



The experimental investigation of width of semi-carbonation zone in carbonated concrete



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HIGHLIGHTS

- Reaction zone of carbonated concrete is determined to chemical component change.
- The partially carbonated zone width was greater than that of pH value variation.
- Carbonated concrete is divided into four zones.
- Carbonation process of concrete is classified into three stages.

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ABSTRACT

An accelerated carbonation experiment was carried out under artificial environment with high CO₂ dosing to investigate the concentration distributions of the Ca(OH)₂ and CaCO₃ by chemical analysis methods as well as the pH value profiles. The results indicate that the distributions of phase components are not consistent with that of the pH value in corresponding zone of carbonated concrete. The cross section of carbonation concrete shows complete carbonation zone, semi-carbonation zone with variable pH value, semi-carbonation zone with constant pH value, and non-carbonation zone. In the light of formation sequence of semi-carbonated zone and completely carbonated zone, the development process of concrete carbonization is divided into three stages: (1) from the beginning of carbonation to the point when pH on concrete surface starts to decrease; (2) from the point when pH on concrete surface starts to decrease to the point when the alkaline component is depleted; (3) from the point when the alkaline component is depleted to any time.

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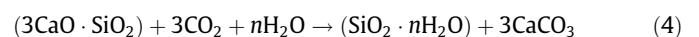
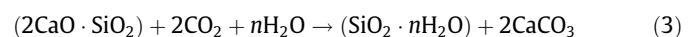
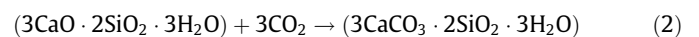
1. Introduction

The corrosion of steel reinforcement is a form of deterioration in reinforced concrete structures. The passivating film that protects steel in concrete is destroyed when corrosion occurs due to the contact of oxygen and water with steel surface. Carbonation is one of the main reasons for passivating film destruction [1–3]. Carbonation is a slow process in which carbon dioxide in the atmosphere (0.03% by volume) diffuses into concrete and alters the hydrated cement paste. Carbonation does not impact the strength of concrete but it affects the pH value change of the solution in concrete pores caused by dissolved CO₂ and its reaction with water and hydrated products of cement. Carbonation reaction begins at

the surface of the concrete and migrates inward progressively with time [4–6].

The carbonation of concrete is a complex physicochemical process. The process takes place in the cement paste component of concrete, whereas fine and coarse aggregates are inert, at least as far as carbonation is concerned.

The solids which react with CO₂ include not only Ca(OH)₂, but also the main strength element of cement paste: calcium silicate hydrate 3CaO·2SiO₂·3H₂O (CSH); unhydrated constituents, 3CaO·SiO₂ (C₃S) and 2CaO·SiO₂ (C₂S) [7,8]. The main carbonation reactions are



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These reactions induce a reduction of the pH of the pore solution to such a value that the steel reinforcement is no longer protected against corrosion. Various indicators have been employed to identify the front of pH change with different pH thresholds, among which phenolphthalein is most commonly used [9]. The carbonation front is able to be identified by simply spraying phenolphthalein indicator dissolved in alcohol onto a freshly fractured concrete surface [10]. The solution is a colorless acid/base indicator, which turns purple when the pH value is above 9. However, this test does not indicate the depth of maximum ingress of CO₂. In reality, the penetrating gas could have reacted at greater depths of the concrete, causing a decrease in the pH-value beyond the carbonation depth indicated by the phenolphthalein spray test. So the propagation of carbonation is more complex than the results suggested by the pH change, with the degree of carbonation (i.e., the pH change) varying with depth rather than being discrete. Because measuring carbonation depth by spraying phenolphthalein indicator is much easier than that by testing the chemical components of the concrete cover. Therefore, spraying phenolphthalein is still an efficient way.

The presence of semi-carbonation zone is introduced to explain this phenomenon by Parrot. Parrot [11] found that the corrosion of steel in concrete took place ahead of the carbonation depth indicated by phenolphthalein. Additionally, the rate of corrosion kept increasing with the carbonation depth until this depth reached certain limit. Three zones are defined according to their carbonation degree by researchers as completely carbonated zone, partially carbonated zone and non-carbonated zone separately [12].

Carbonation changes the phase components of concrete. The Ca(OH)₂ content in the semi-carbonation zone decreases and CaCO₃ content increases gradually with the depth. In this case, the pH value of the concrete gradually increases with the depth into the concrete due to the consumption of Ca(OH)₂. Non-carbonated concrete has a pH value of approximately 12.5–13, whereas the completely carbonated concrete is more neutral with a lower pH value of about 8.5–9.0 [13,14]. From the mechanisms of the corrosion of steel in concrete, the rate of corrosion decreases with pH value, remains constant and approaches zero when the pH values are in the range of 9–11.5, less than 9, and greater than 11.5, respectively [11]. The pH values near the steel drops with the progress of carbonation, and consequently the rate of corrosion increases. The rate of steel corrosion is considered stable in the completely carbonated zone. Therefore, some researchers believed that the variation zone of the pH values was equivalent to the carbonation reaction zone. But strictly speaking, this is true only when the variation zone of phase components of concrete is the same as “the carbonization reaction zone”. Other researchers have reported the pH distribution of carbonated concrete [15,16]. However, to the authors' best knowledge, there has been no discussion on the relationship between the pH distribution and the degree of carbonation in carbonated concrete.

Existence of semi-carbonation zone delays the growth and its rate of complete carbonization zone. The longer the semi-carbonation zone, the slower the rate of complete carbonization is. Most researchers do not consider the partially carbonated zone in concrete carbonation models [4,17–21]. This simplification may lead to the over-estimation of the real carbonation depth. Therefore, accurate determination of the semi-carbonation zone is important to the understanding of diffusion–reaction mechanism of CO₂ in concrete, and to an accurate carbonation model.

The objective to this work is to introduce a new method to determine the semi-carbonation zone based on the chemical compositions. A comparison between this approach and the one commonly used based on pH indicator is conducted by theoretical analysis and experimental validation. In the experiments, the width of semi-carbonation zone in carbonated concrete was

determined using both pH indicator and chemical composition measurement. First, accelerated carbonation experiments were carried out in an artificial environment with high CO₂ concentration. The pH value profiles within the samples were measured using a pH indicator. The length of semi-carbonation zone determined from the variation of Ca(OH)₂ and CaCO₃ content are compared with the results obtained using the pH indicator. The relationship between the distribution of pH values and the degree of carbonation in carbonated concrete are also discussed. Furthermore, a theoretical study is proposed to understand the fact that the carbonation front is gradual and to link it to the kinetics of the chemical reactions which are at stake. In the light of formation sequence of partially carbonated zone and completely carbonated zone, the development process of concrete carbonization is divided into three stages.

2. Theoretical analysis

2.1. Formation of semi-carbonated zone and completely carbonated zone

Concrete is porous material. When carbonation occurs, CO₂ in air penetrated into concrete pores and capillaries filled with air and dissolved into liquid phase, where CO₂ reacted with Ca(OH)₂ in cement gel to form CaCO₃. CaCO₃ selectively precipitated first due to its low solubility. The major hydration products from Portland cement are hydrated calcium silicate (C–S–H) and calcium hydroxide (Ca(OH)₂). The completely hydrated C–S–H in cement paste makes up 50–60 vol% of the solid phase, whereas Ca(OH)₂ makes up 20–25 vol% of solid phase. Along with the carbonation process, although OH[−] in concrete pore solution was consumed continuously, the pore solution was always saturated with Ca(OH)₂ because crystal Ca(OH)₂ dissolved into solution continuously to supply more ions. Since the concentration of OH[−] remained constant, pH value of concrete solution remained relatively high, until all Ca(OH)₂ in solid phase was depleted. Without sufficient OH[−] supply to concrete pore solution from solid Ca(OH)₂, OH[−] concentration decreased due to further consumption in reaction with CO₂, and pH value of concrete pore solution decreased correspondingly. When all Ca(OH)₂ in concrete pore solution was consumed, complete carbonation of concrete was realized. Therefore, the distributions of phase components are not consistent with that of the pH value in corresponding zone of carbonated concrete. The cross section of carbonation concrete in Fig. 1 showed complete carbonation zone, semi-carbonation zone with variable pH value, semi-carbonation zone with constant pH value, and non-carbonation zone.

2.2. CO₂ mass transfer and chemical absorption in concrete

Concrete carbonation is controlled by mass transfer and chemical reactions. The mass transfer rate N_A can be expressed as

$$N_A = K_g a (C_s - C_i) \quad (5)$$

where k_g – gas film mass transfer coefficient; a – specific surface area of cement paste (measured with mercury intrusion porosimetry); C_s – CO₂ concentration in unit volume air on concrete surface; C_i – CO₂ concentration in unit volume air in concrete capillary.

The reaction rate r_A of CO₂ chemical absorption can be expressed as

$$(-r_A) = k C_i \quad (6)$$

where k – reaction rate coefficient.

Under steady state, assuming CO₂ entering the concrete is completely consumed by chemical reactions,

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