



Effect of early carbonation curing on chloride penetration and weathering carbonation in concrete



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HIGHLIGHTS

- Effect of early carbonation on chloride penetration and weathering carbonation is studied.
- Two cyclic exposures are tested including air drying and weathering carbonation.
- Both total and free chloride profile are discussed.
- OPC and fly ash-OPC concrete are tested.

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ABSTRACT

Early carbonation curing of concretes was developed for accelerating the strength gain and promoting carbon dioxide utilization in concrete. However, the effect of early carbonation curing on chloride penetration was not clear. It was reported that weathering carbonation of concrete could increase chloride penetration. The purpose of this paper is to examine if early carbonation curing would also accelerate the chloride penetration and the weathering carbonation depth in concrete during service. Both carbonation cured and hydration cured concretes with and without fly ash were exposed to two severe cyclic conditions: (1) chloride immersion/air drying cycles and (2) chloride immersion/accelerated weathering carbonation cycles. It was found through one-year cyclic tests that total chloride content was actually reduced by more than 50% in concrete subjected to carbonation curing comparing to hydration reference. Reduction in free chloride (water-soluble) in carbonation cured concrete reached more than 60%. It was also found that carbonation cured concrete was not more vulnerable to weathering carbonation. This was attributed to the carbonate-rich surface protective layer which was less permeable, less absorptive, and with comparable pH value, enabling the carbonation cured concretes higher resistance to chloride penetration.

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1. Introduction

Corrosion of steel in concrete is one of major durability issues for reinforced concrete. Deterioration appears to be more severe in harsh marine environment [1]. It is believed that corrosion of steel in concrete is induced by chloride penetration [2]. The cyclic exposure of air drying and seawater wetting increases the ingress of chloride salts [3]. Corrosion occurs once free chloride content reaches the threshold value which varies with pH of pore solution [4]. Another mechanism, known as weathering carbonation corrosion, happens in both coastal and on-land structures. Calcium hydroxide in concrete is reacted with atmospheric carbon dioxide during service life leading to a reduction of pH value [5,6]. When

pH drops to below 9.5, passivation film on steel surface starts to decompose and corrosion starts to initiate [7]. Reinforced concrete simultaneously subjected to both chloride penetration and weathering carbonation is reported to be more vulnerable than that to individual action [8]. Extensive studies show that reduced pH caused by weathering carbonation reduces threshold value of chloride content for corrosion initiation [9]. Accordingly, corrosion of steel rebar starts at lower chloride content when concrete is exposed to weathering carbonation [10]. Corrosion rate is also increased as pH reduces and chloride content increases [11,12]. Some other studies suggest that weathering carbonation accelerates penetration of chloride salt [13,14]. In the meantime, both total chloride content and its penetrating depth are increased. It is also mentioned that as result of global warming and increased concentration of atmospheric carbon dioxide [15–17], carbonation of concrete structures will become more serious [16].

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Unlike weathering carbonation, early carbonation is a curing process intentionally introduced within 24 h after concrete casting. High early strength is observed in a short period [18]. In addition to the accelerated effect on strength gain, early carbonation curing shows improved performance of concrete on exposure to freeze-thaw cycle and sulfate attack [19]. The technique has also received increasing interests due to its capacity of carbon dioxide sequestration [20].

Both weathering carbonation and early carbonation behavior are altered by the presence of fly ash. Concrete incorporated with fly ash becomes more vulnerable to weathering carbonation [21]. Carbonation depth increases as a consequence [22]. This is possibly due to the high water to cement ratio. The same phenomenon is observed in early carbonation. The degree of early carbonation is higher in fly ash concretes comparing to plain concretes and the pozzolanic reaction of fly ash is reduced after early carbonation [23].

The study has shown that early carbonation curing can be applied to reinforced concrete as well [24]. The pH value of early-age carbonated concrete is comparable to hydration reference owing to the subsequent re-hydration. However the effect of early carbonation curing on chloride penetration in concrete is not clear. The purpose of this paper is to investigate if the early carbonation curing would promote the penetration of chloride ions in concrete as was observed by weathering carbonation. The study also examines if the early carbonation curing would increase the weathering carbonation depth in service. In this study, concrete cured by early carbonation is compared chloride penetration and weathering carbonation depth with hydrated concrete. Two exposure conditions are adopted including chloride immersion/air drying cycles and chloride immersion/accelerated weathering carbonation cycles. Both OPC and fly ash-OPC concretes with early carbonation and hydration curing are compared.

2. Experimental program

2.1. Materials and mix proportion

The chemical compositions of Type GU Ordinary Portland Cement (OPC) and fly ash are shown in Table 1. Crushed granite stone and granite sand were used as aggregates. Maximum aggregate size was 12 mm and the fineness modulus of fine aggregate was 3.0. The water adsorption was approximately 4.3% for fine aggregate and 1.6% for coarse aggregate. To avoid chloride contamination from other sources, aggregates had been washed three

Table 1
Chemical composition of cement and fly ash.

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O _(eq)	SO ₃
Cement	63.10	19.80	4.90	2.00	2.00	0.85	3.80
Fly Ash	11.30	54.39	23.65	3.90	1.17	2.91	–

Table 2
Mixture proportion of concrete.

	OPC	FAOPC
Cement, kg/m ³	452	362
Fly ash, kg/m ³	0	90
Fly ash to binder ratio, %	0	20
Water, kg/m ³	181	181
Water to binder ratio	0.4	0.4
Water to cement ratio	0.4	0.5
Coarse aggregate, kg/m ³	1060	1060
Fine aggregate, kg/m ³	680	680
Superplasticizer to cement ratio, %	0.5	0.5
Slump, mm	158	166

times and used in condition of saturated surface dry (SSD). De-ionized water was used instead of tap water to eliminate the possible chlorides from mixing water.

The ratio of water to cementitious materials was chosen as 0.4. Two mixture proportions were used representing OPC and fly ash-OPC concretes. Fly ash was added as 20% replacement of cement by mass in fly ash-OPC concrete. Superplasticizer was used to achieve the slump of about 160 mm for both concretes. The detailed mixture proportions are listed in Table 2.

2.2. Sample preparation and carbonation curing process

Concretes were cast into 100 mm cube steel mold and consolidated on a vibration table. The fresh concrete samples were treated with two different curing methods: early carbonation curing (denoted as C) and conventional hydration curing (denoted as H). For hydration reference, concretes were demolded after 24 h in molds, and further cured in a moisture room at 25 °C and 95% relative humidity (RH). Early carbonation curing of concrete comprised of four steps as shown in Fig. 1: (1) 5–6 h in-mold curing, (2) 5.5 h de-mold conditioning, (3) 12 h carbonation curing and (4) 27 d subsequent hydration.

Since the fresh concrete had high slump, initial hydration before demolding was necessary. The demolding time was dependent on the mixture proportion. For the concrete shown in Table 2, it took approximately 5 h for OPC concrete and 6 h for fly ash-OPC concrete to reach initial set in an open air (25 °C and 60% RH). In step 2, concretes were demolded and left on the bottom plates for fan drying conditioning at wind speed of 1 m/s in ambient condition (50 ± 5% RH, 25 °C). This step was intended to remove about 40% of free water to create paths for carbon dioxide penetration and carbonate precipitation. The conditioned samples were then moved to an enclosed carbonation chamber filled with pure CO₂ gas (99.8% purity) at constant pressure of 5 bar for 12 h. The early carbonation curing setup is shown in Fig. 2. Concrete samples were weighed before and after the 12 h carbonation reaction. Water generated in the chamber by the reaction was collected by absorbent paper and weighed. The CO₂ uptake by mass gain method was calculated using Eq. (1).

$$\text{CO}_2 \text{ uptake (\%)} = \frac{m_2 + m_{\text{water}} - m_1}{m_{\text{cement}}} \times 100\% \quad (1)$$

where m_1 and m_2 are the concrete mass before and after the carbonation reaction, respectively. m_{water} is the mass of water collected in the reaction chamber. m_{cement} is the dry cement mass in carbonated concrete.

Carbonated concretes were surface sprayed by de-ionized water right after carbonation and were moved to moisture room (95% RH, 25 °C) for additional 27 d subsequent hydration. Concrete at the age of 1 d and 28 d were tested for compressive strength. Phenolphthalein solution was sprayed on the split cross section to determine the carbonation depth due to early carbonation curing. A pH meter with flat head (Extech PH110) was used to measure pH at different depth (0–10 mm, 10–25 mm, 25–50 mm) at 1 d and 28 d. The testing area was covered by an absorptive paper (10 × 10 mm) soaked with 100 μL deionized water for 15 min when the diffusion equilibrium was reached. The pH readings were then recorded [25,26]. Concretes after pH measurement were chiselled off at the three depths and crushed into 5–10 mm particle size to determine carbon content through a thermal pyrolysis [24]. About 100 g of concrete samples were heated and measured mass up to 105, 550 and 950 °C. The mass loss between 550 and 950 °C was attributed to decomposition of calcium carbonate and calculated in reference to cement mass to obtain the carbon dioxide content in each depth. Carbon dioxide content was presented as mass percentage of anhydrous cement.

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