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Novel Accelerated Test Method for RH Dependency of Steel Corrosion in Carbonated Mortar

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Abstract

Degradation of reinforced concrete (RC) structures can occur through the carbonation-induced corrosion of reinforcing bars, and this process is a major concern for the durability of RC buildings. Structures located in relatively humid inland environments are especially vulnerable. Therefore, it is important to clarify how relative humidity (RH) affects steel corrosion rates in carbonated concrete. In this study, a novel accelerated test method is presented, which shortens the experimental duration and simplifies the experimental method. A miniaturized specimen was created with 20 × 20 × 40 mm³ dimensions and an effective carbonation depth of only 5 mm. The corrosion rate of rebar in the small mortar specimens was studied at different equilibrium RH conditions, which were controlled using saturated salt solutions. The accelerated carbonation process was found to be much faster than in traditional concrete experiments. Finally, the relationship between water content (as a function of RH) and corrosion rate showed that the corrosion rate of rebar in carbonated mortar has a strong dependency on RH. The relationship between the mortar resistance and the corrosion rate indicated that the corrosion process of rebar in carbonated mortar is under resistive control when RH above 80%, and under anodic control when RH below 80%.

1. Introduction

In recent years, various reinforced concrete structures worldwide have experienced rapid deterioration. The durability of concrete structures, especially those exposed to aggressive environments, is a major concern. Many causes and factors relating to deterioration have been investigated, and rebar corrosion has been identified as a major deterioration source (Alhozaimy 2010; Andrade 2019).

Concrete carbonation due to atmospheric CO₂ is an environmental aggressor that can lead to steel corrosion. The high pH (~13) of concrete provides natural protection against corrosion of the embedded reinforcements by forming a compact insoluble oxide film at the rebar surface. However, carbonation can decrease the concrete pH to approximately 9, destroying the passive oxide layer and initiating active steel corrosion if the rebar is exposed to sufficient oxygen and water (Broomfield 1997; Nasser et al. 2010; Zhou et al. 2014). Relative humidity has also been shown to have a considerable effect on the corrosion behavior of concrete structures both at the initiation and the propagation phases (Glass et al. 1991; Lim et al. 2010; Hussain 2011; Jiang and Yuan 2013). Data reported in several studies (Alonso et al. 1987, 1988) suggest that corrosion rates exponentially increase with increasing water content. Other studies (Gonzalez et al. 1993; López and González 1993) have shown that the maximum corrosion rate occurred at pore saturation values of 60-70%. However, even at the same RH, the corrosion rates of the rebar can be highly variable. The mechanism that relates corrosion rate and relative humidity has not been fully studied. Therefore, a study that clarifies the dependency of steel corrosion on RH in carbonated mortar is necessary.

In traditional steel corrosion experiments in concrete, the samples are typically large in size. As a result, a large amount of time is needed for carbonation to reach the required depth at the rebar surface and for a relative humidity equilibrium to be established. On the other hand, the miniaturized specimens were used in a few papers. Stefanoni et al. embedded the steel wire into a thin mortar specimen acting as a working electrode (Stefanoni et al. 2016). However, the formation of corrosion products during corrosion process may break the steel wires due to the small diameter of the steel wire. García-Alonso et al. put different types of steel rebars in a small mortar specimen to evaluate their different corrosion behaviors (García-Alonso et al. 2007). But the macrocell corrosion may occur among these steel rebars embedded in the same medium due to the different equivalent potentials. Also, because the part of the extended steel rebar outside the mortar exposed to a different environment from the embedded portion of steel rebar, it will influence the accuracy of corrosion rate evaluation. In addition, cracks may occur due to the expansion caused by carbonation or corrosion products,

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which are harmful for homogenous corrosion process and deteriorate the suitable condition for analyzing corrosion rate.

In this study, a novel accelerated test method is presented that overcomes these limitations. A miniaturized sample size can reduce the time required for equilibrium between the inside of a mortar sample and environmental RH. For thin mortar covers, carbonation can reach the required depth quickly, and excessive high CO₂ concentrations are unnecessary. The proposed design of miniature mortar specimen with the embedded steel rebar reflects the practical corrosion situations and the conditions for appropriate electrochemical measurement. To prevent cracking due to the corrosion, polypropylene fibers were applied to the mortar. With this novel design, we studied the corrosion rate of rebar in carbonated samples under changing relative humidity and determined the RH dependency of steel corrosion in carbonated mortar using a faster and simpler method.

2. Experimental methods

2.1 Materials and specimen

Ordinary Portland cement (OPC) was used throughout the experiment. The chemical composition is shown in Table 1, while the mineral composition of OPC is shown in Table 2. The sand/binder ratio was three, and the sand had a maximum particle diameter of 5 mm (properties of the fine aggregates are shown in Table 3). The mortar used for all specimens was cast using the mixture proportions presented in Table 4. Polypropylene fibers (length of 6 mm, diameter of 0.028 mm), as shown in Fig. 1, were used to prevent cracking in the mortar cover caused by carbonation and corrosion products.

The reinforcing bars in the specimens were SGD-400D rebar with a diameter of 10 mm. The rebar was cut to a length of 30 mm which is proper for the mortar size. Each rebar was degreased to remove oil and dust from the surface and allowed to air dry. Conductive wires were welded to each rebar, and a 0.5 mm thick waterproof glue was used to cover the ends. This process is shown in Fig. 2.

The specimens, which included a rebar embedded at the center, were demolded after one day, and sealed in a curing room at 20±1°C for fourteen days before being carbonated. The dimensions of the mortar were designed as 20 × 20 × 40 mm³ with enough top surface to place reference electrode and counter electrode on it during electrochemical measurement process. The configuration for the specimens with reinforcing bars is shown in Fig. 3.

The mortar cover was 5 mm, and 9.42 cm² of the rebar was exposed to mortar.

2.2 Accelerated carbonation

Accelerated carbonation was achieved using a chamber with 60% RH and a CO₂ concentration of 5%. Prior to carbonation, the specimens were kept in an oven at 105°C until they reached a constant weight: when liquid water is evaporated, carbon dioxide can penetrate more easily. Meanwhile, reference specimens under the same conditions were used to verify the carbonation depth during the accelerated carbonation. In addition, non-carbonated specimens were prepared with the same
mixture proportion, size, and the curing conditions as the carbonated ones but without CO2 gas.

Carbonation depths were obtained using a phenolphthalein test on the reference specimens. The phenolphthalein indicator can be used to roughly evaluate pH, and by extension, the carbonation depth. This is a widely used technique in carbonation studies in the field of cementitious materials. In this study, the carbonation process was not investigated; however, sufficient carbonation was required to trigger the corrosion process (Broomfield 1997). To ensure sufficient carbonation of the samples, carbonation took place over two weeks.

2.3 Characterization of the carbonated sample

The specimens were stored at 11% RH and 20°C for 14 days. The part of mortar around steel rebar was taken, and then ground using a ball mill. After grinding, the particles size was checked using a 75 µm sieve. Retained powder, if necessary, was ground manually until all the material passed through the 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 20 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 cm3/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 Bruker AXS D8 ADVANCE powder diffractometer with Cu kα radiation, using a 2θ scan range of 5°-65° and a scan speed of 2°/min.

2.4 RH control for carbonated sample

Following accelerated carbonation, the mortar specimens were subject to variable RH conditions. Different RH conditions were achieved using saturated salt solutions (Table 5) in desiccators at a temperature of 20±1°C (Fig. 4).

The mass of the samples was monitored at regular intervals throughout the exposure period. All samples reached weight equilibration during the process. Once stable conditions were achieved, electrochemical measurements were initiated.

The mass of mortar fragments was measured using a balance with a resolution of 1 mg. The water content by mass w (%) of the fragments in different RH conditions was calculated after the samples reached relative humidity equilibrium, given as

\[ W = \frac{m_{\text{sample}} - m_{\text{dry}}}{m_{\text{dry}}} \]

where \( m_{\text{sample}} \) is the mass of the mortar sample, and \( m_{\text{dry}} \) is the mass after drying at 105°C under vacuum conditions for twenty-four hours.

2.5 Electrochemical measurements

The corrosion of rebar in concrete is a dynamic electro-
chemical process involving the exchange of ions between cathodic and anodic locations on the surface of the rebar (Millard et al., 2001). The corrosion interface can be represented by a simple electronic circuit, known as a Randles circuit (Randles 1947), as shown in Fig. 5. $R_\Omega$ refers to the solution resistance, in this case, the electric resistance of the concrete. $C_{dl}$ denotes double layer capacitance, which is provided by charged ions on the surface of the rebar. $R_p$ is the polarization resistance at the surface of the rebar, which represents the ease with which charged iron ions leave the metal surface and enter solution.

Mortar resistance was measured using a two-electrode setup. Copper plates were placed on two parallel sides of the samples. The contact solution in the experiment was a sponge saturated with a conductive gel. Electrical connection to the LCR meter (IM3533-HIOKI) was established through copper wires. The electric resistance of the mortar can be measured by applying an AC perturbation with an amplitude voltage of 50 mV in the frequency range of 0.1 to 100 kHz. All impedance spectra (Examples of Nyquist plot are provided in the appendix) were fitted to a Randles equivalent circuit using the EIS Spectrum Analyser software (Fig. 5).

The linear polarization technique was used to assess the instantaneous corrosion current. The experimental setup for the electrochemical measurements is shown in Fig. 6. We used a three-electrode system, in which the rebar was the working electrode, a saturated silver/silver chloride electrode (R0303-SHAGHAICHUXI) was the reference electrode, and the stainless steel plate (SUS304) was the counter electrode.

An overpotential ($\Delta E$) ranging from -10 mV to +10 mV was applied for the corrosion potential at a scan rate of 0.1 mV/s. The resulting current $\Delta I$ was recorded. Both over and underpotentials were used to maximize $\Delta E$ while minimizing the disturbance to the rebar potential. According to (Stern 1957), Ohm’s law can be applied if the polarization applied to the rebar does not exceed ±30 mV. Thus, the polarization resistance $R_p$ can be found as

$$R_p = \frac{\Delta E}{\Delta I}$$

(2)

The ohmic resistance of the concrete $R_\Omega$, which was obtained by AC impedance spectra fitting, was subtracted from the overall potentiostatic resistance $R_p^*$ to obtain the real polarization resistance $R_p$ (Millard et al., 2001):

$$R_p = R_p^* - R_\Omega$$

(3)

The corrosion current $I_{corr}$ was then calculated using the Stern-Geary equation (Mansfeld and Oldham 1971),

$$I_{corr} = \frac{\beta a \beta c}{2.3(\beta a + \beta c)} \frac{\Delta E}{R_p}$$

(4)

where $B$ is a constant and can be determined empirically from $\beta a$ and $\beta c$, which are anodic and cathodic Tafel coefficients, respectively. The value of $B$ can be assumed to be 26 mV for actively corroding steel in concrete and 52 mV (Alonso and Andrad 1988) when no significant corrosion develops. Assuming homogeneous corrosion, the corrosion rate $I_{corr}$ can be calculated as

$$I_{corr} = \frac{I_{corr}}{A}$$

(5)

where $A$ is the exposed area of rebar.

3. Results

3.1 Specimen characterization

Carbonation depth was determined by breaking the specimens diametrically and spraying 1% phenolphthalein indicator solution on the broken surface. Figure 7 shows the cross-sections of non-carbonated and carbonated mortar specimens after spraying with the phenolphthalein solution. The carbonation front clearly reached the rebar surface, which was 5 mm away from the mortar surface. Therefore, the carbonation depth was sufficient to activate steel corrosion after the accelerated carbonation process.

Figure 8 shows the diffraction patterns for the non-carbonated and carbonated mortar samples. It was
found that more portlandite remained in the non-carbonated sample, while calcite and aragonite were mainly formed in the carbonated sample. A Rietveld analysis was conducted to quantify the phase composition related to the carbonation, and the results are summarized in Table 6. The percentages reported are the ratios of each phase to each gram of cement.

Figure 9 shows the derivative thermogravimetry (DTG) curves for the non-carbonated and carbonated samples, in which the curves are in terms of grams of cement paste rather than grams of mortar for clear comparison. The carbonated sample had a larger calcite content, which can be observed as a peak at 730°C, while the non-carbonated had a smaller aragonite content, which can be observed as a peak at 580°C. The non-carbonated samples had a greater portlandite content than the carbonated samples, as seen from the peak at around 430°C. Furthermore, the greater content of ettringite decomposed at 90°C in non-carbonated samples.

To evaluate the carbonation conditions, a method for calculating the degree of carbonation $\alpha_c$ is proposed. The degree of carbonation is defined as the ratio of calcium oxide in calcium carbonate, which is formed during the carbonation process, to the total calcium oxide per gram of cement. Therefore, the degree of carbonation can be expressed as

$$\alpha_c(\%) = \frac{R_{cp} \times 56}{(1 - LOI) \times 100} \times \frac{R_0 \times 56}{R_t \times 100}$$

(6)

where $R_{cp}$ is the ratio of calcium carbonate (calcite and aragonite) per gram of carbonated cement paste (Table 6), $R_0$ is the initial ratio of calcium carbonate per gram of cement (Table 2), LOI is the ratio of water loss on ignition, and $R_t$ is the total ratio of calcium oxide per gram of cement (Table 1).

The DTG curve was integrated, and the ratio of phase composition from the XRD analysis was quantified, with the results shown in Table 7. Thus, from these results, the degree of carbonation can be evaluated.

The analysis clearly shows that the degree of carbonation calculated by the XRD analysis and DTG curves were similar, though the degree of carbonation

|                | Calcite | Portlandite | Aragonite | Vaterite |
|----------------|---------|-------------|-----------|----------|
| Non-carbonated sample | 6.80±0.7 | 18.8±0.1  | 0.0       | 0.0      |
| Carbonated sample   | 31.5±0.5 | 3.79±0.8   | 6.64±0.3  | 0.0      |

Fig. 7 Cross-sections (20 × 20 mm²) of reference mortar specimens sprayed with 1% phenolphthalein solution (left: non-carbonated, right: carbonated).

Fig. 8 Diffraction patterns of non-carbonated and carbonated mortar samples (Q: Quartz, C: Calcite, P: Portlandite, Ett: Ettringite, A: Aragonite).

Fig. 9 DTG curves for the non-carbonated and carbonated mortar with the main decompositions labeled (Ett: Ettringite, Mc: Monocarbonate calcium aluminate, CH: Portlandite, A: Aragonite, C: Calcite).
The measured water content for each specimen in different RH conditions is shown in Fig. 10. The water content is a measure of the amount of liquid water present in the pore system, which can act as an electrolyte for the corrosion process. As expected, the water content increased with RH. When the RH was greater than 60%, water began to condense on the surface of the narrow pores, a process known as capillary condensation. When the RH was above 80%, the moisture content significantly increased until reaching a maximum at an RH of 97%.

3.2 Electrochemical results for mortar sample in variable RH conditions

Figure 11 shows the electrical resistance of the mortar and the corrosion rate of the rebar in the carbonated mortar samples under different RH conditions. As expected, a rise in RH was accompanied with a decrease in mortar resistance and an increase in corrosion rate. The mortar resistance remained high and relatively constant for RH values below 74%. However, further increasing the RH to 97% corresponded to a marked decrease in the mortar resistance. In contrast, the corrosion rate increased with increasing RH.

Figure 12 shows the relationship between water content and mortar resistance. Figure 13 shows the relationship between water content and the logarithm of the corrosion rate. The quantitative relationship between the logarithm of corrosion rate and mortar resistance is shown in Fig. 14. The general trend shows that the corrosion rate increased with decreasing mortar resistance; when the RH was below 74%, the mortar resistance remained high and constant in the range of approximately 5500 $\Omega$ cm to 5400 $\Omega$ cm. In this RH range, the corrosion rate was low and independent of mortar resistance.

4. Discussion

4.1 Correlation between water content, mortar resistance, and corrosion rate

Hydroxide ions in the microstructure were primarily transferred by the calcium silicate hydrate, as well as sodium and potassium hydroxides. When water content increases, the ion transfer is facilitated due to the high

| $R_{c}$ | LOI | $R_{c}$ | $R_t$ | $\alpha_t$ (%) |
|---------|-----|---------|------|-------------|
| Calcite | 51.5±0.5 | 6.64±0.3 | 0.248±0.03 | 64.1 | 41.53±0.5 |
| Aragonite | 36.2±0.4 | 3.2±0.1 | 39.33±0.2 |

Table 7 Calculation process for the degree of carbonation.
ion conductivity of dissolved calcium silicate hydrate (Nagao et al. 2020), and thus, the mortar resistance decreases (Fig. 12) and corrosion rate increases (Fig. 13). Therefore, a change in mortar resistance can be attributed to a change in the relative humidity, which is associated with a change in water content in the pore system.

The relationship between the corrosion rate and the mortar resistance (Fig. 14) clarifies the rebar corrosion process. This correlation for an RH above 80% proves that mortar resistance influences the corrosion rate directly, consistent with the previous literature (Dunster et al. 2000; Eden et al. 2004). However, at an RH below 80%, the corrosion rate was independent of the mortar resistance.

Corrosion rate is a parameter that depends on anodic and cathodic reaction processes. Figure 15 shows three controlling mechanisms during the corrosion process according to the different limitations. An anodic control (shown in Fig. 15(a)) occurs when the dissolution of the metal determines the changes in the electrochemical parameters, while a cathodic control (shown in Fig. 15(b)) is present when there is a limiting flux of oxygen.

When ohmic polarization occurs due to the ohmic resistance of the electrolyte, it produces an ohmic potential drop. Fig. 15(c) illustrates resistive control, in which the potential available for corrosion is the difference in potential between the anode and cathode minus the potential drop (Alonso et al. 1988). The key parameter defining the corrosion rate is the presence of water in the pore system of the mortar. When the RH is below 80%, the oxygen content is sufficient at the cathodic site, yet the water content is insufficient to activate the rebar on the anodic site, inhibiting metal dissolution. Thus, it is concluded that the corrosion rate, which remained extremely low, and independent of mortar resistance in these conditions, is under anodic control. For an RH above 80%, sufficient liquid water is present for the corrosion process; the mortar resistance decreases as water content increases, and the corrosion rate increases continuously until an RH of 97%. The maximum corrosion rate at 97% RH suggests that corrosion proceeds under resistive control without oxygen limitations.

To prove this hypothesis, water condensation was also considered. The amount of water contained in the pores of the mortar was in equilibrium with the relative humidity of the atmosphere. The condensation of water in pore systems such as mortar can be described by the Kelvin-Laplace equation (von Helmholtz 1886),

\[ \ln \frac{P}{P_0} = -\frac{2V\gamma}{rRT} \]

where \( P \) is the partial pressure of water vapor, \( V \) is the molar volume of water, \( \gamma \) is the water surface tension, \( r \) is the pore radius, \( R \) is the universal gas constant, and \( T \) is the absolute working temperature. According to this equation, each RH has a matching critical pore radius, below which pores remain water-saturated and above which water will evaporate. The evaporation establishes preferential aeration channels for the penetration of oxygen through vapor-phase diffusion with a coefficient 10^4 times greater than in a liquid phase (Gjørv et al. 1977).

As shown in Fig. 11, when the RH was below 74%, there was insufficient condensed water acting as an electrolyte in the pores (Fig. 10), which inhibited ion transfer in the matrix and resulted in a high and constant resistance. However, an RH below 80% allowed for the formation of a continuous path of open pores from the mortar surface to the rebar, and a portion of the rebar surface remained in contact with air. Therefore, the lim-

Fig. 14 Relationship between corrosion rate and mortar resistance (log (\( i_{corr} \)) versus \( R_\Omega \)).
ited ion transport on the surface of the rebar causes a low corrosion rate. When the RH increased from 74%, the condensed water filled more pores in the mortar microstructure, and the liquid water in the pore system was connected. Thus, in the present study, when the RH ranged from 74% to 97%, the continuity in the liquid water resulted in complete ion transfer channels, and the mortar resistance decreased dramatically. When the RH reached the maximum value of 97%, the condensed liquid water did not fill all the pores around the rebar. A portion of pores remained unsaturated, allowing sufficient levels of oxygen to penetrate through the vapor or gas phase to reach the rebar surface. Consequently, the corrosion rate increased continuously from 80% to 97% RH, with no limitations in oxygen diffusion.

4.2 The accelerated test method
The miniaturized mortar sample described above can be an innovative method for rapidly testing the dependency of carbonation-induced corrosion on RH. As a significant duration is needed for the carbonation process and changes in the exposure conditions to initiate corrosion, the novel accelerated method for minimizing the time required for sample preparation, including casting, curing, accelerated carbonation, and equilibration in different humidity environments, was successful in addressing these limitations. The proposed experimental method can be used to easily test the corrosion rates of mortars with different w/c ratios or cement types using electrochemical tests.

However, as diffusion mechanisms are different between accelerated carbonation laboratory tests and natural carbonation conditions, mortar samples exposed to variable carbon dioxide concentrations may experience different degrees of carbonation and pore size distributions. Thus, an accelerated carbonation test under high carbon dioxide concentrations may not represent natural concrete carbonation conditions (Cui et al. 2015). Additionally, microcracks may form on concrete structures during the natural carbonation process. In this laboratory experiment, this problem was avoided by adding fibers to the mixture. Therefore, future studies are recommended that compare the corrosion behaviors in accelerated and natural carbonation specimens.

5. Conclusion
The electrochemical behavior of rebar embedded in carbonated mortar exposed to different RH conditions was investigated. The degree of carbonation of the mortar was verified by DTG and XRD analysis, and electrochemical parameters including electrical resistance and corrosion current density (corrosion rate) were measured using a potentiostat. The RH dependency of steel corrosion was determined by investigating the correlation between water content (as a function of RH), mortar resistance, and corrosion rate.

A miniaturized mortar sample was built, which provided a novel method to minimize the experiment duration, including mortar sample preparation and reaching equilibration at various relative humidities. All the necessary electrochemical tests were successfully and efficiently carried out to evaluate the corrosion rate of the mortar under different RH conditions.

When the RH is above 80%, the ambient RH has a significant effect on steel corrosion in carbonated concrete. The continuity of the liquid water facilitates ion transfer in the pore system and leads to a decrease in the mortar resistance and a dramatic increase in the corrosion rate.

The corrosion process for rebar in carbonated concrete is under anodic control in RH conditions ranging from 68% to 80% RH and under resistive control from 80% to 97% RH. The water condensation theory by the Kelvin equation further supports this hypothesis.

As there are differences in diffusion mechanisms in the accelerated carbonation laboratory test and natural carbonation conditions, further investigations are recommended to compare the electrochemical and material properties between these two conditions to make the conclusions of this study more practical.

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Appendix

A.1 Fitting of Nyquist plots

Here, an example of the fitting of Nyquist plots in the current study is shown. **Figure A1** shows the Nyquist plots and fitting curves of mortar samples exposed to 97% RH and 68% RH conditions. Mortar resistance is detected according to the intersection of the fitting curve with the horizontal axis.

![Nyquist plots](Fig.A1)