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Cement and Concrete Research 36 (2006) 1760–1767

The experimental investigation of concrete carbonation depth

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Received 13 November 2003; accepted 13 July 2004

Abstract

Phenolphthalein indicator has traditionally been used to determine the depth of carbonation in concrete. This investigation uses the thermalgravimetric analysis (TGA) method, which tests the concentration distribution of $Ca(OH)_2$ and $CaCO_3$, while the X-ray diffraction analysis (XRDA) tests the intensity distribution of $Ca(OH)_2$ and $CaCO_3$. The Fourier transformation infrared spectroscopy (FTIR) test method detects the presence of C–O in concrete samples as a basis for determining the presence of $CaCO_3$. Concrete specimens were prepared and subjected to accelerated carbonation under conditions of 23 °C temperature, 70% RH and 20% concentration of CO_2. The test results of TGA and XRDA indicate that there exist a sharp carbonation front. Three zones of carbonation were identified according to the degree of carbonation and pH in the pore solutions. The TGA, XRDA and FTIR results showed the depth of carbonation front is twice of that determined from phenolphthalein indicator.

Keywords: Carbonation; $Ca(OH)_2$; $CaCO_3$; pH

1. Introduction

Carbonation is known as a neutralizing process, a chemical reaction of $Ca(OH)_2$ and calcium–silicate–hydrate (C–S–H) with CO_2 to form $CaCO_3$ and water [1]. Carbonation reduces the hydroxide concentration in the pore solution, destroying the passivity of the embedded reinforcement bars [2]. The traditional way of determining the depth of carbonation is to spray phenolphthalein indicator onto the surface of a freshly split concrete prism. The solution is a colorless base indicator, which turns purple when the pH is above 9. Past evidences have shown that a partially carbonated zone exists where pH is difficult to detect using the phenolphthalein indicator [3–5]. Papadakis et al. [6] found from both the thermalgravimetric analysis (TGA) and the phenolphthalein colorless depth that there exists a sharp carbonation front for relative humidity (RH) above 50%, but not for results of RH below 50%.

Previous studies have shown that TGA and X-ray diffraction analysis (XRDA) can determine the concentration and relative intensities of $Ca(OH)_2$ and $CaCO_3$, respectively [3,7]. Matsushita et al. [8] proposed the carbonation degree (D_c) of autoclaved aerated concrete as $D_c(\%)=(C-C_o)/(C_{max}-C_o)\times 100$ where C, C_o and C_{max} are the amount of CO_2 in the sample, the amount of CO_2 in the noncarbonated sample and the theoretical amount of CO_2 needed to combine with the total CaO in the sample to form $CaCO_3$, respectively. Fourier transformation infrared spectroscopy (FTIR) has also been used to detect the spectral changes associated with the presence of $Ca(OH)_2$ at the interface between the cement paste and the aggregate [9]. Changes in the FTIR spectra have also been noted for C–S–H that has undergone phase transformations under different stress levels [10]. Carbonation was found in the partly carbonated zone of concrete where the presence of both $CaCO_3$ and $Ca(OH)_2$ were observed, while in greater depth of concrete, $CaCO_3$ is not detected.

Several reports have discussed the deterioration of carbonated concrete, proposing a formula to describe carbonation [2,7,11]. Few studies, however, discuss the relationship between the true carbonation depth of concrete

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doi:10.1016/j.cemconres.2004.07.025

using the TGA, XRDA and FTIR test methods and the colorless depth tested using the phenolphthalein method, concurrently.

This investigation compares the carbonation depths determined from the TGA, XRDA and FTIR methods, with the results obtained using the phenolphthalein indicator. The relationship between the distribution of pH values and the degree of carbonation in carbonated concrete are also discussed.

2. Experimental procedure

2.1. Proportions of the concrete mix

Type I Ordinary Portland Cement (OPC) was used in the concrete mix for the test specimens. Table 1 gives the chemical and physical properties of the cement. The fine aggregate used was river sand with specific gravity, absorption (%) and fineness modulus 2.63, 2.41 and 2.85, respectively. The coarse aggregate used was crushed basalt with nominal grain size of 13 mm, the specific gravity, absorption (%) and fineness modulus were 2.65, 0.89 and 6.13, respectively. Table 2 presents the proportions of constituents in the mix and its compressive strength, for a water–cement ratio of 0.65.

2.2. Casting and curing of test specimens

Twenty-four cylinders (150×300 mm) were cast for this test. After casting, test specimens were covered with plastic sheets, and left in the casting room for 24 h at a temperature of about 23 °C. The specimens were then demolded and placed into a curing room at 23±1.7 °C and 100% RH until testing.

Table 1
Chemical and physical properties of cement

Results of chemical analysis	Cement (%)	Requirements ASTM C150 Type I (%)
Silicon dioxide, SiO_2	21.04	–
Aluminum oxide, Al_2O_3	3.51	–
Ferric oxide, Fe_2O_3	2.84	–
Sulfur trioxide, SO_3	1.78	3.0 max.
Calcium oxide, CaO	68.02	–
Magnesium oxide, MgO	2.03	6.0 max.
Titanium dioxide, TiO_2	0.0	–
Potassium oxide, K_2O	0.53	–
Sodium oxide, Na_2O	0.19	–
Loss on ignition	1.55	3.0 max.
Physical tests		
Air content (%)	7.01	12 max.
Fineness (m^2/kg)	385	280 min.
Autoclave expansion (%)	−0.03	0.8 max.
Specific gravity	3.12	–
Compressive strength (Mpa)		
1-day	9.21	–
3-day	27.94	12.6 min.
7-day	37.23	19.3 min.
28-day	52.11	–

Table 2
Mixture proportions and compressive strength test results

Mixture proportions (W/C=0.65)				
kg/m^3				%
Cement	Water	Coarse aggregate	Fine aggregate	Air content
307	200	1135	745	3.2
Compressive strength (MPa)				
Curing time (days)			After curing and accelerated carbonation (weeks)	
1	7	28	8	16
4.9	18.1	24.2	52.1	56.5

2.3. Accelerated carbonation

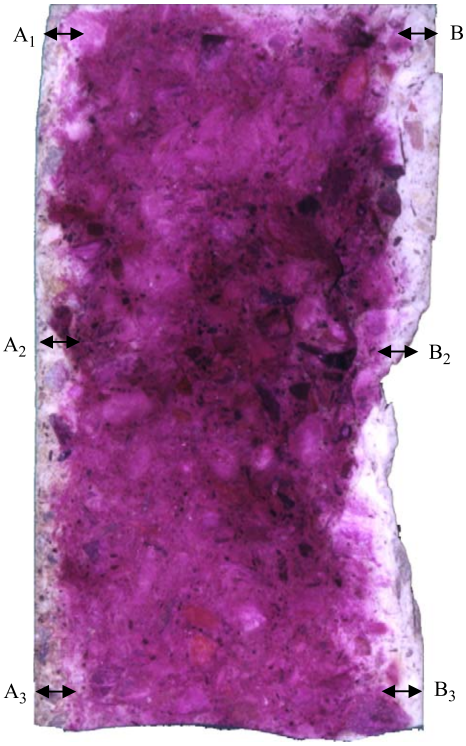
After curing for 28 days, the two ends of each cylinder were coated with epoxy resin to ensure that carbon dioxide could diffuse only into the specimens in a two-dimensional mode. The specimens were transferred to a sealed chamber and subjected to carbonation at 23 °C in temperature, 70% relative humidity and a CO_2 concentration of 20% by volume for 8 and 16 weeks.

2.4. Test of depth of colorless region using phenolphthalein indicator

The test cylinders were taken out of the carbonation chamber at 8 and 16 weeks and split in a tensile splitting test. After splitting the concrete specimens, the freshly split surface was cleaned and sprayed with a phenolphthalein pH indicator. The indicator used was a phenolphthalein 1% ethanol solution with 1 g phenolphthalein and 90 ml 95.0 V/V% ethanol diluted in water to 100 ml [3]. In the noncarbonated part of the specimen, where the concrete was still highly alkaline, a purple-red color was obtained. In the carbonated part of the specimen where the alkalinity of concrete is reduced, no coloration occurred. The average depth 'X_p' of the colorless phenolphthalein region was measured from three points, perpendicular to the two edges of the split face, both immediately after spraying the indicator and at 24 h later. Fig. 1 presents the mean depth of the region of colorless phenolphthalein 'X_p'.

2.5. Thermogravimetric analysis (TGA)

Split face of the concrete specimens was oven-dried prior to taking samples from the split surface in belts of 5-mm width consecutively away from the edge of the split specimen. Sample powder was then obtained using a 0.3-mm sieve. With respect to regions of the split surface where



$$X_p = \frac{A_1 + A_2 + A_3 + B_1 + B_2 + B_3}{6}$$

Fig. 1. Cross-section of a split concrete cylinder specimens after carbonation.

samples were not taken yet, plastic film was used to avoid carbonation of the fresh surface from CO_2 in the ambient air. The powder sample was placed in a crucible designed for use with the thermogravimetric analysis instrument. The temperature was set to increase at a rate of 20 °C/min from room temperature to 1000 °C. Temperature as a function of thermogram was recorded to provide qualitative and quantitative information. The thermogravimetric analysis instrument used was a U.S. SDT 2960 SIMULTANEOUS DSC-TGA, controlled by Thermal Analysis and Rheology software. The test results were analyzed using Thermal Solution analysis software.

The molar concentrations of $Ca(OH)_2$ and $CaCO_3$ were computed based on the thermogravimetric curve determined from the thermogravimetric analysis test for concrete specimens subjected to various periods of accelerated carbonation, and also from the rate of weight loss indicated by the thermogravimetric curve which included the dehydration of $Ca(OH)_2$ and thermal decomposition of $CaCO_3$. Based on the above facts, changes in the amounts of calcium carbonate in concrete specimens under various periods of carbonation could be analyzed [12].

2.6. Powder X-ray diffraction analytical methods

XRD analysis was conducted on a Shimazu Model XD-3. The relative diffraction peaks of $CaCO_3$ and $Ca(OH)_2$ in powder X-ray diffraction analytical spectra of concrete powder dried at 105 °C were compared using an apparatus for powder X-ray diffraction analysis. Its operating conditions were CuKá radiation (40 kV, 100 mA); scan rate: 4°/min; 2θ: 10–70° [12]. From Fig. 5(a), it is evident that there exists a peak for $CaCO_3$ but no peak for $Ca(OH)_2$; hence, this sample could be regarded as fully carbonated. Fig. 5(c) shows there exists only a peak for $Ca(OH)_2$ and no peak for $CaCO_3$; hence, the sample could be regarded as being noncarbonated. Both peaks $CaCO_3$ and $Ca(OH)_2$ exist in Fig. 5(b), which indicates that the sample is partly carbonated [3].

2.7. FTIR spectrum analysis

The test cylinders were split in a tensile test. Powder samples were taken from the split cylinder at depths of 0, 5, 10, 15, 20, 25, 30 and 35 mm measured from the edge of the split surface. The IR spectrum of each powder sample mixed with KBr in a proportion of 1:10 was obtained to facilitate quantitative measurement of the depth of carbonation. The background spectrum of the laboratory environment was scanned before the powder sample was scanned. Carbonation is represented by the transformation of the C=O bonds of CO_2 into C–O bonds in the $CaCO_3$. Thus, a study of the characteristic peak of the C–O functional group in the wave number range of 1410–1510 cm^{-1} would identify the carbonation in concrete [12].

2.8. Test of pH in the pore solutions

Crumbled particles of concrete mortar, sized 2–5 mm, weighing 10–20 g and in aqueous exchange water in a solid to liquid ratio of 1:10, were placed in a sealed container under 15 °C for 20 days, and then filtered through filter paper with a pore size of 0.45 μm. The pH value was tested using a pH microelectrode [12].

3. Results and discussion

3.1. TGA test result

3.1.1. Characteristics of the thermogravimetric analysis curve

In the studies conducted by Sha and Pereira [13], Parrott and Killoh [14] and Papadakis et al. [6], when the concrete specimen was heated to 1000 °C in a TGA instrument, the relationship between the temperature and the weight loss of the specimen is as shown in Fig. 2 [15]. In this figure, the TG curve (thermogravimetric analysis curve) is a curve showing the percentage change in the weight (right vertical axis) as the temperature increases. The first derivative of the thermogravimetric analysis curve (TG curve) with respect to temperature yields the DTG curve, which is also shown in Fig. 2, using the left vertical axis. The positions of the four

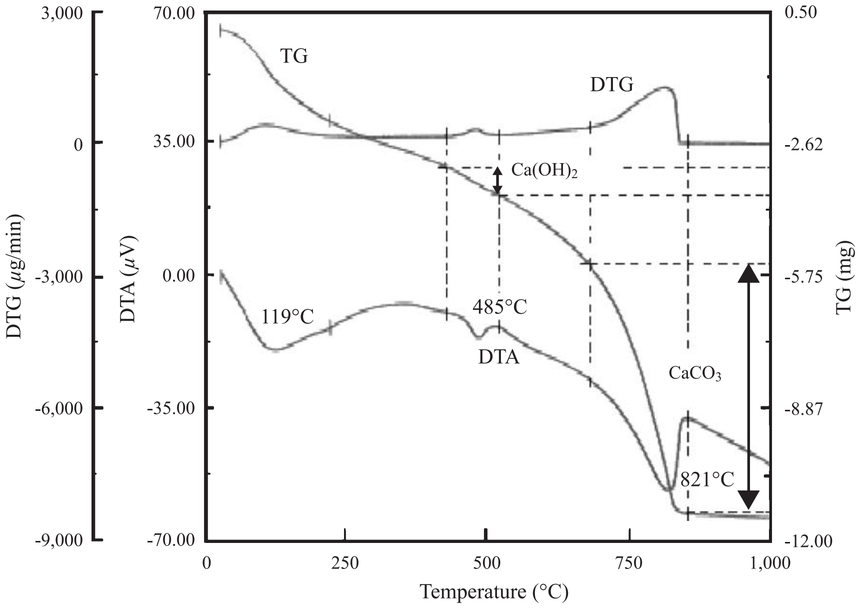


Fig. 2. The concrete characteristic curves of thermogravimetric and differential thermogravimetric analysis with temperature.

peaks in this derivative curve (DTG curve) indicate the percentage weight loss in temperature range of the TG curve. The first peak appears at about 100 °C, when the weight of the water absorbed in the concrete is lost. Absorbed water is present in the large pores of the specimen, and is attached to the walls of the pore by surface tension. The second peak is around 130 °C, where the interlayer water in the concrete is lost. Interlayer water is present in the microscopic pores of the concrete. Very often, the loss of interlayer water will change the sizes of the crystalline structure, without breaking it down. The third peak appears between 425 and 550 °C, and corresponds to the weight loss due to dehydration of calcium hydroxide, as given by Eq. (1). The fourth peak, which is between 550 and 950 °C is caused by the weight lost due to the decomposition of $CaCO_3$ and the consequent releases of CO_2, as described in Eq.

$$Ca(OH)_{2(S)} \rightarrow CaO_{(S)} + H_2O_{(g)} \tag{1}$$

$$CaCO_{3(S)} \rightarrow CaO_{(S)} + CO_{2(g)} \tag{2}$$

3.1.2. Results of thermogravimetric analysis

When concrete specimens are carbonated, the calcium hydroxide in them gradually combines with carbon dioxide to form calcium carbonate. The third and fourth peaks of the DTG curve, representing the thermogravimetric percentage loss in the thermogravimetric analysis test reveal that the weight loss associated with the dehydration of calcium hydroxide and the decomposition of calcium carbonate to carbon dioxide can be determined. From Eqs. (1) and (2), multiplying the two weight losses by the molecular proportions $M_{Ca(OH)_2}/M_{H_2}O$ and M_{CaCO_3}/M_{CO_2}, respectively, yields $M_{Ca(OH)_2}$, $M_{H_2}O$, M_{CaCO_3} and M_{CO_2}, which are the masses of 1 mol of $Ca(OH)_2$, H_2O, $CaCO_3$ and CO_2, respectively. Hence, the proportions by weight of calcium hydroxide and calcium carbonate in the sample can be determined. Figs. 3 and 4 clearly show that as the degree of carbonation increases, more calcium carbonate develops while more calcium hydroxide is exhausted. If the area in which the phenolphthalein indicator is colorless is defined as the depth (X_p) of full carbonation, and the transition point where the concentration distribution of $Ca(OH)_2$ and $CaCO_3$ approaches the horizontal as the carbonation front depth (X_C), then Figs. 3 and 4 show that the depth of the fully carbonated depth is approximately half of the depth of the carbonation front (X_p=1/2 X_C).

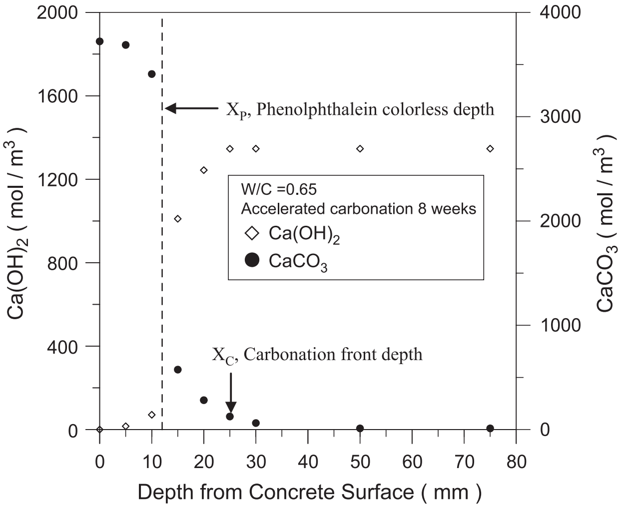


Fig. 3. Relationship between phenolphthalein colorless depth and $CaCO_3$–$Ca(OH)_2$ concentration distributions by TGA of accelerated carbonation for 8-week concrete.

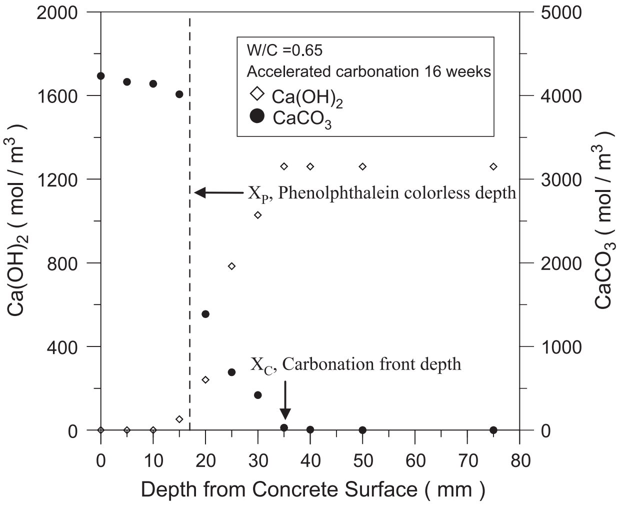


Fig. 4. Relationship between phenolphthalein colorless depth and $CaCO_3$–$Ca(OH)_2$ concentration distributions by TGA of accelerated carbonation for 16-week concrete.

3.2. Analysis of XRDA test results

As shown in Fig. 5, the X-ray test results indicate four main crystalline structures in the concrete sample: quartz, feldspar, $Ca(OH)_2$ and $CaCO_3$. Among these, quartz and feldspar come from aggregates and are basically the same in all samples. However, the contents of $Ca(OH)_2$ and $CaCO_3$ varies with the age of carbonation of the sample [3]. Figs. 6 and 7 present the distributions of $Ca(OH)_2$ and $CaCO_3$ in concrete specimens with a 0.65 water–cement ratio, which had undergone accelerated carbonation for 8 and 16 weeks. At 8 weeks of accelerated carbonation, the surface has not yet been fully carbonated; $Ca(OH)_2$ and $CaCO_3$ are both observed in varying intensities. Notably, the $CaCO_3$ content decreases from the surface to the interior, while the $Ca(OH)_2$ content increases because carbonation is more intensive at the surface than in the interior, such that more $Ca(OH)_2$ has already been converted into $CaCO_3$ at the surface. As the period of carbonation increases, the $CaCO_3$ content increases as the $Ca(OH)_2$ content diminishes. When the accelerated carbonation reaches 16 weeks, all $Ca(OH)_2$ at the surface of the specimens is found to be completely converted into $CaCO_3$. If the transition point where the intensity distribution of $Ca(OH)_2$ and $CaCO_3$ approaches the horizontal is taken as the depth of carbonation front (X_C), then Figs. 6 and 7 show that the depth of the fully carbonated zone is approximately half the depth of the carbonation front (X_p=1/2 X_C).

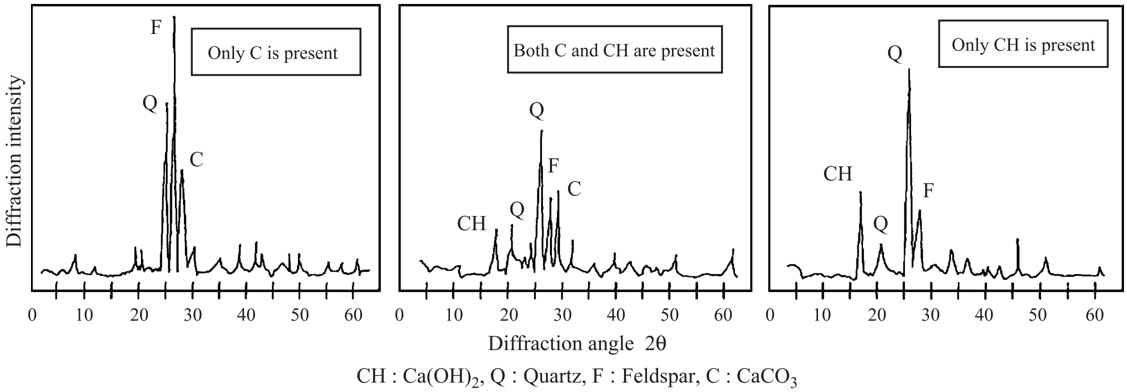
3.3. FTIR spectrum analysis

Carbonation depth is determined from the position of the C–O characteristic peaks relative to the baseline at wave number 1415. Figs. 8 and 9 are the infrared spectra after accelerated carbonation at 8 and 16 weeks. The theory that underlies the FTIR technique can be used to measure the presence of $CaCO_3$ from a level at which the pH is 11.5 to saturation with $CaCO_3$ at pH 8.3. Traditionally, phenolphthalein indicator measures the degree of carbonation at a pH of 9.0; hence, areas of partial carbonation where the pH ranges from 9.0 to 11.5 could not be tested. Consequently, the depths of carbonation in concrete were often underestimated. The test results plotted in Figs. 8 and 9 showed that the depth of carbonation that can be detected using FTIR is twice of that detected using phenolphthalein indicator.

3.4. Results of analysis and discussion of pH value of the pore solution

3.4.1. Definition of carbonation degree of concrete

This study refers to the definition of the degree of carbonation proposed by Matsushita et al. [8], as shown in Eq. (3). The content of calcium carbonate was calculated from the concrete specimens subjected to accelerated carbonation, based on the percentage of the weight loss at each sampling point due to the release of carbon dioxide during heating. The percentage of carbonation at each point can be calculated from Eq. (3). The scope of carbonation in



(a) Completely Carbonated (b) Partly Carbonated Zone (c) Fully uncarbonated Zone

Fig. 5. Results of accelerated carbonation test by X-ray.

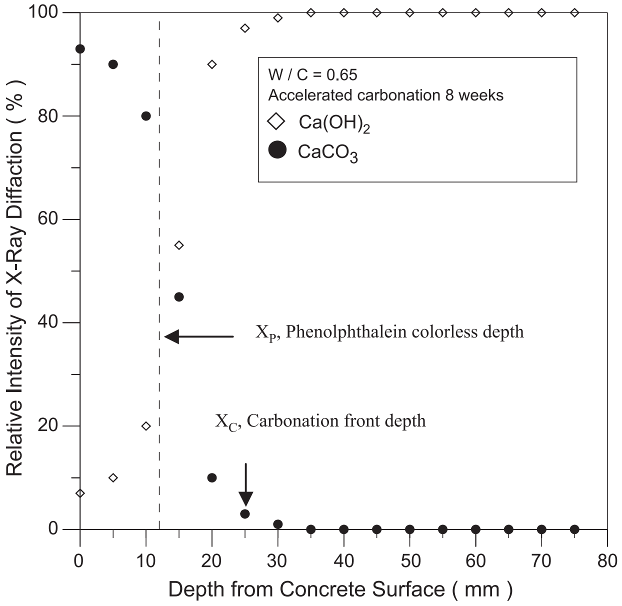


Fig. 6. Relationship between phenolphthalein colorless depth and $CaCO_3$–$Ca(OH)_2$ intensity distributions by X-ray of accelerated carbonation for 8-week concrete.

the specimen and its relative percentage of carbonation can thus be determined accurately (Fig. 10).

$$D_c(\%) = (C - C_o)/(C_{max} - C_o) \times 100 \qquad (3)$$

where $D_c(\%)$=carbonation degree; C_{max}=the theoretical amount of carbon dioxide needed to combine with the total calcium dioxide in the sample to form calcium carbonate; C=amount of carbon dioxide in the sample and C_o=amount of carbon dioxide in the noncarbonated sample.

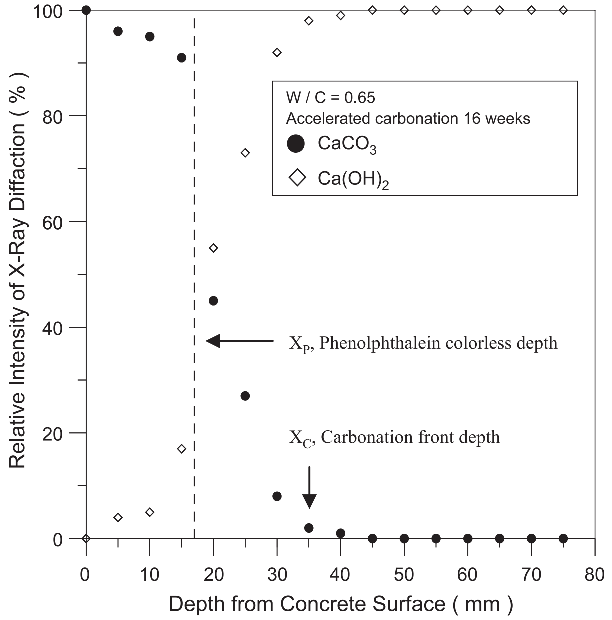


Fig. 7. Relationship between phenolphthalein colorless depth and $CaCO_3$–$Ca(OH)_2$ intensity distributions by X-ray of accelerated carbonation for 16-week concrete.

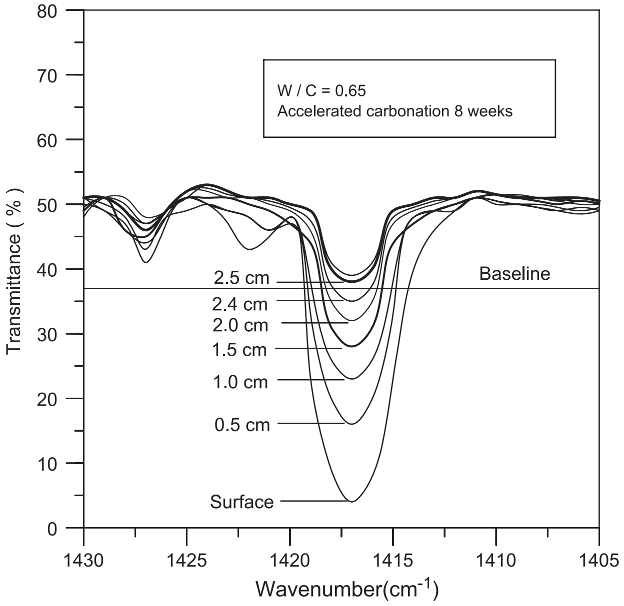


Fig. 8. Carbonation depth determined using FTIR under 8 weeks of accelerated carbonation.

3.4.2. *Degree of carbonation and the respective pH in the pore solution*

A passive protective film of γ-Fe_2O_3, 20–60 Å thick, was formed at the surface of the reinforcing steel bars because of the highly alkaline environment provided by the surrounding concrete. γ-Fe_2O_3 is a dense stable structure pH>11.5, such that neither water nor oxygen can permeate through such a film, leaving the reinforcing bars to be in a passive state and not corroded [16]. Phenolphthalein indicator, which is used conventionally to determine the depth of

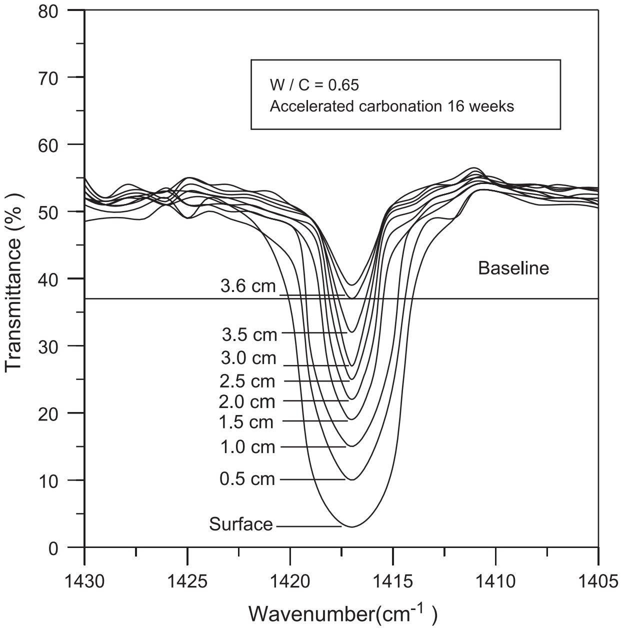


Fig. 9. Carbonation depth determined using FTIR under 16 weeks of accelerated carbonation.

carbonation in concrete, has a pH of approximately 9.0. Fig. 11 shows the relationships between the degree of carbonation in specimens and the respective pH in the pore solution; various relationships are established, as given by Eqs. (4)–(7). When the pH of the pore solution is less than 7.5, the degree of carbonation of the specimen is 100%. When the pH value of the pore solution is between 7.5 and 9.0, the degree of carbonation is 50–100%. When the pH of the pore solution is 9.0–11.5, the degree of carbonation is 0–50%. When the pH of the pore solution exceeds 11.5, the specimen is not carbonated. In this study, some readings were taken from the noncarbonated zone of specimens that has not been subjected to accelerated carbonation. The pH of the samples tested was greater than 11.5.

$$\text{pH}<7.5 \quad D_C = 100 \tag{4}$$

$$7.5<\text{pH}<9.0 \quad D_C = -33.33\text{pH} + 349.98 \tag{5}$$

$$9.0<\text{pH}<11.5 \quad D_C = -20\text{pH} + 230 \tag{6}$$

$$\text{pH}>11.5 \quad D_C = 0 \tag{7}$$

The above results indicate that the zones of carbonation can be identified as fully carbonated, partly carbonated and noncarbonated, according to the degree of carbonation of the concrete, in relation to the respective pH of the pore solution. The fully carbonated zone is defined by the colorless depth of the phenolphthalein indicator. In this area, the degree of carbonation exceeds 50%, and the pH of the pore solution in this zone is smaller than 9.0. In the partly carbonated zone, the presence of both Ca(OH)$_2$ and CaCO$_3$ is observed, and the degree of carbonation in the specimen lies between 0% and 50%. The pH of the pore solution in the partly carbonated zone lies between 9.0 and 11.5. For the noncarbonated zone, the pH of the pore solution is greater than 11.5, and no signs of carbonation are observed.

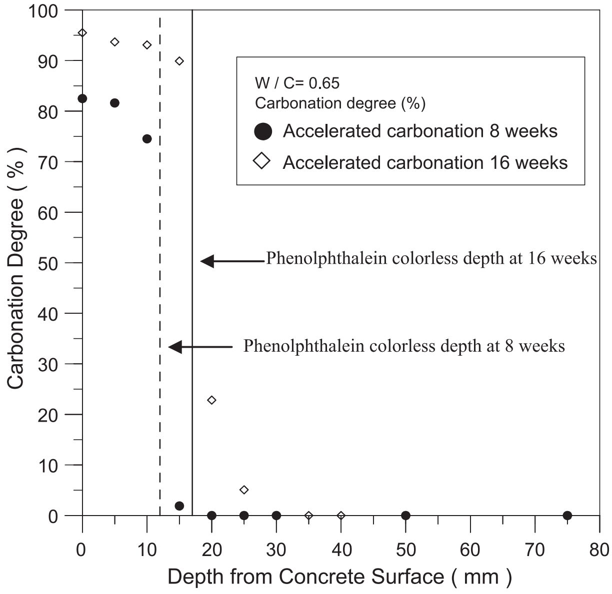


Fig. 10. Relationship between phenolphthalein colorless depth and carbonation degree distribution for concrete under 8 and 16 weeks of accelerated carbonation.

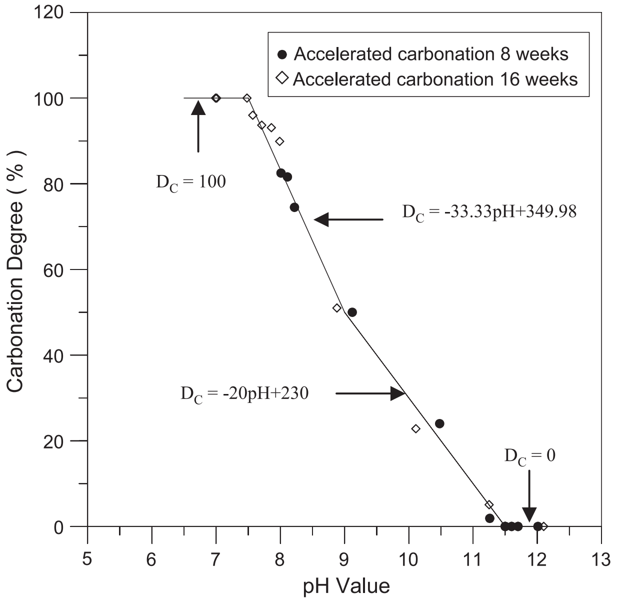


Fig. 11. Relationship between carbonation degree and pH for concrete under 8 and 16 weeks of accelerated carbonation.

4. Conclusions

In this study, the true depth of carbonation in concrete is determined using TGA, XRDA and FTIR instruments, respectively. These results were compared to the depth of colorless region determined using a conventional phenolphthalein indicator. The test results support the following conclusions.

(1) By defining the depth of the colorless region determined using the phenolphthalein indicator in carbonated concrete specimens as X_p, and the depth of carbonation determined from TGA, XRDA and FTIR as X_C, the relationship X_C/X_p is approximately equal to 2.0 (Table 3).

(2) The pH of the pore solution in concrete changes with the degree of carbonation. The boundary pH value using phenolphthalein indicator is generally 9.0, at which the degree of carbonation is 50%. When the pH of the pore solution equals 7.5, the degree of carbonation has reached 100%.

(3) This paper clearly identifies three distinct regions of fully carbonated, partly carbonated and noncarbonated zones in carbonated concrete. The fully carbonated zone is identified with the degree of carbonation greater than 50% (pH<9.0). The degree

Table 3
Relationship between phenolphthalein colorless depths and carbonation front depths obtained from Figs. 3, 4, 6, 7, 8 and 9

Accelerated carbonation time (weeks)	Phenolphthalein colorless depth (X_p, mm)	Carbonation front depth (X_C, mm)			X_C/X_p		
		TGA	X-ray	FTIR	TGA	X-ray	FTIR
8	12	25	25	24	2.1	2.1	2.0
16	17	35	35	35	2.1	2.1	2.1

of carbonation in the partly carbonated zone lies between 0% and 50% ($9.0<\text{pH}<11.5$). The non-carbonated zone is where the test specimen shows no signs of carbonation.

(4) TGA, XRDA and FTIR test methods were used to determine the true depth of carbonation in concrete. Each test method has its advantages, but the resulting depths of carbonation were similar.

(5) Identification of three zones of carbonation in concrete may help in applying concepts that pertain to composite materials to the analysis of the mechanical behaviors of carbonated concrete structures. However, concerning reinforcement corrosion, the pH value using phenolphthalein indicator is a good indication for corrosion initiation.

Notation

C	Amount of carbon dioxide in the sample
C_o	Amount of carbon dioxide in the noncarbonated sample
C_{max}	The theoretical amount of carbon dioxide needed to combine with the total calcium dioxide in the sample to form calcium carbonate
D_c	Carbonation degree
$M_{Ca(OH)_2}$	1 mol of molecular quantity for $Ca(OH)_2$
M_{H_2O}	1 mol of molecular quantity for H_2O
M_{CaCO_3}	1 mol of molecular quantity for $CaCO_3$
M_{CO_2}	1 mol of molecular quantity for CO_2
X_p	Colorless depth of phenolphthalein indicator measurement
X_C	The transition point where the intensity distribution of $Ca(OH)_2$ and $CaCO_3$ approaches the horizontal as the carbonation front

Acknowledgments

The experimental work was carried out in the material laboratory of the South Region Water Resources Bureau, Water Resources Agency, Ministry of Economic Affairs, Pingtung City, Taiwan. The authors gratefully acknowledge the assistance.

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