

Research Article

Chloride Transport Parameters of Carbonated Concrete

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Abstract

Many concrete structures suffer from corrosion of reinforcing steel due to chloride penetration and carbonation, and it is common for both deteriorations to progress simultaneously. Carbonation of concrete significantly changes the properties of concrete, so that cement hydrates is decomposed and the microstructure is also changed. The chloride penetration of carbonated concrete is bound to be significantly different compared to non-carbonated concrete. For this reason, chloride penetration parameters of carbonated cementitious materials were examined in this paper, i.e., (a) surface chloride content, (b) chloride diffusivity, (c) chloride adsorption capacity, and (d) critical chloride content. Each material parameter was calculated from the material parameter model reflecting the change in porosity due to carbonation. Carbonation of concrete converts cement hydrates into Calcite, resulting in almost loss of chloride adsorption capacity. Even though the pH is slightly decreased due to carbonation, critical chloride content of carbonated cementitious materials was calculated, confirming that the reinforcement is very vulnerable to corrosion. Since the main parameters are affected by mixing properties of cementitious materials, the analysis was performed for concrete mixes with arbitrary assumed conditions. Since the parameters change at each cement hydration stage, their time evolution was expressed. This study is expected to be useful to develop a chloride penetration model of carbonated cementitious materials in the future.

Keywords

Carbonation, Chloride Transport, Carbonation, Cement Hydration, Microstructure

1. Introduction

The deterioration of concrete structures is mainly caused by carbonation and chloride penetration in many countries [1, 2]. Although the combined deterioration of concrete due to carbonation and chloride penetration is a common in construction field, many studies on single deterioration have been accomplished, and the combined deterioration was very rare. For marine structures, the combined deterioration of carbonation and chloride penetration occurs mainly in the seawater splash zone and the atmospheric zone caused by flying salts. For urban structures, the slab exposed to de-icing chemicals sprayed for winter is mainly subjected to

the combined deterioration environment. Nevertheless, studies on the combined deterioration are very rare. The mechanism of the combined deterioration is very complicated and not fully understood [3-5].

As the combined deterioration progresses, the concrete structure is deteriorated severely due to the synergistic interaction between carbonation and chloride ions. Critical chloride content of non-carbonated concrete is 0.4%, however, the value of carbonated concrete is 0.2% by cement weight [6]. That is, even a low chloride concentration can lead to severe reinforcement corrosion under the combined deterior-

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ration.

In JSCE, durability guidelines for combined deterioration were set in practical terms, and repair strategy were prepared [3]. However, there has been no study on the identification of the mechanism of the combined deterioration and the evaluation of its lifespan. As shown in Figure 2, it has already been suggested that the rate of degradation is further accelerated by the constant re-diffusion of chloride ions released due to carbonation and concentrated to the non-carbonated zone [7, 8].

Carbonation of concrete is an inevitable phenomenon that occurs in all concrete structures except for concrete permanently submerged in water, and progresses from the concrete surface to the inside over time [9]. Most importantly, it has been universally recognized that alkalinity is lost and reinforcement corrosion is caused. In addition, carbonation of concrete reduces the amount of pores on the surface, resulting in reduced penetration of harmful substances [10, 11].

In the past decades, many studies on the durability design of marine concrete structures have been conducted, however, the results were inconsistent with each other. The analytical approaches had an unclear problem in the effect of individual influencing factor on these parameters. Moreover, studies of carbonation on these parameters are very rare. In order to develop an integrated durability design system for concrete exposed to combined deterioration of carbonation and chloride penetration, it is a necessary to study these parameters in carbonated concrete. The chloride transportation parameters should be reasonably defined based on the material science theory of cement composites.

The purpose of this study is to establish the basic approach to estimate the chloride transportation parameters for the chloride penetration of carbonated concrete by time steps. Carbonation was reflected in the analytical approach that can calculate surface chloride content, chloride diffusivity, and critical chloride content based on the cement hydration model, and the chloride adsorption capacity of Calcite produced by carbonation was experimentally examined.

2. Micro-Structural Properties of Carbonated Concrete

2.1. Pore Structure Change

Carbonation of concrete greatly affects changes of pore structure system, which is connected to the rate of CO₂ diffusion through carbonated concrete. In this study, the change of pore structure system of concrete due to carbonation was limited to OPC concrete. Papadakis et al. proposed the following equation to calculate reduced porosity due to carbonation (ΔV_c), based on the ratio of the volume of Ca(OH)₂ and C-S-H in concrete to calcium carbonate after carbonation [12];

$$\Delta V_c = [\text{CH}] \Delta \bar{V}_{\text{CH}} + [\text{C-S-H}] \Delta \bar{V}_{\text{CSH}} \quad (1)$$

in which, $\Delta \bar{V}_{\text{CH}}$ and $\Delta \bar{V}_{\text{CSH}}$ are equivalent to $3.85 \times 10^{-6} \text{ m}^3/\text{mol}$, $15.39 \times 10^{-6} \text{ m}^3/\text{mol}$, respectively. The above formula is the reduced the amount of pore in carbonated concrete, and the reduced amount of pore (cp) for cement paste can be expressed as follows considering volumetric fraction of the cement paste, V_{cp} , in unit volume concrete.

$$\Delta V_c(\text{cp}) = V_{\text{cp}} \{ [\text{CH}] \Delta \bar{V}_{\text{CH}} + [\text{C-S-H}] \Delta \bar{V}_{\text{CSH}} \} \quad (2)$$

Finally, porosity of carbonated cementitious materials can be described;

$$V_c = V_p - \Delta V_c \quad (3)$$

in which, V_c : porosity of concrete after carbonation, V_p : porosity of concrete before carbonation. Based on the above analysis, the chloride diffusivity of concrete before and after carbonation was calculated.

2.2. Surface Chloride

Surface chloride content can be regarded as an exposure conditional index of cement composite under chloride laden environment. The surface chloride content also depends on porosity of cement composite influenced by mixing proportional properties. In this study, surface chloride content was calculated under the assumption that the concentration of chloride ions in the pore solution of cement composite and seawater reached to chemical equilibrium [13]. General ratio of chemical elements in seawater is 548 mmol for Cl, 470 mmol for Na, 54 mmol for Mg. These elements are mutually combined and present as a complex chloride compound such as NaCl or MgCl₂. Considering the atomic weight, which directly affects the chloride ion concentration, the amount (C_e) of the chloride compound c in seawater is

$$C_e = q_e W_e \quad (4)$$

in which, q_e : amount of element e in seawater (27.45 g/L for NaCl, 4.5 g/L for MgCl₂), W_e : atomic weight (Na: 22.990, Cl: 35.453, Mg: 24.305).

Since the value of the total salt component in seawater is generally regarded as 35 g / l, the weight ratio (Ω_c) of the combined chloride compound c to the total salt component value is

$$\Omega_c = C_e / 35 \quad (5)$$

and the quantity (q_c) of the chloride compound of a unit volume is

$$q_c = \Omega_c \rho_c \quad (6)$$

in which, ρ_c : density of chloride compound c (NaCl: 2.16 g/cm³, MgCl₂: 2.31 g/cm³).

What is ultimately required for the maximum amount of chloride is theoretically the chloride weight (q_c^{Cl}) present in the compound chloride, c , such as NaCl and MgCl₂:

$$q_c^{Cl} = q_c \frac{W_{Cl}}{W_c + W_{Cl}} \quad (7)$$

in which, W_{Cl} : atomic weight of chloride, W_c : atomic weight of chloride compound.

When the chloride ions exist in the capillary pore and the part of the chloride ion reacts with the hydrate of the cement, the maximum chloride content [Cl] in the concrete can be expressed as

$$[Cl]_{\max} = \frac{(q_{NaCl}^{Cl} + q_{MgCl_2}^{Cl}) \cdot d_{sat} \cdot V_p}{W_{conc}} \quad (8)$$

in which, V_{pore} : porosity of concrete (%/100), d_{sat} : degree of saturation of concrete (%/100), W_{conc} : unit weight of concrete. If a concrete pore is fully saturated, the degree of saturation, d_{sat} , is 1, and the maximum amount of chloride ion $[Cl]_{\max}$ in the concrete is equivalent to the surface chloride content, which is an initial value of chloride ion profile with depth from the surface for chloride penetration analysis. Surface chloride content is experimentally an amount of chloride ion existing in powder obtained from concrete surface. Because of the skin effect, only small particles of fine aggregate and cement paste on the surface of concrete exist; however, the existence of coarse aggregate can be negligible. For this reason, porosity, V_{pore} , refers to the pore volume of mortar in Eq. (8). However, actual chloride ions from the concrete powder are measured experimentally, and thus, expressing chloride content relative to concrete weight should be reasonable. The amount of capillary pore in cementitious materials was reflected in the analysis in conjunction with the use of HY-

MOSTRUC. A detailed description of the calculation can be found in Van Breugel [14].

Figure 1 shows the result of the maximum amount of chloride ions when cementitious materials are fully filled with seawater. The amount of chloride ions in saturated concrete increases after concrete casting, and reaches chemical equilibrium after 10 to 15 years of aging [15]. Afterward, the amount of chloride ions is kept constant. This calculation was done for concrete with 20 years. The maximum amount of chloride ions in cementitious materials with a high water/cement (w/c) ratio showed a tendency to increase because the amount of porosity can play a role of space in which permeated seawater is stored. In addition, the maximum amount of chloride ions in cementitious materials tends to decrease with elapsed time.

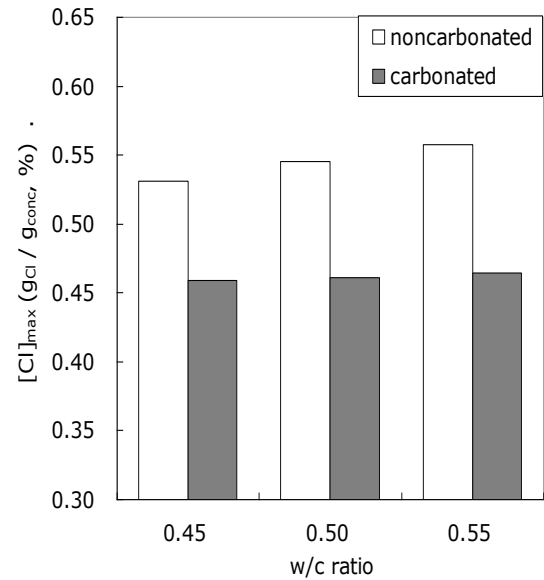


Figure 1. Surface chloride content of concrete with 20 years.

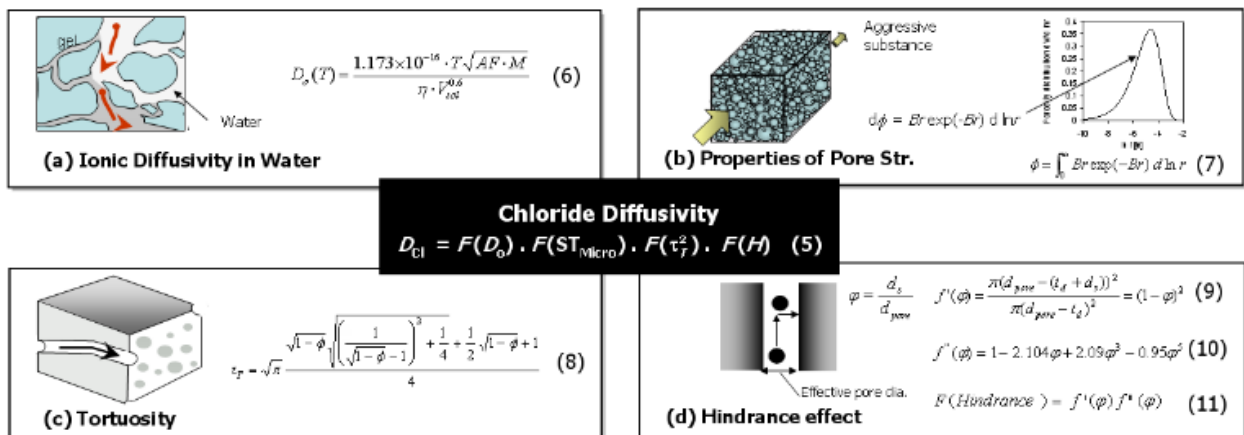


Figure 2. Formulation of chloride diffusivity to consider various affecting parameters (T : Temp., d_{pore} : diameter of pore = $2r$, d_s : solute molecular diameter, B : constant for pore size, D_o : Cl diffusivity in liquid (m²/s), AF : 2.26, M_w : 18.1 g/mol, η : viscosity of water (Pa.s), V_{sol} : molar volume of water at its boiling point (m³/K mol), t_a : thickness of water film on the surface of pore (0 for full saturated condition)).

This is because, as the degree of cement hydration increases, the micro-structural properties of cement paste is developed and the pore volume decreases, thereby reducing the space for storing seawater. However, for carbonated concrete, the surface chloride content decreased considerably. The trend of surface chloride content of concrete with w/c ratio was clear before carbonation. After carbonation, the trend was still found, however, the difference was somewhat unclear.

2.3. Chloride Diffusivity

Carbonation of cement composite can have a significant effect on chloride ions penetration. However, studies on the chloride diffusivity of carbonated cement composite are very rare. Thus, the micro-pore structural characteristics of the cement composite with the hydration stage, the effect of the presence of aggregates, and the ionic movement were considered as major factor functions for calculating diffusivity [16].

The multi-factor function on chloride diffusivity of cement composite is shown in Figure 2; (a) ionic diffusivity in liquid phase, $F(D_o)$, (b) pore volume and micro-pore structural properties of the cement composite, $F(St_{micro})$, (c) tortuosity properties due to pore connection, $F(\tau_T^2)$, (d) hindrance effect due to narrow pore walls, $F(H)$, and so on. In what follows a description of these factors is given

$$D_{Cl(cp)} = F(D_o) \cdot F(St_{micro}) \cdot F(\tau_T^2) \cdot F(H) \quad (9)$$

Ionic diffusivity in water: For saturated concrete, the flow media of chloride ions can be the bulk pore water as the ions can flow through water-filled pores. In this respect, ion diffusivity in bulk fluid was functionalized from the study of Wilke et al. [17].

Micro-pore structural properties of cement composite: The pore size distribution characteristics of cement composite were reflected as shown in Eq. (10). Here, V_p : volumetric fraction of pore with distribution up to pore radius r as a variable, and B : the parameter of the pore distribution function.

$$dV_p = Br \exp(-Br) d \ln r \quad (10)$$

If $\ln r$ replaces x , r is substituted for $\exp(x)$. Eq. (11) is derived as;

$$V_p = B \int_0^\infty \exp(x) \cdot \exp\{-B \cdot \exp(x)\} dx \quad (11)$$

With increasing degree of hydration, the pore volume decreases, mainly at the cost of the volume of the larger pores. Thus, total porosity (V_p) was calculated by HYMOSTRUC for each hydration step, numerical simulation program for cement hydration [14]. A peak point of porosity distribution

B was back calculated from Eq. (11).

Tortuosity of flowline: It was calculated by considering the cement hydrate present per unit volume as one cell, assuming that the cement core is a square, and considering the shape functions of circles and squares. In this study, this method was used for the chloride ionic inflow. To account of the flowline of chloride penetration, tortuosity factor was suggested in previous work of author [17]. Although it makes sense to regard the morphology of cement core as a circular shape, it is assumed to be a square to simplify the fluid streamline. The difference is reflected with shape functional ratio between circle and square and thus, average streamline of tortuosity was depicted as;

$$\tau_T = \sqrt{\pi} \frac{\sqrt{1-V_p} \sqrt{\left(\frac{1}{\sqrt{1-V_p}-1}\right)^2 + \frac{1}{4}} + \frac{1}{4} + \frac{1}{2} \sqrt{1-V_p} + 1}{4} \quad (12)$$

Hindrance effect: The fourth term of Figure 2, $F(H)$, can express hindered diffusion due to interaction of substance and narrow path between pore walls. As the molecular diameter of the harmful substances approaches the pore wall, the transport of the harmful substances through the pore should be disturbed by the narrow pore wall. The function of the disturbed diffusion is described as [18];

$$F(H) = f'(\phi) f''(\phi) \quad (13)$$

Two correction factors, $f'(\phi)$ and $f''(\phi)$, are related to the reduced pore diameter ϕ .

$$\phi = \frac{d_s}{d_{pore}} \quad (14)$$

in which, d_s : kinetic diameter ($\approx 3.34 \times 10^{-8}$ cm for CO_2 gas), d_{pore} : diameter of pore.

The first correlation factor, based on geometrical arguments, can be expressed as;

$$f'(\phi) = \frac{\pi(d_p - (t_d + d_s))^2}{\pi(d_p - t_d)^2} = (1 - \phi)^2 \quad (15)$$

in which, t_d means a twice thickness of adsorbed layer. The second correlation factor can be expressed as Renkin equation;

$$f''(\phi) = 1 - 2.104\phi + 2.09\phi^3 - 0.95\phi^5 \quad (16)$$

The effect is effective to depict disturbed diffusion rate due to consequence of narrow pore diameter and collision of CO_2 gaseous molecules to each other.

Figure 3 shows the comparison of chloride diffusivities between carbonated and non-carbonated concrete. Carbonation of concrete influence directly (b) the amount of pores and pore structural properties of the cementitious materials, (c) tortuosity of flowline due to the connection of the pores, (d) the hindrance delay effect due to narrow pore walls, except for (a) the ionic diffusivity in the liquid phase. That is, the reduced pore volume leads not only more complicated ionic flowline, but also more severe hindrance effect. This leads significant reduction of chloride diffusivity of carbonated concrete.

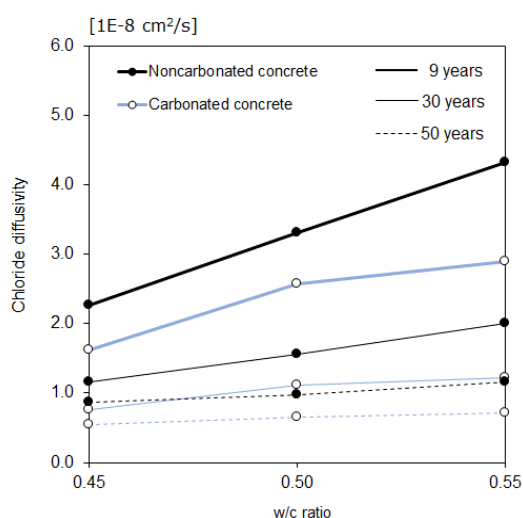


Figure 3. Effect of carbonation on chloride diffusivity.

2.4. Chloride Adsorption

Adsorption of chloride ions in cement composite is activated by the physicochemical interaction between hydrates and chloride ions [19, 20]. In this study, cement hydrates were synthesized to obtain the chloride ion adsorption capacity of the hydrate. For calcium hydroxide (CH) and Calcite, chemical products with a chemical purity of 99% or higher were used. For C_3S , $CaCO_3$ and SiO_2 were stoichiometrically mixed to obtain artificially synthesized C_3S . The C_3S mixed with water was cured to synthesize C-S-H with Ca/Si 1.50. For C_3A , $CaCO_3$ and Al_2O_3 were stoichiometrically mixed. The C_3A mixed with water was cured to synthesize Mono-sulfate ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 10H_2O$, AFm). Ettringite ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$, AFt) was synthesized with $Ca(OH)_2$ and $Al_2(SO_4)_3$.

Experimental apparatus was designed to conduct chloride ions adsorption of synthesized hydrates in liquid phase for 3 months. For artificial pore water, de-ionized water with NaOH vs. KOH ratio of 1:28 was prepared. The pH of the water was 13.5 ± 0.1 . NaCl was added in the artificial pore water to obtain a chloride aqueous solution of 0.1 mol/L.

Figure 4 shows the chloride adsorption capacity of hy-

drates before carbonation of hydrates. AFm phase had the strongest chloride adsorption capacity and this was overwhelmingly high compared to any other hydrates. C-S-H phase had 2nd adsorption capacity, which was 1/100 of the AFm phase. The CH phase and AFt phase adsorbed 0.0016 to 0.0019 mmol per hydrate, which was so slight and meant practically no adsorption capacity.

Many hydrates are converted to Calcite phase as they are carbonated [2]. Thus, AFm and C-S-H phase with a Ca/Si 1.5, which had an excellent chloride adsorption capacity before carbonation, were exposed to a carbonation accelerator to form carbonated AFm and carbonated C-S-H phase. The result was compared to pure Calcite phase, as shown in Figure 5. After carbonation, AFm and C-S-H phase lost chloride adsorption capacity significantly. The chloride adsorption capacity of Calcite phase was almost negligible. Although the carbonated AFm and C-S-H phase had somewhat higher chloride adsorption capacity than pure Calcite phase, it was not meaningful practically. It can be concluded, therefore, that the chloride adsorption capacity of carbonated concrete can be almost negligible.

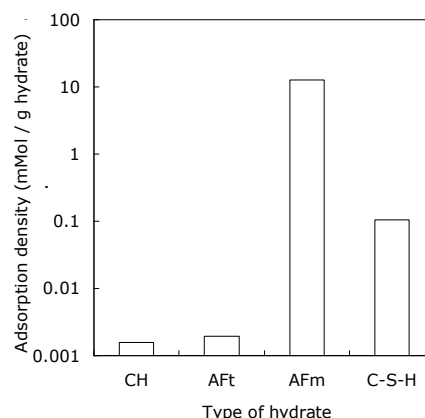


Figure 4. Chloride adsorption density of hydrates before carbonation.

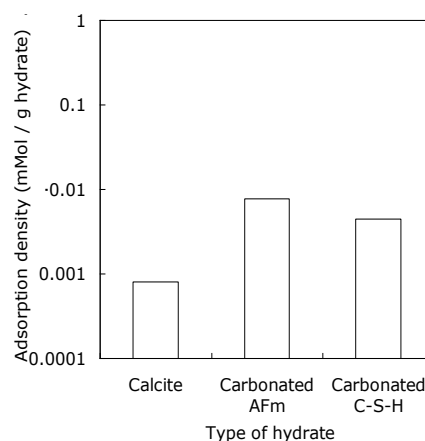


Figure 5. Chloride adsorption density of hydrates after carbonation.

2.5. Critical Chloride Content

Carbonation of concrete greatly affects not only the pH of the pore water but also the critical chloride content. However, there are very few studies that have suggested the critical chloride content of carbonated concrete. In the case of non-carbonated concrete, the critical chloride content was regarded as about 0.40% by cement weight, however, the value decreases significantly as carbonation proceeds [6]. When carbonation occurs, the passivation film on the surface of the reinforcement created by the alkalinity properties of the cement paste is destroyed, so that the reinforcement easily corrodes even with a small amount of chloride ions.

In general, the pH of the pore water in concrete has a value of around 13 to 14, however, when carbonation proceeds, reinforcement corrosion starts at a pH of around 11.5 [21]. In this study, pH up to 11.5 was regarded as the pH of the pore water of carbonated concrete, and the OH^- concentration to depict the decrease of pH values due to carbonation was reflected.

$$[\text{Cl}^-(\text{aq})]_{cr} = \frac{\begin{cases} -(0.348 + 0.424 \log [\text{OH}^-]) [\text{OH}^-] & \text{for pH} < 12.5 \\ 0.287 [\text{OH}^-] & \text{for pH} \geq 12.5 \end{cases}}{\phi_w \cdot V_{cp} \cdot W_{conc}} \frac{MW_{OH}}{MW_{Cl}} \quad (19)$$

in which, $[\text{Cl}(\text{aq})]_{cr}$: critical chloride content (g/g_{conc}), W_{conc} : unit weight of concrete ($= 2,350 \text{ kg/m}^3$), MW_{OH} : molar weight of OH ($= 17.01 \text{ g/mol}$), MW_{Cl} : molar weight of Cl ($= 35.45 \text{ g/mol}$), ϕ_w : volumetric fraction of water in pore system.

The results of calculating the critical chloride content of carbonated concrete are shown in Figure 6. Carbonated concrete showed a tendency to increase in the critical chlorine content over time as in the noncarbonated concrete. In addition, even when the pH was slightly decreased to about 12.5 due to carbonation, the critical chloride content was significantly decreased. This is because, as can be seen from Eq. (19), when the pH is 13.0 or less, the OH^- concentration decreases significantly. In particular, when the pH is lowered to 11.5, the critical chloride content has an extremely low value, close to 0, because the passivation film on the reinforcement surface is already destroyed by carbonation, regardless with chloride ions. That is, since carbonated concrete is already depassivated, the critical chloride content is no longer an indicator to determine reinforcement corrosion. Reinforcement corrosion should be very severe under even very low chloride content.

Meanwhile, the tendency of a significant decrease in the critical chloride content due to carbonation in concrete with a low w/c ratio was noticeable, compared to concrete with a high w/c ratio. This is because concrete with a low w/c ratio contained a high volume of cement and produced a high amount of OH^- ions. Furthermore, the decrease in pH and

In this study, based on the experimental results of Gouda [22], Eq. (17) and (18) with the bilinear functions were derived and pH 12.5 was the boundary value of bilinear relationship between $[\text{Cl}^-]/[\text{OH}^-]$ and pH. This pH boundary condition has a similar meaning as Hausmann proposed a critical chloride content of 0.60 $[\text{Cl}^-] / [\text{OH}^-]$ within the pH 12.5 [23].

$$[\text{Cl}^-] = -(0.348 + 0.424 \text{ pH}) [\text{OH}^-], (11.5 < \text{pH} < 12.5) \quad (17)$$

$$[\text{Cl}^-] = 0.287 [\text{OH}^-], (\text{pH} \geq 12.5) \quad (18)$$

The concentration of hydroxyl ions in cement pore water can be measured only at laboratory by means of chemical analysis and it is difficult to measure it in general concrete laboratories and field measurements. For this reason, best should be expressed as a chloride content by weight of cement or concrete from a practical point of view. Finally, the critical chloride content was derived as in Eq. (19).

OH^- values due to carbonation was relatively high, compared to concrete with a low w/c ratio. However, like noncarbonated concrete, carbonated concrete with a high w/c ratio also showed a high critical chloride content.

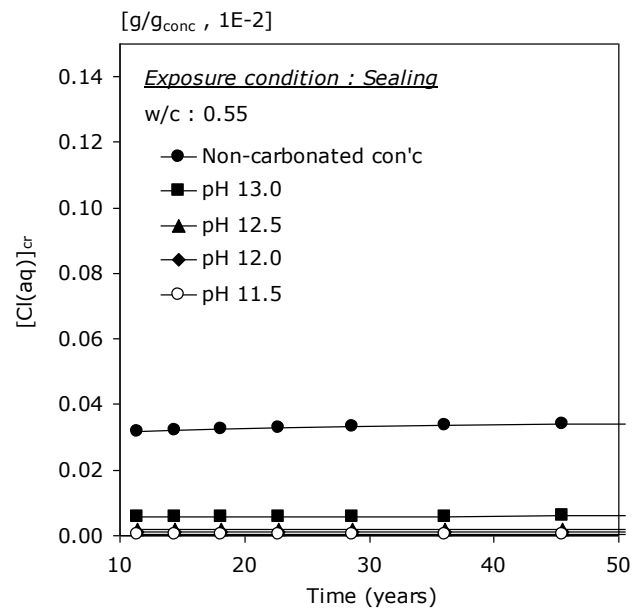


Figure 6. Effect of carbonation on critical chloride content of concrete with w/c 0.55.

3. Conclusions

(1) This study investigated the effects of carbonation of concrete on chloride penetration parameters which depends on the mixing conditions, hydration, micro-structural properties of cement composite, and so on. Physical and chemical changes due to carbonation significantly affected the chloride transportation parameters of cement composite.

(2) Carbonation of concrete greatly reduces the amount of surface chloride content and chloride diffusivity due to the reduction of pores, and converts cement hydrates into Calcite, resulting in almost loss of chloride adsorption capacity. Even though the pH is slightly decreased due to carbonation, furthermore, critical chloride content is greatly reduced, confirming that the reinforcement is very vulnerable to corrosion.

Author Contributions

In-Seok Yoon is the sole author. The author read and approved the final manuscript.

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Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

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