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- Technical Paper -

Estimating the effect of early-age carbonation curing of concrete on air permeability and surface resistance using NDTs

Kumar AVADH*1, Kengo SEKI*2, Takeshi TORICHIGAI*3

ABSTRACT

Curing pre-cast concrete products under high temperature, low relative humidity, and elevated CO_2 levels, facilitates the storage of CO_2 as $CaCO_3$, thereby transforming the concrete into a carbon sink. To ensure consistent quality of such products, it is essential to ascertain the depth to which CO_2 has been stored. This study investigated the feasibility of using Torrent permeameter and Wenner probe as non-destructive testing (NDT) methods to determine the effect of early-age carbonation curing on concrete's air permeability and electrical surface resistance, respectively, and their variation with respect to carbonation depth.

Keywords: Carbonation Curing, Carbonation Depth, CO_2 Utilization, Non-Destructive Testing (NDTs), Wenner Probe

1. INTRODUCTION

With cement production accounting for almost 8% of global CO_2 emissions, efforts to reduce concrete's carbon footprint have gained momentum, particularly through the reduction of cement content by incorporating industrial by-products like blast furnace slag (BFS) and fly ash etc. Another promising approach is early-age carbonation curing, where concrete containing non-hydraulic calcium silicate-based carbonation binders such as γ-C_2S is cured in a high CO_2 environment instead of water, allowing it to become a carbon sink. Combining these strategies has shown to further decrease concrete's carbon emissions [1,2].

Previous researches indicate that the carbonation curing of concrete at elevated temperatures, low relative humidity, and low initial strength facilitates CO_2 storage [2,3]. Due to the high water content during early age, the concrete needs to be dried to allow CO_2 to penetrate. As the water dries, CO_2 enters and dissolves in the pore water of the concrete and reacts with calcium hydroxide and calcium-silicate-based hydration products, precipitating $CaCO_3$ and lowering the pH. With the increase in the duration of carbonation curing, the depth to which concrete has dried increases along with carbonation depth and the amount of $CaCO_3$ precipitated, which alters the porosity and degree of water saturation of concrete. Given the dependence of carbonation rate on multiple factors such as materials used, water-cement ratio and carbonation curing conditions etc., it is important to develop and implement a robust quality assessment and control (QA/QC) framework to ascertain the amount of CO_2 stored in concrete products ensuring that the products are of uniform quality and have achieved prescribed CO_2 storage.

At the laboratory scale, the amount of CO_2 stored is expressed in terms of carbonation depth and the amount of $CaCO_3$ formed. As per current standards, the depth of carbonation in concrete is measured destructively using pH indicators such as phenolphthalein, while the quantity of $CaCO_3$ produced is determined through the utilization of analytical techniques, including TG-DTA, TOC, and XRD-Rietveld analysis etc. However, their application is constrained to the experimental level due to the necessity for expertise, cost and the dependence on pre-

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*1 Research Engineer　Kajima Technical Research Institute　Dr. Eng. JCI Member
*2 Senior Research Engineer　Kajima Technical Research Institute　JCI Member
*3 Chief Research Engineer　Kajima Technical Research Institute　Dr. Eng. JCI Member

treatment processes [4,5].

Natural carbonation has been shown to modify the pH, porosity and electrical resistance of concrete. Previous studies have utilized non-destructive testing (NDT) methods, including the Torrent method for air permeability and the Wenner method for surface resistivity measurements, to assess carbonation depth in concrete specimens subjected to 28 days of underwater curing followed by carbonation curing, simulating natural carbonation in existing structures [6]. Studies show that due to the precipitation of $CaCO_3$ on carbonation, the air permeability decreases due to pore clogging. Additionally, as $CaCO_3$ is an insulator, its increase in concrete pores, increases the electrical resistance of concrete. Hence, the application of these NDTs to develop a QA/QC framework for determining the depth of carbonation in early-age carbonation-cured products produced using low-carbon concrete needs to be investigated. Furthermore, in precast concrete factories, mix proportions and curing conditions are generally uniform, hence the variation in values measured using NDTs is also expected to be reduced. However, research on the application of these methods to evaluate the changes in early-age carbonation-cured concrete remains lacking. Furthermore, since the effects of carbonation may vary based on the type of binder used, it is essential to investigate the applicability of these methods to low-cement concrete specimens that incorporate industrial by-products as cement replacements [7].

This study aims to investigate suitability of various NDTs to predict carbonation depth in early age carbonation-cured low-cement concrete specimens. The relationship between change in air permeability measured using the Torrent method and surface electrical resistivity measured using the Wenner method with respect to carbonation depth was evaluated. The study was conducted on three distinct mix designs to determine the impact of carbonation curing low cement concrete on these properties.

Table 1: Physical properties of material used

Symbol	Name	Density	SSA*
		(g/cm^3)	cm^2/g
OPC	Ordinary Portland Cement	3.16	3310
γ-C_2S	Di-Calcium Silicate	2.85	1500
BFS	Blast Furnace Slag	2.80	4000
S	Crushed Sand	2.61	-
G	Gravel	2.65	-

*SSA: Specific surface area

2. EXPERIMENTAL SCHEME

2.1 Materials

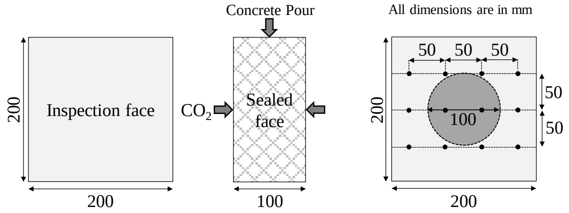
OPC as the primary binder with BFS and γ-C_2S as its replacement in low cement concrete mixes were used in this study. Their properties along with that of sand and gravel used to make concrete specimens are shown in Table 1.

2.2 Mix Proportion

The mix proportions considered in this study are shown in Table 2. Each concrete mix had the same water to powder ratio of 50%. The mix N consisted solely of cement while BB had 50% of cement replaced with BFS. In G30, the cement content was reduced to 35% compared to mix N, with the remaining 65% comprising of 35% BFS and 30% non-hydraulic binder γ-C_2S. Mix BB and G30 are low cement concrete. The target slump and air content were 15 ± 2.5 cm and 4.5 ± 1.5%, respectively for all the mixes.

2.3 Concrete specimen and curing conditions

Two distinct types of specimens were produced in



(a) Geometry (b) Points for NDTs

Figure 1: Details of specimen

Table 2: Mix proportion of concrete

Name	W/P*	W/C	Unit Weight (kg/m^3)						WR	AEA	Slump	Air
	(%)	(%)	W	C	BFS	γ-C_2S	S	G	(%×P)	(A)	(cm)	(%)
N	50	50.0	175	350	0	0	813	958	1.0	1.5	15.0	5.1
BB	50	100.0	175	175	175	0	807	951	0.8	2.0	15.5	4.8
G30	50	142.3	175	123	123	104	807	951	1.4	1.5	15.0	5.6

*P = C+BFS+γ-C_2S WRA=Water Reducing Agent AEA = Air Entraining Agent 1A = 0.001%×P this study. The first were cylindrical specimens with a diameter of 100 mm and a height of 200 mm, to assess the progress of compressive strength. The second type was rectangular specimens, measuring 200 mm×200 mm×100 mm as shown in Figure 1. Concrete was poured into the molds from the 100 mm×200 mm face to ensure consistent quality in the two square inspection faces. After casting, the specimens were cured at 20°C and 60% relative humidity (RH) for 24h and then demolded. Subsequently the four rectangular sides (100 mm×200 mm) were sealed with aluminum tape to prevent CO_2 permeation, allowing only the two square inspection faces to be exposed.

Specimens were then carbonation-cured at 50°C, 50% RH, and 80% CO_2 concentration for 1d, 7d and 28d as shown by A, B and C, respectively in Table 3. Additionally, one specimen was cured for 7d at 50°C and 50% RH similar to B but in the absence of CO_2, denoted as condition BN. In total, four rectangular specimens were produced for each mix.

Table 3: Details of carbonation curing conditions

Name	Temp. (°C)	RH (%)	CO_2 (%)	Curing time
A	50	50	80	1d
B	50	50	80	7d
C	50	50	80	28d
BN	50	50	0	7d

2.4 Tests Conducted

(1) Compressive Strength

The compressive strength was assessed using cylindrical specimens with diameter of 100 mm and height of 200 mm as per JIS A 1108:2018. Tests were conducted following demolding after 1d of casting, as well as after 1, 7, and 28 d of carbonation curing.

(2) Carbonation Depth using pH indicator

To assess the depth of carbonation after the specified carbonation curing duration, the rectangular specimens were cut in the transverse direction to expose the cross-section, and phenolphthalein pH indicator was sprayed. The depth of carbonation was measured in accordance with JIS A 1152:2018, defined as the distance from the specimen's boundary to the dark pink-colored region at 10 mm intervals, followed by calculating the average of all measurements.

(3) Coefficient of air permeability

The impact of carbonation depth on the air permeability of concrete was evaluated using the Torrent method, which utilizes the double chamber method. It measures the pressure change in the inner chamber and calculates the coefficient of air permeability (kT) using (1). The test was performed by positioning the suction chamber at the center of the rectangular specimen, as indicated by the dotted circle of 100 mm in Figure 1(b). It was conducted multiple times on the carbonated surface of the specimen until a stable value was achieved.

$$kT = \left(\frac{V_c}{A}\right)^2 \frac{\mu}{2\varepsilon P_a}\left[\frac{ln\left(\frac{P_a+\Delta P}{P_a-\Delta P}\right)}{\sqrt{t}-\sqrt{t_0}}\right]^2 \tag{1}$$

kT = Torrent air permeability coefficient (m^2)
Vc = Volume of inner cell (m^3)
A = Cross-sectional area of inner cell (m^2)
μ = Viscosity of air (= 2×10^{-5} Ns/m^2)
ε = Estimated porosity of concrete (= 0.15)
P_a = Atmospheric Pressure (N/m^2)
ΔP = pressure rise in inner cell at the end (N/m^2)
t = Test duration (s)
t_0 = Test start time (60 s)

(4) Surface Resistivity

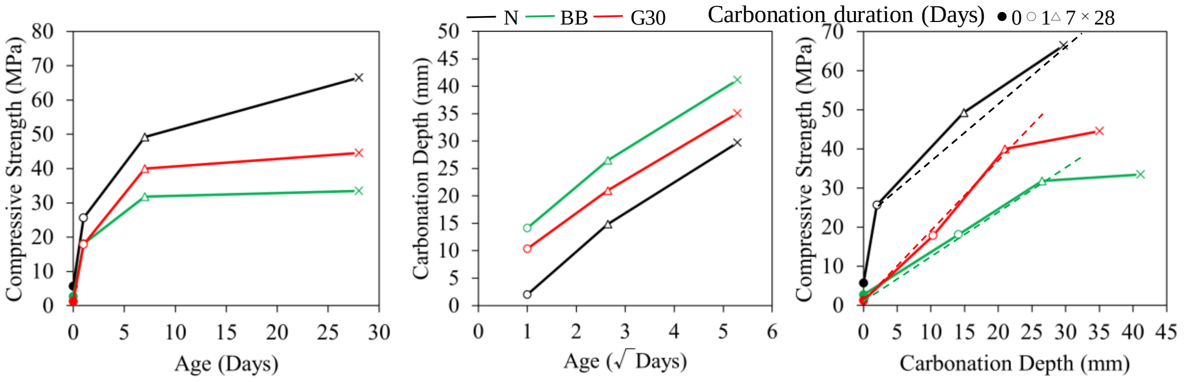
The influence of early-age carbonation curing on concrete surface resistivity was assessed using a Wenner method. The concrete resistivity is calculated using (2). The voltage difference and current intensity are measured through the four electrodes integrated into the device at points shown with black dots along the three dotted lines in Figure 1(b). An average of these values was calculated and considered for analysis.

$$\rho = 2\pi a\frac{V}{I} \tag{2}$$

ρ= Concrete resistivity (kΩcm)
a = spacing between two electrodes in m (=5 cm)
V = Voltage (V)
I = Current intensity (mA)

3. RESULTS AND DISCUSSION

This section discusses the results of the experiments conducted as a part of this study. Throughout this section, the results displayed in plots represent the outcomes corresponding to mixes N, BB and G30, in black, green and red colored lines, respectively. Additionally, the symbols "filled circle", "hollow circle", "hollow triangle" and "cross"



(a) Compressive strength

(b) Carbonation depth

(c) Change in compressive strength with carbonation depth

Figure 3: Investigating the relation between compressive strength and carbonation depth

correspond to carbonation curing durations of 0, 1, 7 and 28d, respectively.

3.1 Compressive strength and carbonation depth

Figure 3(a) illustrates the compressive strength of the cylindrical specimens on the y-axis and the duration of carbonation curing on the x-axis. The results show that the compressive strength of mix N consistently increases as carbonation curing progresses. The mix BB, which contains 50% less cement than N, exhibits a lower compressive strength of 31.8 MPa at 7d with a negligible increase to 33.5 MPa after 28d. For mix G30, which contains only 35% of cement compared to N and 30% non-hydraulic binder γ-C_2S, the compressive strength reaches to 40 MPa after 7d and 44.5 MPa after 28d. However, like BB, the strength gain from 7d to 28d is minimal.

Figure 3 (b) shows carbonation depth on the y-axis and the square root of carbonation duration on the x-axis with images in Figure 2. The results reveal that mix N, containing only cement, has the slowest carbonation rate, while BB is the fastest and G30 lies in between. As compressive strength is related to the porosity of concrete, mixes with higher compressive strength, have lower porosity, resulting in a slower rate of carbonation, which is in line with previous studies [2].

The plot in Figure 3(c) shows the relation between compressive strength in y-axis and carbonation depth on x axis. For mix N, after 1d of carbonation curing, the compressive strength jumps to 25.6 MPa, however the average carbonation depth is only 2 mm. Following this compressive strength increases linearly as shown by black dotted line. As for low cement mixes BB and G30, a linear correlation is apparent right from the beginning till 7d of carbonation curing. Subsequently, the increase in compressive strength is negligible. Hence, the compressive strength test on cylindrical specimens, as per JIS 1108, is related to carbonation depth. However, its application for low cement concrete is limited to early-age carbonation curing within 7 days. Since compressive strength testing is a standard QA/QC practice, this method can be easily adapted to assess carbonation depth.

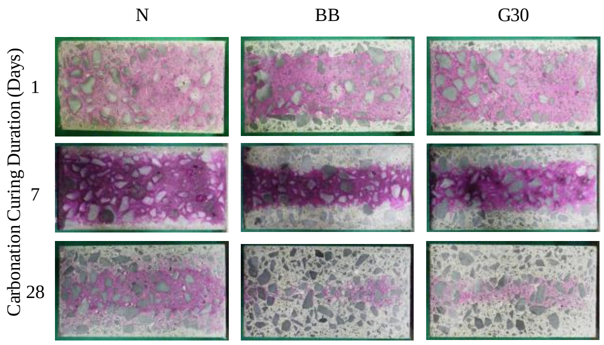


Figure 2: Images of cross-section sprayed with phenolphthalein after carbonation curing

3.2 Coefficient of Air permeability

Figure 4 shows kT on the y-axis and carbonation depth on the x-axis. For mix N, after 1d of carbonation curing, kT increased as the carbonation depth reached 2 mm. This increase can be attributed to the combined effects of curing at elevated temperature and reduced humidity, which caused the concrete to dry. As carbonation depth increased, kT decreased, indicating pore clogging due to $CaCO_3$ precipitation.

Mix BB demonstrated an increase in kT, with a more pronounced rise than observed in mix N after 1d of carbonation curing. Due to slower reactivity and lower compressive strength, a greater degree of drying was observed in BB. Carbonation curing for 7d lead to a decrease in the value of kT, indicating pore clogging. However contrary to mix N, carbonation curing of mix BB for 28d, exhibited a slight rise in kT, showing that the drying effect may have outweighed the pore clogging. Previous research [7] has shown that open porosity of concrete containing blast furnace slag coarsens on carbonation, which may also increase air permeability. Therefore, a detailed investigation of the porosity is necessary to better understand the obtained result.

In the case of mix G30, containing non-hydraulic binder γ-C_2S, the values of kT exhibited a similar increase after 1d of carbonation curing due to drying. As the carbonation curing continued, a notable decrease in air permeability was observed owing to pore clogging.

The early-age carbonation curing of concrete includes two distinct processes: the drying of concrete pores, which increases air permeability, and the precipitation of $CaCO_3$, which reduces air permeability. Furthermore, depending upon the mix proportion of the low-cement concrete, the changes in porosity of concrete on carbonation curing may also impact air permeability. Therefore, applying air permeability as a parameter and the Torrent method as an NDT to determine carbonation depth needs in-depth understanding and investigation.

3.3 Surface Resistivity

The relationship between carbonation depth and surface resistivity was quantified utilizing a Wenner probe. Figure 5 illustrates the variation in surface resistivity on the y-axis and carbonation depth on x-axis. In all three mixes N, BB, and G30, an increase in surface resistivity was observed with a progressive increase in carbonation depth. The increase was particularly notable in mixes BB and G30 in comparison to mix N, given that these are low-cement mixes with lower strength resulting in greater drying depth and carbonation depth. Excluding the surface resistivity value at 0 mm carbonation depth (before the start of carbonation curing), a linear increase in surface resistivity was observed with respect to carbonation depth for each mix. The linear equations displayed in Figure 5 shows a significant correlation with R^2 values of 0.96, 0.99, and 0.98 for mixes N, BB, and G30, respectively. The slope for mixes N, BB, and G30 were found to be 3.2, 8.2, and 12, respectively.

To further confirm if the increase in surface resistivity is due to both drying and carbonation and not just drying, specimens of all the mixes were cured under the BN condition as well. Under condition BN, the specimens were cured for 7d at the same temperature and relative humidity as B but without CO_2. The results, as illustrated in Figure 6, demonstrate concrete specimens cured in the absence of CO_2 show lower surface resistivity compared to those cured in the presence of CO_2. Hence, precipitation of $CaCO_3$ an insulator in concrete pores increases the surface resistance. As carbonation curing progresses, the combined effect of the increase in both the depth of drying and the amount of calcium carbonate precipitated in pores increases the electrical resistivity of concrete with respect to carbonation depth.

In conclusion, a linear relationship was observed between surface resistivity and carbonation depth in not

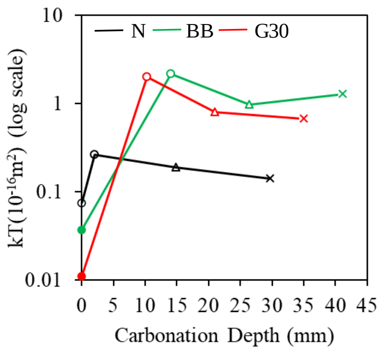


Figure 4: Change in air permeability

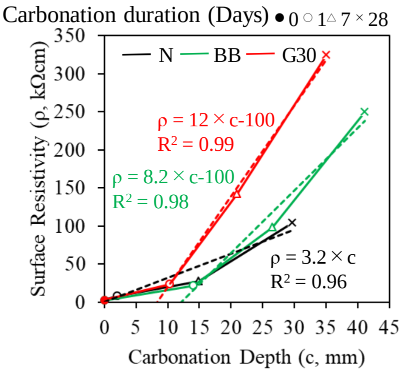


Figure 5: Change in Surface Resistivity

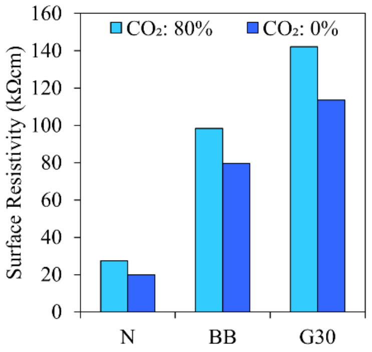


Figure 6: Effect of curing without CO_2 on surface resistance

just concrete made using only cement but low-cement concrete as well. However, further investigation on an increased number of specimens of different sizes is required to determine its reliability.

4. Conclusion

The effect of drying of concrete cover and pore clogging due to precipitation of $CaCO_3$ in concrete subjected to early-age carbonation curing was investigated using Torrent permeameter and Wenner probe NDT methods. The following conclusions were derived from the study.

1. As linear correlation was observed between compressive strength and carbonation depth, it is possible to use compressive strength as a method for QA/QC of carbonation depth. However, it cannot be applied to low cement concrete specimen which are carbonation-cured for more than 7d.
2. The coefficient of air permeability does not vary linearly with respect to carbonation depth and the trend seems to depend on the mix proportion of concrete.
3. The surface resistivity, as determined by Wenner resistivimeter, demonstrates a linear increase with carbonation depth, irrespective of the concrete mix. This strong correlation clearly demonstrates the suitability of surface resistivity measurement as a non-destructive testing (NDT) method for the estimation of carbonation depth.

Further investigation is required to ascertain the influence of carbonation conditions and size of specimens, in the relation between carbonation depth and surface electrical resistance to assess the reliability of the method. Analytical investigation in terms of measurement of the amount of calcium carbonate and change in porosity within concrete specimens is necessary in the future to understand the underlying mechanism of the observed results.

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