



Article Experimental Study on Effective Chloride Diffusion Coefficient of Cement Mortar by Different Electrical Accelerated Measurements

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Abstract: This study investigated the effective chloride diffusion coefficient of cement mortar with different water-to-cement ratio (w/c) under electrical accelerated migration measurement. The cumulative chloride concentration in anode cell solution and the cumulative chloride concentration drop in the cathode cell solution was measured by RCT measurement and the results were further used to calculate the chloride diffusion coefficient by Nordtest Build 355 method and Truc method. The influence of w/c on cement mortar's chloride coefficient was investigated and the chloride diffusion coefficient under different determination methods were compared with other researchers' work, a good consistency between this work's results and literatures' results was obtained. The results indicated that the increased w/c of cement mortar samples will have a higher chloride diffusion coefficient. The cumulative chloride concentration drop in the cathode cell solution will have deviation in early stage measurement (before 60 h) which will result in overestimation of the effective chloride diffusion coefficient.

Keywords: effective chloride diffusion; cement mortar; electrical accelerated test; water-to-cement ratio (w/c)

1. Introduction

The resistance of cementitious materials against chloride contamination is one of the most crucial design parameters for reinforced concrete structures. The passive film of steel rebar would break down when chloride ions reach its surface and exceed the threshold and induce corrosion and cracking of concrete structures [1]. It has been widely acknowledged that the chloride diffusion coefficient is the primary factor in the normal chloride-bearing corrosive environment [2–4]. In order to reliably predict the service life and improve the durability of concrete structures, it is of importance to determine the chloride diffusion coefficient of cementitious materials precisely.

Over the past several decades, different chloride diffusion coefficient measurement methods have been proposed including 90 days ponding test (AASHTO T259 [5]), and bulk diffusion test (ASTM C1556 [6]), these traditional chloride diffusion coefficient measurements are time-consuming, labor-intensive, and/or prone to measurement errors when practically assessed in the sites. Electrical accelerated chloride diffusion coefficient methods have also been developed such as the rapid test methods (NEL) [7], rapid chloride migration test (RCM) [8] and modified non-contact electrical resistivity measurement (MN-CM) [9,10]. In comparison to the above-mentioned traditional methods, electrical accelerated measurement has the advantages of short testing duration, convenient operation, and high adaptability to the various assessment conditions [11]. There are some other commonly used rapid tests which could provide an indication of the chloride transport resistant of cementitious materials such as electrical migration based test (NT Build 492 [12]) and a



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). steady state chloride conduction test [13]. All of which aforementioned methods can be used to determine the chloride resistance of cementitious materials, indirect relationships might exist between different chloride diffusion coefficient measurements' results [14], even though the results from different methods cannot be directly compared [15,16].

The interconnected pore network of concrete can transport the ionic species i in the pore solution which follows the Nernst-Plank equation as expressed in Equation (1) [17]

$$-J_i = D_i \frac{\partial C_i}{\partial x} + \frac{Z_i F}{RT} D_i C_i \frac{\partial E}{\partial x} - C_i v_i$$
(1)

where J_i is the ionic flux of species $i \pmod{\text{cm}^2/\text{s}}$, D_i is the effective diffusion coefficient of species i in the concrete (cm²/s), C_i is the concentration of species i in the pore solution as a function of location $x \pmod{\text{cm}^3}$, Z_i is the valence and v_i is the convection velocity of the ionic species $i \pmod{\text{s}}$, F is Farady's constant (96487 C/mol), R is the universal gas constant (8.314 J/mol/K), T is the absolute temperature (K) and E is the electrical voltage (V).

Each item in Equation (1) has a specific transportation mechanism, in which is the ion movement under the concentration gradient which is controlled by Fick's first law, the ion movement driven by the electrical potential and the ion convection under the pressure gradient, density difference of fluid etc.. For electrical accelerated chloride diffusion test, the concentration gradient and pressure gradient are negligible, and the movement of chloride ion is only controlled by electrical field. Adopting with the aforementioned assumption, the Equation (1) can be rewritten as Equation (2) [17]:

$$-J(x) = \frac{ZF}{RT} D_{eff} c \frac{\partial E(x)}{\partial x}$$
(2)

The chloride diffusion coefficient of electrical accelerated test methods can be calculated by solving Equation (2).

In electrical accelerated chloride diffusion measurement, the chloride ions will be transported from cathode cell to anode cell under electrical field. Thus, the chloride ion concentration change in anode and/or cathode cell can be an indication of chloride diffusion coefficient. Nordtest Build 355 [18] adopted the electrical accelerated chloride migration measurement and the effective chloride diffusion coefficient (D_a) can be calculated by measuring the cumulative anode chloride concentration as in Equation (3) which is derived from Equation (2) [19]

$$D_a = \frac{RTLV_a}{ZFEc_0A} \cdot \frac{\Delta c_a}{\Delta t}$$
(3)

where *L* is the thickness of slice sample (0.05 m); V_a represents the volume of anode cell (2.1 L); E is the applied voltage (22 V); c_0 is the initial chloride ion concentration in cathode cell (52.6 g/L); *A* is the cross section area of the slice sample (6.36 × 10⁻³ m²). $\Delta c_a / \Delta t$ is the slope of the cumulative chloride ion concentration in anode cell versus time curve.

NT Build 355 method assumes that when chloride ion transport through the sample and reaches the anode cell, it is in steady-state diffusion. For some low-permeability cementitious materials, it might require couple of days for chloride ions to transport through the sample and reach the anode cell as reported in literature [20]. Truc et al. [21,22] proposed that the chloride concentration drop in cathode cell can be used to calculate the effective chloride diffusion coefficient since the chloride will penetrate into the sample at the beginning of the test and the chloride ion concentration flux in anode cell is constant and therefore independent of the interaction between chloride ion and samples [21]. Then, the effective chloride diffusion coefficient (D_c) can be determined by the chloride concentration drop in cathode as Equation (4)

$$D_c = \frac{RTLV_a}{ZFEc_0A} \cdot \frac{\Delta c_c}{\Delta t} \tag{4}$$

where $\Delta c_c / \Delta t$ is the slope of the cumulative chloride ion concentration drop in cathode cell versus time curve. In this work, the effective chloride diffusion coefficient of cement mortar with different w/c under electrical accelerated measurement was determined by NT Build 355 method and Turc method. The results of effective chloride diffusion coefficient obtained from these two methods were compared, the influence of the linear regression starting time point was investigates. In addition, the chloride binding capacity for different w/c cement mortar was investigated.

2. Experimental Program

2.1. Materials and Mixture Proportions

The cement used in this work was P.I. 52.5 cement (corresponding to CEM I 52.5 cement) meets the Chinese standard GB 175-2007 [23] with specific surface of $365 \text{ m}^2/\text{kg}$, the chemical composition had physical properties of cement are presented in Table 1. The natural river sand with a fineness modulus of 2.49 was used in this work as fine aggregate, the absorption of the fine aggregate was 2.28%, the weight ratio between fine aggregate and cement was 2, the gradation information of the fine aggregate is shown in Figure 1. The distilled water was used as mixing water. Cement mortar with 5 different w/c was prepared, polycarboxylic superplasticizer (SP) was used in this work to have a similar workability of cement mortar, the details of the mixture and workability are presented in Table 2.

Table 1. Chemical composition (% by mass) and fineness of the cement.

CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	MgO	SO_3	K ₂ O	Na ₂ O	LoI	Fineness (m ² /kg)
64.47	20.87	4.87	3.69	2.13	2.52	0.65	0.11	0.77	368.9



Figure 1. Fine aggregate gradation.

Table 2. Mixture composition for cement mortar $(kg/m^3, SP \text{ content was by the mass of cem})$	nent)
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Mix	w/c	Cement	Mixing Water	Fine Aggregate *	SP (%)	Slump Flow/mm
А	0.55	615	338	1230	0.00	240
В	0.50	634	317	1269	0.20	230
С	0.45	655	295	1310	0.50	230
D	0.40	678	271	1355	0.70	230
Е	0.35	701	245	1403	1.00	190

* Fine aggregate was in saturated surface dry (SSD) condition.

2.2. Sample Preparation

Each mixture was mixed for 2 min in a planetary-type mixer at 45 rpm, then followed by a high speed (90 rpm) for 1 min. The ready-mixed cement mortars were cast into cylinder molds with the dimension of φ 100 mm × 150 mm. Each mixture was prepared with 3 specimens. After 24 h, the cylinder mold was removed, and all specimens were cured in water under 23 ± 2 °C for 28 days.

Slice samples with the dimension of $\varphi 100 \text{ mm} \times 50 \text{ mm}$ were cut from the middle portion of the cylinder specimens at designated age for chloride diffusion test. The lateral surface of the slice samples was coated with epoxy to eliminate the chloride ion loss from the lateral surface. Before measurement, all slice samples were vacuumed for 3 h in a vacuum chamber and then followed by saturated Ca(OH)₂ injection and immersion for 18 h. The slice samples were then removed from the solution and placed between two symmetrical cells as shown in Figure 2.





Figure 2. The configuration of chloride diffusion test setup: (**a**) configuration of the test setup. (**b**) samples under test.

2.3. Effective Chloride Diffusion Test

All tests were conducted in an environmental chamber with a stable temperature of 25 °C. For each mixture, three slice samples underwent the accelerated chloride diffusion test to evaluate the chloride diffusion coefficient. Similar test principle was described in ASTM C1202 [24]. The cathode cell of the test setup was filled with 5% (by mass) NaCl solution while the anode cell of the setup was filled 0.3 M NaOH solution. The two cells were connected to a direct current (DC) power station with 22 V voltage through two brass electrodes to form a steady state electrical field so as to accelerate the diffusion of chloride

ion across the slice sample. The chloride ion concentration in anode cell and cathode cell were measured periodically by a rapid chloride test (RCT) measurement, the error of the measurement was less than 5%, and the measurement was calibrated by 5.0×10^{-4} and 5.0×10^{-3} M NaCl solution, respectively, the reliability of the RCT measurement in this work could be found in our companion work [25]. The RCT measurement time interval of mixtures A, B and C was 6 h since the high w/c sample usually have a porous microstructure which will result in a fast ion diffusion. The measurement time interval of mixtures D and E was 12 h as low w/c samples associated with slow ion diffusion.

At the designated measurement time point, 20 mL solutions from cathodic and anodic cells were taken out, the solution from cathodic cell was diluted 100 times and then stored in a 2.5 flask, the anodic cell solution was diluted 20 times and stored in a 500 mL flask. The diluted solutions were stored for 20 min until the solutions' temperature reached the chamber temperature ($25 \,^{\circ}$ C). The chloride ion concentration in cathodic and anodic cells were then determined by RCT device. After the solutions were taken out from the cathodic and anodic cells, 20 mL of 5% NaCl solution and 0.3 M NaOH solution were replenished into the cathodic and anodic cells, respectively. Since the volume of the cells are 2.1 L, the solution volume changing for each time's measurement was less than 1%.

3. Results and Discussion

3.1. Cumulative Chloride Concentration in the Anode Cell

The cumulative chloride concentrations in anode cell for different mixtures are shown in Figure 3a–e. It can be found that for a given w/c, three duplicate samples' cumulative chloride concentration in anode cell showed high repeatability. For Nordtest Build 335 method, the $\Delta c_a / \Delta t$ (slope) is determined by linear regression when chloride reaches steady state diffusion. In this research, the steady state diffusion starting point was determined when the measured cumulative chloride concentration was firstly higher than 0.1 g/L. The steady state diffusion starting time was concluded in Figure 4. A very good correlation between w/c and steady state diffusion starting time point can be obtained in Figure 4. The steady state diffusion starting time point decreases with the increase of w/c which is reasonable since the higher w/c sample usually associate with a porous microstructure [26] and is easier to result in steady state diffusion than low w/c sample.

The anode cumulative chloride concentration for each mixture's three duplicates' linear regression result is presented in Figure 5. It can be seen that the regression slope $(\Delta c_a / \Delta t)$ decreases with the decrease of w/c which indicates the lower chloride diffusion coefficient for a lower w/c cement mortar sample, which is reasonable since the low w/c cement mortar has a denser bulk than high w/c cement mortar samples. The linear regression parameters of all mixtures were concluded in Table 3, the fitting parameters of all mixtures were higher than 0.95 which indicate that all measurements were in steady state diffusion.

3.2. Cumulative Chloride Ion Concentration Drop in Cathode Cell

The cumulative chloride concentration drop in the cathode cell of all mixture samples are presented in Figure 6a–e. For each mixture, three duplicate samples' cumulative chloride concentration ion drop in cathode cell showed high repeatability. The steady state diffusion starting time point for all mixtures are 24 h which indicates a relative rapid chloride concentration drop in cathode compared with the cumulative chloride concentration in anode. The reason for the faster chloride concentration reduction in the cathode than in the anode can be attributed to the absorption of chloride ions by cement mortar samples, as will discussed in the following section, and the slow transport speed of chloride ions in cement mortar's pore network.



Figure 3. Cumulative chloride concentration in anode cell: (a) w/c = 0.55 cement mortar samples, (b) w/c = 0.50 cement mortar samples, (c) w/c = 0.45 cement mortar samples, (d) w/c = 0.40 cement mortar samples, (e) w/c = 0.35 cement mortar samples.



Figure 4. The relationship between w/c and steady state diffusion starting time point.



Figure 5. Anode cumulative chloride concentration linear regression results of all mixtures.

Fab	le 3.	L	inear	regression	parameters	for anoc	le ch	loric	le concentration o	f al	ll mixtures.
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Mix	Linear Regression Equation	R ²
А	y = 0.132x - 2.677	0.9967
В	y = 0.071x - 2.097	0.9898
С	y = 0.038x - 2.970	0.9993
D	y = 0.023x - 2.645	0.9986
E	y = 0.016x - 2.362	0.9932

The averaged cumulative chloride concentration drop in cathode cell and the linear regression results were concluded in Figure 7, the regression parameters were presented in Table 4. It can be found in Figure 7 that the higher w/c, the higher linear regression slope which indicates a higher chloride diffusion coefficient for high w/c samples. But in the early stage of the measurement, the relationship of chloride concentration drop slope in cathode cell for different mixtures did not follow the w/c decrease or increase (i.e., mixture A and B in first 36 h, mixtures C and D in first 60 h), the reason for the inconsistence might be caused by the electrochemical reaction in cathodic electrode and the chloride binding by cement mortar at early measurement. At the early stage of the experiment, the chloride diffusion was in a non-steady state. The electrochemical reaction in the cathodic solution temperature could influence the RCT test result. In steady state diffusion, the temperature of the cathodic solution was stable, and the chloride concentration reduction in the cathode cell was consistent with w/c. Besides this, some chloride ions were bonded into the cement

mortar micro-pore walls in non-steady state diffusion, but these parts of chloride ions cannot be taken into consideration in the steady state chloride diffusion calculation. When the cement mortar reached steady-state diffusion, the cement mortar was saturated with chloride ions, and the chloride ion concentration was consistent with w/c. It is worth noting that the initial NaCl concentration in the cathode chamber was 50g/L, which indicated that the Cl⁻ concentration was 30.3 g/L. The measurement error of the RCT device was around 5%, so the initial chloride concentration reduction in the cathode cell might also be caused by a measurement error in the RCT device.



Figure 6. Cumulative chloride concentration drop in cathode cell: (a) w/c = 0.55 cement mortar samples, (b) w/c = 0.50 cement mortar samples, (c) w/c = 0.45 cement mortar samples, (d) w/c = 0.40 cement mortar samples, (e) w/c = 0.35 cement mortar samples.



Figure 7. Cathode cumulative chloride concentration drop linear regression results of all mixtures.

Table 4. Linear regression parameters for cathode chloride concentration drop of all mixtures.

Regression Rule	Mix	Linear Regression Equation	R ²
	А	y = 0.139x - 0.430	0.9619
	В	y = 0.076x + 1.190	0.9818
Rule 1: Starting from	С	y = 0.042x + 0.010	0.9813
24 h	D	y = 0.027x + 1.033	0.9661
	Е	y = 0.018x + 0.106	0.9803
	А	y = 0.131x + 2.404	0.9218
	В	y = 0.070x + 2.112	0.9770
Rule 2: Starting from	С	y = 0.039x + 0.545	0.9838
60 h	D	y = 0.021x + 1.391	0.9949
	E	y = 0.016x + 0.138	0.9721

In this part, two linear regression rules were adopted to analyze the influence of the early stage of the chloride concentration reduction deviation. The first rule is to run the regression starting with the 24 h data, and the second rule is to run the regression starting with the 60 h data. The linear regression results and parameters are shown in Table 4. The linear regression parameters of all mixtures were higher than 0.90, which also indicate that all samples were in steady state diffusion condition. Most of the R² values under the second regression rule are lower than the first regression rule which is caused by the lesser fitting data points. All mixtures' slope under the second regression rule are lower than the first regression rule are lower than the first regression rule are lower than the second regression rule are lower than the second regression rule are lower than the first regression rule are lower than the

3.3. The Effective Chloride Diffusion Coefficients Comparison

The effective chloride diffusion coefficient determined by Nordtest Build 355 and Truc methods are presented in Figure 8, the values of effective chloride diffusion coefficient determined by NTB 355 method (D_a), Truc method regression rule 1 (D_{c1}) and Truc method regression rule 2 (D_{c2}) and the standard deviations are concluded in Table 5. It is obviously that the chloride diffusion coefficient increases with the w/c as the high w/c sample usually has a more porous microstructure which is easier for chloride diffusion. It is interesting that the Truc method results with the first regression rule has a higher calculated chloride diffusion coefficient while the diffusion coefficients under second regression rule are very close to NTB 355 method results as showed in Figure 9. As discussed before, in early stage of the measurement, the chloride diffusion was not stable, part of the chloride ion was absorbed by the cement mortar, which was not considered in Truc first regression rule. After 60 h, all samples reached the steady state diffusion, as a result, the chloride diffusion coefficient under second regression rule showed similar results with the NTB 355 method.



Figure 8. Chloride diffusion coefficient with different determination methods.

Table 5. The calculated effective chloride diffusion coefficient of all mixtures.

Mix	w/c	${D_a \over (imes 10^{-12} \text{ m}^2/\text{s})}$	Standard Deviation	${D_{c1} \atop (imes 10^{-12} {m^2/s})}$	Standard Deviation	${D_{c2}} \over (imes 10^{-12} \text{ m}^2/\text{s})$	Standard Deviation
А	0.55	13.4	0.4523	14.1	0.7465	12.3	0.7056
В	0.50	7.2	0.2351	7.7	0.7512	7.1	0.6542
С	0.45	3.9	0.5215	4.3	0.6545	3.9	0.4310
D	0.40	2.3	0.6845	2.7	0.5896	2.1	0.1024
Е	0.35	1.6	0.1656	1.8	0.3458	1.7	0.1105



Figure 9. Chloride diffusion coefficient difference with NTB 355 and Truc methods (D_{c1} denotes the chloride diffusion coefficient determined by Truc method, linear regression was started from 24 h; D_{c2} denotes the chloride diffusion coefficient determined by Truc method, linear regression was started from 60 h; D_a denotes the chloride diffusion coefficient determined by NTB 335 method).

The effective chloride diffusion coefficients determined in this work were further compared with other researcher's work [9,27–29] with the same mixtures as showing in Figure 10. It can be seen from Figure 10 that the relationship between w/c and effective chloride diffusion coefficient shows good consistency, the fitting parameter R^2 is as high as 0.9976 which indicate a good agreement between the effective chloride coefficient obtained in this work and the literature.



Figure 10. Comparison of effective chloride diffusion coefficient in this research with other researcher's work.

3.4. Chloride Binding Capacity

The chloride concentration drop in the cathode includes the bonded chloride ion by cement mortar and the free chloride ions that diffused into the anode as measured by NT Build 355 method. In this way, the bonded chloride ion can be determined by the difference of chloride ion concentration between anode and cathode cells. As discussed before, the steady state diffusion starting time point in anode and cathode cells varied with different mixtures. In this part, the chloride concentration difference determination time point was determined when both anode and cathode cells reached the steady state diffusion. The chloride ion concentration in a cement mortar sample can be determined by Equation (5):

$$C_p = (C_c - C_a) \frac{V_a}{V_p} \tag{5}$$

where C_p denotes the chloride ion concentration in cement mortar sample (g/L); C_c denotes the chloride concentration in the cathode cell in steady state diffusion (g/L); C_a denotes the chloride concentration in anode cell (g/L); V_a denotes the volume of anode and cathode cell (L); V_p denotes the volume of cement mortar sample (L).

The chloride ion concentrations in cement mortar sample for all mixtures are concluded in Figure 11. It can be seen that the chloride ion concentration in cement mortar sample increases with the increase of w/c, which is reasonable since the less dense bulk of high w/c sample is easier for chloride ion penetrate into the pore network compared with low w/c sample.



Figure 11. Chloride ion concentration in cement mortar sample.

The chloride binding capacity of cement mortar under equilibrium chloride profile can be calculated by the following equation [8,30,31]:

$$C_b = C_p - 35.45C_0\rho \tag{6}$$

where C_b is the bonded chloride (g/L), C_0 is the chloride concentration in the cathode cell (mol/L), ρ denotes the porosity of cement mortar (%), which can be estimated according to Ref. [25].

The porosity and chloride binding capacity are presented in Figure 12. It can be seen that the porosity increases with the increase of w/c since the higher w/c samples have more consumable water which can be used for hydration during the curing process, as a result, the higher w/c sample will leave more micropores after hydration. The chloride binding capacity reasonably increases with the increase of w/c.



Figure 12. Chloride binding of different w/c mixtures.

4. Conclusions

In this study, the chloride diffusion coefficient of cement mortar was experimental investigated by NT Build 355 method and Truc methods. The influence of w/c on the chloride diffusion coefficient was evaluated and the difference between NT Build 355 method and Truc method was evaluated. Following conclusions were obtained:

- 1. The steady state diffusion starting time point of NT Build 355 method decreases with the increase of w/c while the Turc method has similar starting time point for different mixture.
- 2. The cumulative chloride concentration drop in the cathode cell showed deviation in first 60 h which will lead to overestimate of the chloride diffusion coefficient for Turc method while the linear regression result after 60 h measurement is very close to NT Build 355 method result.
- 3. The chloride diffusion coefficient and chloride binding capacity of cement mortar increases with the increase of w/c which can be attributed to the higher w/c would result in a porous bulk.

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