

The investigation of pH threshold value on the corrosion of steel reinforcement in concrete

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(Received March 21, 2016, Revised December 20, 2016, Accepted December 24, 2016)

Abstract. The aim of this study is to investigate the pH threshold value for the corrosion of steel reinforcement in concrete. A method was designed to attain the pH value of the pore solution on the location of the steel in concrete. Then the pH values of the pore solution on the location of steel in concrete were changed by exposing the samples to the environment (CO₂ 5%, RH 40%) to accelerate carbonation with different periods. Based on this, the pH threshold value for the corrosion of steel reinforcement had been examined by the methods of half-cell potential and electrochemical impedance spectra (EIS). The results have indicated that the pH threshold value for the initial corrosion of steel reinforcement in concrete was 11.21. However, in the carbonated concrete, agreement among whether steel corrosion was initiatory determined by the detection methods mentioned above could be found.

Keywords: carbonation, corrosion, steel reinforcement, pH threshold

1. Introduction

Apart from structural design failures, corrosion of steel reinforcement is an important cause of concrete degradation (Pradhan 2014). It is well known that the steel reinforcement in concrete is normally in a passive state against corrosion due to a thin iron oxide layer by the high alkaline environment of the concrete pore solution. Carbonation itself does not cause the deterioration of concrete, but it changes the pH of the pore solution in the concrete (Leemann *et al.* 2015). As soon as the pH of the pore solution on the location of the steel exceeds a critical level, i.e., pH threshold value, the film will be destroyed (which is called depassivation) and the corrosion is to be initiated.

The pH threshold value for the corrosion of steel reinforcement is essential for the durability and service-life prediction of reinforced concrete by carbonation. Based on this, numerous investigations have been performed on the pH threshold value. However, up to now, no agreement on the reported values is attained. For example, the pH threshold value decided by Papadakis is 10 (Papadakis 2000) and in simulated NaHCO₃ solution, the pH threshold value is between 8.3 and 9.4 (Huet *et al.* 2006). Other literature indicated that the pH threshold value on the corrosion of steel reinforcement in concrete is 11.5. The different pH threshold value is related to that the composition of the solution on the location of the steel reinforcement influences the pH threshold value (Huet *et al.* 2006). Unfortunately, there were only a few researches about

the pore solution composition in carbonated concrete. Therefore, the pH threshold values decided by the simulated solution used in literatures, such as Ca(OH)₂ solution and the mixed solution by Ca(OH)₂, KOH and NaOH, may be incorrect. It was difficult to utilize those reported pH threshold values to predict the life span of reinforced concrete. A further study was required to examine the pH threshold value on the corrosion of steel reinforcement, so as to obtain a reliable pH threshold value.

Based on this, the pH of the pore solution on the location of the steel reinforcement should be determined firstly. Unfortunately, to the best of our knowledge, only a few researches about the decreasing pH of pore solution in concrete during carbonation process can be found (Pu *et al.* 2012, Behnood *et al.* 2016 and Kouřil *et al.* 2010). As the carbonation in concrete was consecutive, it was difficult to obtain the pore solution at designated carbonation depth. Thus, it is needed to study the pore solution pH on the location of the steel reinforcement during carbonation process in concrete.

This paper focuses on the pH threshold value of the pore solution for steel corrosion in carbonated concrete. This study had been performed in two sections. First, a method was designed to calculate the pore solution pH on the location of the steel. Then the pore solution pH values on the location of the steel in reinforced concrete had been prepared by carbonation. Second, based on the first step, two detection methods, i.e., half-cell potential and EIS, had been applied and the pH threshold value for the corrosion of steel reinforcement had been examined.

2. Experimental procedures

2.1 Material

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Table 1 Chemical compositions of cement (%)

| Oxide | SiO ₂ | Al ₂ O ₃ | Ca O | Fe ₂ O ₃ | Mg O | Na ₂ O | K ₂ O | SO ₃ | Ig .loss |
|-------|------------------|--------------------------------|-------|--------------------------------|------|-------------------|------------------|-----------------|----------|
| % W/W | 22.97 | 9.34 | 53.98 | 3.1 | 1.35 | 0.12 | 0.91 | 3.99 | 4.16 |

NO. 42.5 ordinary Portland cement manufactured in China was used. The chemical composition of the cement was showed in Table 1. The river sand with a fineness modulus of 2.8 and crushed stone with the maximum size of 20 mm were used as fine and coarse aggregates, respectively. The concrete with the cement content of 500 kg/m³, fine aggregate content of 680 kg/m³, coarse aggregate content of 1167 kg/m³ and a water-cement ratio (W/C) of 0.6 was prepared using distilled water. A cylindrical steel bar with a diameter of 10 mm and length of 6 cm was used. The exposed area of each steel bar was 12.5 cm² and the remaining areas had been sealed. The chemical composition (in% by mass) was 0.22% C, 0.30% Si, 0.65% Mn, 0.05% S, 0.045% P and the residual Fe.

2.2 Calculation of the pore solution pH

Specimens of dimension 10 cm diameter by 15 cm in height were manufactured. Standard curing periods was 27 days, trial age was 28 days. The cylinders were finally cut, perpendicular to their axis, into 3-mm-thick discs, as shown in Fig. 1(the detailed step was discussed by Pu *et al.* 2012). Before carbonation, the discs were dried at 5°C for 24 hours. Then the discs were exposed to a CO₂ enriched atmosphere of 5% CO₂ at a relative humidity of 40% with a temperature of 20°C in an accelerated carbonation chamber for: 3 days, 6 days, 9 days, 12 days, 15 days, 21 days, 28 days.

At different periods of exposure, the specimens were removed from the carbonation chamber. A procedure was developed to allow the pore solution to be obtained from the carbonated concretes. This procedure was described below: First, the specimens were converted to a saturated, surface-dry condition. The method was taken as fallow: the specimens were put on platforms above distilled water in sealed tanks and an air humidifier was worked intermittently (5 minutes per hour). The specimens were weighted daily until practically constant weights were achieved.

After the specimens were saturated, ten specimens were stacked together to form a cylinder (approximately 30 mm in height) and the pore solution was expressed at a pressure of up to 300 MPa which was described elsewhere (Behnood *et al.* 2016).

When sufficient pore solution of the carbonated specimens was obtained, the pH of pore solution was measured using PHS-3C at the temperature of 25°C. The results were given as averages of three replicates.

The same carbonated specimens were dried at 50°C for 24 hours. Then the specimens were ground to powder using a mortar and pestle. Later, an amount of powdered specimen was suspended in distilled water with a water-solid ratio of 100. After 96 hours, the pH of the suspension was measured using PHS-3C at the temperature of 25°C. The results were given as averages of three replicates. Then

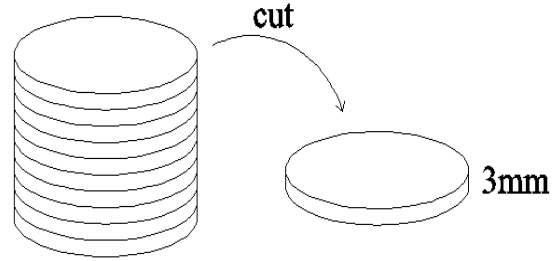


Fig. 1 Experimental setup used for the carbonation of concrete

the relationship between the two methods of measuring pH, the suspension pH and the pore solution pH, was established. So the pore solution pH of carbonated concrete can be calculated simply by the suspension pH.

2.3 Tests in carbonated concrete

Prior to application, the steel bars were degreased in acetone and then washed in distilled water. The exposed area of each steel bar was 12.5 cm² and the remaining area was sealed by means of hot-shrinkable tube. To obtaining more accurately the results, three concrete specimens at an identical level were prepared. Besides, two identical steel bars were wholly embedded in each concrete specimen with cover depth of 10 mm. Standard curing periods was 27 days, trial age was 28 days. The specimens were dried at 50°C for 24 hours. Then the samples were exposed to a CO₂ enriched atmosphere of 5% CO₂ at a relative humidity of 40% and a temperature of 20°C in an accelerated carbonation chamber. During the carbonation process, a certain quality powder near the steel surface was drill down by milling machine. Then the pore solution pH could be calculated by process 2.2. Five levels of pore solution pH value were supplied during carbonation, ranging around from 12.5 to 8.5. After carbonation, the concrete samples were cured at ambient temperature for three months. Then the pore solution pH values near the steel surface in concrete were measured again by the above measurements.

Half-cell potentials were measured for all the samples using a copper-copper sulfate reference electrode (SCE), in accordance with ASTM C-876. Before EIS measurement, the concrete samples were transferred into a simulated solution which had the same pH values comparing with the pore solution pH of the carbonated concrete near the steel. The saturated calomel electrode and platinum electrode had been connected to work as a reference and auxiliary electrode, respectively. However, the EIS in the frequency range from 10 mHz to 1 MHz was performed. Moreover, the perturbing signal applied was 5 mV. The electrical resistance of concrete (i.e., IR drop) had been compensated by using positive feedback technique. The polarization resistance (Rp) obtained by EIS had been introduced into the Stern-Geary equation to calculate the corrosion current (I_{corr}). The Stern-Geary equation was: I_{corr}=B/Rp. At this time, the Stern-Geary constant B was considered to be 26 mV, too. Furthermore, consider the pH value where the corrosion current density of steel bar is higher than 1 mA/m² as the pH threshold value (Xu *et al.* 2009).

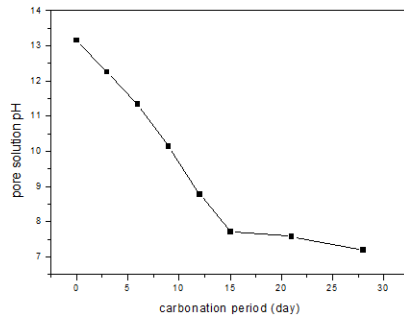


Fig. 2 The pore solution pH profile of the concrete during the carbonation process

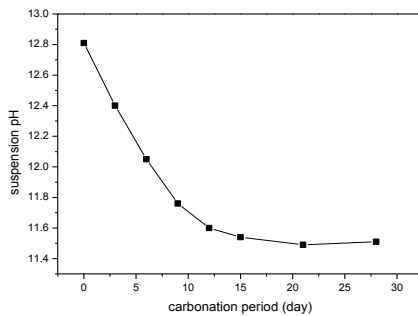


Fig. 3 The suspension pH profile of the concrete during the carbonation process

3. Result and discussion

3.1 Determination of the pore solution pH

The pore solution pH profile was plotted against degree of carbonation, as showed in Fig. 2. The profile showed a decrease in pH due to carbonation and the pore solution pH values of the concrete mainly decreased in the 15 days. After 15 days' carbonation, the variation of the pore solution pH values became stable which was only about 5% comparing with the final value. The reason for this variation was likely to be due to the conversion of carbonate to bicarbonate (Huet *et al.* 2006). So it was thought that the concrete was wholly carbonated on the 15th day (at pH 7.85).

The suspension pH profile of the concrete with different degree carbonation was shown in Fig. 3. The suspension pH values mainly decreased during the 15 days. After about 15 days carbonation, the variations of the suspension pH values became constant which were only about 0.2% comparing with the final value. Although the pH of the suspension with small water-solid ratio was similar with the pore solution pH, it was difficult to obtain sufficient powdered specimen from the carbonated concrete at designated carbonation depth (McPolin *et al.* 2009). So a large water-solid ratio was suitable in this case.

The two methods of measuring pH, the suspension pH and the pore solution pH, were compared in Fig. 4. This relationship showed that it was possible to use the suspension pH to assess the degree of carbonation, with a reasonably acceptable degree of confidence, without pore solution extraction tests. A fitting equation from the curves was obtained

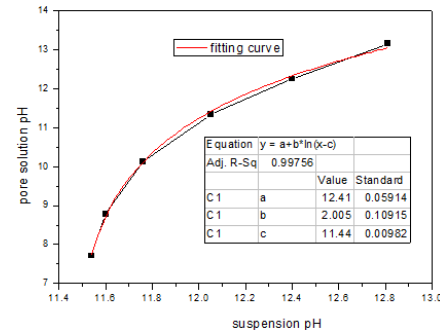


Fig. 4 Pore solution pH vs suspension pH

Table 2 The suspension pH and calculated pore solution pH

| suspension pH | 11.58 | 11.67 | 11.85 | 12.07 | 12.51 |
|------------------|-------|-------|-------|-------|-------|
| pore solution pH | 8.51 | 9.44 | 10.62 | 11.5 | 12.55 |

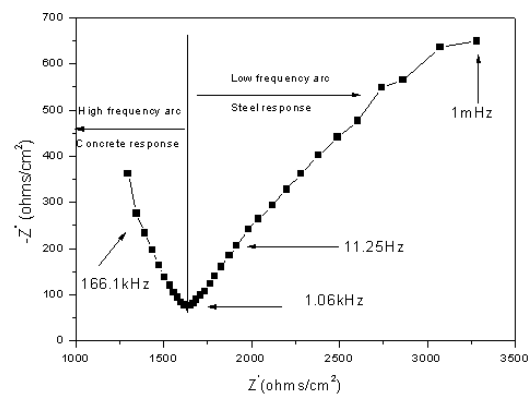


Fig. 5 Representative Nyquist plot for the steel reinforcement in the carbonated concrete

$$pH_{\text{pore solution}} = 12.41 + 2.005 \times \ln(pH_{\text{suspension}} - 11.44)$$

The graph showed that there was a strong correlation between the two pH measurements, where the R^2 value was 0.997. This relationship gave confidence in the use of suspension pH as a method of inferring the pH of the pore solution. But the fitting equation cannot be used elsewhere, the fitting equation may change with changing of the concrete parameters. Once the pore solution pH value was need, the fitting equation should be established first.

Table 2 showed the suspension pH values of the concrete near the steel surface and the pore solution pH values calculated by the fitting equation during carbonation process. The results were given as averages of three replicates which were cured at ambient temperature for three months.

3.2 pH threshold value in concrete

The EIS spectra of the concrete are complex and have more than one time constant. The EIS spectra of the concrete can be divided into three parts: high frequency region, middle frequency region and low frequency region. Nyquist plot of EIS for the steel reinforcement in the carbonated concrete in this work shown in Fig. 5 was representative.

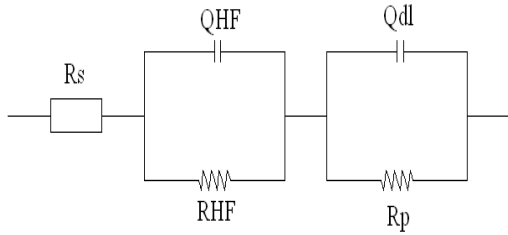


Fig. 6 Equivalent circuit for the EIS simulation of steel reinforcement in the carbonated concrete

The high frequency region of the spectra (over 10^5 Hz) was related to the ion concentration in the pore solutions, to the porosity and pore size distribution (Despas *et al.* 2014, Dong *et al.* 2016, Ortega *et al.* 2015 and Villat *et al.* 2010). The middle frequency region (10^4 –10 Hz range) was related to the dielectric properties of a layer formed on the steel surface and reputed to be constituted by corrosion products (Han *et al.* 2012), or by a protective precipitated calcium hydroxide or a lime-rich product (Cruz *et al.* 2013, Zhang *et al.* 2014 and Dong *et al.* 2014). The low frequency region (lower than 10 Hz) was related to the Faradaic corrosion process occurring on the embedded steel electrodes (Duarte *et al.* 2014).

The complete equivalent-circuit model simulating the EIS spectra was shown in Fig. 6, where: R_s was the electrolyte resistance in the pore solution, and the parallel circuit (RHF, QHF), were associated with the dielectric properties of concrete. Besides, the constant phase element (Qdl) was associated with the fractal nature of the electrode interface or heterogeneity of the steel surface (Xu *et al.* 2009) and R_p was the polarization resistance. The Qdl (CPE) can be calculated by the equation: $Y_{CPE} = Y_0(i\omega)^n$, where n generally ranged between 1 and 0.5. The frequency ranging from 10 mHz to 10 Hz had been chosen in this study in order to obtain the polarization resistance (Li *et al.* 2001).

The half-cell potentials and polarization resistances (R_p) with different pH values during carbonation process were shown in Table 3. Generally, the half-potentials and R_p decrease with the decrease of the pH values, which showed an upgraded risk of corrosion for the steel reinforcements during carbonation process. However, the samples with an identical pH value display a different half-cell potentials and polarization resistances (R_p), which had been related to the stochastic nature of corrosion for steel reinforcement in a concrete (Dong *et al.* 2014).

The half-cell potentials for all the samples plotted with the pH values of the pore solution in carbonated concrete were shown in Fig. 7. In the samples with the pore solution pH value higher than 12.55 by mass of binder, all the half-potentials of six steel reinforcements were more positive than -200 mV SCE, according to ASTM C-876, which indicated that no corrosion for all the samples happened at this time. At the pore solution pH value of 11.5 by mass of binder, the half-cell potentials for four steel reinforcements in six samples fall in the range of -200 to -350 mV SCE, according to ASTM C-876, which suggested a possible occurrence of active corrosion for them. Furthermore, when the pH values were lower than 10.62, there were half-cell

Table 3 Values of half-cell potential, polarization resistance obtained by electrochemical impedance spectra (EIS) for all the samples

| pH value of the pore solution | Half-cell potential (mv) | Polarization resistance (Q cm) |
|-------------------------------|--------------------------|--------------------------------|
| 12.55 | -174 | 6.55E+06 |
| | -160 | 8.88E+06 |
| | -180 | 7.29E+06 |
| | -168 | 1.29E+07 |
| | -172 | 6.29E+06 |
| | -142 | 8.29E+06 |
| 11.5 | -287 | 2.70E+05 |
| | -357 | 2.89E+05 |
| | -309 | 2.26E+05 |
| | -300 | 4.73E+05 |
| | -363 | 1.02E+06 |
| | -324 | 3.20E+06 |
| 10.62 | -420 | 1.19E+05 |
| | -405 | 2.06E+05 |
| | -432 | 2.12E+05 |
| | -426 | 1.00E+05 |
| | -382 | 8.34E+04 |
| | -395 | 3.30E+05 |
| 9.44 | -418 | 6.38E+04 |
| | -445 | 6.60E+04 |
| | -501 | 5.50E+04 |
| | -436 | 5.58E+04 |
| | -447 | 5.95E+04 |
| | -488 | 5.20E+04 |
| 8.51 | -547 | 3.21E+04 |
| | -488 | 2.94E+04 |
| | -450 | 3.65E+04 |
| | -521 | 3.31E+04 |
| | -492 | 3.60E+04 |
| | -479 | 4.40E+04 |

potentials more negative than -350 mV for all the steel reinforcements, according to ASTM C-876, which indicated that active corrosion had been initiated. By fitting the relationship in the exponential equation between the half-cell potentials and pH values, and according to ASTM C-876, where active corrosion was initiated at -350 mV, pH threshold values can be calculated. Therefore, the pore solution pH threshold value concluded by the detection method of half-potential in this work was 11.16.

The corrosion current densities for the steel reinforcements had been obtained by introducing the polarization resistances (shown in Table 5) into Stern-Geary equation. The impedance spectra obtained for all the samples with different pH display a similar shape. Generally, Nyquist plots depict a capacitive loop of depressed semicircles. Besides, as the increase of pH level, the diameter of semicircle has been gradually increased,

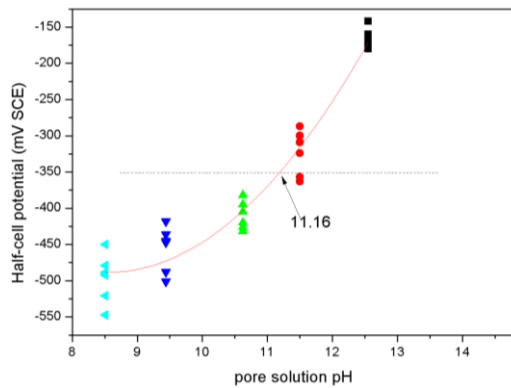


Fig. 7 Half-cell potentials for all the samples changed with the increase of pore solution pH values in the carbonated concrete

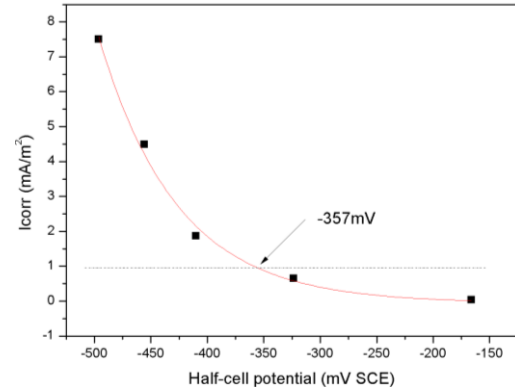


Fig. 9 Corrosion current densities changed with the half-cell potentials for the samples in carbonated concrete

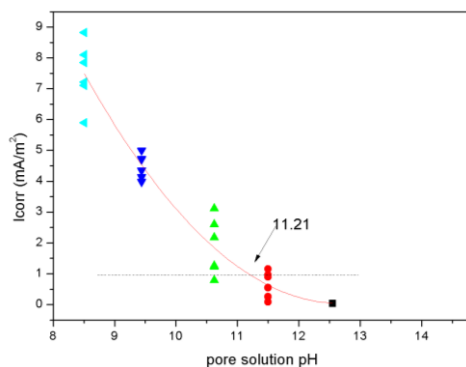


Fig. 8 Corrosion current densities changed with the pore solution pH values for the samples in carbonated concrete, obtained by EIS

which indicate an upgraded risk of corrosion for the steel reinforcements in concrete. At the same time, the polarization resistance (R_s) for each sample obtained is inconsistent with that calculated by EIS at the same pH level. However, the difference becomes little when the pH level in the sample is decreased to 9.44. Moreover, the samples at an identical pH level display a different half-cell potentials and polarization resistances (R_s), which have been attributed to the stochastic nature of corrosion for steel reinforcement in a concrete.

The corrosion current densities of six steel reinforcements at the same pH value, had been plotted with pH values, as shown in Fig. 8. With the decrease of pH values, the corrosion current densities and corrosion risk of steel reinforcement raised. However, no sudden shift of corrosion current density was clearly indicated. The reason may be attributed to that the corrosion current indicated in Fig. 8 was not monitored continuously on an identical sample, but measured on the different ones (Xu *et al.* 2009). Moreover, the stochastic nature of corrosion may influence the result, too. Furthermore, by fitting the relationship in the exponential equation between the corrosion current densities and pH values, and consider the pH where the corrosion current density of steel bar was higher than 1 mA/m² as the pH threshold value (Xu *et al.* 2009), the pH value could be calculated. The pH threshold value concluded by the detection method of EIS was 11.21.

At the same time, the half-cell potentials for the samples plotted with the corrosion current densities which were an average of six steel reinforcements respectively were showed in Fig. 9. With the decrease of pH values, the corrosion current density and corrosion risk of steel reinforcement raised. Where the corrosion current density of steel bar was higher than 1 mA/m², the half-cell potential can be calculated. The concluded half-cell potential was -357 mV. According to ASTM C-876, if the potentials were lower than -350 mV SCE, reinforcing steel corrosion was occurring. It can be detected that results decided by the methods of half-cell potential and EIS, applied to diagnose the corrosion condition of steel reinforcement in a concrete in carbonated concrete, were approximate.

From the above results, we can conclude that the pH threshold values decided by two different detection methods were similar. Considering the safety of structural, the pore solution pH threshold value for the corrosion of steel reinforcement concluded by 11.21 may be suitable. The pore solution pH threshold value for the corrosion of steel reinforcement concluded by the electrochemical impedance spectra was 11.21, which was higher than the value 11.16 that decided by half-cell potential. The results may be due to the factor that the two methods were by means of different electrochemical mechanism and parameters, which may influence pH threshold value. Besides, the pH values studied in the test were not monitored continuously on the samples, which may increase the error of the fitting equation by which the pH threshold value was concluded. Another reason was the stochastic nature of corrosion for steel reinforcement in a concrete affected by several factors (such as moisture content in the concrete, temperature, type of cation, O₂ content in the sample, blended materials, chemical composition of steels and their finishing, etc.). Otherwise, Green Rusts formed by the presence of Fe (II) ions in concrete were common corrosion products and they transform either to goethite or to lepidocrocite (Duff *et al.* 2004). The stochastic nature of transformation may also influence the results. Both of the pH threshold values detected by methods of EIS and half-cell potentials were lower than 11.5. This was not in accordance with the Pourbaix diagram of iron (Fe/H₂O). Previous results of the literature indicated that the transition conditions depend

probably not only on the pH but also on the carbonate content of the interstitial solution (Huet *et al.* 2006).

4. Conclusions

The pH threshold value on the corrosion of steel reinforcement in concrete had been investigated. The pH threshold value for the corrosion of steel reinforcement was 11.21. Agreement among whether steel corrosion was initiatory determined by the detection methods mentioned above could be found. The half-cell potential threshold value for the corrosion of steel reinforcement concluded by the detection method of EIS was -357 mV, which was approximate to the value of -350 mV from ASTM C-876.

Acknowledgments

The research described in this paper was financially supported by the Opening Foundation of State Key Laboratory of Green Building Materials (YA-533).

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