# Experimental determination of chloride threshold values for reinforcement corrosion in concrete: Experiences form the lab





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## ABSTRACT

The reliability of all commonly used service life models for chloride exposed concrete structures is highly dependent on the applied input parameters of the models, e.g. the chloride threshold value for initiation of reinforcement corrosion. Since 2011, a number of different experiments have been conducted at Danish Technological Institute with the main purpose of developing an accelerated in-lab test method for determination of chloride threshold values. In this paper we report on our experiences from the experimental work.

Key words: Chloride ingress, corrosion, chloride threshold values, testing.

## 1. INTRODUCTION

The chloride threshold value is the minimum concentration of chloride at reinforcement depth that is able to initiate corrosion of the steel, e.g. in a concrete structure. When attempting to model the expected service life of a reinforced concrete structure, a relatively small change in the chloride threshold value will generally have a marked effect on the calculated service life. Therefore, a commonly accepted test method for a reliable and precise determination of chloride threshold values is greatly needed, but such a method is currently not available. A series of tests have been carried out at Danish Technological Institute with the aim of developing such a method. This work has served as useful input for the RILEM Technical Committee 235-CTC, which was formed in 2009 with the purpose of addressing the problem with the lack of a generally accepted test method of determination of chloride threshold values.

## 2. EXPERIMENTAL SETUP

A series of experiments was initiated in 2011 at Danish Technological Institute with the purpose of testing an in-lab method for determination of chloride threshold values in reinforced concrete. Part of the test design is based on work by /Nygaard & Geiker 2005/. The basic idea of the experiments is to expose a number of concrete specimens with cast-in rebars to a 6 wt% NaCl solution and subsequently detect the onset of reinforcement corrosion in one of two ways: (1) by observation of a significant drop in the electrochemical potential of the rebar measured against a reference electrode (open circuit approach) or (2) by observation of a significant increase in the

current required to maintain a specific potential of the rebar measured against a reference electrode (potentiostatic approach). Two different potentials is used in the potentiostatic setup, one typical for submerged conditions (-125 mV vs. CSE) and one representing splash-zone conditions (+125 mV vs. CSE). In both setups the chloride concentration is measured at the depth of the rebars when corrosion onset is observed, thus giving the chloride threshold value.

### 2.1 Preparation of concrete specimens

Eight cubic concrete specimens (P1 to P8), each containing four rebars, were cast from the same batch of concrete with an equivalent water/cement ratio of 0.45 (activity factor 0.5 for fly ash) and a binder composed of 75 wt% of CEM I 52,5 N Portland cement (RAPID<sup>®</sup> cement from Aalborg Portland, Denmark) and 25 wt% of pulverized fly ash (type B4 from *E*mineral A/S). After casting each specimen was cut with a diamond saw to produce the shape displayed in Figure 1(a-c). This was done to reduce the cover thickness to 5 mm (four specimens for open circuit approach) or 15 mm (two specimens for open circuit approach) or 15 mm (two specimens for open circuit approach) and the purpose of the epoxy coating was to ensure a unidirectional chloride ingress during exposure.



Figure 1 - Top view (a), front view (b) and side view (c) of concrete specimens from specimen series P. Each specimen of the series contains four rebars and all surfaces are sealed with an epoxy coating, except the 'cover surfaces', i.e. the surfaces to be exposed to the chloride solution. The dashed lines indicate the position of the rebars below the concrete surface and the two arrows in part (c) indicate the position of the 'cover surfaces'.

The rebars were prepared from ribbed steel (Ø 10 mm) and two of the rebars to be cast in each concrete specimen were cleaned using citric acid ( $C_6H_8O_7$ ) and subsequently repassivated in a solution saturated with Ca(OH)<sub>2</sub>, whereas the remaining rebars were used as delivered, with some surface rust on them. After 28 days of curing the eight concrete specimens were exposed to an external chloride source by placing two specimens in each of four identical plastic tanks (23 x 27 x 15 cm) containing 7 liters of a 6.0 wt% NaCl solution, thus immersing the specimens to a depth of 10 cm. The chloride solutions were made using water from the tap.

### 3. **RESULTS**

In the open circuit setup corrosion onset has been observed for one rebar (after ~940 days of exposure) at the time of writing (March 2014). This was identified by a sudden and significant drop in the measured potential of about 150 mV. After having observed that the potential remained at this new and lower level for at least seven days, the chloride concentration was measured at the depth of the rebar by profile grinding, thus giving a chloride threshold value of 0.73 wt% of binder. It is noted, that the corrosion onset was observed for one of the rebars,

which were cast into the concrete specimen in "as-received condition". The free potentials of the non-corroding rebars are in the range between approximately -150 and -50 mV vs. CSE.

In the fixed potential setup with +125 mV vs. CSE corrosion onset was observed for two rebars in the same specimen after 835 and 853 days of chloride exposure, respectively. About two weeks after corrosion onset, the rebars were disconnected from the setup and the chloride concentrations were determined at the rebar depths by profiles grinding. The chloride content was very high when measured directly on the corroding specimens after two weeks of high current flowing between the corroding rebars and the potentiostat. Therefore, the chloride content was also measured after a few more weeks in the same concrete specimen at a third rebar where corrosion onset had not been observed. At this rebar a much lower concentration (0.41 wt% of binder) was measured. A number of additional chloride ingress profiles were also determined at earlier exposure times with the purpose of following the general progress of the chloride ingress in the specimens. All the measured profiles are displayed in Figure 2. Two of the specimens used for the profile grinding were also utilized to prepare thin section for optical microscope investigations, which revealed the presence of a thin (~10  $\mu$ m) and quite dense layer of calcite on the exposed surface of both investigated specimens.



Figure 2 – Chloride profiles measured on concrete specimens that have been exposed to a 6.0 wt% NaCl solution. The chloride contents were determined according to the procedure given in /NT BUILD 208 1984/. The exposure time is indicated for each profile in the legends along with information on the type of experimental setup: OC = Open Circuit setup and FP = Fixed Potential setup. The elevated chloride contents in the two profiles measured after 862 and 871 days are believed to be a result of chloride migration after corrosion onset in the potentiostatic setup (see text for explanation).

#### 4. **DISCUSSION**

Despite concrete cover thicknesses of only 5 mm for most of the specimens and a prolonged exposure time of more than 900 days, corrosion onset has only been detected for one out of 8 rebars with 5 mm cover in the open circuit setup and for two out of 16 rebars with 5 mm cover in the fixed potential setup. This result was quite unexpected, since the test method was

designed in such a way, that corrosion onset was expected to occur within a few months after the first chloride exposure. Nevertheless, it seems reasonable that active corrosion has not been widely observed during the first 900 days, since the measured chloride profiles have demonstrated a slow ingress of chlorides into the specimens. This might be partly due to (1) the presence of a thin and dense calcite crust on the exposed specimen surfaces, which may have introduced some blocking effect on the ingress of chloride ions, and (2) the high proportion of fly ash in the binder of the concrete, a material which has previously been reported to result in a refinement of the pore structure of the cement paste, as well as enhancement of the chloride binding capacity, thus leading to a higher resistance of the concrete against ingress of chloride ions /Dhir & Jones 1999/. We have encountered similar problems with prolonged exposure times without corrosion onset and slow ingress of chloride in concrete specimens in connection with our participation in the RILEM TC 235-CTC group, which involves a Round Robin test of a newly proposed method for determination of chloride threshold values /Sørensen *et al.* 2012/.

The high chloride contents measured after corrosion onset in the potentiostatic setup is believed to be a result of chloride migration into the sample in the period between corrosion onset and disconnection from the potentiostat, because a high electrical current was required to maintain the fixed potential during this two week period. This notion was supported by the fact that a much lower chloride concentration was measured subsequently in the same specimen at a third rebar for which corrosion had not been detected. Consequently, it is important that the rebar is disconnected from the potentiostat straight after registration of corrosion onset if the chloride threshold value should be determined by measurement directly on the corroding specimen.

### 5. CONCLUSIONS

Two experimental setups for measuring chloride threshold values in chloride exposed concrete specimens with cast-in rebars have been tested in the lab: One involving an open circuit approach and another with a potentiostatic approach. The tested concrete had an equivalent water/cement ratio of 0.45 (water/powder ratio approx. 0.40) and a binder consisting of ordinary Portland cement blended with 25% fly ash. In the open circuit setup a chloride threshold value of 0.73% (by weight of binder) has been measured at a free potential of approx. -50 mV vs. CSE. In the potentiostatic setup a chloride threshold value of 0.41% (by weight of binder) has been measured at a fixed potential of +125 mV vs. CSE. These results are initial results from an ongoing experiment.

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