 14 days before impregnation.

4. Impregnation is carried out on all surfaces (grit- blasted form surfaces).

5. Impregnation curing time is between 7 and 28 days, depending on how long period the agent needs.

6. Chlorides are determined on two parts of the specimens; 0-20 mm from the surface and 20-50 mm from the surface. (Combined samples from all specimens).

The most important difference is the use of grit-blasted form surfaces and not sawn surfaces.

The Finnish method also includes a classification system including class ++++, class +++ class ++ and class +. For class +++ the amount of chlorides in the impregnated samples (0-20 mm depth) shall be ≤ 8% of the amount of chlorides in the references. The corresponding requirement for classes +++, ++ and + are ≤ 15%, ≤ 20% and ≤ 100% respectively.

6.3.1   Influence of pre-conditioning and impregnation curing period on the filter effect

As found with the Swedish method in 6.1.1, longer pre-conditioning may lead to an increased filter effect around 6 %-units. Here, we have a further complication in that the impregnation curing period is not fixed but may vary between 7 and 28 days in 65% RH. We here have the same kind of ambiguity as discussed regarding the Norwegian method when it comes to certain products which show a better effect at 7 days than at 14 and 28 and others that have a positive development of the performance from 7 to 28 days.

6.3.2   Influence of type of surface.

As discussed in 4.3.3.2.3 a form surface has a somewhat altered composition compared to a sawn surface due to the wall effect. Based on averages of data obtained on specimens with the w/c-ratio around 0.45 in the studies presented there, around 6 %-units higher value is obtained with form surfaces compared to sawn surfaces

Apart from the data from VTT showed in Figure 19, we have not had the possibility to see any other data obtained with the Finnish method.

Converted to FE_{25} obtained with the new method and taking into account the influences of longer curing time, type of surface and deduction of initial chlorides (see 5.5.1.2), the requirement levels of the Finnish method will correspond to approximately:

−   0.82 (=1-0.08-0.06-0.06+0.02)   for class ++++
−   0.75 (=1-0.015-0.06-0.06+0.02)   for class +++
−   0.70 (=1-0.20-0.06-0.06+0.02)   for class ++

7   COMPARISON WITH FIELD EXPERIENCE

There are some well-documented field studies of the chloride blocking effect of impregnation products which are the main basis for this comparison. In some cases the same products as in this project are used, but not always. All are though silane or siloxane based products. It is not easy to know whether the products are exactly the same or not for several reasons: The product may be sold under a specific name for a longer period even though there may be minor changes made to it during this period. Exactly the same product is often sold under different trade names by different companies. The trade name may also be changed over time, even though it is basically the same product.

All hydrophobic impregnation agents in the three field studies described below are characterised in Table E1 in Annex E together wit the products used within this project.

Selander (Selander, 2010) Paper VII, exposed impregnated samples (sawn surfaces) to an urban express road environment in the Eugenia tunnel in Stockholm for 5 years. The impregnation was a liquid with 100% silane (Product P5). A concrete with w/c = 0.45 was used. There is a clear effect of the impregnation on the chloride content. The chloride level at the surface is in the treated samples somewhat higher than in the untreated ones (approx. 0.85% and 0.6% per cement weight, respectively), but decreases steadily towards the interior and is 0 at 20 mm depth. The maximum chloride level in untreated samples (approx. 1.3% per cement weight) occurs at a depth of 5 mm. The value at the surface is less than half of the maximum. From 5 mm the level decreases steadily down to 0 at around 35 mm depth. This is a typical form for chloride profiles obtained in surfaces of real structures submitted to fluctuating air and moisture conditions, see for example (Boubitsas, et al., 2014) and (Costa & Appleton , 1999).

When expressed as a filter effect calculated down to 25 mm it becomes approximately 0.65. This encompasses the whole chloride profile in the treated samples but not in the untreated ones. If the whole profile is used (about 35 mm) the filter effect becomes around 0.68.

In the field study reported in (Malaga, 2015), two impregnation products applied on four different types of concrete were exposed to a real road environment at an exposure site close to the busy motorway, Riksväg 40, in Borås, Sweden and tested after 5 years exposure. The impregnation products were the same gel as used in this project (P2) and a liquid (product P6). Three of the concretes had a w/c = 0.45. Two ordinary concretes, one with air entrainment (5% air) and one without were included in this study. The third one was a self-compacting concrete without air. This concrete had a higher cement content (435 kg/m^3 compared to 375 kg/m^3 for the ordinary concretes), a higher amount of superplasticiser and it contained 110 kg/m^3 of limestone filler. A concrete with w/c = 0.70 was also included. As regards chloride intrusion into the ordinary 0.45-concretes, the two mixes behaved in a similar way. Both showed the same type of profiles as in (Selander, 2010) reported above and both products diminish the chloride content significantly. The filter effects (0-30 mm) are all within the range 0.57-0.69. When it comes to the self-compacting concrete the effect was much smaller, especially for the liquid P6 where the filter effect is just 0.15. Product P2 obtained a filter effect of 0.43 with this kind of concrete. The reason for this decrease in efficiency when applied on self-compacting concrete is not clear. When it comes to the concrete with w/c = 0.70 the reference samples were totally broken down after 5 years, so the filter effect could

not be determined. However, the fact that the impregnated samples could still be tested is a proof that the impregnation really has an effect on the durability, even though it is not the chloride blocking effect that stands for this improvement. On this type of concrete the liquid product P6 has been more effective; more than twice as much total chloride has penetrated the samples impregnated with P2 than those treated with product P6.

A field investigation of different surface treatments exposed for ten years in a marine environment (bottom side of a concrete quay slab and underlying beams with upper surface around 2 m above sea level repaired with either an ordinary concrete or a sprayed concrete with w/c = 0.39 and 5% silica fume) is presented in (Rodum & Lindland, 2012). On site investigation of the sprayed concrete however indicated a w/c-ratio around 0.5. Among these, three treatments consisted of treatment with different hydrophobic impregnations only. One of the products used was product P2 used in this project. Evaluation of the chloride blocking effect after ten years of the treatments were made by the following grading: None – Noticeable – Evident – Very Evident. On the bottom side of the quay slab the chloride blocking effect of all the used hydrophobic impregnations was evaluated as "Very evident". On the beams the effects were moderated and with P2 the registered effects were between "Noticeble" and "Very Evident", depending on location.

(Schueremans, et al., 2007) have investigated an harbour quay impregnated above and in the tidal zone with a solvent free silane (isobutyltriethoxysilane) at several occasions up to twelve years after treatment. Due to variations in the composition of the impregnated concrete at the different locations where samples have been taken a precise filter effect can not be determined. However, a filter effect after 12 years, estimated on the given chloride profiles in that paper, is somewhere between 0.5 and 0.6 for both in the tidal zone and above the tidal zone.

In Table 2, the comparison between results obtained by the new method and with existing test methods and the three well-documented field experiences are summarized. As can be seen impregnations have substantially decreased the chloride content in structures after 5-10 years field exposure for ordinary concretes with w/c-ratio around 0.45. The decrease, i.e. the filter effect measured on field structures, is in the vicinity or even higher than the filter effect obtained with the new method. Thus, the new method seems to reflect rather well the situation in real structures after at least 5 years exposure.

Table 2: Performance of products P1 to P6 in tests and field exposure.

Product	Finnish test (Class. see 5.3.2)	Norwegian test (Req. level ≥ 0.75)		Swedish test (Req. level ≤ 0.15)		Pre-study	Round robin exercise as FE_{25}			Field exposure		
		SVV lab.	SINTEF lab.	w/c= 0.45	w/c= 0,70	as FE_{20}	VTT	SINTEF	CBI	Eugenia tunnel (5 years)	RV 40 (5 years)	Quay Sursöya (10 years)
P1/P3	+++[1]			0.115	0.134	0.58	0.62	0.61	0.61			
P2	++++	0.82	0.79	0.195	0.055	0.64	0.69	0.69	0.66		Clear effect. FE_{25}=0.62-0.67	*On slab:* Very evident / *On beams*: Noticeable to very evident
P4	+++[1][2]			No test	0.107		0.60	0.49	0.54			
P5										Clear effect FE_{25} = 0.65		
P6				0.094	0.055						Clear effect FE_{25}=0.57-0.69	

[1] Based on tests with the Swedish method and w/c=0.70, classification questionable

[2] Tested as part of the project with the Finnish method, did not qualify for ++

Colour coding: Green (Passed the test / FE ≥ 0.60 / estimated to have a very evident effect)

Yellow (0.50 ≤ FE < 0.60 / estimated to have at least noticeable effect)

Red (Did not pass the test / FE < 0.50)

8   RECOMMENDATIONS FOR REQUIREMENT LEVELS

Based on the conclusions drawn from the comparison between the new method and the Swedish and Norwegian methods 6.1 and 6.2 an appropriate minimum FE_{25} level could be 0.65. However, there are many uncertain points and the conclusions are based on only few data in the comparison so this value can not be taken for absolute. When also the field data are considered a level around 0.60 seems to be more appropriate for a really well-performing hydrophobic impregnation. FE_{25} = 0.50 could perhaps be a suitable level for a product which still blocks the chloride but not to such a high degree as a really well-performing product.

A final decision on appropriate requirement levels are up to the owners of construction works or relevant authorities to decide, depending on their needs for their specific structures.

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ANNEX A – FINAL METHOD DESCRIPTION

A.1. Short description of the test method

Concrete specimens are prepared by sawing 100 mm cubes into two halves, three cubes per test series. The sawn surfaces are defined as exposure faces. Three halves are treated with the hydrophobic impregnation to be tested and the other three halves are kept as untreated references. The specimens are exposed submerged in 15% NaCl-solution for 56 days. After exposure, the chloride ingress is determined by profile grinding and the total amount of penetrated chlorides is calculated. The filtering effect (in %) of the hydrophobic impregnation is determined based on the ratio between the amount of penetrated chlorides into treated and non-treated concrete specimens.

A.2 Preparation of the specimens

The test specimens shall be prepared from cast 100 mm concrete cubes.

The concrete used shall be of the type MC (0.45) according to EN 1766. The cement used shall be of the type CEM I 42.5 or CEM I 52.5 and the maximum aggregate size shall be 10 mm.

For each hydrophobic impregnation to be tested, 3 treated specimens (half-cubes) shall be tested together with 3 corresponding reference specimens (half-cubes) from the same batch. Up to 3 hydrophobic impregnations can be tested simultaneously with the same reference series (non-treated). The standard procedures for mixing and casting shall be used. The cubes shall be demolded after 1 day and thereafter cured in water at (20±2)°C until the age of 28 days.

At the age of 28 days, the specimens shall be prepared by sawing the cubes into two halves (Figure A.1) with a water-cooled diamond saw. The sawn faces shall be carefully scrubbed with a soft brush under running tap water immediately after cutting in order to remove the cutting slurry from the as-cut surfaces. The exposure to tap water shall be as short as possible.
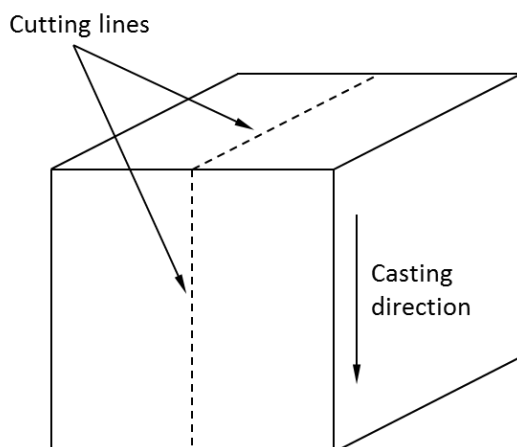
Figure A.1: Illustration of cutting direction.

When still wet, each specimen shall be judged visually with respect to a homogeneous distribution of aggregate and content of encapsulated air. Specimens that are abnormally inhomogeneous and/or have abnormally many air bubbles or air voids ≥ Ø5 mm shall be rejected.

After cutting, the specimens shall be left to dry, placed with the sawn surfaces facing down on a damp tight substrate (plastic sheet), at (20±2)°C and (65±5)% RH for 2-4 hours. After 2-4 hours drying, all surfaces, except the cut surface, shall be coated with 2 layers of epoxy resin. During coating, the surface to be impregnated shall be lifted up from the underlay/table, for instance by placing the specimens on hollow cylinders with the sawn surface facing down. The specimens are then left to harden overnight at (20±2)°C and (65±5)% RH with the sawn surface facing down.

The specimens to be treated with the hydrophobic impregnation shall be wrapped using a band (30 mm wide) of adhesive neoprene, protruding 5-10 mm from the cut surface (Figure A.2). This is to guarantee a homogeneous distribution of the hydrophobic impregnation and preventing it from running off the test surface. The inner edges shall then be sealed with moisture resistant contact glue (e.g. Casco Kontaktlim). The specimens shall be stored at (20±2)°C and (65±5)% RH for preconditioning. Care should be taken to avoid contamination of the exposure face during sealing.
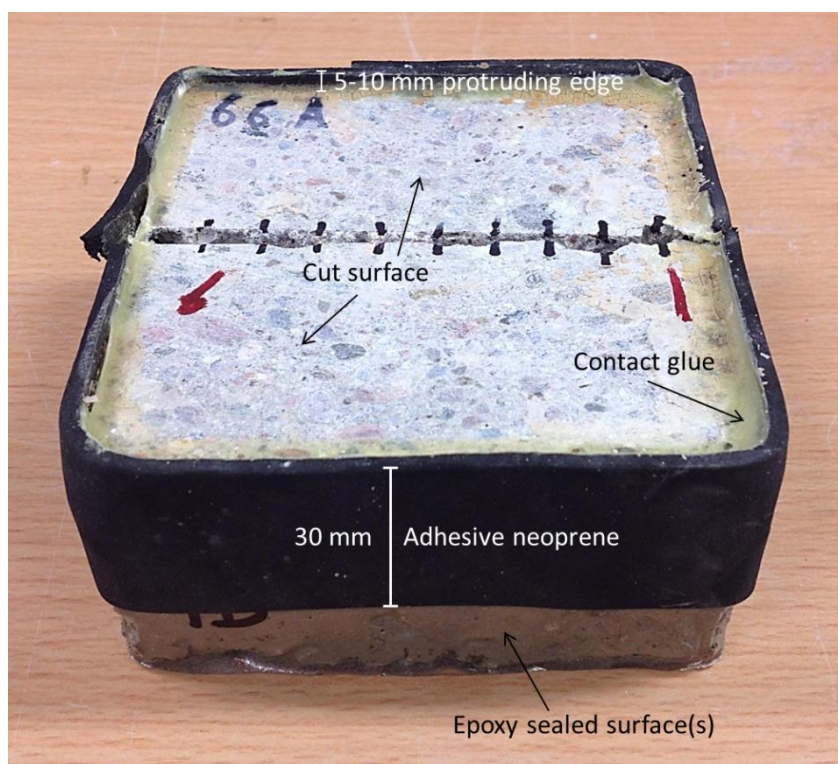
Figure A.2: Illustration of test specimen.

A.3 Preconditioning of the specimens

After preparation, the specimens shall be preconditioned at (20±2)°C and (65±5)% RH for 7 days, with the exposure face upwards.

A.4 Application of the hydrophobic impregnation

After 7 days at (20±2)°C and (65±5)% RH, the specimens to be treated shall be placed on top of a scale, in the horizontal position with the sawn surface facing up. The scale is counterbalanced and the hydrophobic impregnation is applied to the test surface, in the amount (in grams) recommended by the supplier. The product shall be applied and homogeneously distributed using a non-absorbent soft brush. The weight of the applied product shall be recorded in grams, to the second decimal.

A.5 Curing or the hydrophobic impregnation

After application of the hydrophobic impregnation, the treated specimens shall be conditioned at (20±2)°C and (65±5)% RH for 28 days.

A.6 Storage of reference specimens

After preconditioning at (20±2)°C and (65±5)% RH for 7 days, the reference specimens shall be continually stored at (20±2)°C and (65±5)% RH for further 28 days, separated from the treated specimens. Care should be taken that the reference specimens are not contaminated by airborne hydrophobic impregnations.

A.7 Exposure to chlorides

An aqueous NaCl solution is prepared to a concentration of 150±1 g NaCl per litre of solution. The NaCl concentration of the solution shall be checked before and after 14 and 28 days of exposure, and if necessary adjusted within 150±1 g NaCl per litre solution.

At the beginning of the exposure, the temperature of the solution shall be (20±2)°C. Exposure shall be carried out in a room in which the climate is controlled at (20±2)°C.

Reference and treated specimens shall be immersed in the saline solution, in separate containers. The containers should be completely filled with solution and closed with a lid. The volume of NaCl-solution should not be less than 12,5 ml/cm^2 exposed concrete surface. The exposure period shall last for 56 days and the solution in the containers shall be stirred once a week. The date and time of the start and termination of the exposure shall be recorded.

A.8 Removal from the chloride solution and storage until grinding

When the specimens are removed from the NaCl-solution, the excess solution on the surface shall be carefully wiped off with a cloth moistened in the NaCl-solution and squeezed in order to remove all excess liquid.

If it is not possible to grind the specimens directly thereafter, the specimens shall be tightly sealed in plastics and stored at 5±2 °C.

A.9 Grinding and drying of the concrete powder

The specimens shall be ground within 7 days from the moment they are taken up from the NaCl-solution. All parallel specimens (same hydrophobic impregnation/reference) shall be ground the same day. The grinding of the treated samples and their corresponding references shall be carried out within 48 hours.

Concrete powder shall be ground in layers with an area of at least 25 cm^2 down to a depth of 25 mm. Grinding should not be performed within 10 mm from the edge of the test surface.

It is recommended to use 5 mm thick layers. The layer between 20 and 25 mm of both impregnated specimens and references can normally be used to determine the initial chloride content of the concrete.

The date and time of grinding shall be recorded.

After grinding the ground samples shall be oven-dried in 105±5 °C.

The ground samples shall, before and after oven-drying, be kept protected from humidity and CO_2 until the chloride analysis is carried out.

A.10 Chloride analysis

The acid soluble chloride content in each layer shall be determined according to EN 14629 (CEN, 2007) (or another method with the same or better precision), as percent of the oven-dry concrete mass. The chloride content shall be given with three significant numbers.

For expression of chloride contents in other units, for example per volume, the oven-dry density of the concrete determined in accordance with EN 12390-7 should be used

A.11 Expression of results

For each layer, the total chloride ingress shall be calculated according to the following expression:

$$Cl_i = [Cl_{t,i} - Cl_0]$$

where:

Cl_i is the total chloride ingress in layer *i* (in % or g per oven-dry concrete mass[*])

$Cl_{t,i}$ is the total chloride content in layer *i* (in % or g per oven-dry concrete mass[*])

Cl_0 is the chloride background level (in % or g per oven-dry concrete mass[*]);

The total chloride ingress over the depth considered (normally 25 mm) in one specimen is then calculated as:

$$Cl_n = \left[\sum_0^d Cl_i \cdot x_i \right] \cdot \frac{1}{d}$$

where:

Cl_n is the total chloride ingress over the analysed depth (d) of specimen *n* (in % or g per oven-dry concrete mass[*])

d is the total analysed depth (in mm)

x_i is the thickness of layer *i* (in mm)

The efficiency of the treatment, expressed in terms of filter effect based on the chloride content down to a depth of 25 mm from the surface, FE_{25}, shall then be determined using the following equation:

$$FE_{25} = Filter\ Effect = [1 - \frac{\sum Cl_{a,treated}}{\sum Cl_{a,reference}}]$$

where:

$Cl_{a,treated}$ is the average chloride content (Cl_n, depth =25 mm) of the three parallell specimens treated with one hydrophobic impregnation product (in % or g per oven-dry concrete mass[*]);

$Cl_{a,refernce}$ is the average of the chloride contents (Cl_n, depth =25 mm) of the three parallell reference specimens (in % or g per oven-dry concrete mass[*]);

If other total depths are used for specific purposes, the number in FE_{25} shall be changed accordingly.

[*] *Other units may be used. The same unit shall be used throughout the calculations.*

ANNEX B – REVIEW OF EXISTING METHODS

Table B.1 : Review of existing methods to determine the chloride blocking effect.

	Swedish AMA 10	Norwegian SINTEF	Finnish Old SILKO	Finnish SILKO	Danish NT Build 489 (Paint systems)	EN 13369 (Adapted[1])
Castings (per series)	Minimum 2 cubes 100*100	3 cubes 100*100	6 cubes 100*100	6 cubes 100*100	Prisms 90*100*400 mm	7 cast or cut cylinders diameter ≥ 100 mm(same)
Concrete	According to EN 1766; w/c= 0,40; 0,45 or 0,70; CEM I 42/5 R	According to NS 3099, w/c= 0,45	CEM II A 275 kg/m^3, w/c 0.75, max grain size 8 mm and air content 6%	EN 1766 C(0,45)	MC 45 according to EN 1766, further detailed in procedure	The concrete repair material (Concrete according to EN 1766, w/c=0,45)
Concrete curing	Water cured till 28 days age	Minimum 28 days, water curing, can be much higher (no max. given)	95±5 % RH and 20±2°C up to 28 days	In 95 % RH for 28 days + grit blasting at the age of 28 d	Covered till 7 days (at 50±5 % RH and 23±2°C). Prisms shall be demolded at age 1-7 days	Water cured till 28 days age Vacuum saturation (In water 28 days, no vacuum sat)
Test specimens	Discs 100*100*20 mm from central parts of cubes	Cubes sawn in 2 parts (A treated, B ref) Sides sealed	The 6 cubes	The 6 cubes	4 prisms (90*50*200 mm) are sawn from each main prism at age 7 days	The 7 cast or cut cylinders (same)

	Swedish AMA 10	Norwegian SINTEF	Finnish Old SILKO	Finnish SILKO	Danish NT Build 489 (Paint systems)	EN 13369 (Adapted[1])
Pre-conditioning of test specimens.	65 % RH, 14 days	Ca(OH)$_2$-water saturated 1 hour in 50% RH before application	70±5 % RH and 20±2°C from 28 to approx. 56 days	65 % RH, 14 days	Sides sealed at age 8-9 days, exposure face protected against moisture exchange. Stored until age 14 days	Not relevant (60 % RH, 14 days)
Application of the impregnation product	All sides of discs	To sawn surface	To sandblasted surfaces, all sides of the cubes	All sides of the cubes	To sawn surface at age 15(-16) days.	Not relevant (All sides of the specimens?)
Curing of the impregnation	65 % RH, 14 days	50 % RH, 2 days (7 days for coatings)	According to manufacture's instruction, thereafter 14 days at 70±5 % RH and 20±2°C	65 % RH, 7 days (→28 days depending on how long period the hydrophobic impregnation agent needs)	At 50±5 % RH and 23±2°C until concrete age 21 days, thereafter: 14 days at 50±5 % RH and 23±2°C, 7 days at 50±5 % RH and 38±2°C, 7 days with cycling 4 h drying and 2 h immersion in water	Not relevant (60 % RH, 14 days)

	Swedish AMA 10	Norwegian SINTEF	Finnish Old SILKO	Finnish SILKO	Danish NT Build 489 (Paint systems)	EN 13369 (Adapted[1])
Age of test specimens at start of exposure	≈56 days	Minimum 30 days, can be much higher (no max)	70 days + x days (curing time specified by the manufacturer)	≈49 days (→70 days depending on how long curing period the hydrophobic impregnation agent needs)	49 days	28 + sat period (≈56 days)
Exposure to chloride	3 treated and 3 untreated discs are immersed in 15 % NaCl-solution	3 treated and 3 untreated specimens are exposed in a salt spray chamber 4 hours spraying with 3 % NaCl and 4 hours drying	3 treated and 3 untreated cubes are cyclic exposed: 7 days in 15 % NaCl-solution, 7 days in air of 70±5 % RH and 20±2°C	3 treated and 3 untreated cubes are immersed in 15 % NaCl-solution	3 treated and 3 untreated prisms are cyclic exposed: 2 h in 16,5 % NaCl-solution and 4 h air of 50±5 % RH and 23±2°C and wind 1.3±0.7 m/s	6 specimens immersed in 3% Na-solution, 1control in (same or in 15 % NaCl-solution)
Chloride ingress from	Sawed surface	Sawed surface	Form surface	Form surface	Sawn surface	Troweled surface(same)
Exposure time	56 days	42 days	56 days (4 cycles)	56 days	30 ± 1 weeks	28days, 3 months, 6 months 2 specimens taken out at the same occasion (same)

	Swedish AMA 10	Norwegian SINTEF	Finnish Old SILKO	Finnish SILKO	Danish NT Build 489 (Paint systems)	EN 13369 (Adapted[1])
Taking of samples for chloride measure-ment	50 mm bored core, 2,5 mm are grind off from the exposed surfaces The chloride content of the rest of the test specimen is determined	Concrete is ground from the exposed surface in layers of 5 mm (treated) or 10 mm (untreated), to a total depth of 15-20 mm. The total amount of chloride ingress is calculated, as g/m^2	Concrete powder is drilled from the vertical sides of the cubes (casting face on the top) at depths 0-20 mm and 20-50 mm (combined samples from all 3 cubes)	Depths 0-20 mm & 20-50 mm (combined samples from all 3 cubes)	Grinding of 8 layers with thickness 3-5 mm (untreated) and 4-6 layers á 1 mm (treated) Number of layers and layer thickness must be adjusted to the expected profiles	20 mm annulus grind off at the perimeter (same)
Type of chloride measured		Acid-soluble chloride content - by spectrophotometric method or potentiometric titration	Water-soluble chloride content (in addition calc. of weight changes in treated and untreated cubes)		Acid-soluble chloride content – by Volhard titration (or potentiometric titration)	In increments from troweled surface: 0-2 mm; 4-6 mm; 8-10 mm Background level measured on control specimen (same)
Chloride measure-ment method	SP 0433 or equivalent	SINTEF procedure (spectrophotometric) or NPRA procedure 14.643 (potentiometric)	Mohr method	EN 1744-1	NT Build 208	EN 14629 (or SP0433 or equivalent)

	Swedish AMA 10	Norwegian SINTEF	Finnish Old SILKO	Finnish SILKO	Danish NT Build 489 (Paint systems)	EN 13369 (Adapted[1])
Requirement	Cl content of treated specimens ≤ 15 % of Cl content of the untreated	Minimum filter effect of treatment =75 %		Grading criterions: + ≤ 100 % of untreated ++ ≤ 20 %; +++ ≤ 15 % ++++ ≤8 % for depth 0-20 mm Different grades for different applications (normally +++)	Calculation of diffusion coefficients and equivalent concrete thickness for the paint system. (No requirements given)	No requirement given (Need to be established)

ANNEX C – PRE-STUDY

C.1 Individual results of penetration depth

Impregnation depth, mm

	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{min}	d_{max}	Average	Std.dev.
P1d-S-28-1	3,3	4,3	3,8		3,0	4,4	3,2	4,6	4,2	3,0	4,6	3,9	0,6
P1d-S-28-2		4,6	5,4	4,8	5,4	5,3	5,3	5,2		4,6	5,4	5,1	0,3
P1d-S-28-3	3,1	4,5	4,2	5,7		5,0	5,2	5,2		3,1	5,7	4,7	0,9
												4,6	**0,6**

	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{min}	d_{max}	Average	Std.dev.
P2d-S-28-1	3,0	7,5	3,8	4,1	4,1	3,9	4,1	4,5	4,3	3,0	7,5	4,4	1,2
P2d-S-28-2	3,9	5,2	4,3	4,5	6,0	5,6	4,6	5,0	5,1	3,9	6,0	4,9	0,7
P2d-S-28-3	4,1	4,7	4,7	5,7	4,9	5,0	5,3	5,9	5,5	4,1	5,9	5,1	0,6
												4,8	**0,8**

P1-S-7-1	P1-S-7-2	P1-S-7-3	Average	St.dev.
4,75	5,75	5,1	5,2	0,5
P1-C3-7-1	**P1-C3-7-2**	**P1-C3-7-3**		
4,15	3	2	3,1	1,1
P2-S-7-1	**P2-S-7-2**	**P2-S-7-3**		
5,5	5,5	4,85	5,3	0,4
P2-C3-7-1	**P2-C3-7-2**	**P2-C3-7-3**		
3,5	4,35	2,5	3,5	0,9

C.2 Individual results of chloride ingress through sawn surfaces

		7 days								14 days								28 days							
		R-S-7-1	R-S-7-2	R-S-7-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-S-14-1	R-S-14-2	R-S-14-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-S-28-1	R-S-28-2	R-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
		Cl %	Cl %	Cl %				133	0	Cl %	Cl %	Cl %				129	0	Cl %	Cl %	Cl %				124	0
1	0-2	0,601	0,554	0,620	0,592	0,034	6	28		0,450	0,451	0,459	0,454	0,005	1	21		0,409	0,365	0,401	0,392	0,023	6	18	
3	2-4	0,689	0,638	0,648	0,658	0,027	4	31		0,445	0,488	0,500	0,478	0,029	6	22		0,470	0,487	0,500	0,486	0,015	3	23	
5	4-6	0,585	0,542	0,498	0,542	0,043	8	25		0,433	0,437	0,463	0,444	0,016	4	21		0,446	0,465	0,490	0,467	0,022	5	22	
8	6-10	0,366	0,341	0,315	0,340	0,026	8	32		0,350	0,345	0,375	0,356	0,016	4	33		0,354	0,350	0,380	0,361	0,016	4	34	
12,5	10-15	0,143	0,125	0,123	0,130	0,011	8	15		0,197	0,191	0,225	0,204	0,018	9	24		0,172	0,203	0,223	0,199	0,025	13	23	
17,5	15-20	0,022	0,022	0,021	0,022	0,000	1	2		0,052	0,057	0,088	0,066	0,020	30	7		0,022	0,046	0,048	0,039	0,014	36	4	
		R-C3-7-1	R-C3-7-2	R-C3-7-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-C3-14-1	R-C3-14-2	R-C3-14-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-C3-28-1	R-C3-28-2	R-C3-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
		Cl %	Cl %	Cl %				128	3	Cl %	Cl %	Cl %				126	2	Cl %	Cl %	Cl %				116	6
	0-2	0,633	0,698	0,664	0,665	0,032	5	31		0,595	0,563	0,601	0,587	0,020	3	28		0,402	0,420	0,442	0,421	0,020	5	20	
	2-4	0,654	0,661	0,664	0,660	0,005	1	31		0,601	0,584	0,668	0,618	0,044	7	29		0,440	0,472	0,475	0,462	0,019	4	22	
	4-6	0,523	0,507	0,506	0,512	0,010	2	24		0,473	0,494	0,520	0,496	0,024	5	23		0,398	0,424	0,431	0,418	0,017	4	20	
	6-10	0,326	0,323	0,308	0,319	0,009	3	30		0,322	0,327	0,339	0,329	0,009	3	31		0,320	0,355	0,323	0,333	0,020	6	31	
	10-15	0,088	0,106	0,081	0,092	0,013	14	10		0,107	0,118	0,144	0,123	0,019	16	14		0,177	0,187	0,173	0,179	0,007	4	21	
	15-20	0,023	0,017	0,024	0,021	0,004	18	2		0,018	0,018	0,024	0,020	0,004	17	2		0,034	0,044	0,034	0,037	0,006	16	4	
		R-C7-7-1	R-C7-7-2	R-C7-7-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-C7-14-1	R-C7-14-2	R-C7-14-3	Avg	Std	COV (%)	g/m²	Filter (%)	R-C7-28-1	R-C7-28-2	R-C7-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
		Cl %	Cl %	Cl %				131	1	Cl %	Cl %	Cl %				122	5	Cl %	Cl %	Cl %				117	5
	0-2	0,660	0,585	0,682	0,643	0,051	8	30		0,450	0,417	0,437	0,435	0,017	4	20		0,393	0,388	0,361	0,381	0,017	4	18	
	2-4	0,633	0,643	0,707	0,661	0,040	6	31		0,464	0,502	0,454	0,473	0,026	5	22		0,458	0,442	0,434	0,445	0,012	3	21	
	4-6	0,547	0,560	0,529	0,545	0,016	3	26		0,419	0,436	0,401	0,419	0,017	4	20		0,413	0,388	0,420	0,407	0,017	4	19	
	6-10	0,342	0,345	0,322	0,336	0,013	4	31		0,337	0,369	0,337	0,348	0,019	5	33		0,334	0,318	0,326	0,326	0,008	3	30	
	10-15	0,086	0,108	0,100	0,098	0,011	11	11		0,178	0,211	0,201	0,197	0,017	9	23		0,200	0,192	0,201	0,198	0,005	2	23	
	15-20	0,020	0,017	0,017	0,018	0,001	7	2		0,030	0,046	0,053	0,043	0,012	27	5		0,065	0,058	0,051	0,058	0,007	12	6	

(Row group label: **R** spanning all three blocks on the left)

	P1-S-7-1 Cl %	P1-S-7-2 Cl %	P1-S-7-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-S-14-1 Cl %	P1-S 14-2 Cl %	P1-S-14-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-S-28-1 Cl %	P1-S-28-2 Cl %	P1-S-28-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)
							63	53							59	54							52	58
0-2	0,489	0,441	0,495	0,475	0,030	6	22		0,417	0,423	0,408	0,416	0,008	2	19		0,233	0,280	0,247	0,253	0,024	9	12	
2-4	0,361	0,347	0,391	0,366	0,022	6	17		0,334	0,324	0,315	0,324	0,010	3	15		0,249	0,271	0,252	0,257	0,012	5	12	
4-6	0,235	0,242	0,253	0,243	0,009	4	11		0,234	0,249	0,208	0,230	0,021	9	11		0,225	0,224	0,214	0,221	0,006	3	10	
6-10	0,103	0,109	0,103	0,105	0,003	3	9		0,107	0,109	0,122	0,113	0,008	7	10		0,142	0,142	0,143	0,142	0,001	0	13	
10-15	0,023	0,019	0,021	0,021	0,002	10	2		0,020	0,025	0,030	0,025	0,005	20	2		0,039	0,036	0,033	0,036	0,003	7	4	
15-20	0,006	0,014	0,016	0,012	0,005	45	1		0,015	0,015	0,018	0,016	0,002	9	1		0,016	0,017	0,019	0,017	0,002	10	1	

	P1-C3-7-1 Cl %	P1-C3-7-2 Cl %	P1-C3-7-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-C3-14-1 Cl %	P1-C3-14-2 Cl %	P1-C3-14-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-C3-28-1 Cl %	P1-C3-28-2 Cl %	P1-C3-28-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)
							64	50							64	49							49	58
0-2	0,538	0,534	0,488	0,520	0,027	5	24		0,458	0,488	0,481	0,475	0,016	3	22		0,271	0,232	0,237	0,247	0,021	9	11	
2-4	0,381	0,358	0,383	0,374	0,014	4	17		0,335	0,374	0,350	0,353	0,019	6	17		0,248	0,225	0,229	0,234	0,012	5	11	
4-6	0,245	0,234	0,230	0,236	0,008	3	11		0,246	0,247	0,256	0,250	0,006	2	12		0,216	0,196	0,204	0,205	0,010	5	10	
6-10	0,100	0,081	0,096	0,092	0,010	11	8		0,102	0,122	0,110	0,111	0,010	9	10		0,135	0,132	0,146	0,138	0,007	5	13	
10-15	0,018	0,018	0,015	0,017	0,002	11	1		0,020	0,027	0,025	0,024	0,003	14	2		0,031	0,030	0,040	0,034	0,006	17	3	
15-20	0,023	0,014	0,023	0,020	0,005	26	2		0,015	0,018	0,016	0,016	0,001	7	1		0,012	0,014	0,007	0,011	0,004	32	1	

	P1-C7-7-1 Cl %	P1-C7-7-2 Cl %	P1-C7-7-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-C7-14-1 Cl %	P1-C7- 14-2 Cl %	P1-C7-14-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)	P1-C7-28-1 Cl %	P1-C7-28-2 Cl %	P1-C7-28-3 Cl %	Avg	Std	COV (%)	g/m²	Filter (%)
							56	57							48	60							39	66
0-2	0,475	0,463	0,460	0,466	0,008	2	22		0,233	0,219	0,243	0,232	0,012	5	11		0,174	0,200	0,216	0,197	0,021	11	9	
2-4	0,348	0,316	0,338	0,334	0,017	5	16		0,232	0,228	0,220	0,227	0,006	3	11		0,194	0,186	0,222	0,201	0,019	9	9	
4-6	0,208	0,212	0,197	0,206	0,008	4	10		0,178	0,202	0,181	0,187	0,013	7	9		0,160	0,151	0,188	0,166	0,020	12	8	
6-10	0,074	0,093	0,064	0,077	0,015	19	7		0,317	0,105	0,091	0,171	0,127	74	16		0,107	0,087	0,115	0,103	0,014	14	9	
10-15	0,006	0,017	0,017	0,013	0,006	45	1		0,022	0,029	0,022	0,025	0,004	17	2		0,034	0,028	0,023	0,028	0,005	19	3	
15-20	0,016	0,014	0,015	0,015	0,001	9	1		0,016	-0,008	0,016	0,008	0,014	170	0		0,016	0,020	0,016	0,017	0,002	13	1	

P2-S

	7 days								14 days								28 days							
	P2-S-7-1	P2-S-7-2	P2-S 7-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-S-14-1	P2-S 14-2	P2-S 14-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-S-28-1	P2-S-28-2	P2-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
	Cl %	Cl %	Cl %				34	75	Cl %	Cl %	Cl %				49	62	Cl %	Cl %	Cl %				44	64
0-2	0,287	0,296	0,286	0,290	0,005	2	13		0,292	0,257	0,296	0,282	0,022	8	13		0,262	0,254	0,293	0,270	0,021	8	13	
2-4	0,192	0,200	0,183	0,192	0,008	4	9		0,240	0,226	0,261	0,242	0,018	7	11		0,222	0,251	0,243	0,239	0,014	6	11	
4-6	0,108	0,106	0,120	0,111	0,008	7	5		0,202	0,178	0,211	0,197	0,017	8	9		0,173	0,194	0,190	0,186	0,011	6	9	
6-10	0,039	0,035	0,036	0,037	0,002	5	3		0,132	0,100	0,135	0,122	0,019	16	11		0,077	0,101	0,104	0,094	0,015	16	8	
10-15	0,021	0,020	0,015	0,019	0,003	18	2		0,039	0,028	0,038	0,035	0,006	18	4		0,024	0,022	0,031	0,026	0,005	19	2	
15-20	0,021	0,017	0,016	0,018	0,003	15	2		0,013	0,017	0,018	0,016	0,003	19	1		0,016	0,016	0,017	0,016	0,001	3	1	

P2-C3

	7 days								14 days								28 days							
	P2-C3-7-1	P2-C3-7-2	P2-C3-7-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-C3-14-1	P2-C3-14-2	P2-C3-14-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-C3-28-1	P2-C3-28-2	P2-C3-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
	Cl %	Cl %	Cl %				33	74	Cl %	Cl %	Cl %				56	56	Cl %	Cl %	Cl %				40	65
0-2	0,315	0,289	0,397	0,334	0,057	17	16		0,509	0,531	0,463	0,501	0,035	7	24		0,258	0,231	0,242	0,244	0,013	5	11	
2-4	0,185	0,178	0,202	0,188	0,012	6	9		0,315	0,362	0,259	0,312	0,051	16	15		0,205	0,196	0,192	0,198	0,006	3	9	
4-6	0,101	0,096	0,035	0,078	0,037	47	3		0,176	0,216	0,163	0,185	0,028	15	9		0,192	0,177	0,110	0,160	0,044	28	7	
6-10	0,026	0,041	0,019	0,029	0,011	40	2		0,048	0,091	0,068	0,069	0,022	31	6		0,126	0,108	0,080	0,105	0,023	22	9	
10-15	0,022	0,011	0,020	0,018	0,006	31	1		0,015	0,037	0,019	0,024	0,012	51	2		0,020	0,027	0,018	0,022	0,004	21	2	
15-20	0,005	0,023	0,023	0,017	0,010	61	1		0,013	0,017	0,017	0,016	0,002	16	1		0,014	0,019	0,015	0,016	0,003	17	1	

P2-C7

	7 days								14 days								28 days							
	P2-C7-7-1	P2-C7-7-2	P2-C7-7-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-C7-14-1	P2-C7-14-2	P2-C7-14-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-C7-28-1	P2-C7-28-2	P2-C7-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
	Cl %	Cl %	Cl %				28	79	Cl %	Cl %	Cl %				41	67	Cl %	Cl %	Cl %				37	69
0-2	0,289	0,218	0,316	0,275	0,051	18	13		0,266	0,283	0,294	0,281	0,014	5	13		0,239	0,221	0,227	0,229	0,009	4	11	
2-4	0,169	0,119	0,182	0,156	0,033	21	7		0,197	0,223	0,223	0,215	0,015	7	10		0,205	0,189	0,183	0,193	0,011	6	9	
4-6	0,087	0,061	0,074	0,074	0,013	17	3		0,154	0,170	0,166	0,163	0,008	5	8		0,158	0,154	0,146	0,153	0,006	4	7	
6-10	0,032	0,019	0,020	0,024	0,007	30	2		0,077	0,091	0,083	0,083	0,007	8	7		0,088	0,082	0,082	0,084	0,004	4	7	
10-15	0,020	0,016	0,017	0,018	0,002	12	2		0,019	0,021	0,019	0,020	0,001	4	2		0,020	0,013	0,021	0,018	0,004	24	2	
15-20	0,017	0,014	0,017	0,016	0,002	13	1		0,015	0,014	0,014	0,014	0,001	6	1		0,017	0,012	0,015	0,014	0,003	18	1	

C.3 Individual results of chloride ingress through form surfaces

		R-S-28-1	R-S-28-2	R-S-28-3	Avg	Std	COV (%)	g/m²	P1-S-28-1	P1-S-28-2	P1-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2-S-28-1	P2-S-28-2	P2-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
		Cl %	Cl %	Cl %				124	Cl %	Cl %	Cl %				52	58	Cl %	Cl %	Cl %				44	64
1	0-2	0,409	0,365	0,401	0,392	0,023	6	18	0,233	0,280	0,247	0,253	0,024	9	12		0,262	0,254	0,293	0,270	0,021	8	13	
3	2-4	0,470	0,487	0,500	0,486	0,015	3	23	0,249	0,271	0,252	0,257	0,012	5	12		0,222	0,251	0,243	0,239	0,014	6	11	
5	4-6	0,446	0,465	0,490	0,467	0,022	5	22	0,225	0,224	0,214	0,221	0,006	3	10		0,173	0,194	0,190	0,186	0,011	6	9	
8	6-10	0,354	0,350	0,380	0,361	0,016	4	34	0,142	0,142	0,143	0,142	0,001	0	13		0,077	0,101	0,104	0,094	0,015	16	8	
12,5	10-15	0,172	0,203	0,223	0,199	0,025	13	23	0,039	0,036	0,033	0,036	0,003	7	4		0,024	0,022	0,031	0,026	0,005	19	2	
17,5	15-20	0,022	0,046	0,048	0,039	0,014	36	4	0,016	0,017	0,019	0,017	0,002	10	1		0,016	0,016	0,017	0,016	0,001	3	1	

	RF-S-28-1	RF-S-28-2	RF-S-28-3	Avg	Std	COV (%)	g/m²	P1F-S-28-1	P1F-S-28-2	P1F-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)	P2F-S-28-1	P2F-S-28-2	P2F-S-28-3	Avg	Std	COV (%)	g/m²	Filter (%)
	Cl %	Cl %	Cl %				119	Cl %	Cl %	Cl %				41	66	Cl %	Cl %	Cl %				38	68
0-2	0,558	0,507	0,471	0,512	0,044	9	24	0,332	0,315	0,296	0,314	0,018	6	15		0,322	0,492	0,372	0,395	0,087	22	18	
2-4	0,413	0,436	0,428	0,426	0,012	3	20	0,197	0,178	0,170	0,182	0,014	8	8		0,158	0,169	0,154	0,160	0,008	5	7	
4-6	0,361	0,397	0,394	0,384	0,020	5	18	0,165	0,139	0,138	0,147	0,015	10	7		0,118	0,130	0,110	0,119	0,010	9	5	
6-10	0,313	0,326	0,320	0,320	0,007	2	30	0,111	0,062	0,062	0,078	0,028	36	7		0,045	0,068	0,048	0,053	0,012	23	5	
10-15	0,196	0,188	0,184	0,189	0,006	3	22	0,043	0,017	0,019	0,026	0,015	55	3		0,016	0,013	0,010	0,013	0,003	22	1	
15-20	0,045	0,060	0,042	0,049	0,009	19	5	0,015	0,015	0,017	0,016	0,001	8	1		0,015	0,013	0,012	0,013	0,002	12	1	

ANNEX D – TEST METHOD USED IN THE ROUND ROBIN EXERCISE

D.1 Description of the test method for chloride blocking effect for the round robin exercise

D.1.1   Short description of the test method for the chloride blocking effect

Concrete specimens are prepared by sawing 100 mm cubes into two halves, three cubes per test series. The sawn surfaces are defined as exposure faces. Three halves are treated with the product to be tested and the other three halves are kept as untreated references. The specimens are exposed submerged in 15% NaCl-solution for 56 days. After exposure, the chloride ingress is determined by profile grinding and the total amount of penetrated chlorides is calculated. The filtering effect of the hydrophobic agent is determined based on the ratio between the amount of penetrated chlorides into treated and non-treated concrete specimens, in %.

D.1.2   Preparation of the specimens

The specimens are prepared from 100 mm concrete cubes, three cubes per test series. The concrete used shall be of the type MC (0.45) according to EN 1766. The cement used shall be of the type CEM I 52,5) and the maximum aggregate size shall be 10 mm.

For each hydrophobic agent to be tested, 5 treated specimens (half-cubes) shall be tested together with 5 corresponding reference specimens (half-cubes) from the same batch. If more than one product is to be tested simultaneously, the same reference series (non-treated) can be used for all products as long as all specimens come from the same batch.

standard procedures for mixing and casting shall be used. The specimens shall be demolded after 1 day and thereafter cured in water at (20±2)°C.

At the age of 28 days, the specimens shall be prepared by sawing the cubes into two halves (Figure D.1) with a water-cooled diamond saw. The sawn faces shall be carefully scrubbed with a soft brush under running tap water immediately after cutting in order to remove the cutting slurry from the as-sawn surfaces.
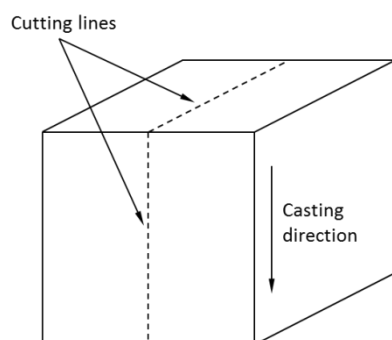
Figure D.1: Illustration of cutting direction.

When still wet, each specimen shall be judged visually with respect to a homogeneous distribution of aggregate and content of encapsulated air. Specimens that are abnormally inhomogeneous and/or have abnormally many air bubbles or air voids ≥ Ø5 mm shall be rejected.

After sawing, the specimens shall be left to dry, placed with the sawn surfaces facing down on a damp tight substrate (plastic sheet), at (20±2)°C and (60±10)% RH for 2-4 hours. After 2-4 hours drying, all surfaces, except the sawn surface, shall be coated with 2 layers of epoxy resin and left to harden overnight in (20±2)°C and (60±10)% RH with the sawn surface facing down.

The specimens to be treated with hydrophobic agent shall be wrapped using a band (30 mm wide) of adhesive neoprene, protruding 5-10 mm from the cut surface (see Figure D.2). This is to guarantee a homogeneous distribution of the hydrophobic agent and preventing it from running off the test surface. The inner edges shall then be sealed with moisture resistant contact glue (e.g. Casco Kontaktlim). Care should be taken during sealing in order to avoid contamination of the exposure face.
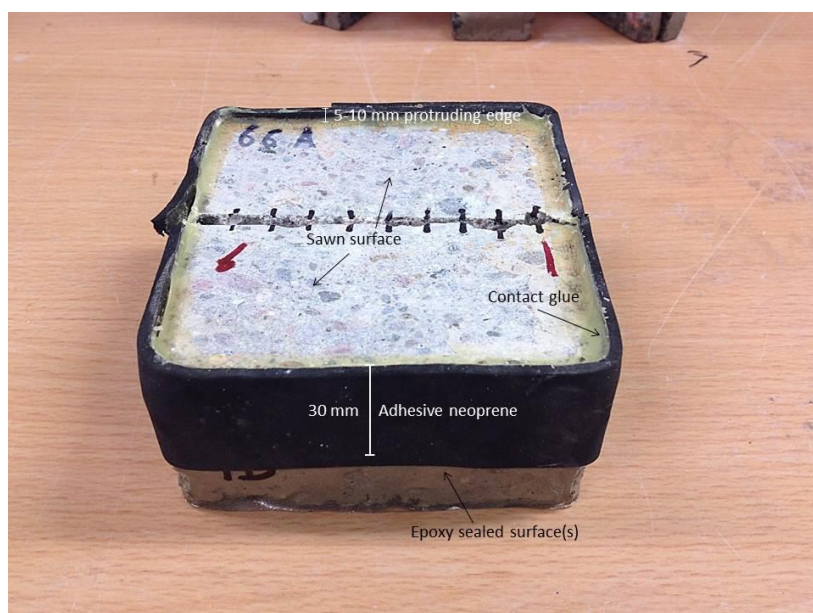
Figure D.2: Illustration of test specimen.

D.1.3 Preconditioning of the test specimens

After preparation, the specimens shall be preconditioned at (20±2) °C and (60±10) % RH for 7 days, with the exposure face upwards.

D.1.4 Application of the hydrophobic agent

After 7 days at (20±2)°C and (60±10)% RH, the specimens to be treated shall be placed on top of a scale, in the horizontal position with the sawn surface facing up. The scale is counterbalanced and the hydrophobic agent is applied to the test surface, in the amount (in grams) recommended by the supplier. The product shall be applied and homogeneously distributed using a non-absorbent soft brush. The weight of the applied product shall be recorded in grams, to the second decimal.

D.1.5 Curing of the hydrophobic agent and conditioning of reference specimens

After application of the hydrophobic agent, the treated specimens shall be conditioned at (20±2)°C and (60±10)% RH for 28 days.

After preconditioning at (20±2)°C and (60±10)% RH for 7 days, the reference specimens shall be continually stored at (20±2)°C and (60±10)% RH for further 28 days, separated from the treated specimens. Care should be taken that the reference specimens are not contaminated by airborne hydrophobic agents.

D.1.6 Exposure to chlorides

An aqueous NaCl solution is prepared to a concentration of 150±1 g NaCl per litre of solution. The NaCl concentration of the solution shall be checked before and after 14 and 28 days of exposure, and if necessary adjusted within 150±1 g NaCl per litre solution.

At the beginning of the exposure, the temperature of the solution shall be (20±2)°C. Exposure shall be carried in climate controlled room at (20±2)°C.

Reference and treated specimens shall be immersed in the saline solution, in separate containers. The exposure period shall last for 56 days and the solution in the containers shall be stirred once a week. The date and time of exposure start and exposure stop shall be recorded.

D.1.7 Sampling and chloride analysis

Sampling shall be carried out immediately after the exposure is terminated. The specimens shall be removed from the solution, surface dried with absorbing paper and profile ground. If immediate grinding is not possible, the specimens shall be sealed in plastic bags and stored at (5±2)°C until profile grinding is executed. Profile grinding shall be carried within 7 days after the exposure is terminated.

Concrete powder shall be ground in the following layers: 0-2; 2-4; 4-6; 6-10; 10-15; 15-20 and 20-25 mm. The date and time of grinding shall be recorded.

The acid soluble chloride content in each layer shall be determined by potentiometric titration according to AASHTO-T260 (or another method with the same or better precision), as percent of the dry sample mass. The chloride content shall be given to the third decimal.

D.1.8 Determination of the CaO content

On one specimen per each product and one reference specimen the calcium content in the sampled powder shall be determined according to the following procedures or another method with the same of better precision. The calcium content shall be given to the second decimal in percent of the dry sample mass.

Determination of Calcium Content after AASHTO T 260, Procedure A

Calcium selective electrode and calomel or Ag/AgCl reference electrode are used. The volume increment of titrant (0.1 N EDTA) is set to 0.1 ml.

After titration for chloride ions, 5 ml of 1:2 diluted triethanolamine is added to the sample solution and the pH-value of the solution is adjusted to >12 using NaOH. The calcium content in a sample can be calculated by the following equation:

$$CaO, \% = \frac{5.608VN}{W} \cdot f$$

Where: V = volume of titrant at endpoint, ml;

               N = normality of titrant, N = 0.1 N;

               f = titration factor, f = 1.03~1.05 (based on Tang's experience);

               W = weight of the sample, g.

D.1.9   Expression of results

The total chloride ingress, in g/m^2, shall be calculated per dry sample weight with deduction of the background. The efficiency of the treatment, expressed in terms of filter effect can then be determined using following equation:

$$Filter\ Effect\ (\%) = [1 - \frac{Cl(g/m^2)_{treated}}{Cl(g/m^2)_{reference}}] \times 100$$

D.2 Individual results compressive strength and dry density

	Compressive strength					Dry density				
	1	2	3	Average	Std-dev.	1	2	3	Average	Std-dev.
VTT*	65,9	66,6	66,3	66,3	0,4	2380	2380	2370	2377	6
Sintef**				67,7					2256	
CBI**	62,4	62,62	65,01	63,3	1,4	2241	2228	2220	2230	11
* Measured surface dry density										
** Measured surface dry density										

D.3 Individual results of penetration depth

D.3.2   Testing according to EN 1504-2 on specimens with w/c=0,45

D.3.2.1   Impregnation depths obtained at VTT

	Penetration depth, mm								
	Product P2			Product P3			Product P4		
Specimen	P2-1	P2-2	P2-3	P3-1	P3-2	P3-3	P4-1	P4-2	P4-3
Average	9,88	8,18	11,44	7,88	8,15	6,74	4,79	3,89	4,63
Mean value	9,83			7,59			4,44		

D.3.2.2   Impregnation depths obtained at SINTEF

Product	Product P2			Product P3			Product p4		
Specimen	1	2	3	1	2	3	1	2	3
Penetration depth, mm Min/max	3,5/ 5,0	4,0/4, 5	4,0/4, 5	3,5/4, 5	3,5/4, 5	3,5/ 4,0	2,5/ 3,5	2,0/ 3,0	2,0/ 3,5
Average	4,3	4,1	4,7	4,0	4,0	3,7	2,9	2,6	2,7
Mean value	4,4			3,9			2,7		
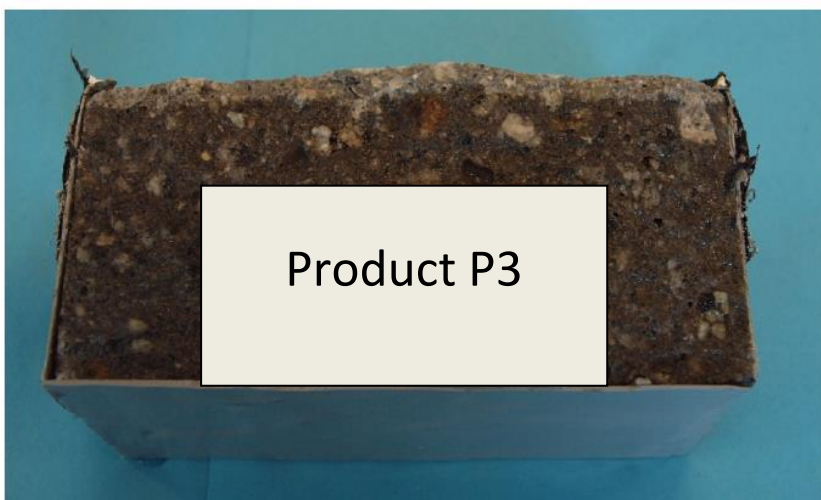
Figure D.3: Photo of one penetration depth specimen per product from SINTEF.

D.3.2.3   Impregnation depths obtained at CBI

Impregnation Depth (mm)

Product P2	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{min}	d_{max}	Avg	Stdev
P2d-1	6,3	7,9	6,9	9,3	9,4	8,4	6,5	5,9	4,7	4,7	9,4	7,3	1,6
P2d-2	8,7	6,7	5,6	6,8	4,0	10,0	9,0	6,9	5,9	4,0	10,0	7,1	1,9
P2d-3	6,0	7,6	10,6	9,3	11,9	7,3	8,5	4,4	5,0	4,4	11,9	7,8	2,5
												7,4	2,0
Of all												7,4	2,0

Impregnation Depth (mm)

Product P3	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{min}	d_{max}	Avg	Stdev
P3d-1	3,9	3,4	6,1	5,7	15,4	13,8	12,7	12,5	12,0	3,4	15,4	9,5	4,7
P3d-2	7,8	7,3	9,3	6,8	6,1	9,0	7,8	5,1	4,5	4,5	9,3	7,1	1,6
P3d-3	5,0	7,6	6,5	6,5	10,3	7,9	8,2	8,7	7,5	5,0	10,3	7,6	1,5
												8,1	2,6
Of all												8,1	3,1

Impregnation Depth (mm)

Product P4	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{min}	d_{max}	Avg	Stdev
P4-1	3,9	6,2	5,1	4,1	3,6	8,6	7,8	7,8	6,6	3,6	8,6	6,0	1,9
P4-2	4,6	5,5	6,8	6,1	6,0	4,1	4,1	5,0	4,3	4,1	6,8	5,2	1,0
P4-3	9,3	8,2	3,5	5,8	8,0	8,1	5,9	2,9	3,8	2,9	9,3	6,2	2,4
												5,8	1,7
Of all												5,8	1,8

Figure D.4: Photo of one penetration depth specimen per product from CBI.

D.3.3 Testing with the drop method

D.3.1 VTT results

	P2-4	P3-4	P4-4
0-2 mm	yes	yes	yes
2-4 mm	yes	yes	yes
4-5 mm	yes	yes	yes
5-6 mm	yes	no	yes
6-7 mm	no	no	yes
7-8 mm	no	no	no

D.3.3.2   SINTEF results

Depth, mm	Evaluation (6 = totally water repellent, 0 = totally water absorbent)		
	Product P2	Product P3	Product P4
0-2	6	6	6
2-3	6	6	5
3-4	6	5	3
4-5	4	4	2
5-6	1	2	1
6-7	0	1	0

Figure D.5: Photo from drop test at SINTEF, Product P2.

Figure D.6: Photo from drop test at SINTEF, Product P3.
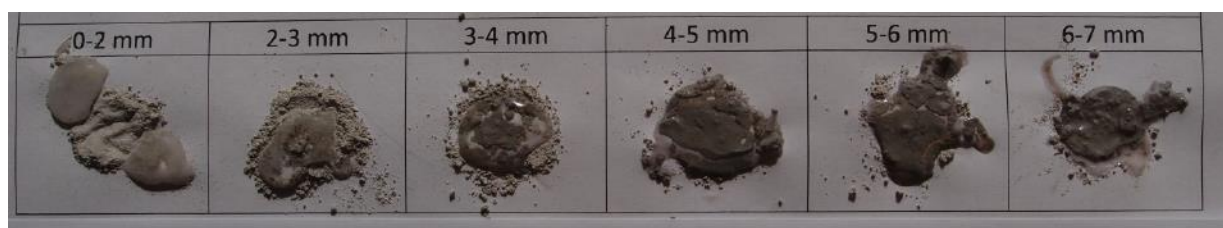
Figure D.7: Photo from drop test at SINTEF, Product P4.

D.3.3.3   CBI results

	Evaluation (6 = totally water repellent, 0 = totally water absorbent)		
Depth, mm	Product P2	Product P3	Product P4
0-2	6	6	6
2-3	6	6	6
3-4	6	6	6
4-5	5	4	2
5-6	4	1	1
6-7	2	0	1

Figure D.8: Photo from drop test at CBI, product P2.

Figure D.9: Photo from drop test at CBI, Product P3.

Figure D.10: Photo from drop test at CBI, product P4.

D.4 Individual results of chloride ingress

D.4.1 VTT results

Concrete Dry Density (kg/m3):		2380
Cl Background Level (% of dry sample mass):		0,010

Reference / Product P2

Depth (mm)		REF1	REF2	REF3	REF4	REF5	Avg	Std	COV (%)	g/m²		P2-1	P2-2	P2-3	P2-4	P2-5	Avg	Std	COV (%)	g/m²	Filter (%)
1	0-2	0,768	0,678	0,837	0,739	0,691	0,743	0,064	9	35		0,586	0,872	0,615	0,489	0,711	0,654	0,145	22	31	
3	2-4	0,632	0,625	0,688	0,647	0,646	0,647	0,024	4	30		0,118	0,279	0,174	0,087	0,181	0,168	0,073	44	8	
5	4-6	0,472	0,488	0,536	0,516	0,509	0,504	0,025	5	24		0,047	0,089	0,064	0,023	0,057	0,056	0,024	43	2	
8	6-10	0,355	0,341	0,360	0,347	0,351	0,351	0,007	2	32		0,018	0,016	0,017	0,010	0,018	0,016	0,003	21	1	
12,5	10-15	0,170	0,138	0,166	0,169	0,159	0,160	0,013	8	18		0,013	0,017	0,005	0,014	0,014	0,013	0,004	35	0	
17,5	15-20	0,026	0,013	0,014	0,023	0,022	0,020	0,006	30	1		0,012	0,015	0,012	0,006	0,011	0,011	0,003	27	0	
22,5	20-25	0,015	0,008	0,005	0,010	0,011	0,010	0,004	38	0		0,008	0,012	0,009	0,008	0,012	0,010	0,002	20	0	
										140										41	71

Product P3 / Product P4

Depth (mm)		P3-1	P3-2	P3-3	P3-4	P3-5	Avg	Std	COV (%)	g/m²	Filter (%)	P4-1	P4-2	P4-3	P4-4	P4-5	Avg	Std	COV (%)	g/m²	Filter (%)
1	0-2	0,749	0,888	0,756	0,832	0,546	0,754	0,130	17	35		0,652	0,726	0,688	0,819	0,632	0,703	0,074	10	33	
3	2-4	0,242	0,270	0,261	0,227	0,118	0,224	0,061	27	10		0,256	0,269	0,276	0,311	0,234	0,269	0,028	10	12	
5	4-6	0,093	0,089	0,090	0,052	0,044	0,074	0,024	32	3		0,122	0,109	0,117	0,110	0,098	0,111	0,009	8	5	
8	6-10	0,023	0,023	0,027	0,010	0,014	0,019	0,007	36	1		0,040	0,019	0,021	0,024	0,021	0,025	0,009	34	1	
12,5	10-15	0,018	0,015	0,011	0,007	0,009	0,012	0,005	40	0		0,017	0,004	0,016	0,016	0,012	0,013	0,005	41	0	
17,5	15-20	0,011	0,019	0,010	0,005	0,007	0,010	0,005	50	0		0,014	0,006	0,014	0,008	0,014	0,011	0,004	36	0	
22,5	20-25	0,014	0,010	0,010	0,006	0,010	0,010	0,003	29	0		0,013	0,005	0,008	0,023	0,012	0,012	0,007	57	0	
										50	65									52	63

D.4.2 SINTEF results

Concrete Dry Density (kg/m3):			2256		
Cl Background Level (% of dry sample mass):			0,005		

Chloride as % of dry sample mass

Depth (mm)		Reference									Product P2									
		REF1	REF2	REF3	REF4	REF5	Avg	Std	COV (%)	g/m²		P2-1	P2-2	P2-3	P2-4	P2-5	Avg	Std	COV (%)	g/m²	Filter (%)
1	0-2	0,572	0,580	0,562	0,629	0,615	0,592	0,029	5	26		0,543	0,579	0,476	0,541	0,559	0,540	0,039	7	24	
3	2-4	0,572	0,543	0,533	0,571	0,539	0,552	0,019	3	25		0,139	0,152	0,156	0,154	0,163	0,153	0,009	6	7	
5	4-6	0,494	0,438	0,417	0,495	0,462	0,461	0,034	7	21		0,047	0,060	0,055	0,041	0,063	0,053	0,009	17	2	
8	6-10	0,349	0,360	0,339	0,373	0,328	0,350	0,018	5	31		0,008	0,011	0,022	0,021	0,024	0,017	0,007	42	1	
12,5	10-15	0,156	0,127	0,112	0,159	0,125	0,136	0,021	15	15		0,014	0,005	0,009	0,005	0,005	0,008	0,004	52	0	
17,5	15-20	0,031	0,025	0,005	0,029	0,025	0,023	0,010	45	2		0,005	0,010	0,007	0,005	0,009	0,007	0,002	32	0	
22,5	20-25	0,005	0,005	0,005	0,005	0,006	0,005	0,000	9	0		0,005	0,005	0,005	0,005	0,017	0,007	0,005	73	0	
										120										35	71

Depth (mm)		Product P3										Product P4									
		P3-1	P3-2	P3-3	P3-4	P3-5	Avg	Std	COV (%)	g/m²	Filter (%)	P4-1	P4-2	P4-3	P4-4	P4--5	Avg	Std	COV (%)	g/m²	Filter (%)
1	0-2	0,572	0,563	0,591	0,623	0,524	0,575	0,036	6	26		0,611	0,604	0,660	0,700	0,686	0,652	0,043	7	29	
3	2-4	0,225	0,222	0,210	0,188	0,181	0,205	0,020	10	9		0,285	0,290	0,274	0,280	0,283	0,282	0,006	2	13	
5	4-6	0,146	0,133	0,137	0,106	0,114	0,127	0,017	13	6		0,209	0,188	0,191	0,186	0,179	0,191	0,011	6	8	
8	6-10	0,047	0,046	0,050	0,033	0,030	0,041	0,009	22	3		0,100	0,087	0,094	0,082	0,080	0,089	0,008	9	8	
12,5	10-15	0,018	0,005	0,014	0,005	0,005	0,009	0,006	66	0		0,025	0,020	0,032	0,021	0,005	0,021	0,010	48	2	
17,5	15-20	0,005	0,005	0,005	0,005	0,005	0,005	0,000	0	0		0,005	0,011	0,005	0,005	0,005	0,006	0,003	43	0	
22,5	20-25	0,005	0,012	0,005	0,005	0,014	0,008	0,004	54	0		0,009	0,005	0,005	0,007	0,005	0,006	0,002	29	0	
										44	63									60	50

D.4.3 CBI results

Concrete Dry Density (kg/m3): 2230
Cl Background Level (% of dry sample mass): 0,005

Chloride as % of dry sample mass

Depth (mm)		REF1	REF2	REF3	REF4	REF5	Avg	Std	COV (%)	g/m²		P2-1	P2-2	P2-3	P2-4	P2-5	Avg	Std	COV (%)	g/m²	Filter (%)
				Reference											Product P2						
1	0-2	0,597	0,656	0,598	0,621	0,569	0,608	0,033	5	27		0,546	0,542	0,478	0,542	0,624	0,546	0,052	9	24	
3	2-4	0,629	0,611	0,635	0,606	0,604	0,617	0,014	2	27		0,188	0,206	0,177	0,209	0,198	0,196	0,013	7	8	
5	4-6	0,534	0,513	0,544	0,479	0,490	0,512	0,028	5	23		0,073	0,096	0,081	0,072	0,087	0,082	0,010	13	3	
8	6-10	0,360	0,373	0,389	0,316	0,344	0,356	0,028	8	31		0,021	0,022	0,019	0,025	0,020	0,021	0,002	11	1	
12,5	10-15	0,120	0,135	0,145	0,114	0,122	0,127	0,013	10	14		0,010	0,010	0,017	0,015	0,011	0,013	0,003	24	1	
17,5	15-20	0,011	0,013	0,015	0,011	0,016	0,013	0,002	16	1		0,010	0,010	0,011	0,012	0,013	0,011	0,001	11	1	
22,5	20-25	0,013	0,009	0,008	0,034	0,020	0,017	0,011	65	1		0,010	0,028	0,011	0,030	0,017	0,019	0,009	49	2	
										124										41	67

Depth (mm)		P3-1	P3-2	P3-3	P3-4	P3-5	Avg	Std	COV (%)	g/m²	Filter (%)	P4-1	P4-2	P4-3	P4-4	P4-5	Avg	Std	COV (%)	g/m²	Filter (%)
				Product P3												Product P4					
1	0-2	0,769	0,531	0,571	0,624	0,542	0,608	0,097	16	27		0,593	0,690	0,706	0,645	0,660	0,659	0,044	7	29	
3	2-4	0,243	0,222	0,276	0,255	0,214	0,242	0,025	10	11		0,289	0,326	0,313	0,300	0,337	0,313	0,019	6	14	
5	4-6	0,099	0,093	0,124	0,108	0,085	0,102	0,015	15	4		0,164	0,154	0,149	0,153	0,160	0,156	0,006	4	7	
8	6-10	0,091	0,018	0,031	0,039	0,010	0,038	0,032	84	3		0,052	0,030	0,041	0,035	0,031	0,038	0,009	24	3	
12,5	10-15	0,012	0,009	0,013	0,015	0,008	0,012	0,003	25	1		0,014	0,015	0,016	0,012	0,013	0,014	0,002	11	1	
17,5	15-20	0,010	0,009	0,011	0,012	0,006	0,010	0,002	21	1		0,013	0,013	0,012	0,011	0,012	0,012	0,001	7	1	
22,5	20-25	0,007	0,008	0,014	0,008	0,008	0,009	0,003	31	0		0,013	0,011	0,011	0,011	0,011	0,011	0,001	6	1	
										46	63									55	56

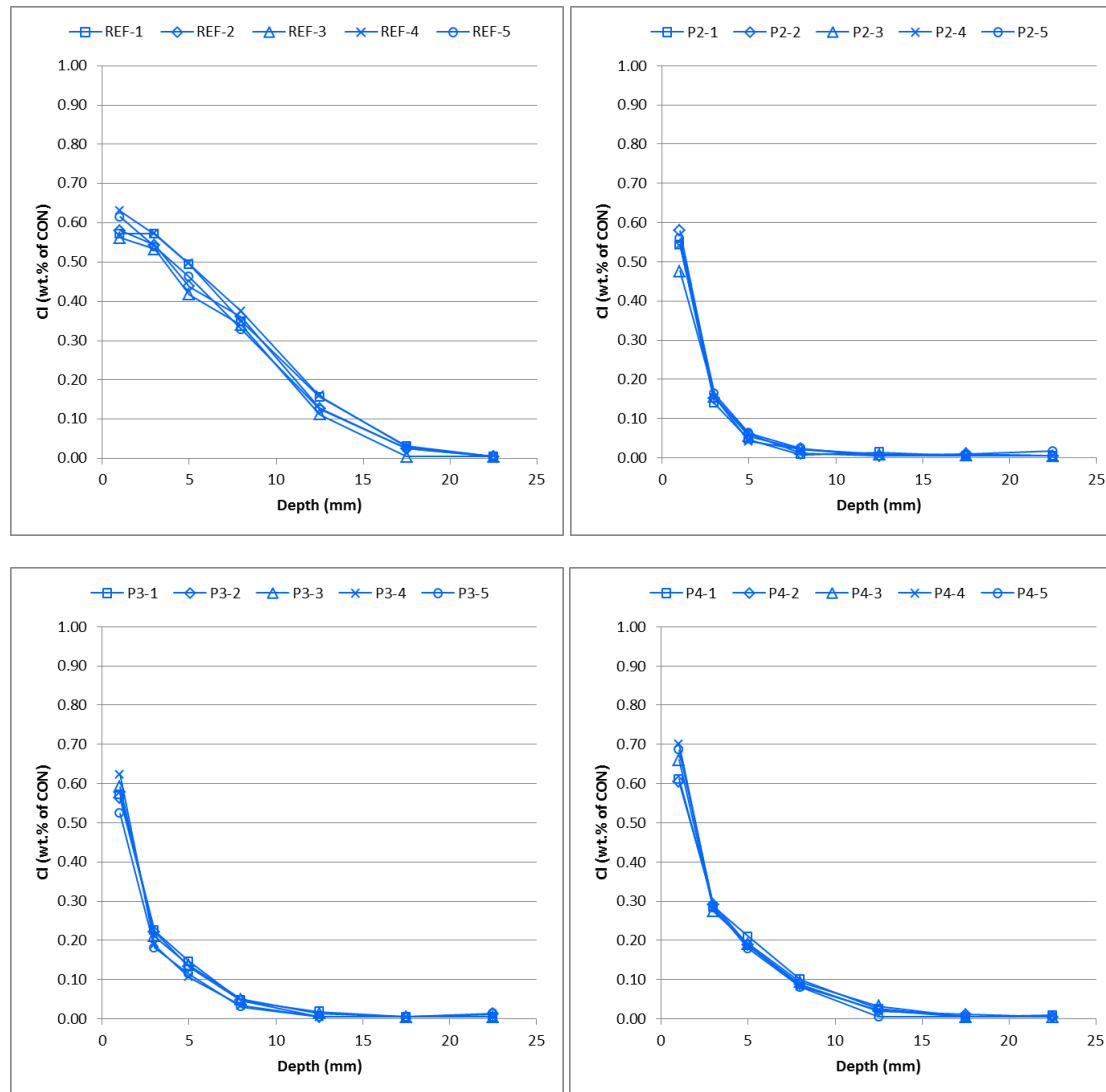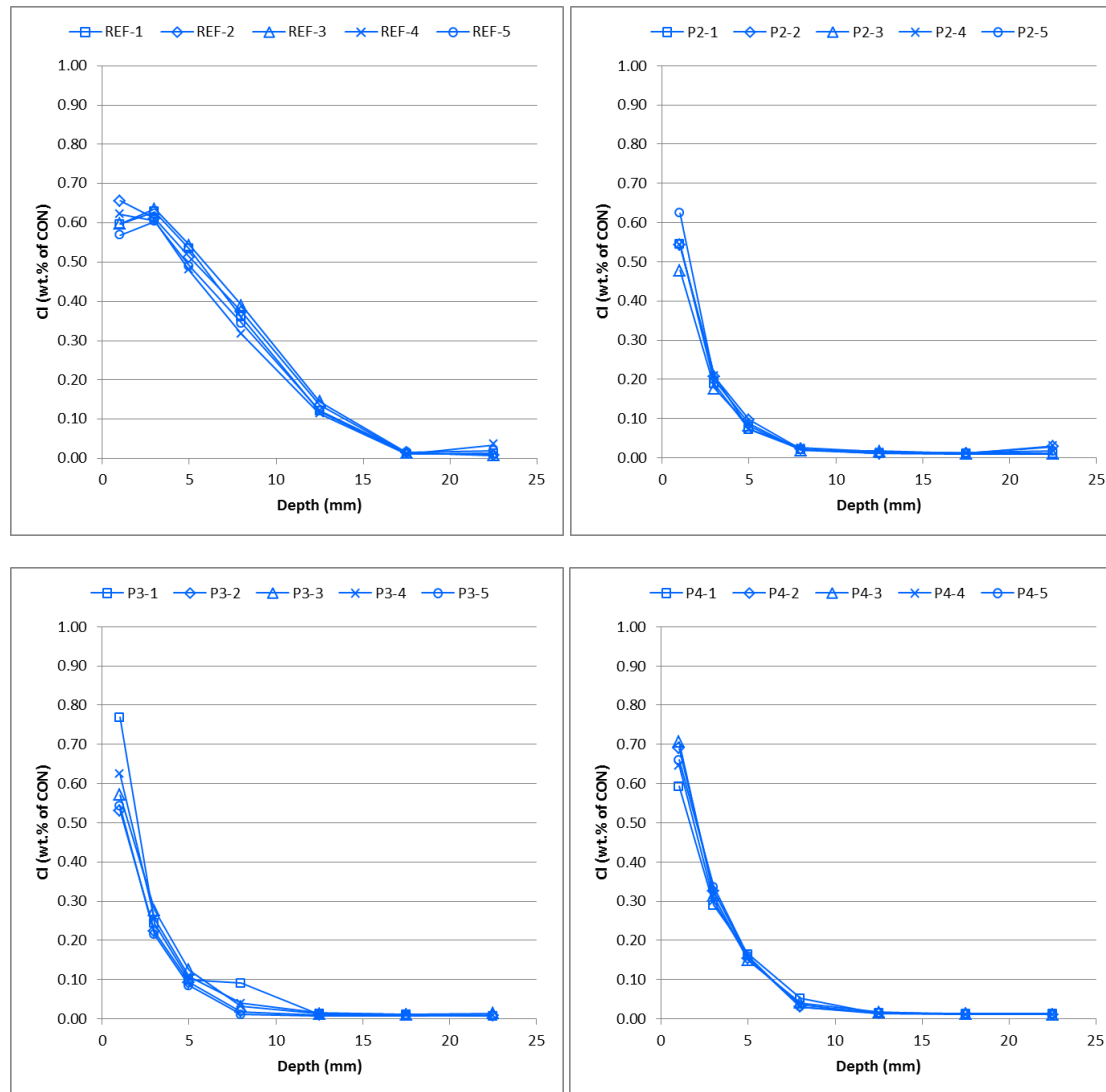## D.4.4 Individual chloride profiles – VTT

D.4.5 Individual chloride profiles – Sintef

D.4.6　　　　Individual chloride profiles – CBI

D.5 Individual results of calcium profiles

	Depth (mm)	VTT		Sintef				CBI			
		REF1	P3-1	REF1	P2-1	P3-1	P4-1	REF1	P2-1	P3-1	P4-1
1	0-2	11,68	11,00	9,32	9,06	9,07	9,77	10,19	10,19	10,30	9,76
3	2-4	11,93	11,93	9,77	9,65	9,51	10,26	10,63	10,16	10,11	10,36
5	4-6	12,31	12,31	10,77	9,42	9,58	10,67	10,74	10,12	10,63	10,69
8	6-10	12,31	12,05	9,83	10,18	9,62	10,25	10,97	9,93	10,30	10,36
12,5	10-15	12,31	12,45	9,66	10,14	10,75	10,75	10,76	10,39	10,06	9,51
17,5	15-20	12,45	11,92	9,72	9,34	9,91	11,03	11,08	10,25	11,26	10,24
22,5	20-25	12,31	12,45	9,58	9,80	9,91	10,74	10,77	10,29	10,89	10,25

ANNEX E – MATERIAL DATA

Table E1: Characteristics of the used hydrophobic impregnation products

Product	Form	Active sub-stance	Active sub-stance	Pen. depth [1]	Recom-mended amount	Recom-mended number of coats	Den-sity	Increase of drying rate coefficient [2]	Freeze-salt resistance [3]	Water absorption [3]	Alkali resistance [3]	Chloride blocking effect
		type	%	mm	g/m^2		kg/l	%				
P1/P3	liquid	silane	99%	≥ 10	about 150 per coat	2-3 wet in wet	0,900	> 30%	OK	OK	OK	AMA [4] approval
P2	gel (contains. bentonite)	silane	90%	≥ 10	225-900	1	0,90	> 30%	OK	OK	OK	AMA [4] approval
P4	cream	silane	80%	≥ 10	200-300 per coat	1-2	0,900	> 30%	OK	OK	OK	AMA [4] approval
P5	liquid	triethox yisoocty lsilane	100 %	≥ 10	90-135 per coat	1	0,90	> 30%	not declared	OK	not declared	AMA [4] approval
P6	liquid	octyl triethox y-silane	98 %	< 10	not given (until surface is saturated)	not given (until surface is saturated)	0,875	not declared	not declared	OK	OK	AMA [4] approval 2002

[1] Tested according to EN 1504-2 (w/c=0,70)

[2] Tested as specified in EN 1504-2

[3] Requirement given in EN 1504-2

[4] Approval according to existing Swedish method as given in AMA (Svensk byggtjänst, 2010).

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