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COMPARISON OF SELECTED METHODS FOR MEASUREMENT OF THE CONCRETE ELECTRICAL RESISTANCE TO CHLORIDE PENETRATION

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ABSTRACT

The paper compares selected experimental measurement techniques for evaluation of concrete ability to resist chloride penetration. There are three procedures of evaluation of concrete diffusion coefficient compared. The paper is aimed at the comparison of diffusion coefficient based on the chloride profiling and electrical properties of concrete, namely electrical conductivity and resistivity. There are discussed methods of rapid chloride penetration test as well as surface measurement of electrical resistivity using Wenner probe. The comparison aim is to obtain input data for a finite element numerical model for probabilistic calculation of durability of reinforced concrete bridge deck exposed to the chloride laden environment.

Keywords: concrete, diffusion coefficient, chlorides, electrical resistivity, wenner probe, experiment.

INTRODUCTION

Evaluation of the diffusion of chloride ions is important with respect to durability of reinforced concrete structures exposed to corrosion inducing chloride laden environment. Since concrete is material that strengthen during its maturity process, thus concrete properties are time dependent.

It is even more significant in case of an ability to resist ingress of chlorides comparing to mechanical properties. Both the diffusion coefficient decreases as well as chloride ingress rate in undamaged concrete decreases with the progress of concrete maturity (see 0, [2], [3] and [3]). Thus, effective evaluation of the effect of concrete long-term aging is current topic.

Knowledge of the diffusion coefficient allows describing the penetration of chlorides in concrete to the level of a steel reinforcement. A prepared diffusion coefficient is one of the key input parameters into numerical model for the estimation of durability of concrete structures (e.g. bridge decks [5], [6], [8] and [9]). It is therefore desirable to obtain the parameter of concrete, both sufficiently accurately and efficiently.

Basis for the selection of suitable methods for determining the diffusion coefficient may be found e.g in summaries [10], [11] and [12]. The traditional way of determination of the chloride ability to penetrate into concrete is to analyze the chloride profile during long time sample surface treatment with an aqueous NaCl solution 0 (modifications NT BUILD 208 [13]).

High labor and time demand lead to development of more effective procedures such as accelerated penetration of chloride [14] and the electrical resistivity measurements of concrete [15], [2] and [16]. There are results of diffusion coefficient computed based on chloride penetration as well as electrochemical procedures however the sources comparing both approaches are scare. Moreover comparison of both approaches is vital for our laboratory with respect to the effective evaluation of concrete ability to resist chloride ingress as well as calculation of diffusion coefficient suitable for the numerical modelling of chloride induced corrosion related durability problems.

The paper that is translation and modification of [17] is focused on comparison of the results of a chloride profile analysis and the measurement of resistivity on the high performance concrete specimens with respect to chloride ingress modelling.

CHLORIDE INGRESS MODELS

The corrosion of steel reinforcement is primarily controlled by the diffusion of chlorides. The effect of hydraulic pressure and capillary suction may be neglected in case of the structures of interest.

The diffusion is the most common way in which chloride ions are brought into contact with the reinforcement of concrete bridges decks. The process of chlorides penetration through concrete as a function of depth and time can then be modelled with the aid of Ficks 2nd law of diffusion (see for example [18], [10] and [5]), as is generally accepted.

Constant diffusion coefficient in time

The governing differential relation can be solved using Cracks solution applied to the problem of diffusion in concrete in [19]. A polynomial development can help in the numerical solution to the chosen differential equation as shown in Equation (1).

It needs to be noted that for the more complicated boundary conditions, the numerical solution of governing differential equation shall be applied. See e.g. [20], [21], and in case of concrete with crack concrete with crack [6], [22], [9]).

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$$C_{z,t} = C_0 \left\{ 1 - \frac{2}{\sqrt{\pi}} \sum_{n=0}^{14} \frac{(-1)^n \left(\frac{z}{\sqrt{4 D_c t}} \right)^{2n+1}}{n!(2n+1)} \right\} \quad (1)$$

Where are

- $C_{z,t}$-concentration of chlorides [% by mass of total cementitious materials (%/cem)],
- C_0-concentration of chlorides [% by mass of total cementitious materials (%/cem)] at the surface layer of concrete,
- D_c-apparent diffusion coefficient of chloride ions in concrete [m^2/s],
- t-chloride exposition time [s],
- z -reinforcement depth [m],

Diffusion coefficient considering aging effect

Due to the long term curing of concrete the diffusion coefficient is a time dependent parameter. Reformulation of Equation (1) in order to respect the prolonged effect of concrete maturity was introduced in 0 and [3]. Respective formula is given bellow:

$$C_{z,t} = C_0 \left\{ 1 - \frac{2}{\sqrt{\pi}} \sum_{n=0}^{14} \frac{(-1)^n \left(\frac{z}{\sqrt{4 \frac{D_{c,i}}{1-m} t^{(1-m)}}} \right)^{2n+1}}{n!(2n+1)} \right\} \quad (2)$$

Where are:

- $C_{z,t}$-concentration of chlorides [% by mass of total cementitious materials] at time t [years],
- C_0-concentrationof chlorides[% by mass of total cementitious materials (%/cem)] at the surface directly inside the concrete,
- $D_{c,i}$-teoretical diffusion coefficient in the age of 1 sec [m^2/s],
- t-chloride exposition time [s],
- z -reinforcement depth [m],
- m-aging factor [-].

The value of the diffusion coefficient in selected time such as value in theoretical age of 1 sec may be computed using Equation (3). This equation was introduced in [3] and reformulated to one given bellow in [23].

$$D_c(t) = D_{c,ref} \cdot \left(\frac{t_{ref}}{t} \right)^m \quad (3)$$

In equation (3) there are:

- $D_{c(t)}$-effective diffusion coefficient for the selected age [m^2/s],
- $D_{c,ref}$-diffusion coefficient [m^2/s] obtained at aget_{ref},
- t-chloride exposition time [s] ,
- t_{ref}-age at the measurement of $D_{c,ref}$[s],
- m-aging factor [-].

Diffusioncoefficient as well as aging factor given in formulas (1-3) are among key parameters in chloride ingress modelling. The laboratory evaluation of diffusion parameters is described in following sections.

CHLORIDE PROFILE ANALYSIS

To analyze the diffusion coefficient, several methods based on penetration and profiling may be selected [10]. The modified NORDTEST NT Build 443 [13] is selected herein. For a suitable period of time (at least 35 days), concrete specimens are immersed in saline solution. This test of natural diffusion gives, thanks to a very high gradient of values of the diffusion coefficient $D_{nssd.}$ (non-steady state diffusion coefficient), enough data for the creation of a curve of the measured chloride profile.

Since the suitable tool for grinding the surface of the concrete and dust collection according to NT Build 443 is not available, the process is modified. Sampling of concrete powder in respective layers of chloride profile is conducted by drilling. It is approach prescribed in 0. Hence it is necessary to extend the period of exposure in salt solution.

The exposure extension allows the differences between the individual profile layers to show up. There is difference in a method for treating the samples during maturing between 0 and [13]. AASHTO T259 recommends samples to dry at controlled humidity and temperature, while NT Build 443 stores samples saturated throughout the maturing.

The preparation of samples is given bellow:

- the test specimens used are of sufficient diameter (approx. 100 mm) and a height (approx. 60 mm),
- newly concreted samples are allowed to mature for 28 days in a bath of lime-water,
- the curing container is hermetically sealed and filled to the brim, thus carbonation of water is avoided,
- the tested surface is cut so that it is smooth and clean,
- untested surface are closed by epoxy-coating sealing,

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- the samples are stored in a solution of 165 g of NaCl per liter of water for at least 90 days,
- the salt solution is mixed once a week and replaced once per five weeks,
- sampling (grinding or drilling) must be made at a sufficient distance from the edge of the sample,
- concrete powder is obtained from several layers, thusdescribing the distribution of chloride concentration along the height,
- powder from 6 layers is extracted and analyzed,
- according to NT Build 443 must be 5 grams of concrete powder from each layer.

The amount of chloride in the concrete powder obtained can be determined by potentiometric titration (see, e.g. [24], [2] and [25]). Chloride content is being determined according to standard EN 14629 (Determination of chloride content in hardened concrete) [24] and potentiometric titration with a standard solution of $AgNO_3$. Potentiometric titration curve is then evaluated with the help of the first derivative, where the equivalence point is determined, which corresponds to the consumption of the titration agent $AgNO_3$. Cl- content of the sample is calculated as the percent ratio of the weight of chlorides and concrete. The test is rather long lasting, labor intensive and is not able to effectively capture the effect of aging of concrete.

When the concentration of chlorides in given depths are available than the measured chloride profile is approximated via Equation (1) with the D_c calculated by the method of least squares for an equation (4).

$$S_j = \sum_i^N \Delta C^2_{(i)} = \sum_i^N \Delta\left(C_{m(i)} - C_{c(j,i)}\right)^2 , \quad (4)$$

where:

- $S_{(j)}$-sum of the squares in j-th iteration [(% by mass)^2],
- N -number of layers in profile,
- $\Delta C_{(i)}$-difference between the measured and calculated concentration of chlorides N-th layer [% by mass of total cementitious materials],
- $C_{,m(i)}$-measured concentration of chlorides [% by mass of total cementitious materials],
- $C_{,c(j,i)}$-calculated concentration of chlorides in j-th step of iteration in i-th layer [% by mass of total cementitious materials].

It is worth mentioning that the unknown parameters are both diffusion coefficient as well as surface chloride concentration.

MEASUREMENT OF ELECTRICAL RESISTIVITY

Observed correlation between the passage of electric current and a diffusion coefficient [2] and [10] allows to save time and resources in case of the assessment of concrete ability to resist the ingress of chlorides comparing to the chloride profiling that takes weeks of chloride ponding, and hours of profiling and titration.

The accelerated chloride penetration tests (RCPT, AASHTO T277 [14] and ASTM C1202 [26]) is also more labor intensive because it requires couple of days and preparation of samples is still labor intensive.

Other electrochemical method is based on the measurement of electrical resistivity ρ (AASTHTO TP-95 [15] and [29]). The measurements itself takes minutes, which is extremely fast in comparison with penetration tests [30] and [13] or even RCPT test [14] and [26].

Four-point method

One of the options for the measurement of electrical resistivity is the surface measurement using an instrument called Wenner probe (Figure-1).The advantage of the method is rapid handling of instruments and samples. The method is nondestructive so the repeated measurement is possible in order to obtain the development of diffusion in time.

There is also theoretical possibility of measurement on the existing structure. The reading may be taken in situ however limitation is caused by the presence of steel reinforcement in concrete. It is worth noticing that the surface resistivity shall be converted to bulk resistivity using geometric correlation relationships [31]. Conversion may be avoided and precision of reading improved if bulk conductivity is measured instead of surface resistivity [32].

Figure-1. The concrete cylinder samples and the Wenner probe.

The Wenner probe consists of four electrodes with spacing of approx. 5 cm. External electrodes applie electric current and the internal electrodes measure the difference in electric potential.

This method of measurement may unfortunately exhibit a relatively large scatter, partly due to the heterogeneity of the test material, and also due to the application of rather uncontrollable contact conditions. The contact between the electrodes and the concrete is maintained via wet sponge where electrical connection is

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influenced by the level of contact pressure and sponge saturation.

After the samples are casted, they are cured in a water bath or in a limewater. When a lime bath is used, the container is filled with a saturated lime solution up to the edge and closed so it is airtight. The samples are tested under saturated surface dry condition. Resistance to the passage of current for each of the cylinders is tested lengthways 8 times, while test is repeated 2 times at each of four selected sides.

If the electrical resistance is obtained than the diffusion coefficient may be computed. The evaluation of the diffusion coefficient may be computed via several procedures (see e.g. 0, [2], [27], [28] and [16]). The resulting diffusion coefficients correlated even though there are differences in actual values depending on the choice of respective procedure.In presented work, the diffusion coefficient is computed according to Nernst-Einstein relationship (5) applicable for porous materials such as concrete [28]:

$$D = \frac{RT}{Z^2 F^2} x \frac{t_i}{\gamma_i C_i \rho_{BR}}, \tag{5}$$

where are:

- D-diffusion coefficient [m^2/s],
- R-universal gas constant [J/K.mol],
- T-absolute temperature [K],
- Z -valence of ions [-],
- F-Faradays constant [C/mol],
- t_i-transport number of chloride ions [-],
- γ_i-activity coefficient of chloride ions [-],
- C_i-the concentration of chloride ions [mol/m^3],
- ρ_{BR}-bulk electric resistivity [Ωm].
- Molar concentration of chloride ions C_i for a water solution can be determined as follows:

$$C_i = m/n \times 1000, \tag{6}$$

where:

- C_i-molar concentration of chloride ions [mol/m^3],
- m -the weight of chlorides in 100 ml of suitable solution [m],
- n -molal constant [mol].

The coefficient of activity γ_i is selected herein as 1, which corresponds to one of the first measurements of the electric characteristics of concrete for calculating the diffusion coefficient [2] or the diffusion coefficient derived on the basis of the article by [32]. However, also Ghosh in his work [29] reports an approach which leads to the calculation of the activity coefficient γ_i through a parameter I:

$$I = \frac{1}{2} \sum mZ^2, \tag{7}$$

where are:

- Z-charge of ions [mol/m^3],
- m-molality [-].

Above mentioned parameter $I$is subsequently used to calculate the coefficient of activity after the solution of following logarithm equation:

$$-\log \gamma = AZ^2 [\frac{\sqrt{I}}{1+\sqrt{I}} - 0.2xI], \tag{8}$$

where:

- A-is an empirical constant [-] (A equalsto 0.5094 at room temperature),
- Z - valence of chloride ions that is equal to 1 [-].

When the diffusion coefficient is obtained during the concrete aging, the aging factor m may be derived to describe the ability of concrete to improve the resistance of concrete to ingress of chloride. The parameter m is computed via curve-fitting of measured diffusion coefficients to Equation (3).

LABORATORY EXPERIMENT

The aim of the experiments is to analyze the diffusion coefficient for selected high performance concrete (HPC) reinforced bridge deck [33]. It is need to be noted that the batch proportion and admixtures are confidential. Since, the goal of the research is comparison of diffusion coefficient evaluation via two testing options, the actual mix design does not have significant influence on the comparison. The testing procedures are analysis of chloride profile and measuring of the electrical resistivity.

Samples information

There were prepared 6 samples, labelled FN, The samples were concreted 133 days before testing of chloride profile analysis. Their dimensions are: diameter / length circa 103/204 mm. The samples were cured according to NT BUILD 443 [13] in a saturated solution of $Ca(OH)_2$.

Three samples were used to evaluate the chloride profile. The remaining samples were used to measure electrical resistivity.

Chloride profile analysis on samples

Three samples that were selected for the chloride profiling were analyzed also for the electrical resistivity (FN4, FN5 and FN6) first. The cylinders were sliced after the measurement of the resistivity. The slices of height 6 cm were prepared. Three sides of the samples were provided with an epoxy coating. Surfaces exposed to saline were cleaned. Samples were immersed in the bath and airtight (Figure-2).

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Figure-2. Samples submerged in the chloride solution.

Profiles of chloride concentration are shown in Figure-3. The chloride profiles are then used in iterative search for the unknown surface chloride concentration C_0 and diffusion coefficient D_c. The iterative solution of equation (4) via least square method is applied. In the calculation, it is assumed that the concentration of chlorides in the background is zero

RESULTS

The resulting values of chloride profiling-based calculated values of the diffusion coefficients are given in Table-1. However the table contains corrected values because the actual diffusion coefficient is time dependent and thus long term penetration is able to compute effective value from the exposure period. Diffusion coefficient for the age of 133 days D_{c133} is 4.80×10^{-12} [m²/s]. This value is corrected for the aging effect to the 28 days age using the relationship (3) and and aging factor m = 0.39. After the corrections, the D_{c28} is 8.80×10^{-12} [m²/s]. The aging factor evaluation will be described below):

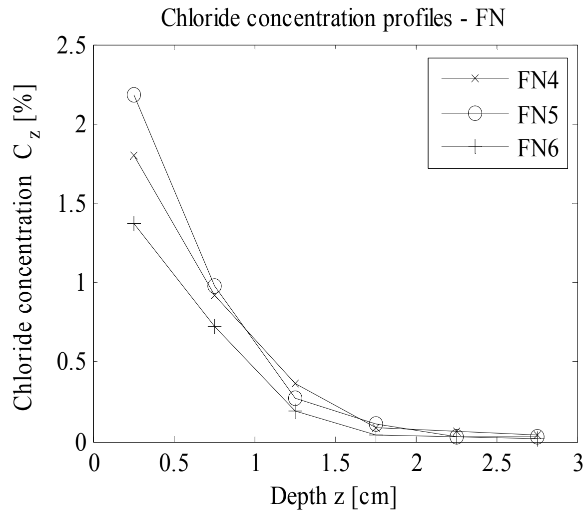

Figure-3. Chloride concentration profiles of studied high performance concrete FN.

The Figure-4 shows measured and approximated chloride profile from one of slices. It needs to be noted that the number of profile depths was lower than 8 (recommendation of [13]). The reason was very low concentration of chlorides at higher depths.

The surface resistivity ρ_{SR} was measured for all six samples on September 30, 2014. The measurement of resistivity continued later on cores that were not used at chloride profile measurement (FN 1-3). Thus the development of diffusion coefficient D_c in time is obtained (3).

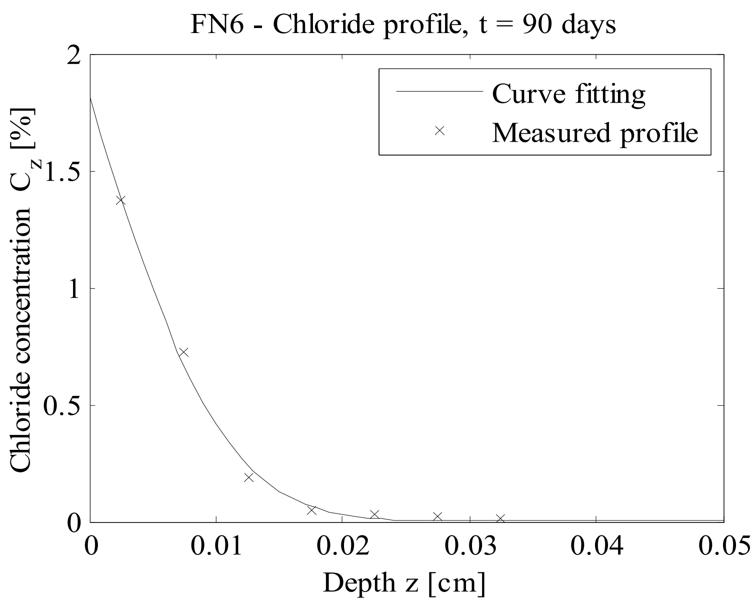

Figure-4. Profile of chloride concentration for the sample FN6 with measured data and curve fit.

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Table-1. Surface chloride concentration and diffusion coefficient for the age of 133 days for the exposed 90 days of chloride penetration.

Exposure *t* [days] 90	**Surface concentration C_0** [% weight.]	**Diffusion coefficientD_{c133}** [m^2/s]	**Diffusion coefficient for m = 0.388D_{c28}**[m^2/s]
FN4	2.33	4.98×10^{-12}	9.11×10^{-12}
FN5	2.50	4.98×10^{-12}	9.11×10^{-12}
FN6	1.81	4.46×10^{-12}	8.16×10^{-12}
Average value		4.80×10^{-12}	8.80×10^{-12}

Table-2. Electrical resistivity and respective diffusion coefficients.

Body	**30. 9. 2014**		**Surface resistivity ρ_{SR}** [kΩcm]	**Bulk resistivity ρ_{BR}** [kΩcm]	**Diffusion coefficient D_c** [ms^{-2}]
	28 days	Diameter	33.54	14.07	3.65×10^{-12}
FN	48 samples	Standard deviation	2.14	0.90	0.226×10^{-12}
		Variation coefficient	0.06	0.06	0.062

Table-3. The effect of the coefficient of activity γ_i of chloride ions on the resulting diffusion coefficient obtained with the help of electric resistivity.

Body	**30. 9. 2014**		**Diffusion coefficient D_c**	**Diffusion coefficient D_c**
			[$10^{-12}\times$ms^{-2}]	[$10^{-12}\times$ms^{-2}]
			$\gamma_i = 1$	$\gamma_i = 0.692$
	28 days	Diameter	3.65	5.28
FN	48 samples	Standard deviation	0.226	0.327
		Variation coefficient	0.062	0.062

The values of surface resistivity corrected to bulk resistivity values according to [31] are given in Table-2. The resulting diffusion coefficient, given in Table-3, is computed in two variants, considering the chloride ions activity coefficient ($\gamma_i = 0.692$) and without its consideration ($\gamma_i = 1$). Analysis of time-dependent diffusion coefficient $D_{c(t)}$ obtained via analysis of concrete resistivity measurements ρ_{SR} allowed for the evaluation of coefficient of aging *m* computation. Average diffusion coefficients obtained for selected maturity ages are given in Table-4. The resulting aging factor obtained via formula (3) curve fitting [32] to measured data. The least square method was applied and $m = 0.388$.

Computed *m* factor and formula (3) was also applied on the results in Table-1 (see on page 5) in case of the chloride profiling-based results.

Table-4. Diffusion coefficient $D_{c(t)}$derived from surface resistivity ρ_{SR} considering chloride activity coefficient $\gamma_i = 1$.

Date	**30.9.2014**	**14.10.2014**	**29.10.2014**	**13.11.2014**	**2.12.2014**	**6.1.2015**
Age *t* [days]	28	42	57	72	91	126
Diffusion coefficient $D_{c(t)}$ [10^{-12}ms^{-2}]	3.64	3.27	2.66	2.40	2.22	2.20

CONCLUSIONS AND DISCUSSIONS

The article is dealing with the evaluation of durability related concrete material parameters. There are discussed two approaches for the computation of diffusion coefficient applicable for the numerical modelling of chloride ion ingress to concrete. Two studied approaches are chloride profiling and electrical resistivity measurement.

Diffusion coefficient obtained from chloride profiling recalculated for the age 28 days is higher than values obtained for the same age via electrical resistivity measurements. There are two reasons affecting the difference. First one is the progressive ingress of chlorides throughout entire penetration period while the concrete's maturity is changing. Thus at the beginning, chloride penetrates faster than at the end of penetration period. This leads to higher diffusion coefficient at the end of penetration period comparing to one computed from the instant measurement. The other reason is the effect of chloride ion activity coefficient. Application of this coefficient in calculations of diffusion coefficient according to electrical resistivity measurement lead to better match of compared values (profiling × electrical resistivity).

It needs to be noted that the comparison of long term chloride testing and fast electrical resistivity measurements shows match however the difference in results obtained in our laboratory deserve attention in further research.

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