- Technical Paper -

### EVALUATION OF ELECTRICAL RESISTIVITY OF CARBONATED MORTAR UNDER DIFFERENT RH CONDITIONS

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### ABSTRACT

An experimental investigation was carried out to study the effect of carbonation degree on mortar electrical property. The Ordinary Portland Cement mortar specimens with different w/c ratios of 0.5, 0.6, 0.7 were accelerated carbonated in different carbonation duration of 0, 15, 30, and 50 days and then stored in six relative humidity (RH) conditions. Alternative current (AC)impedance method was performed to estimate the electrical resistivity of mortar. Results showed that different carbonation durations corresponding to different carbonation degrees. The effect of RH on electrical resistivity of mortar increase as RH decrease. Keywords: carbonation, RH, electrical resistivity, carbonation degree

### 1. INTRODUCTION

Carbonation is a chemical reaction of portlandite and calcium-silicate-hydrate (denoted as C-S-H) with carbon dioxide, resulting in calcium carbonate and water. Carbon dioxide penetrates concrete by diffusion. The decrement of the concrete initial alkalinity (pH = ~13) is one of the most significant consequences of carbonation. This fact is related to the depassivation of steel in reinforced concrete, contributing to the risk of rebar corrosion[1].

Due to the carbonation of concrete, mineralogical and microstructural changes occur. The main hydrates (portlandite and C-S-H) dissolution and calcium carbonate precipitation lead to the modification of physical properties in the carbonated zone. It is widely acknowledged that calcium carbonate precipitation leads to total porosity using mercury intrusion porosimetry[2] and generally leads to changes in transport properties[3][4].

Electrical resistivity is another important property of carbonated concrete that can be defined as the resistance of the concrete against the flow of an electrical current flow through the concrete[5]. It is influenced primarily by the ionic nature of the solution and the amount of water inside the pores of concrete. Electrical resistivity is largely depended on the microstructure properties of concrete, such as pore size and shape of the interconnections. Thus, the electrical resistivity of concrete is used as an indirect index to evaluate the corrosion possibility of embedded steel[6].

### 2. EXPERIMENTAL METHODS

### 2.1 Materials and Specimen

Ordinary Portland cement (OPC) was used throughout the experiment. The sand/binder (S/B) ratio was two, and the size of quartz sand was 0.2-1.7 mm (UBE INDUSTRIES 5A). The mortar used for all specimens was cast using the mixture proportions presented in Table 1.

Table 1 Mixture proportions for the mortar								
W/C	S/C	Water	Cement	Sand				
(%)	3/C	$(kg/m^3)$	$(kg/m^3)$	$(kg/m^3)$				
50	2	269	1346	673				
60	2	315	1261	631				
70	2	356	1186	593				

All the specimens were mixed until reached good fluidity in standard laboratory conditions with  $20\pm1^{\circ}$ C and cast in standard prism steel mold ( $40\times40\times160$  mm). Specimens were demoulded after one day and sealed in a curing room at  $20\pm1^{\circ}$ C for twenty-eight days. After the curing period, the samples were cut into slices by cutter for faster carbonation. The configuration for such slices (dimensions of  $3-4\times15\times40$  mm) is shown in Fig. 1.



# Fig. 1 Specimen configuration 2.2 Accelerated Carbonation

Accelerated carbonation was achieved using a chamber with 60% RH and a CO<sub>2</sub> concentration of 5%. Prior to carbonation process, all the specimens were kept in an oven at 105 °C for twenty-four hours. This process was intended to remove all the evaporable

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water from mortar and gas can easily penetrate the inside of specimen, aiming to enhance the rate of carbonation and homogenous carbonation.

The specimens with three W/C ratios were placed in carbonation chamber for 0, 15, 30, and 50 days to obtain different carbonation degrees. The specimens were denoted as 0.5-nc, 0.5-15d, 0.5-30d, 0.5-50d, 0.6-nc, 0.6-15d, 0.6-30d, 0.6-50d, 0.7-nc, 0.7-15d, 0.7-30d, 0.7-50d.

# 2.3 Characterization of the Carbonated Specimens

The specimens were stored at 11% RH and 20 °C for 14 days. The specimens were ground using a ball mill and particle size of powder was checked using a 75  $\mu$ m sieve. The powder was analyzed using a Bruker TG-DTA-2000SA to quantify changes in the amount of calcium hydroxide and calcium carbonate after carbonation. Approximately 40 mg of powder was placed in an alumina crucible. The weight was monitored while the powder was heated from 25 °C to 1000 °C at 10 °C/min in a nitrogen atmosphere flowing at 100 cm<sup>3</sup>/min.

The sample powder was combined with corundum powder (10 mass%) as a standard reference. To identify the phase compositions, powder X-ray diffraction (XRD) was conducted using a Rigaku MiniFlex powder diffractometer with Cu k $\alpha$  radiation, using a 2 $\theta$  scan range of 5°-65° and a scan speed of 2°/min.

### 2.4 RH Controlling

Following accelerated carbonation for three different duration of 15, 30, and 50 days, the mortar specimens were placed under various RH conditions. Different RH conditions were achieved using saturated salt solutions (Table 2) in desiccators at a temperature of  $20\pm1$  °C (Fig. 2)

Table 2 Relative humidity in the presence of saturated solution at 20±1 °C.

Salt solution	$K_2SO_4$	KNO <sub>3</sub>	KCl	KBr	KI
RH (%)	97.3	93.6	84.5	80.9	68.9



Fig. 2 Method for RH controlling of the specimens using saturated salt solutions in sealed containers.

The mass of the samples was monitored at regular intervals throughout the exposure period. Once all samples reached mass equilibration during the process, the electrical resistivity of mortar samples was measured.

### 2.5 Electrical Resistance Measurement

An LCR meter (IM3533-HIOKI) was used to obtain resistance over the frequency range of 0.1 Hz to 100 kHz by applying an AC perturbation with an amplitude voltage of 50 mV. Coaxial cables connected the measuring equipment to the electrodes. The electrode configurations used within the experimental program comprised two-point measurements, that is two external plate-electrodes.

In this arrangement, two copper plates with acrylic plates backing were placed against the two opposite faces of the mortar specimen. The faces were thoroughly cleaned with emery paper to remove any deposits that may have accumulating during storage. With reference to the schematic shown in Fig. 3, intimate contact between the specimen and the electrodes was achieved by means of a sponge (approximately 2 mm thick), which had been saturated with conductive gel. For two-point measurements using the LCR meter, leads from the current (high) and potential (high) connections were coupled at one of the electrodes, and leads to current (low) and potential (low) connections were coupled at the other electrode (refer to Fig. 3).



## Fig. 3 LCR arrangement for two-point electrical measurement

As the parallel plate ensure volume electrical conduction, the resistivity of the mortar specimen,  $R_{\Omega}$ , could then be evaluated[7]:

$$R_{\Omega} = \frac{\rho_{\Omega} \cdot A}{L} \tag{1}$$

where,

 $\rho_{\Omega}$  : mortar resistance measured by LCR

*A* : cross-sectional area of the specimen

L : distance between the electrodes

### 3. RESULTS

### 3.1 Specimen Characterization

	Specimen	Calcite	Aragonite	Ettringite	Portlandite	Vaterite
Non- carbonated	0.5-nc	2.8	0	3.4	4.6	0
	0.6-nc	5.7	0	3.5	6.5	0
	0.7-nc	3.8	0	5.4	8.5	0
Carbonated	0.5-15d	4	0.3	2.9	2.01	0
	0.5-30d	11.5	0	1.5	0.64	0
	0.5-50d	14	0.7	1.8	1.12	0
	0.6-15d	16	0.02	1.7	0.73	0
	0.6-30d	10.1	2.4	1.7	0.87	0
	0.6-50d	14.2	2.9	1.7	1.65	0
	0.7-15d	3.6	0.07	1.8	0.42	0.25
	0.7-30d	10.8	2.1	1.6	0.82	0
	0.7-50d	23	7.2	2.0	2.33	0

Table 3 Phase composition determined through XRD/Rietveld analysis of the cement paste (mass%)



Fig. 4 Carbonation degree of specimens for different carbonation duration calculated by XRD and TG

A Rietveld analysis was conducted to quantify the phase composition related to the carbonation according to the XRD diffraction patterns of mortar with three different w/c ratios for 0-, 15-, 30-, and 50day carbonation duration, and the results are summarized in Table 3.

Phase changes upon carbonation were identified by comparing the DTG-curves. The mass loss due to the decomposition of portlandite (CH) in the temperature range of 400 °C to 550 °C is determined using the tangential method, and the mass loss due to the decomposition of carbonates (CC) in the temperature range of 550 °C to 750 °C. The amounts of portlandite and calcium carbonate are calculated according to Eq. 2 and 3. The temperatures indicated here are only informative, the exact boundaries were determined based on the derivative curves[8]:

$$CH = (m_{450} - m_{550}) \times \frac{74}{18} \tag{2}$$

$$CC = (m_{550} - m_{750}) \times \frac{100}{44} \tag{3}$$

where,

т : weight loss at the indicates temperature 74 g/mol: molar mass of Ca(OH)<sub>2</sub>, 18 g/mol: molar mass of H<sub>2</sub>O 44 g/mol: molar mass of CO2 *100 g/mol*: molar mass of CaCO<sub>3</sub>.

With the quantified ratio of phase composition by XRD and TG analysis, a method for calculating the degree of carbonation  $\alpha_c$  is proposed to evaluate the carbonation conditions. The degree of carbonation is defined as the ratio of calcium oxide in calcium carbonate formed during the carbonation process to the initial calcium oxide per gram of cement. Therefore, the degree of carbonation can be expressed as:

$$\alpha_{c}(\%) = \frac{\frac{R_{cp}}{1 - LOI} \times \frac{56}{100} - R_{c}^{0} \times \frac{56}{100}}{R_{t}} \times 100$$
 (4)  
where

where,

 $R_{cp}$ : ratio of calcium carbonate (calcite, aragonite and vaterite) per gram of carbonated cement paste (Table 3)

 $R_C^0$ : initial ratio of calcium carbonate per gram of cement

*LOI*: ratio of water loss on ignition  $R_t$ : initial ratio of calcium oxide per gram of cement.



Fig. 5 Electrical resistance of the mortar specimens of different w/c ratio at different carbonation duration

3.2 Electrical Resistivity of Mortar Specimens in Various RH Conditions

Electrical resistivity of carbonated mortar specimens with different carbonation duration of 0, 15, 30, 50 days was measured. Fig. 5 describes the electrical resistivity evolution of the mortar specimens with different w/c ratios under various RH conditions over four carbonation duration. It shows that the electrical resistivity of mortar decreases with





increasing environmental RHs in general; when the RH range is below 80%, the resistivity decreases from 68% until 80% generally; when RH further increases to 97%, marked decrease is shown in electrical resistivity.

Before carbonation (carbonation duration=0), the mortar with the lowest w/c ratio (w/c=0.5) had the highest electrical resistivity while having the lowest electrical resistivity after the carbonation process under various RH conditions.

### 3.3 Electrical Resistivity of Mortar Specimens at Different Carbonation Duration

Fig. 6 shows the electrical resistivity of the mortar specimens at different carbonation duration under various RH conditions. It demonstrated that the electrical resistivity increases as carbonation increases under various RH conditions. Before carbonation, the obvious higher electrical resistivity ( $R_{\Omega}$ ) was shown in the mortar with a 0.5 w/c ratio at 68, 84, 94 and 93% RH conditions. However, the mortar specimen with a 0.5 w/c ratio had the lowest electrical resistivity during the carbonation process, while the mortar specimen with a 0.7 w/c ratio had the highest electrical resistivity.

### 4. DISCUSSION

Electrical resistivity in concrete is a reversed function of conductivity and is related to the diffusivity of ions in the pores. the measurement of electrical resistivity is considered to be a very attractive method for evaluating the properties of geomaterials and for the quality control of concrete because such nondestructive testing (NDT) is simple, rapid, and costeffective[9].

Moisture within the pore is essential for the passage of electrical current. The conductivity in mortar is influenced by the presence of water in the capillary pores, which contains dissolved salts and acts as an electrolyte, then resistivity can be determined. When water content increases, the ion transfer is facilitated due to the high ion conductivity of dissolved calcium silicate hydrate[10], and thus, the electrical resistivity decreases with increasing RH (Fig. 5). Take into account the theory of water capillary condensation in porous material (described by Kelvin-Laplace equation[11]), which defines the maximum pore radius in which capillary condensation is possible at a given relative humidity. When the RH is below 80%, there were not enough pores in mortar to condense sufficient water acting as an electrolyte in the pores, resulting in a high and constant electrical resistivity. When the RH increases from 80%, the pores filled by condensed water increase to facilitate interconnection of electrolyte in the mortar microstructure. Thus, in the present study, when the RH ranges from 80% to 97%, the continuity in the liquid water resulted in complete current flow channels, and the electrical resistivity of mortar decreases dramatically.

Furthermore, electrical conduction flows

through the fluid contained in the pores within the mortar, so resistivity is influenced by the total volume of entrapped air voids and capillary pores in the mortar and by pore size and connectivity.

Before carbonation, more hydration products and fine pores contained in mortar with lowest W/C ratio (W/C=0.5) inhibit formation of water transfer channels through matrix, resulting in largest electrical resistivity of mortar, while larger pore volume provides current channel inside mortar with higher W/C ratio (W/C=0.7) (Fig.6 when carbonation duration=0 day). This results is in agreement with the experimental results presented in previous paper[12].

After carbonation, A solid volume increase of around 11% in conversion of  $Ca(OH)_2$ to CaCO<sub>3</sub>[13][14]causes an expansion of solid volume and a reduction of porosity. Another factor is that the hydration of cement continues over time when RH is above 80%. Hydrated compounds of cement continue to be formed over time, which densify the microstructure of mortar. resulting in products that fill mortar pores[15]. Current flow channel is inhibited by finer pores, which explains the increase of resistivity during carbonation duration under each RH condition (Fig. 6 from dash line). Therefore, for noncarbonated mortar, the effect of RH condition on mortar resistivity depends on pore size distribution, while such effect depends on the carbonation situation of carbonated mortar.

When specimens start to be carbonated, the calcite carbonate precipitated inside pores of mortar, thus the carbonation degree increases dramatically analyzed by TG and XRD. With the increasing of carbonation duration, precipitation of carbonation products narrows the pores, carbonation coefficient decreases, carbonation degree increases generally when carbonation duration is over 30 days (Fig. 4). Such results are in agreement with the change of electrical resistivity of mortar shown in Fig. 6: the electrical resistivity of the mortar increases significantly when the carbonation duration increases from 0 to 30 days due to the reduced porosity by carbonation products and hydrated compounds (RH>80%). the electrical resistivities of mortar were similar when carbonation duration comes to 30 and 50 days. Similar results were shown in the Fig.5: when carbonation duration is above 30 days, the electrical resistivity of mortar with each w/c ratio keep relative stable at different RH conditions (although several exceptions happened in mortar with 0.7 w/c ratio); on the other hand, the effect of moisture content on the electrical resistivity of mortar is more noticeable when carbonation duration is below 30 days. It can be explained by the stable carbonation degree of mortar when carbonation duration is above 30 days

It should be noticed that smaller carbonation degree shown in the mortar with smaller W/C ratio attribute to lower gas diffusivity (Fig. 4), which makes the larger pores shown in the carbonated mortar with W/C ratio of 0.5. Thus, larger volume of pores leads to

higher conductivity and lower electrical resistivity of carbonated mortar with W/C ratio of 0.5 (Fig. 6), while the electrical resistivity of carbonated mortar with W/C ratio of 0.7 changes to be highest due to the highest carbonation degree during carbonation duration.

Finally, considering the electrical resistivity difference under lower RH conditions (RH=68%, 74%) is small, while the resistivity difference is larger under relative higher RH conditions (RH=80%, 84%, 93%, 97%) among the mortar with different w/c ratios and carbonation degree. So, the effect of RH condition on the electrical resistivity of mortar is more noticeable than that of porosity and w/c ratio of mortar.

### 5. CONCLUSIONS

(1) Carbonation degree is higher in the carbonated mortar with a higher w/c ratio because of higher carbonation coefficient caused by higher porosity.

(2) Electrical resistivity decreases with increasing RH. When the RH is below 80%, electrical resistivity of mortar decreases stays high and stable due to insufficient condensed water in the pores, When the RH is above 80%, electrical resistivity decreases dramatically due to more condensed and connected water.

(3) Electrical resistivity of carbonated mortar increases significantly when carbonation duration is below 30 days due to precipitation of carbonation products and formation of hydration products, but gradually increases when carbonation duration is above 30 days due to the lower gas diffusivity.

(4) For non-carbonated mortar specimens, electrical resistivity is higher in the mortar with lower W/C ratio due to the lower porosity. For carbonated mortar specimens, lower electrical resistivity is shown in mortar specimens with a lower w/c ratio due to the smaller carbonation degree.

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