Comparison of Protective Properties of Concretes with Low Alkali Cement

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Abstract. Diffusion process determines the rate of chloride ions penetration in concrete and therefore diffusion coefficient values play a major role in the evaluation of protective properties in concrete with reference to reinforcement. This paper describes tests on protective properties of concrete cover made of ordinary concrete and different types of low alkali cement. The tests on four types of concrete mix which differed in a type of used cement was performed. The following types of cement were applied: in concrete C1 - cement CEM I 42.5 R characterised by high heat of hydration, rapid increase in strength and high early strength. In concrete C2 – cement CEM III/A 32.5 N-LH/HSR/NA characterised by high resistance to chemical aggression. In concrete C3 – cement CEM I 42.5 N/SR3/NA with high chemical resistance, particularly to sulphate corrosion, low content of Na2O alkali, tricalcium aluminate C3A and aluminium oxide Al2O3. In concrete C4 – cement CEM IV/B (V) 32.5 R – LH/NA characterised by a stable increase in strength, increased strength during longer stages of concrete curing. Protective properties of concrete cover for reinforcing steel were specified by determining values of chloride diffusion coefficients describing flow rate of chloride ions in wet concrete. Chloride diffusion coefficients were determined on the basis of the thermodynamic model of chloride flow in the electric field. Migration tests in the electric field were used to determine diffusion coefficients. Then, distribution of chloride concentrations in diffusion tests with the forecast distribution of chloride concentrations, taking into account the obtained values of chloride concentrations was compared. Consequently, reliable values of diffusion coefficients were determined. Also the forecast durability of concrete structure made of tested types of concrete was specified.

1. Introduction
Chloride induced corrosion was recognised as one of the major cause of degradation of reinforced concrete structures [1]. Chloride ions, responsible for the corrosion of the steel, can be provided by a marine environment or by deicing salts for example. Corrosion may start as far as oxygen and water are available. Corrosion of reinforcing steel can reduce the structure's lifetime, i.e. the period of time during which the structure has to maintain minimum performance (structural safety and serviceability) [2].

Corrosion of reinforcing steel may begin when the chloride ions concentration near the surface reaches critical value of 0.4% of the mass of cement that is contained in concrete. During the progression of corrosion the volume of reaction products increases what causes cracking and loosening of concrete coating [3]. Diffusion process determines the rate of chloride ions penetration in
concrete and therefore diffusion coefficient values play a major role in the evaluation of protective properties in concrete with reference to reinforcement.

The differences in diffusion coefficient values obtained with taking into account changes of chlorides concentration, indicate that to describe and analyse the process we should use theoretical thermodynamic models, on the basis of which the equation and a converse equation of chloride migration in an electric field can be obtained. It allows us to determine theoretically justified averaging of the results obtained in an experiment [4].

In this work thermodynamic model of chlorides flow in concrete pore solution under influence of an electric field that was presented in [4] was used. The results of our own tests run in accordance with theoretical solution allowed to determine meaningful diffusion coefficient value in concretes with different types of cements coming from one producer. The main goal of presented research was comparing the influence of different types of cement in concrete composition on protective properties of concrete coating in regard to reinforcing steel in environment containing chloride ions. Based on migration tests in an electric field diffusion coefficient values characterising chloride ions flow rate in wet concrete were determined. The forecast durability of concrete structure made of tested types of concrete was also specified.

2. Experimental plan, materials and mixture composition
Tests of chloride ions penetration under the influence of an electric field were planned in order to calculate diffusion coefficient value based on theoretical model presented in work [4]. Then the verification of calculated diffusion coefficient values were obtained through running tests in diffusion conditions. The forecast durability of concrete structure made of tested types of concrete was also specified. Similar procedure was used when the protective properties of SCC concretes with ISF slag [5] and in woks [6], [7], [8], [9] were compared. The tests on four types of concrete mix which differed in a type of used cement was performed. The following types of cement were applied: in concrete C1 - cement CEM I 42.5 R characterised by high heat of hydration, rapid increase in strength and high early strength. Due to these characteristics its use reduces production process of prefabricated elements, allows the increase in formwork rotation and decreases costs of energy consumption in the process of increasing speed of elements maturation. Besides making of prefabricated elements CEM I 42,5 R can be used to prepare BWW, SCC and AAC concretes and for use in road and bridge construction and communications engineering. CEM I 42,5 R if dosed in accordance with the standard [10] can be used in conditions of aggressive influence of environment on concrete and reinforcement except classes XA2 and XA3, i.e. chemical aggression caused by sulphate (except the ones of sea origin) [11].

In concrete C2 – cement CEM III/A 32.5 N-LH/HSR/NA characterised by high resistance to chemical aggression (particularly sulphate aggression), low heat of hydration, moderate dynamics of strength increase at the initial stage and satisfactory dynamics of strength increase during other stages, low shrinkage, lighter colour and very good workability. Use of this cement allows producing concrete or prefabricated elements with high leakproofness and resistance for chemically aggressive environment (especially for sulphate corrosion). It minimalises the risk of concrete cracking in case of the occurrence of reactive silica powder in aggregate while due to the low heat of hydration reduces the risk of shrinkage cracks what allows us to build massive constructions and increases their durability. This type of cement is designed to produce concretes and mortars in accordance with [8], also ones that are exposed to chemical aggression (i.a. to make floors in animal stables, manure pads, flues, retaining walls). It is also designed for production of concretes for constructions, monolithic elements, hydraulic constructions (e.g. dams), sewage plants, seeage barriers, tunnels, production of concretes for floors and architectural concrete [11].
In concrete C3 – cement CEM I 42.5 N/SR3/NA with high chemical resistance, particularly to sulphate corrosion, low content of Na₂O alkali, tricalcium aluminate C₃A and aluminium oxide Al₂O₃, standard dynamics of strength increase at the beginning and high strength during later stages. Cement CEM I 42.5 N/SR3/NA is designed for building bridges, roads and airports surfaces, hydraulic and industrial constructions, reinforced prefabricated elements exposed to chemically aggressive environments and animal stables. Use of this cement allows to produce concrete or prefabricated elements with high resistance for chemically aggressive environments, increases safety during and after construction works and minimises the risk of concrete cracking in case of the occurrence of reactive silica powder in aggregate, reduces the risk of shrinkage cracks which reduces the risk of corrosion [11].

In concrete C4 - cement CEM IV/B (V) 32.5 R – LH/NA characterised by a stable increase in strength, increased strength during longer stages of concrete curing, low heat of hydration which significantly reduces the risk of shrinkage cracks, good plasticity, workability and pumpability of concrete mix, reduced tendency towards the formation of carbonate blooms and stable quality parameters. Cement CEM IV/B (V) 32.5 R - LH/NA is designed to produce ordinary concrete classes from C8/10 to C30/37, mortar, self-compacting concrete, lean concrete, concrete used in soil stabilization, concrete used in massive constructions. The use of CEM IV/B (V) 32.5 R - LH/NA increases concrete and mortar resistance for chemically aggressive environments, improves leakproofness, allows to prepare most types of ordinary concrete, has good plasticity, workability, allows to transport the mixture due to prolonged workability, while due to special additives improves pumpability of concrete mix, has reduced tendency towards the formation of carbonate blooms, reduces the risk of shrinkage cracks and makes the production process easier because of its stability and versatility. Cement CEM IV/B (V) 32.5 R - LH/NA if dosed in accordance with the standard [8] can be used in concrete class X0 (no aggressive influence of environment), XC1, XC2 (corrosion caused by carbonatation), XA1, (corrosion caused by chemical aggression of natural soil and groundwater) [11].

| Table 1. Cement properties. |
|-----------------------------|
| Parameters                  | CEM I 42,5 R | CEM III/A 42,5 N-LH/HSR/NA | CEM I/ N/SR3/NA | CEM IV/B (V) 32,5 R – LH/NA |
| Constituent, % mass         |              |                            |                 |                            |
| SiO₂                        | 19.38        | 29.08                      | 21.15           | 30.18                      |
| Al₂O₃                       | 4.57         | 6.30                       | 3.93            | 11.92                      |
| Fe₂O₃                       | 3.59         | 1.37                       | 5.14            | 4.72                       |
| CaO                         | 63.78        | 48.82                      | 63.34           | 41.95                      |
| MgO                         | 1.38         | 4.36                       | 1.28            | 1.72                       |
| K₂O                         | 0.58         | 0.73                       | 0.39            | 1.43                       |
| Na₂O                        | 0.21         | 0.34                       | 0.21            | 0.39                       |
| Eq. Na₂O                    | 0.59         | 0.82                       | 0.47            | 1.33                       |
| Surface, cm²/g              | 3.26         | 2.74                       | 2.61            | 2.65                       |
| Density, g/m³               | 0.069        | 0.066                      | 0.058           | 0.057                      |
| Setting time, min           | 3535         | 4760                       | 3067            | 4457                       |
| Compression                 |              |                            |                 |                            |
| Beginning                   | 3.09         | 2.97                       | 3.18            | 2.65                       |
| End                         | 160          | 238                        | 210             | 200                        |
| Flexural strength, MPa      | 2 d          | 30.3                       | 14.9            | 24.7                       |
|                            | 28 d         | 56.9                       | 57.6            | 54.1                       |
| Water demand, %             | 27.6         | 31.6                       | 27.8            | 29.0                       |
The detailed chemical composition and properties of the used cements is given in Table 1. Three types of natural rounded aggregate were utilized for mix creation: sand, 0÷2 mm; gravel, 2÷8 mm; and gravel, 8÷16 mm. All created mixes were characterized by a water/cement ratio of 0.5. The detailed compositions of mixes are presented in Table 2. All created mixes were characterized by a water/cement ratio of 0.5. The detailed compositions of mixes are presented in Table 2.

### Table 2. Composition and compressive strength of concrete mixtures.

| Constituent                   | C1   | C2   | C3   | C4   |
|-------------------------------|------|------|------|------|
| Cement                        | 324.1|      |      |      |
| Aggregate                     | 1931.2|     |      |      |
| Water                         | 166.7|      |      |      |
| w/c                           | 0.5  |      |      |      |
| Compressive strength f<sub>cm</sub> | 54.2 | 49.5 | 58.4 | 46.5 |
| Volume weight γ<sub>b</sub>, kg/m³ | 2271 | 2269 | 2258 | 2280 |

### 3. Testing methods

In presented at work [4] the thermodynamic model of chlorine penetration into the concrete ion flows in the pore liquid were analyzed. Based on the expression determining the diffusion coefficient of chloride ions $D' = 1/Q$ – which is the reverse of the diffusion resistance of the entire tested concrete zone with the range

$$
D' = \frac{\bar{J}^{'1}(a)\Delta t}{\bar{z}'F\bar{U}_g\left[\rho_1^{-1} + \rho_2^{-1} + ... + \rho_n^{-1}\right] \Delta t - B}
$$

In this expression, $\bar{J}^{'1}(a)$ is the value of the mass flow of chloride ions passing through the plane situated at a distance $x = a$, $\bar{\rho}_i^{-1}$ are the averaged in time $\Delta t$, mass densities of ion $Cl^-$ at midpoints of consecutive intervals $[0, g]$, $[g, 2g]$, $...$, $[(n-1)g, a]$. The first component of the denominator defines the stationary part of the chloride ion flows, while the second component B – defines non-stationary part. In this expression, $z'$ is the ion valence, $R = 8.317$ J/mol·K – universal gas constant, $F = 96 487$ C/mol – the Faraday constant, $U$ – the voltage between the electrodes, $h$ – the specimen height. The stationary conditions correspond to the flows in a nearly ideal situation which can occur inside the solution. In the real concrete structure, the flow is disturbed by the electrostatic interactions that make the ion movements more difficult and the near surface phenomena, such as adsorption at pore walls, exchange with the solution and electroosmosis. The flow of diffusion is complicated by the processes of binding the chloride with the matrix elements of cement – mainly $Ca(OH)_2$ which leads to the formation of alkaline salt type of $Ca(OH)_2\cdot CaCl_2 \cdot 2H_2O$ and celite $C_3A$ that is transformed in so-called Friedel salt $3CaO\cdot Al_2O_3 \cdot CaCl_2 \cdot 10H_2O$ insoluble in alkaline medium. The changes caused by these processes are together expressed by the non-stationary influence [4].

According to the relation (1), the contribution of the non-stationary influence was estimated proportionally to the component expressing the stationary part. By increasing in sequence the proportionality factor by 0.1 within $\omega = 0.1 ÷ 0.5$ range, the value of the diffusion coefficient of chloride ions was estimated taking into account the non-stationary course of the migration – Table 11.
The reliable value of the diffusion coefficient $D^1_m$ was determined by comparing the distribution of the mass density of chloride ions calculated according to the well-known solution for the diffusion

$$\rho^1_{\text{cal}}(x,t) = \rho^1_{0,\text{cal}} \left( 1 - \frac{x}{2\sqrt{D^1_m t}} \right),$$

with, mass densities of these ions determined during the diffusion tests at time $t_3 = 90$ days and $t_4 = 180$ days. In the dependence (2), $\rho^1_{\text{cal}}$ is the calculated mass density of chlorides at the edge of the element, erf – the Gauss error function, $t$ – time.

To determine the most convergent calculated and experimental results, the mean square error was calculated

$$s = \sqrt{\frac{\sum_{i=1}^{n} [\rho^1_{\text{cal}}(x,t) - \rho^1(x,t)]^2}{n-1}},$$

between the expected values and the ones obtained from the diffusion tests at time $t_3 = 90$ days and $t_4 = 180$ days. In the above formula, $n$ means a number of experimental results.

For tests, 12 cylindrical specimens with a diameter of 100 mm and a height of 50 mm were prepared from concrete C1. And the same number of specimens were prepared from concrete C2, C3, C4 respectively. The tests started after three months from concreting. Containers with 3% solution of NaCl were tightly attached to the top surface of cylinders. Six samples made from each type of concrete were exposed to the electric field inducing the migration of chloride ions, whereas the diffusion of these ions, without the impact of the electric field, was observed in the remaining six ones made of any type of concrete. Ions were migrating concurrently in three specimens in parallel in the electric circuit. Before the tests, the specimens were kept in distilled water for 72 hours in order to improve the electrical conductivity of concrete. The soaked specimens were connected to the source of direct current at a voltage $U = 18$ V. The test was conducted in two time intervals: $t_1 = 24$ h and $t_2 = 48$ h. NaCl solution was replaced every 24 h. During the whole period of tests, the temperature of solution was constant – about 20 °C. During the tests on diffusion, three specimens made from any type of concrete were treated with chloride solution for $t_3 = 90$ days, the next three specimens for $t_4 = 180$ days. The level of NaCl solution was maintained at 150 mm over the surface of a specimen. After completing migration and diffusion measurements, the equipment elements were disassembled and the specimens were left under ambient conditions for 72 h. Subsequently, the specimens were ground layer by layer. The ground concrete was collected from consecutive depths of each specimen. Distilled water was added to the ground concrete in a 1:1 wt ratio. The dense solution was stirred every few hours and then passed through a filter after 24 h. The washing out procedure was repeated three times. Washing out was repeated two times [4]. The experimental solutions were subjected to chemical analysis, and the concentration $c^1$ of chloride ions in the solution was determined by argentometric titration according to the Mohr method [12].

4. Results and discussion

On the basis of measurements of mass density distribution $\rho^1$ of chloride ions migrating in concrete under the influence of the electric field, a reliable value of the diffusion coefficient was determined using the dependence (1). The results of calculations are summarized in table 3.
Table 3. The list of calculated results of concretes C1, C2, C3, C4 for the diffusion coefficient of ions Cl⁻

| Zone       | D₁₅s = 10⁻¹² [m²/s] | D₁₅ns = 10⁻¹² [m²/s] | Non-stationary influence |
|------------|----------------------|-----------------------|--------------------------|
|            |                      |                       | 10%  | 20%  | 30%  | 40%  | 50%  |
| C1         | 1.36                 | 1.49                  | 1.76 | 1.90 | 2.03 | 2.71 |
| C2         | 0.46                 | 0.50                  | 0.59 | 0.64 | 0.69 | 0.91 |
| C3         | 4.73                 | 5.20                  | 6.15 | 6.62 | 7.09 | 9.46 |
| C4         | 2.25                 | 2.48                  | 2.93 | 3.16 | 3.38 | 4.51 |

In Figure 1, the values of the calculated theoretical diffusion coefficient (D₁) of tested concretes are presented. The values of the diffusion coefficient achieved by the theoretical model (based on migration results) range from 0.46x10⁻¹² m²/s to 4.73x10⁻¹² m²/s. Concrete C2 (with CEM III/A 42,5 N-LH/HSR/NA addition) is characterized by the smallest value of diffusion coefficient. Concrete C1 (with CEM I 42,5 R addition) is characterized by 296% higher diffusion coefficient than the composite C2. In case of concrete C4 (with CEM IV/B (V) 32,5 R – LH/NA addition), the difference reaches to 465%. In case of concrete C3 (with CEM I/ N/SR3/NA addition), the difference reaches to 1028%. One has to keep in mind that the larger the value of diffusion coefficient, weaker the protective properties of concrete coating of rebars.

Figure 1. Values of diffusion coefficient achieved by the theoretical model (based on migration results)

In Figure 2, the values of distribution of mass densities of chlorides ρ₁ achieved through real diffusion (after t₃ = 90 days and t₄ = 180 days) and calculated using the theoretical model are presented. Theoretically calculated distributions of mass densities of chlorides were obtained using the values of diffusion coefficient presented in Figure 1.

Figure 2a illustrates the calculated and experimental distributions of the mass density of chloride ions in concrete C1. A dotted line indicate the results calculated on the assumption that the diffusion coefficient corresponds to the stationary conditions. Whereas the solid lines indicates the results calculated on the assumption that the diffusion coefficient Dₙₐ = 1.49·10⁻¹² m²/s is reliable at the time t₃ = 90 days the diffusion coefficient Dₛ = 1.36·10⁻¹² m²/s is reliable at the time t₄ = 180 days.
Figure 2c illustrates the calculated and experimental distributions of the mass density of chloride ions in concrete C3. A dotted line indicates the results calculated on the assumption that the diffusion coefficient corresponds to the stationary conditions. Whereas the solid lines indicate the results calculated on the assumption that the diffusion coefficient $D_{ns} = 5.20 \cdot 10^{-12} \text{ m}^2/\text{s}$ is reliable at the time $t_3 = 90$ days the diffusion coefficient $D_s = 4.73 \cdot 10^{-12} \text{ m}^2/\text{s}$ is reliable at the time $t_4 = 180$ days.

In the case of C2 and C4 concrete, the reliable values of diffusion coefficients are equal to those obtained at steady-state both after the 90 days and 180 days of testing - Figures 2b and 2d.

The protective properties of concrete coating of rebars were determined by calculating the increase of the concentration of chloride ions. It was assumed that the cast element is in a form of a slab with concrete coating of $x_1 = 20$ mm and $x_2 = 40$ mm. The critical value of 0.4% (by the weight of cement) of chloride ion concentration was adopted as a corrosion trigger. The calculations of mass density of chloride ions at the contact zone of steel reinforcement with a concrete coating were conducted according to a well-known equation for the diffusion (4).

Subsequently, the mass density of chloride ions $\rho_1$ was a point of reference for a constant mass density of cement $\rho_{cem}$ in tested concretes, to establish the concentration $c_1$ of chloride ions:

![Figure 2](image-url)
\[ c^1(t) = \frac{\rho^1(x = (20;40),t)}{\rho_{cem}} \times 100\% \]  

On the edge of the concrete, the chloride concentration \( c^1 = 0.8\% \) (by weight of cement) was adopted. In Figure 3, the estimated technical life span of concretes structures is presented. Comparing the value of the estimated chloride concentration to its critical value, when the corrosion is triggered, allows defining the expected technical life span of the concretes structure. It should be noted that the concrete structure made out of mix C3 - with CEM I/ N/SR3/NA addition, (characterized by diffusion coefficient \( D^1 = 4.73 \times 10^{-12} \text{ m}^2/\text{s} \)) would be the most prone to corrosion. Only after 3 years and 12 years of service, the corrosion may occur in structures with concrete coating of 20 and 40 mm, respectively. The concrete structure made out of mix C2 - with CEM III/A 42,5 N-LH/HSR/NA addition, (characterized by diffusion coefficient \( D^1 = 0.46 \times 10^{-12} \text{ m}^2/\text{s} \)) would be the most resistant to the penetration of chloride ions. In this case of concrete, the corrosion may occur after 32 and 122 years of operational use for concrete coating of 20 and 40 mm, respectively. According to European standard EN 1992-1-1, newly erected concrete structures should be characterized by durability that allows 50 years of service. In the case of tested concretes, only one mix (concrete C2 with CEM III/A 42,5 N-LH/HSR/NA addition) fulfill this requirement.

**Figure 3.** Time dependent changes of chloride concentration in concrete-steel contact zone for two thicknesses (\( x_1 \), and \( x_2 \)) of concrete C1, C2, C3, C4 coatings of steel reinforcement

5. Conclusions

Among analysed concretes, C2 which is made with CEM III/A 42,5 N-LH/HSR/NA cement has the best protective properties against chloride ions penetration. Next one is C1 concrete, made with standard Portland cement CEM I 42,5 R. Next is concrete C4 made with CEM IV/B (V) 32,5 R – LH/NA. The weakest protective properties characterise concrete made with cement CEM I/ N/SR3/NA. Concrete C1 made with CEM I 42,5 R is almost three times worse than C2 made with CEM III/A 42,5 N-LH/HSR/NA. While C4 with cement CEM IV/B (V) 32,5 R – LH/NA is five times less resistant for chlorides penetration than concrete C2. The worst protective properties has concrete C3 with cement CEM I/ N/SR3/NA and the are over ten times worse than protective properties of concrete C2.

This protective properties influence the forecast durability of reinforced concrete structure made of tested types of concrete. With the coating thickness of 20 (40) mm time after which chloride ions concentration near the surface reaches critical value of 0,4% of the mass of cement that is contained in concrete is: 3 (12) years in concrete C3, 7 (27) years in concrete C4, 11(44) years in concrete C1 and 32(122) years in concrete C2.
What is more, the best resistance for chemically aggressive environments i.a. sulphate corrosion characterises blast furnace cements CEM III, i.a. CEM III/A 32,5N-LH/HSR/NA, CEM III/A 42,5N-HSR/NA or CEM III/A 52,5N-NA.

Making the right choice of cement requires not only the knowledge about its basic properties like: constancy of volume, time of setting, speed of strength increase, exotherm of setting and hardening but also knowledge about protective properties against chemical sulphate and chloride aggression.

The best way to avoid damage of concrete that is caused by reaction between alkali and reactive silica powder is avoiding the use of reactive aggregate. If it is not possible low alcali NA cement should be used. Based on obtained results cement CEM III/A 42,5 N-LH/HSR/NA is the best to use in constructions exposed to aggressive environment.

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