

C-1-3 Nordic method for testing hydrophobic impregnations for concrete, with regard to prevention of chloride ingress

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ABSTRACT: Chloride induced corrosion of reinforcement is the major cause of degradation of 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 have been using different national methods to evaluate the performance of this type of products meaning that the same CE-marked product needed to meet different requirements. Therefore 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 was initiated. 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 defining 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 $\text{Ca}(\text{OH})_2$ -solution or not should be further investigated. The project resulted in the Nordic method NT Built 515 Hydrophobic impregnation for concrete prevention of chloride ingress - filter effect.

KEY-WORDS: Concrete, surface impregnation, Nordic method, chloride penetration.

BACKGROUND, AIM AND SCOPE

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, i.e. their chloride blocking effect.

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

- 1) Analysis of existing test standards (national and international)
- 2) A pre-study to evaluate the influence of different tests parameters
- 3) A round robin exercise involving three laboratories (one in each country).

Results obtained with the new method should also be compared to results obtained by the existing methods and field experience, in order to propose suitable requirements levels.

THE REVIEW OF EXISTING METHODS

A thorough comparison of the existing methods and review of relevant literature allowed defining which test parameters could be directly used in the formulation of the new method and which ones required further studies. A common feature of all existing methods was that the chloride ingress in specimens with hydrophobic impregnations was compared to the ingress in untreated specimens. All methods include the following steps:

- 1) Casting, curing and further preparation of specimens
- 2) Pre-conditioning of specimens before impregnation
- 3) Curing of the impregnation
- 4) Exposure to chlorides
- 5) Determination of the chloride contents
- 6) Expression of the results

However, in all steps there are large differences between the methods, such as the type of specimen used, testing age, storing conditions and length of both, the pre-conditioning and the curing period, methods in how to expose the specimens to chlorides, chloride concentrations and the way to express the result and the requirement.

A vital part of the method is the exposure to chlorides: Permanent submersion, intermittent submersion and salt-spraying are applied in the existing methods each with different NaCl-concentration in the solutions. Based on reviewing these methods it was decided to use permanent submersion in a solution with a 15 % NaCl-concentration in the new method.

Another issue that was decided at an early stage was that chloride profiles should be determined and the chloride blocking effect should be expressed in form of a “filter effect”, FE, defined as:

$$\text{Filter Effect} = FE = 1 - \frac{Cl_I}{Cl_R}$$

where Cl_I is the amount of chloride ions in the treated samples and Cl_R is the amount of chloride ions in the reference samples: For both sample sets the same depth profile steps should be taken. Cl_I and Cl_R can also be seen as the area under the chloride content profile of the impregnated and reference samples respectively (Fig. 1). Both, reference and treated sample show always higher concentration on the surface of the sample (Fig. 1) due to since most of the hydrophobic agents cannot completely prevent chloride penetration.

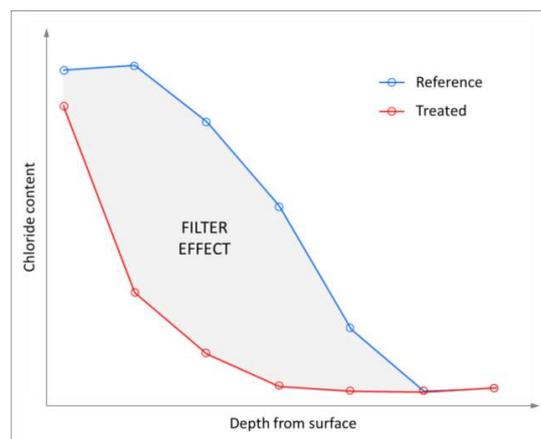


Fig. 1. Schematic representation of the filter effect.

Due to the fact that the method is primarily aimed to classify hydrophobic impregnations for the use on infrastructure constructions a $w/c = 0.45$ was defined as a requirement for the concrete substrate.

THE PRE-STUDY

Some questions needing further studies during the first phase were:

- 1) What type of surface should be used: Formwork surface or a cut surface?
- 2) Should the test surface be pre-wetted with $\text{Ca}(\text{OH})_2$ -solution or not?
- 3) Is it necessary to determine the CaO-content in order to determine the cement content in samples or is it sufficient to give the chloride content per oven-dry concrete mass?
- 4) For how long should the specimens be pre-conditioned before impregnation in a relative humidity around 60 %?
- 5) For how long should the specimens be cured after impregnation?

With regard to the two last questions, three different pre-conditioning types were investigated; exposure to 65 % RH for 7 days with no pre-wetting (**S**); pre-wetting with $\text{Ca}(\text{OH})_2$ -solution for 3 days followed by storage of 3 days (**H**) or 7 days (**C**) at 20 °C, 65 % RH. Three impregnation curing periods were used: 7, 14 and 28 days. Two different types of hydrophobic impregnations were tested, one gel with bentonite clay and 80 % silane (**P2**) and one silane liquid (99 % silane, **P1**).

The results showed that the application of the impregnation to formwork surfaces led to somewhat better chloride blocking effect than when applied on a cut surface. Despite this, it was decided to use cut surfaces in the method, since it is much easier to obtain reproducible surface characteristics that way and represents also a “worst case” scenario. A formwork surface will be affected by the formwork material, release agent, curing conditions etc.

Pre-wetting the surface with $\text{Ca}(\text{OH})_2$ -solution before impregnation was found slightly beneficial for 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 and it increased the number of experimental steps it was decided to exclude such a treatment. Moreover, pre-wetting with $\text{Ca}(\text{OH})_2$ -solution is not representative of what happens in field.

After curing both, reference and treated specimens, were immersed in 15 % NaCl-solution in separate containers for 56 days. 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 °C before dry grinding on a lathe with a diamond tool. 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 chloride analysis. The acid soluble chloride content in each layer was determined by potentiometric titration according to AASHTO-T260 [1] and expressed in wt.-% of sample (oven dry concrete).

It was found that with the specific concrete used ($w/c = 0.45$, aggregate size 0/10 mm, binder CEM I) the determination of the CaO content did not contribute to the robustness and reproducibility of the method and could be omitted. The filter effects obtained with the different pre-conditionings and subsequent periods in 65 % RH are shown in Fig. 2. Generally the filter effect increased with the impregnation curing time. It is not clear which factor was most dominant; the continuous condensation of the hydrophobic impregnation or the continuous cement hydration. However, with the gel product (P2) the filter effects after 7 days of curing in 65 % RH were surprisingly large for all pre-conditionings. In the case of P2 treated concretes a thin remnant gel residue layer was on the surface which provided an extra hydrophobic barrier against chlorides as long as it existed. In order to exclude the effect of such short-lived phenomenon and give the hydrophobic impregnations enough time to distribute and condensate properly, the curing period prior to exposure to chlorides was specified to be 28 days.

In the pre-study it was found that the handling of the specimens between taking them up from the exposure bath and the chloride analysis has a large influence on the obtained chloride profiles. If wiping off the excess chloride solution from the test surface is carried out with a dry absorbing tissue, some of the chloride solution in the pore structure may be sucked out, reducing the measured chloride content in the surface layer. This will influence the total chloride level in the specimens and possibly also the measured filter effect. If the wet specimens, wrapped in plastic, are stored for longer periods before grinding, redistribution of chlorides within the specimen will take place through diffusion. If the redistribution only occurs within the analysed depth, the total chloride content and the filter effect will not be affected. Nevertheless, it is essential to specify the handling during these steps more precisely and avoid long storage periods so that the chloride profile existing when the specimen is taken up from the bath is preserved until the analysis is carried out.

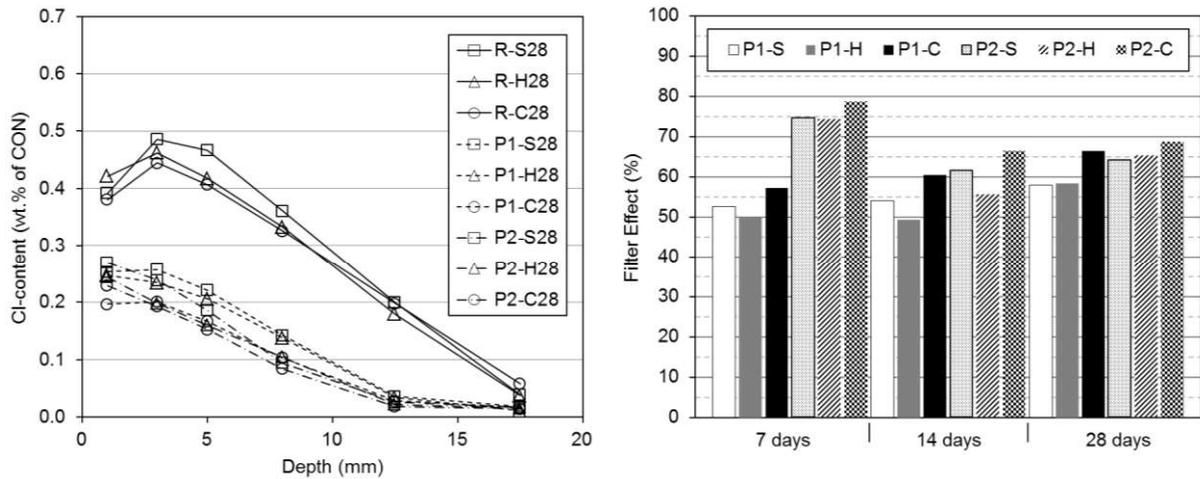


Fig.2. Chloride profiles measured for series cured for 28 days after application of the hydrophobic agent (left). Filter effect for different curing times of the agents (right). Each data point is the average of 3 specimens.

THE ROUND-ROBIN EXERCISE

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. All parts of the tests, from the casting to the analysis, were to be carried out by the different laboratories themselves with local materials and based on the first version of the test method. Tests were carried out with three hydrophobic impregnations (P2, P3 & P4). The method can be briefly described as follows:

- 1) Concrete (w/c = 0.45, aggregate size 0/10 mm, CEM I) specimens were prepared by cutting 100 mm cubes into two halves at the age of 28 days; three cubes per test series
- 2) After cutting the specimens were pre-conditioned in 20 °C, 65 ±10 % RH for 7 days
- 3) The cut surfaces were defined as exposure faces; three halves were treated with the hydrophobic impregnation to be tested and the other three halves were kept untreated as reference
- 4) Both references and impregnated samples were then kept at 20 °C, 65 ±10 % RH for an additional 28 days
- 5) After that, the specimens were submerged in 15 % NaCl-solution for 56 days
- 6) After exposure, the chloride ingress was determined by profile grinding within 7 days down to a depth of 25 mm.

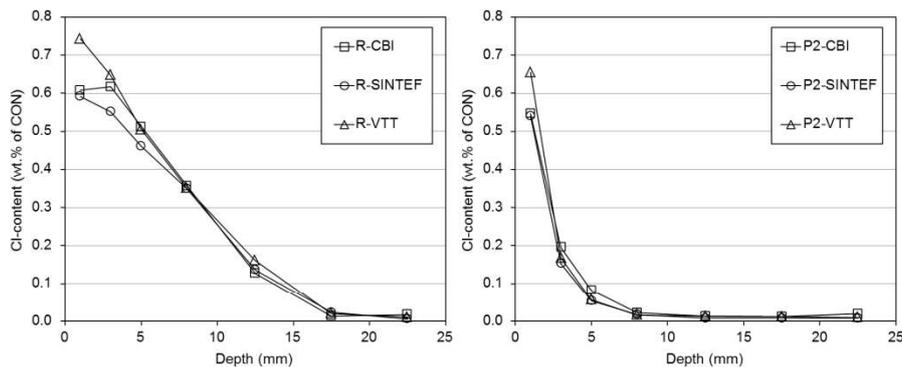


Fig.3. Chloride profiles in the references and in specimens with the P2 hydrophobic impregnation obtained at the three laboratories.

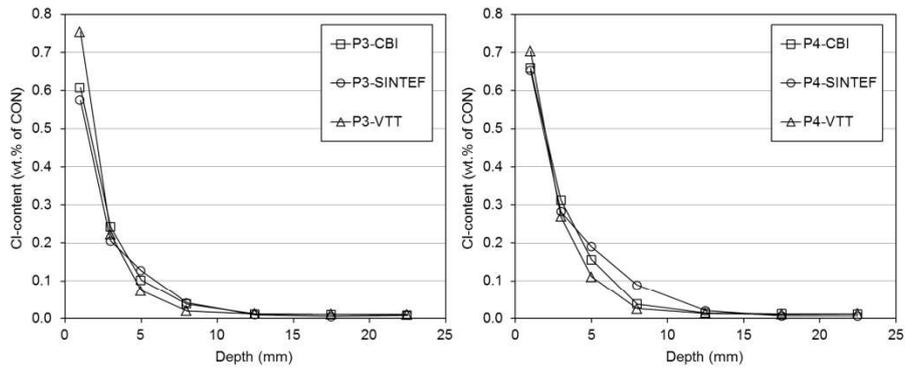


Fig.4. Chloride profiles in the P3 and P4 hydrophobic impregnations obtained at the three laboratories.

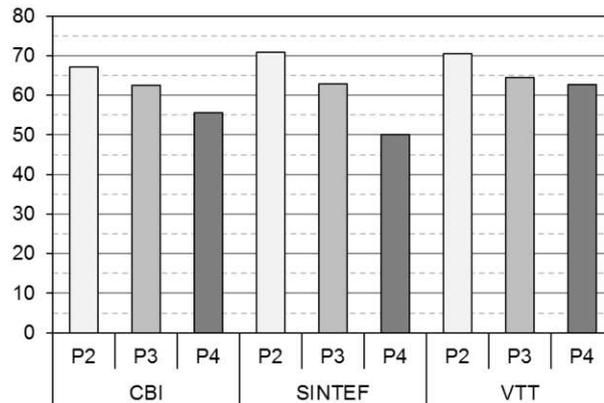


Fig.5. Filter effect obtained at the three laboratories the three hydrophobic impregnations (P2, P3 & P4)

Despite some minor deviations in the tests conditions within the different laboratories, such as the cement brand, the pre-conditioning and curing environment, highly reproducible results were obtained. The major causes for the discrepancies of the results were differences in the actual relative humidity before and after impregnation and the moisture content of the powder samples for chloride analysis. One of the hydrophobic impregnations (P4) also seemed to give more unstable results than the other two. Chloride profiles obtained at the three laboratories for the references and the three products are shown in Fig. 2, Fig. 3 and the filter effects in Fig. 4.

COMPARISON WITH EXISTING METHODS AND FIELD EXPERIENCE

It is not difficult to correlate the results with the new method (FE_{0-25} , the index in FE refers to the profile depth range) to results obtained with the existing Swedish method [5], when a concrete with $w/c = 0.45$ has been used. Taking into account that 2.5 mm of the outermost surface is taken away and the 20 mm sample has been exposed to chlorides at both surfaces this will be equal to the relative chloride content of the sample between 2.5 and 17.5 mm depth. The difference between this content and the one obtained between 2 and 20 mm (FE_{2-20}) will only be marginal, since beyond 17.5 mm the chloride content in both the impregnated specimens and the references is very close to 0. The requirement of the existing Swedish method [5] corresponds to that $FE_{2-20} \geq 0.85$. Using all the results from the round robin exercise, it can be shown that a FE_{2-20} equal to 0.85 corresponds to a FE_{0-25} around 0.65. It is also rather easy to relate the results obtained with Finnish method [6] to the filter effect of the new method. However, the Finnish method was recently modified and we have had difficulties to find any results acquired with it.

The requirements level when the Norwegian method [7] is used is $FE=0.75$. It is not as easy to relate the results from the Norwegian method to the new method as for the Swedish or Finnish method, since the differences in the procedures are much greater. According to [2] somewhat higher filter effects are obtained when salt-spraying is used for the chloride exposure compared to permanent submersion. The Norwegian method uses much shorter periods for pre-conditioning and exposes the cut surface to lime-water after cutting. Lengthening the preconditioning period and omitting the exposure to lime-water of the cut surface both diminishes the value of the filter effect. Moreover, the Norwegian method uses shorter periods for the conditioning after application. As can be seen from the pre-study the influence of prolonging the conditioning periods also influences the filter effect,

however not in the same way for all products. On the whole the $FE_{0.25}$ obtained for a certain product will be lower than the filter effect obtained with the Norwegian method.

Based on the comparison and considerations above, a preliminary assumption of a requirement level for the protection against chloride ingress of hydrophobic impregnation agents can be around $FE_{0.25}=0.65$ for the new method.

The influence of hydrophobic impregnations on the chloride ingress has also been the subject of several field studies, both in a road environment and a marine environment. However, finding results which can be compared is not without problems. There are many hydrophobic agents on the market, sometimes exactly the same product is sold under several different trade names. In many cases, products that were used in earlier studies do no longer exist or has been substantially modified. Thus, results for exactly the same product obtained with the different methods and from field studies are not easy to find. Another parameter that varies in the different studies is the amount of product applied and the conditions under which they were applied. Test results with the products P1, P2, P3 and P4 obtained with the different methods and the outcome of a couple of field evaluations are summarized in Table 2.

Table 2. Classification and performance of different products.

Product	Existing Swedish Method	Existing Norwegian Method	Pre-Study $FE_{0.20}$	Round Robin $FE_{0.25}$			Marine Environment after 10 years ³⁾	Road Environment after 5 years ⁴⁾
				CBI	SINTEF	VTT		
P1	Approved		55				Clear effect	
P2	¹⁾	Approved	63	66	69	68	Clear effect	Clear effect
P3	Approved			61	61	62		
P4	²⁾			54	49	60		

¹⁾ Did not pass at w/c=0.45; very good performance at w/c=0.70
²⁾ Not tested with w/c=0.45; approved when tested with w/c=0.70
³⁾ Data from [4]
⁴⁾ Data from [5]

CONCLUSIONS

The proposed method to classify hydrophobic impregnations with regard to protection against chloride ingress gives sufficiently reproducible results. Even though, the form of the obtained chloride profiles vary somewhat when determined at different laboratories, this does not influence the parameter chosen to describe the efficiency of the products, the filter effect, more than marginally. Since the filter effect is based on the amount of chloride in impregnated samples in relation to the amount of chlorides in un-impregnated samples, the variations in procedures causing differences in chloride profiles, influences the references and impregnated samples to the same degree. However, the reproducibility of the method may be enhanced if some parts of the procedures are made more precise.

Even though the method uses experimental conditions, such as submersion in a solution with unnaturally high concentration of NaCl and application of impregnations to sawed surfaces, the results seem to be relevant when compared to results with other test methods with more realistic conditions and field experiments. The new method was published already by Nordtest under NT Nuuld 515 [8].

ACKNOWLEDGEMENT

The financial support of the Swedish Transport Administration, Norwegian Public Roads Administration and Finnish Transport Agency under grant agreement TRV 2012/75824 is acknowledged.

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