



Chloride penetration in concrete exposed to cyclic drying-wetting and carbonation



Hailong Ye^{a,c,*}, Xianyu Jin^a, Chuanqing Fu^{a,b}, Nanguo Jin^a, Yibin Xu^a, Tao Huang^a

^a College of Civil Engineering, Zhejiang University, Hangzhou 310027, PR China

^b College of Civil Engineering and Architecture, Zhejiang University of Technology, Hangzhou 310034, PR China

^c Department of Civil and Environmental Engineering, The Pennsylvania State University, 3127 Research Drive, State College, PA 16801, United States

HIGHLIGHTS

- Concrete performance exposed to a combination of various deterioration mechanisms.
- Chloride penetration in concrete under cyclic drying-wetting and carbonation.
- Effects of supplemental cementitious materials and flexural loading are presented.
- Coupled physical-chemical interaction between environments and concrete materials.

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ABSTRACT

In this paper, the chloride penetration process in concrete exposed to a cyclic drying-wetting and carbonation environment was experimentally characterized. In order to better represent the concrete in service, chloride penetration tests were performed on concrete with various levels of flexural loading and incorporation of supplemental cementitious materials (i.e. blast-furnace slag and low calcium fly ash). The result shows that the chloride penetration profile in concrete is a result of multiple interactive deteriorating mechanisms and dependent on the properties and stress status of concrete itself. Carbonation induces considerable chloride (both free and bound) redistribution in concrete, rendering chloride ions ingress from carbonated region towards un-carbonated region. The incorporation of supplemental cementitious materials makes concrete more vulnerable to carbonation-induced chloride movements due to its less amount of portlandite. The increased magnitude of flexural loading results in higher chloride content in concrete probably by forming some micro-cracks.

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1. Introduction

The corrosion of reinforced concrete (RC) structures has been a major issue for infrastructures, such as offshore and harbor structures, pavement, and bridges [1–3]. The initiation of corrosion typically originates from a high chloride concentration in pore solution of concrete which destroys the initially-formed passive film around reinforcement, when RC structures are exposed to marine environments or deicing slats [4]. Due to the significance of this problem, tremendous investigations regarding the chloride penetration process into concrete were conducted over decades [5–9]. According to previous research, intermitted drying-wetting

cycle is the most unfavorable condition for RC structure, since it accelerates the chloride ingress considerably [7–9].

In order to better understand the deterioration mechanisms of RC structure under chloride attack, as well as accurately predict its service-life performance, it is essentially important to capture the status of in-service concrete and exposed environments. For example, the concrete in RC structure is typically under stressed status with various natures (e.g. tension, compression, and shear) and magnitudes. Therefore, emerging studies regarding the influence of loading on chloride penetration in saturated/non-saturated concrete have been conducted by many researchers [9–14]. In addition, considering that most existing RC structures always serve with micro- or macro-cracks, some attempts to illuminate the effects of crack characteristics on the chloride penetration in concrete was also made by numerous investigators [15–17]. Furthermore, modern concrete was intentionally designed with significant amount incorporation of supplementary cementitious

* Corresponding author at: Department of Civil and Environmental Engineering, The Pennsylvania State University, 3127 Research Drive, State College, PA 16801, United States.

E-mail address: huy131@psu.edu (H. Ye).

materials (SCM), including blast-furnace slag, fly ash, and silica fume. There were some recent studies trying to elucidate the influence of SCM on the chloride penetration process in concrete subjected to drying-wetting cycles [18,19].

Besides a better representation of in-service modern concrete itself, characterizing the realistic exposed deteriorating environments is however more challenging. Most RC structures were experiencing multi-deteriorating actions simultaneously over its entire service life, and the combination of them may even more detrimental than any single deterioration process alone. For instance, when chloride attack is accompanied with carbonation under wetting-drying cycles, it may even further increase the risk of corrosion in RC structures. Recently, there are several studies regarding the influence of carbonation on chloride penetration in concrete. For instance, Tumidajski et al. found that carbonation has a minor effect on the chloride diffusivity of ordinary Portland cement-based concrete, but increases the chloride diffusivity for concrete with blast-furnace slag [20]. However, Lee et al. found that the ratio of water-soluble chloride (i.e. free) to the acid-soluble chloride (total) content is higher for the case of accelerated carbonation than that of natural air exposure [21]. They reported that the addition of fly ash can reduce the chloride content at natural carbonated condition, but increases chloride content when accelerated carbonated [21]. Saeki reported that the carbonation of AFm phases (i.e. a family of hydrated calcium aluminate phases) in cement can transform some originally fixed chloride ions (i.e. bound) to free one [22]. Considering that Saeki investigated this problem using synthetic AFm phases in an artificial pore solution, it would be questionable on further application in realistic modern concrete.

On the other hand, both carbonation and chloride salt crystallization (during drying) can potentially modify the properties of concrete (e.g. pore structure, hydrated phase assemblage, binding capability of hydrates), which further affect the penetration process for carbon dioxide and chloride ions simultaneously. In other words, the chloride penetration and carbonation process involves a coupled physical-chemical interaction between environments and dynamic responses of concrete materials [9]. For example, it was reported that the chloride-bearing crystals can physically block the pores which contributes to a reduction of carbonation depth [23]. The free chloride is hygroscopic adsorbing moisture into concrete, blocking the porosity of concrete, and retarding the carbonation reaction. In addition, carbonation can significantly coarsen the pore structure and reduce the porosity, which may retard the chloride penetration forward as well [24,25]. A recent study indicates that the chloride diffusivity is lower for carbonated samples than non-carbonated, which reduces initial amount of chloride content and benefits the chloride resistance [26]. With respect to the chemical impacts of carbonation on chloride penetration, as aforementioned, it is still unclear whether these physical modifications can override the chemical effects and partially benefit to retardation of corrosion initiation. Therefore, controversial findings were reported in literature, and more investigation is needed regarding the influence of carbonation on chloride penetration into modern concrete.

In the field, structural concrete is always stressed and simultaneously affected by multi-degradation mechanisms, possibly with a synergetic effect on deterioration. However, a systematic study characterizing concrete performance with various SCMs and stress status, as attacked by a combination of cyclic drying-wetting, chloride, and carbonation, was rarely reported. This study aims to contribute to a better understanding of the service life performance of in-service RC structures exposed to a combination of various deterioration mechanisms. In particular, the chloride penetration process in concrete with/without a considerable amount of SCMs (i.e. blast-furnace slag and low-calcium fly ash), with/without stress, and with/without carbonation at drying-wetting cycles, was investigated.

2. Experimental program

2.1. Materials and mixing proportion

The ASTM Type I ordinary Portland cement (OPC), with a 28-day compressive strength of 42.5 N/mm² (MPa), was used in this study. The mineral composition of OPC is listed in Table 1. The alkali content (denoted as Na₂O + 0.658K₂O) is <0.60%, and the fineness is 350 m²/kg. The ASTM C989 Grade 100 ground granulated blast-furnace slag with the fineness of 450 m²/kg, and ASTM C618 Class F fly ash were used as SCMs. The fine aggregate was river sand with a fineness modulus of 2.64. The coarse aggregate was crushed gravel with a maximum size of 20 mm and continuous grading ranges from 5 mm to 20 mm. The water was from tap water. In addition, a polycarboxylate-type superplasticizer was used.

In order to investigate the influence of SCMs on the chloride penetration resistance, two types of mixing proportion for concrete with various contents of SCMs were used, as shown in Table 2. Mixture PC is a pure OPC-based concrete and Mixture FS has considerable amounts of Class F low-calcium fly ash and blast-furnace slag, while both of them have the same water-to-binder (i.e. OPC + SCMs) ratio and volume fraction of aggregates. To prepare reinforced concrete samples (to be able to sustain various magnitudes of flexural loads), a series of ϕ 10 HPB 235 bars (the characteristic value of yielding strength is 235 N/mm², according to Chinese Standard GB 50010-2002) were used as the reinforcements.

2.2. Sample preparation

The configuration of sample was a prism with dimensions of 100 mm × 100 mm × 460 mm. All samples were casted and demolded after 24 h, followed by the moisture curing at 100% relative humidity (RH) and 20 ± 5 °C for 28 days. After moisture curing, every two samples (on PC and one FS) were put together as one loading group, and were loaded using a back-to-back four-point loading scheme as shown in Fig. 1(a). To apply flexural loads, a designed loading device which can exert sustaining loads on samples during the entire tests was implemented. As elaborated in a previous study [12], the magnitude of loads was regressively correlated to the length of springs attached to the end of samples, as shown in Fig. 1. Prior to loading, the loading magnitude and length of springs was calibrated using universal testing machines. Therefore, in order to apply targeted magnitude of loads, the screws (see Fig. 1) were mechanically adjusted to reach certain length of springs.

For each mixture (PC or FS), totally 39 samples was casted. 3 of them was used for testing the ultimate loading capability, 12 samples were remained un-loaded, 12 samples were loaded with 30% of the ultimate loading capability, and 12 samples were loaded with 60% of the ultimate loading capability. The ultimate loading capability for PC and FS mixtures was measured to be 13.2 kN and 12.7 kN, respectively.

2.3. Exposure condition

After curing and loading, all samples were experiencing three different types of exposure as shown in Table 3. In particular, the samples were first immersed into a highly-concentrated 15% NaCl solution for four days (as shown in Fig. 1(b)). Afterwards, they were surface-dried before putting into an oven which was programmed to 60 °C for two days. Then, they were selectively moved to an accelerated carbonation chamber which was programmed to a CO₂ concentration of 20 ± 3%, temperature of 20 ± 5 °C, and 70 ± 5% RH. More specifically, one third of samples (with same mixture proportion and loading magnitude) were exposed to natural air as control (i.e. exposure condition I), while one third were accelerated carbonated for two days (i.e. exposure condition II), and another one third were accelerated carbonated for four days (i.e. exposure condition III). In addition, in order to realize one-dimension chloride penetration, merely the two opposite surfaces (i.e. the two faces experiencing pure flexural loadings as shown in Fig. 1) were exposed to chloride attack, while other surfaces were coated with high melting point paraffin.

2.4. Free chloride content

The chloride concentration in concrete as a function of depth was measured for each sample after 2, 4, 6, 8, and 10 wetting-drying-carbonation cycles (i.e. one cycle is as indicated in Table 3). The concrete powders were drilled with an interval of 2 mm for the first 10 mm away from exposed surface, then with an interval of 5 mm in the range of 10–30 mm. In each position, the concrete powders collected from three holes were mixed to avoid the influence of aggregates. The powder with a weight of 15 g (passing through 0.63 mm sieve) was oven-dried at 105 ± 5 °C for two hours before cooling down and conducting the chloride content measurements.

Table 1
Mineral composition of ordinary Portland cement (mass%).

Mineral composition	C ₃ S	C ₂ S	C ₃ A	C ₄ AF	Gypsum
Content	55.5	19.1	6.5	10.1	5.0

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