A R C H I T E C T U R E C I V I L E N G I N E E R I N G

The Silesian University of Technology



ANALYSIS OF CHLORIDE DIFFUSION AND MIGRATION IN CONCRETE PART II – EXPERIMENTAL TESTS

ENVIRONMENT

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Abstract

The theoretical model of chloride ion penetration into the concrete under the electric field and the diffusion coefficient of these ions determined on the basis of the converse task of the migration equation were presented in the first part of the paper. Now, the theoretical results are referred to our own laboratory studies. Taking into account experimentally obtained distribution of chloride concentrations under the electric field, the diffusion coefficient was determined. The distribution of the chloride concentrations was calculated on the basis of that coefficient. And then, it was compared with the measurement results for concentrations of chlorides penetrating into the concrete via diffusion. The lowest mean square error between the experimental and calculated distributions of mass densities indicated the reliable value of the chloride diffusion coefficient.

Streszczenie

W pierwszej części pracy przedstawiono model teoretyczny wnikania jonów chlorkowych do betonu pod wpływem działania pola elektrycznego oraz na podstawie zadania odwrotnego równania migracji określono współczynnik dyfuzji tych jonów. Obecnie wyniki teoretyczne nawiązano do własnych badań laboratoryjnych. Uwzględniając doświadczalnie otrzymany pod wpływem pola elektrycznego rozkład stężenia chlorków wyznaczono współczynnik dyfuzji. Na podstawie tego współczynnika obliczono rozkład stężenia chlorków, który porównano z wynikami pomiaru stężenia chlorków wnikających do betonu dyfuzyjnie. Najmniejszy błąd średniokwadratowy między rozkładami gęstości masy uzyskanymi doświadczalnie i obliczeniowo wskazał miarodajną wartość współczynnika dyfuzji chlorków.

Keywords: Experimental tests; Diffusion of chlorides; Migration of chlorides in the electric field; Reliable diffusion coefficient.

1. COURSE AND RESULTS OF STUDIES

The experimental studies on the penetration of chloride ions under the electric field and diffusion were planned so as to use the obtained results to calculate the diffusion coefficient on the basis of the theoretical solution presented in the paper [1] and verify the calculated values.

For tests 12 cylindrical specimens with a diameter of 100 mm and a height of 50 mm were prepared from concrete class C12/15 with w/c = 0.6. The concrete

mixture was made of Portland cement CEM I 32.5R (233.5 kg/m³), mineral aggregate with the fraction $0 \div 8 \text{ mm}$ (2059.5 kg/m³) and tap water (147.5 l/m³). The chemical composition of cement is shown in Table 1.

Lateral surfaces of the specimens were covered with epoxy resin in order to provide one-way flow of chloride ions. The tests started after three months from concreting.

Containers with 3% solution of NaCl were tightly

Table 1. The results of chemical analysis of cement "S.A. Górażdże Cement" CEM I 32.5 R Class

Lp.	Component	Content [%]
1	2	3
1.	Moisture in 105°C	0.31
2.	Loss on calcination in 900°C	2.25
3.	SiO ₂	19.14
4.	Al ₂ O ₃	6.37
5.	Fe ₂ O ₃	3.07
6.	CaO	59.55
7.	MgO	4.30
8.	SO ₃	3.9
9.	The rest of this: K ₂ O i Na ₂ O	1.11
	TOTAL	100



attached to the top surface of cylinders. Six specimens 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.

Ions were migrating concurrently in three specimens in parallel in the electric circuit illustrated in Fig. 1. The stainless steel cathode 2 with its size adjusted to the section of the tested element was placed in the tank 1. The elements were placed on the wet sponge with platinum plated titanium mesh anode 5 underneath. Before the tests, the specimens were kept in distilled water for 72 hours in order to improve the electrical conductivity of the concrete. The soaked specimens were connected to the source of direct current 6 at a voltage U = 18 V.

The duration of chlorides migration in three specimens was $t_1 = 24$ hours, whereas in other three specimens $t_2 = 48$ hours. NaCl solution was replaced every 24 hours. During the whole period of tests, the temperature of solution was constant, at about 20°C. During the tests on diffusion, three specimens were

treated with chloride solution for $t_3 = 180$ days, and other three specimens for $t_4 = 360$ days. The solution was maintained a the same level equal to 60 mm above the concrete surface.

After completed migration and diffusion, the equipment elements were disassembled, and the specimens were left under ambient conditions for 72 hours. Then, the grinded concrete was collected by layers of thickness g = 2 mm from 9 consecutive depths of each specimen with "Profile Grinding Kit" of German Instruments AS company. The grinded concrete from corresponding three similar specimens was combined to form the representative material for averaged values. The water extracts were made from this material.

Distilled water was added to the grinded concrete in a 1:1 weight ratio. The dense solution was stirred every several hours per 24 hours, and then passed through a medium sized filter. The filtration was supported by vacuum from the suction flask connected with the electric pump. About $20 \div 50 \text{ cm}^3$ of filtered liquid was obtained from each portion of the dense solution. Washing out was repeated three times.

The experimental solutions were subjected to chemical analysis and the concentration $c^1 \text{ [mg/dm^3]}$ of chloride ions in the liquid was determined by argentometric titration according to the Mohr method – Tables 2 and 3. Argentometric method allows to obtain correct results in the environment with a pH = $6.5 \div 10$. Since the analyzed water extracts were characterized by a pH = $9.09 \div 11.90$, so before titration as recommended by the standard [2] solutions was adjusted to pH = $7.0 \div 9.0$ by adding a small amount of sulfuric acid.

As the grinded concrete was washed out with water three times in a 1:1 weight ratio, volume V_s of the experimental liquid was estimated on the basis of water volume weight $\gamma_w = 1 \text{ kg/dm}^3$

$$V_{s} = \frac{m_{s}}{\gamma_{w}} = \frac{3m_{c}}{\gamma_{w}},$$
 (1)

where m_s is the mass of the solution, m_c – the mass of grinded concrete.

The mass m^1 of chloride ions in volume V_s of the solution was determined from the relation

$$m^{1} = \frac{3c^{1}m_{c}}{\gamma_{w}}.$$
 (2)

Taking into account unavoidable losses of the material during concrete grinding, the volume V_c of the

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Table 2.	
Concentration c^1 in the experimental solution and mass density ρ^1 in concrete - migration of ions Cl^- under the electric field	

Location acc. to	of layers Fig. 2	Cl ⁻ concentrati [mg/	on in solution c ¹ dm ³]	Density of mass C [kg	cl^{-} in concrete ρ^{1} /m ³]
Interval [mm]	Interval [mm] Comp. coordinate [mm]		t ₂ = 48 h	$t_1 = 24 h$	t ₂ = 48 h
0 ÷ 2	1	1260.96	1510.00	9.57	11.46
$2 \div 4$	3	702.72	1278.20	5.33	9.70
4 ÷ 6	5	314.88	1146.3	2.39	8.70
6 ÷ 8	7	184.32	1018.45	1.40	7.73
8 ÷ 10	9	165.12	898.45	1.25	6.82
10 ÷ 12	11	157.44	874.50	1.19	6.64
12 ÷ 14	13	130.56	867.80	0.99	6.59
14 ÷ 16	15	138.24	859.34	1.05	6.52
16 ÷ 18	17	134.40	858.90	1.02	6.52

Table 3.

Concentration c^1 in the experimental solution and mass density ρ^1 in concrete – diffusion of ions Cl^-

Location acc. to	of layers Fig. 2	Cl ⁻ concentrati [mg/	on in solution c ¹ dm ³]	Density of mass C [kg	cl^{-} in concrete ρ^{1} /m ³]
Interval [mm]	Comp. coordinate [mm]	$t_3 = 180 \text{ days}$	$t_4 = 360 \text{ days}$	$t_3 = 180 \text{ days}$	$t_4 = 360 \text{ days}$
0 ÷ 2	1	2138.9	2142	16.23	16.26
2 ÷ 4	3	937.6	1937.6	7.12	14.71
4 ÷ 6	5	768	1768	5.83	13.42
6 ÷ 8	7	595.2	1595.2	4.52	12.11
8 ÷ 10	9	687.4	1387.2	5.22	10.53
10 ÷ 12	11	695	1275.2	5.28	9.68
12 ÷ 14	13	625.9	1265.4	4.75	9.60
14 ÷ 16	15	652.8	1252.2	4.95	9.50
16 ÷ 18	17	541.4	1243.6	4.11	9.44

concrete was determined on the basis of its volume weight γ_{c}

$$V_{c} = \frac{m_{c}}{\gamma_{c}}, \qquad (3)$$

and then, the mass density ρ^1 of chloride ions was quantified

$$\rho^{1} = \frac{m^{1}}{V_{c}} = \frac{3c^{1}\gamma_{c}}{\gamma_{w}}.$$
(4)

During the accompanying tests, the volume weight of concrete was quantified $\gamma_c = 2530 \text{ kg/m}^3$. The quantification results for the mass density ρ^1 of chloride ions migrating in the concrete specimens exposed to the electric field are presented in Table 2, whereas the similar results for diffusion of these ions are listed in Table 3.

2. QUANTIFICATION OF THE DIFFU-SION COEFFICIENT OF IONS CI⁻

On the basis of the distribution measurements of mass density ρ^1 of chloride ions migrating in the concrete under electric field, the reliable value of the diffusion coefficient was obtained using the relation derived in the paper [1]

$$D^{1} = \frac{\overline{j}^{1}(a)a\Delta t}{\frac{z^{1}FUg}{RTh}\left[\overline{\rho}_{1}^{1} + \overline{\rho}_{2}^{1} + \dots + \overline{\rho}_{n}^{1}\right]\Delta t - \int_{0}^{a}Q_{x}\left[\rho^{1}(x, t + \Delta t) - \rho^{1}(x, t)\right]dx},$$
(5)

where $\overline{j}^{-1}(a)$ is the averaged in time t value of the mass flux of chloride ions flowing through the plane situated at a distance x =a from the specimen edge, $\overline{\rho}_{1}^{-1}, \overline{\rho}_{2}^{-1}$, and $\overline{\rho}_{n}^{-1}$ are averaged in time t mass densities of ion Cl⁻ in individual sampling depths of the material. The first term of the dominator determines the stationary part of chloride ion flows, whereas the second one refers to the non-stationary part.



Computational scheme: a) mass density in isolated layers, b) computational zones

At first, experimental results were grouped into a computational zone I with a = 8g = 16 mm range that included eight sampling depths of the material – Fig. 2. Fig. 2a illustrates the distribution of the mass density of chlorides in consecutive sampling depths. Results obtained during the migration duration $t_1 = 24$ hours are marked with light colour, whereas dark colour indicates the results for the migration duration duration $t_2 = 48$ hours.

Taking into account the averaged mass densities of chloride ions in individual sampling depths

n = 1, 2, ..., 8

$$\bar{\rho}_{n}^{1}(t) = 0.5 \rho_{n}^{1}(t), \quad t = t_{1} = 24 \text{ h}, \quad t = t_{2} = 48 \text{ h}, \quad (6)$$

the averaged value of the mass density in the whole computational zone I was determined

$$\bar{\rho}_{1}^{1}(t) = 0,5[\rho_{1}^{1}(t) + \rho_{2}^{1}(t) + ... + \rho_{8}^{1}(t)], \quad t = t_{1}, \quad t = t_{2}.$$
(7)

The averaged mass flux of chloride ions flowing out from the boundary area of the computational zone I was determined on the basis of the definition, and the mass density ρ_9^1 of chloride ions present in the last layer n = 9 outside the zone I was calculated

$$\bar{j}^{1}(a) = \frac{m^{1}}{At} = \frac{g}{t}\rho_{9}^{1}, \quad t = t_{1}, \quad t = t_{2},$$
 (8)

where m¹ is the mass of chlorides flowing through the section of the area A at time t.

For the dominator of the expression (5), the term describing the non-stationary part of chloride ion flows is assumed to be proportional to the term expressing the stationary course

$$\int_{a}^{a} Q_{x} \left[\rho^{1}(x, t + \Delta t) - \rho^{1}(x, t) \right] dx \cong$$

$$\cong \omega \frac{z^{1} FUg}{RTh} \left(\overline{\rho}_{1}^{1} + \overline{\rho}_{2}^{1} + \dots + \overline{\rho}_{n}^{1} \right) \Delta t, \qquad (9)$$

where ω is the proportionality factor.

The conditions similar to the stationary ones ($\omega = 0$) were assumed and the expression (5) was reduced to

$$D_s^{I} = \frac{\overline{j}^{I}(a)a}{\frac{z^{I}FUg}{BTh}\overline{\rho}_{I}^{I}(t)}.$$
 (10)

The quantification results are presented in Table 4.

By analysing the next computational zone II with a = 7g = 14 mm range, the averaged value of the mass density of chloride ions present in it were quantified

$$\bar{\rho}_{II}^{I}(t) = 0.5 \left[\rho_{1}^{I}(t) + \rho_{2}^{I}(t) + \dots + \rho_{7}^{I}(t) \right], \quad t = t_{1}, \quad t = t_{2}, \quad (11)$$

along with the averaged mass flux of these ions at the boundary area of the zone II

$$\bar{j}^{1}(a) = \frac{g}{t}(\rho_{8}^{1} + \rho_{9}^{1}), \quad t = t_{1}, \quad t = t_{2}, \quad (12)$$

which is expressed by the mass density of ions Cl^- present in layers n = 8 and n = 9 outside the zone II. Then, the diffusion coefficient under stationary conditions was quantified

$$D_{s}^{I} = \frac{\overline{j}^{I}(a)a}{\frac{z^{I}FUg}{RTh}\overline{\rho}_{II}^{I}(t)}.$$
(13)

The calculations on the basis of consecutively isolated zones $III \div VIII$ were performed analogously. The quantification results are presented in Table 4.

The diffusion coefficient of chloride ions quantified

		1	-100.000	D^{1} 1012			$D_{ns}^{1} \cdot 10^{12} [m^{2}/s]$		
Zone	t [h]	ρ ¹ [kg/m ³]	$j^{-1}(a) \cdot 10^{6}$ [kg/m ² s]	$[m^2/s]$		Non	-stationary influ	ience	
				1 1	10%	20%	30%	40%	50%
т	24	11.59	2.36	1.14	1.26	1.49	1.60	1.94	2.29
1	48	32.08	7.55	1.32	1.45	1.72	1.85	2.24	2.64
п	24	11.07	4.79	2.13	2.34	2.76	2.98	3.61	4.25
11	48	28.82	15.09	2.57	2.83	3.34	3.60	4.37	5.14
	24	10.57	7.08	2.82	3.10	3.67	3.95	4.79	5.64
111	48	25.52	22.72	3.75	4.12	4.87	5.24	6.37	7.49
117	24	9.97	9.85	3.46	3.81	4.50	4.85	5.89	6.93
IV	48	22.21	30.40	4.80	5.28	6.24	6.72	8.16	9.60
N 7	24	9.35	12.75	3.83	4.21	4.98	5.36	6.51	7.66
v	48	18.80	38.29	5.72	6.29	7.43	8.00	9.72	11.43
X / I	24	8.65	15.99	3.89	4.28	5.06	5.45	6.62	7.78
VI	48	14.93	47.24	6.66	7.32	8.66	9.32	11.32	13.32
1/11	24	7.45	21.52	4.05	4.46	5.27	5.67	6.89	8.10
VII	48	10,58	57.31	7.60	8.36	9.88	10.64	12.92	15.20
VIII	24	4.79	33.87	4.96	5.46	6.45	6.95	8.44	9.93
	48	5.73	68.54	8.39	9.23	10.91	11.75	14.26	16.78
		Mean value	5^{1}	4.19	4.61	5.45	5.87	7.12	8.38

Table 4.					
The list of calculated	results for t	he diffusion	coefficient of	of ions C	1-

under the stationary conditions was the lowest $D_s^1 = 1.14 \cdot 10^{-12}$ m²/s when the analysis was conducted on the basis of the isolated zone I with a = 16 mmrange and the migration time $t_1 = 24$ h was included. The of highest value this coefficient $D_s^1 = 8.39 \cdot 10^{-12} \text{ m}^2/\text{s}$ was determined by calculations according to the scheme including the zone VIII of a = 2 mm and the migration time $t_2 = 48 \text{ h}$. As there was not a strong tendency indicating the reliable result, the arithmetic mean of the diffusion coefficient value was determined on the basis of the results from the analysis conducted in all computational zones at both migration times - Table 4.

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 electro-osmosis. 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 $Ca(OH)_2$ ·CaCl₂·2H₂O and celite C₃A that is transformed in so-called Friedel salt 3CaO·Al₂O₃·CaCl₂·10H₂O insoluble in alkaline medium [3]. The changes caused by these processes are together expressed by the non-stationary influence.

According to the relations (5) and (9), 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 $\omega = 0.1 \div 0.5$ range, the value of the diffusion coefficient of chloride ions D_{ns}^1 was estimated taking into account the non-stationary course of the migration – Table 4.

The comparison of calculated values of the diffusion coefficient indicates the importance of the non-stationary influences. Assuming that the non-stationary term in the dominator of the expression (5) constitutes 10% of the component expressing the stationary process resulted in a slight increase of the coefficient by $11\% - D_{ns}^1 = 1.1D_s^1$. However, the non-stationary influence of 50% determined in the same way caused

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Table 5.

Calculated results for mass density ρ^1_{cal} of chlorides and mean square error s between these values and the results of the diffusion tests ρ^1

$ar{D}^1 \cdot 10^{12}$ [m ² /s]	t [days]				Coordina	te x [mm]				s
	v [uujo]	3	5	7	9	11	13	15	17	
4.10	180	9.12	7.61	6.21	4.95	3.86	2.93	2.17	1.57	1.75
4.19	360	16.20	14.38	12.63	10.97	9.42	7.99	6.70	5.55	1.71
4.61	180	9.22	7.78	6.43	5.20	4.12	3.19	2.42	1.79	1.69
4.01	360	16.33	14.59	12.91	11.31	9.80	8.41	7.14	5.99	1.55
5 45	180	9.40	8.06	6.79	5.63	4.58	3.66	2.87	2.20	1.65
5.45	360	16.54	14.93	13.37	11.87	10.45	9.12	7.89	6.76	1.43
5.97	180	9.48	8.18	6.95	5.81	4.78	3.86	3.07	2.39	1.66
5.67	360	16.63	15.08	13.57	12.11	10.73	9.43	8.21	7.10	1.45
7.12	180	9.66	8.47	7.34	6.27	5.29	4.40	3.61	2.91	1.73
7.12	360	16.85	15.43	14.05	12.71	11.42	10.20	9.05	7.97	1.67
0.20	180	9.80	8.70	7.64	6.64	5.70	4.84	4.06	3.36	1.87
0.30	360	17.01	15.71	14.42	13.18	11.97	10.81	9.71	8.67	1.97

a 2-fold increase in the value of the diffusion coefficient $(D_{ns}^1 = 2.0 D_s^1)$.

The reliable value of the diffusion coefficient D_{ns}^1 was determined by comparing the distribution of the mass density of chloride ions calculated according to the well-known solution for the diffusion equation

$$\rho_{cal}^{l}(x,t) = \rho_{0,cal}^{l}\left(1 - \operatorname{erf} \frac{x}{2\sqrt{D_{ns}^{l}t}}\right), \quad t = t_{3}, \quad t = t_{4}, \quad (14)$$

with, previously presented in Table 3, mass densities of these ions $\rho^1(x, t)$ determined during the diffusion tests at time $t_3 = 180$ days and $t_4 = 360$ days. In the dependence (14), $\rho^1_{0,cal}$ is the calculated mass density of chlorides at the edge of the element, erf – the Gauss error function, t – time.

The method described in the paper [4] was used to determine boundary values of the mass density $\rho_{0,cal}^1 = 11.5 \text{ kg/m}^3$ at time $t_3 = 180$ days and $\rho_{0,cal}^1 = 19.0 \text{ kg/m}^3$ at time $t_4 = 360$ days.

On the basis of the dependence (14), the value of the mass density ρ_{cal}^1 in points at the same location as the coordinates of layers used in the experimental tests was calculated taking into account the mean values of the diffusion coefficient \overline{D}^1 .

To determine the most convergent calculated and experimental results, the mean square error [5] was calculated

$$s = \sqrt{\frac{\sum_{i=1}^{n} \left[\rho_{cal}^{1}(x,t) - \rho^{1}(x,t) \right]^{2}}{n-1}}, \quad t = t_{3}, \quad t = t_{4}, \quad (15)$$

between the expected values $\rho_{cal}^1(x, t)$ and the ones obtained from the diffusion tests $\rho^1(x, t)$ at time $t_3 = 180$ days and $t_4 = 360$ days.

In the above formula, n means a number of experimental results. In accordance with the recommendations [6], the value of the mass density of chlorides ρ^1 corresponding to the coordinate x = 1 mm was excluded while calculating the mean square error s. The quantification results are presented in Table 5.

The analysis of calculated and experimental results revealed that in both cases regarding the chloride ion diffusion lasting for $t_3 = 180$ days and $t_4 = 360$ days, the lowest value of the mean square error s was obtained at the diffusion coefficient $\overline{D}_{ns}^1 = 5.45 \cdot 10^{-12} \text{ m}^2/\text{s}$ which was estimated taking into account 20% of the non-stationary influence. The indicated value of the diffusion coefficient can be regarded as reliable because the calculated distributions of the concentration $\rho_{cal}^1(x, t)$ were the closest to the experimental ones $\rho^1(x, t)$.

Figure 3 illustrates the calculated and experimental distributions of the mass density of chloride ions. Dashed line indicates the results calculated on the assumption that the diffusion coefficient \bar{D}_s^1 =4.19·10⁻¹² m²/s corresponds to the stationary conditions, whereas the solid line indicates the results calculated on the assumption that the diffusion coefficient \bar{D}_{ns}^1 =5.45·10⁻¹² m²/s is reliable. The attention



Experimental and calculated distributions of mass densities of chlorides. Description in the text

should be paid to a discrepancy between the measured and calculated values in points at a largest distance from the boundary (x = 15 mm, x = 17 mm) which could be caused by the imperfect course of the studies, and particularly by the difficulties in maintaining the constant conditions for the actions of chloride solution on the concrete specimens over a long period of time, that is, the calendar year.

3. CONCLUSIONS

The diffusion coefficient of chloride ions in the concrete is the most frequently determined on the basis of the long-lasting experimental studies justified by Fick's laws. Such tests are considerably difficult to perform in case of high performance concrete. That is why an attempt has been recently made to apply the electric field, whose action still is not satisfactorily explained, into the evaluation of the resistance against the chloride ion penetration.

This paper, preceded by the article [1], presents the consistent theoretical and experimental model of chloride ion migration under the electric field in the concrete and the method of determining the reliable value of the diffusion coefficient of these ions. The theoretical description includes both ions Cl⁻ and the presence of the main components of the pore liquid solution - ions OH-, Na+, K+, Ca²⁺ and the inert concrete skeleton along with the aqueous solution [1]. The flow processes were expressed by the global mass balance of all components of the representative volume element isolated from the concrete. The diffusion equation was derived on the basis of the simplifying assumptions of the centre of gravity of the representative volume element, the lack of chemical reactions, and according to Fick's first law, the simplest form of material constants. Electric properties of ionic components of the pore solution were expressed by the electrostatic forces proportional to the intensity of the electric field. After taking into account the relations defined by Faraday's and Ohm's laws, the migration equation for all main ions in the solution filling the concrete pores under the applied voltage was derived. The converse task of the migration equation, limited to only one component, was formulated in order to determine analytically the diffusion coefficient of chloride ions. ENGINEERING

C I V I L

The experimental studies, planned in accordance with the theoretical solution presented in the first part of the article [1], were conducted for the concrete specimens to determine the distributions of chloride concentration induced by the migration in the electric field and diffusion under original conditions. The diffusion coefficient of chloride ions including the non-stationary influence was determined on the basis of these studies and the theoretical solution.

The performed analysis indicated that the short-lasting studies on the migration in the electric field could be used to determine the reliable value of the diffusion coefficient of the chloride ions, and then to calculate the concentration distributions of those ions in the concrete. The presented method can be useful to evaluate the permeability of chlorides through the high performance concrete, whose compact structure makes the performance of diffusion tests difficult.

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