

Research Article

In Situ Determination of the Transport Properties of Near-Surface Concrete Using AC Impedance Spectroscopy Techniques

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The durability of existing concrete structures has increasingly attracted widespread attention in recent years. The phenomenon of performance degradation is often associated with the intrusion of hazardous ions from outside. As the first barrier to external substances intrusion, the near-surface concrete plays an important role in durability. So the performance of in-service concrete structures often depends on the transport properties of the near-surface concrete. Accordingly, information on service conditions and life prediction can be obtained by testing these transport properties. In this paper, an *in situ* method for chloride ion diffusion coefficient determination is proposed based on the relationship between the alternating current impedance spectroscopy parameters and the chloride ion diffusion coefficient. By a rational design, the new method can synthetically reflect the transport properties of near-surface concrete and is not affected by the presence of the reinforcing bar. In addition, the experimental results show that the method is in good agreement with “PERMIT” migration test which has been widely used. The proposed method is less time consuming and nondestructive and has good reproducibility.

1. Introduction

Durability of civil engineering structures has attracted worldwide attention. Concrete is predominantly used in construction within worldwide. In order to predict the service life of existing concrete structures, some important parameters such as chlorine ion diffusion coefficient must be obtained, especially for concrete structures exposed to deicing salt or marine environment. Laboratory test methods for permeability determination are generally suitable for a concrete block prepared in the laboratory. Drilled cores cannot reflect well the nature of in-service concrete structures because the field pouring, vibrating, and curing conditions of concrete are different from those of the laboratory; core samples drilled from structural members will bring damage resulting in microstructure change; and existing concrete structure always bear a certain load. Moreover, these methods aim to study the entire test block rather than the near-surface concrete. The near-surface concrete is the primary barrier to harmful substances, thereby determining the durability of

reinforced concrete structures to a certain extent. For these reasons, research and development of the chloride ion permeability test method applicable to *in situ* testing are particularly important. Researchers have proposed many field test methods. Some methods are for liquid intrusion [1–8], while some are for gas permeation [7–9] and electromigration [10, 11]. The gas permeation tests have high sealing requirements and are susceptible to concrete humidity. These methods also have poor discrimination ability for high-performance concrete. Liquid intrusion tests are simple to perform, and the required test equipment is also simple. However, their ability for high-performance concrete distinction is poor. These methods are apt to be influenced by temperature and humidity, which greatly reduce their applications. Chloride ion migration tests have reliable theory, but their testing process and data processing are complicated.

A testing method for chloride ion diffusion coefficient determination based on the alternating current (AC) impedance spectroscopy technique is established by Wu et al. by choosing an equivalent circuit model with a clear physical

meaning, using the Einstein–Smoluchowski equation, introducing a high concentration of chloride ion solution, and considering the correction of decreased impedance because of mineral admixture [12]. The AC impedance technique is applied to the field test in this paper through many considerations.

The *in situ* near-surface transport property measurement is quite different from that of the laboratory block test. First, the electrode arrangement is different. In the laboratory test, two stainless steel electrodes are attached to two opposite surfaces of the fully saturated specimen. However, one option for the *in situ* situation is the preembedding of an electrode in the concrete and attaching another electrode on the concrete surface. On the one hand, the diffusion coefficient equation of the laboratory test can be directly adopted. On the other hand, the measured point is fixed and the embedded electrode is susceptible to vibrating during concrete pouring, which results in errors that cannot easily be eliminated. The second method is arranging two electrodes on the concrete member surface, which will cause change of the conductivity cell constant. The influence of reinforcing bar on the impedance test is not clear. The advantage of this method is that the test location can be freely selected. The latter is used in this paper.

Using surface-contacted electrode arrangement will introduce several issues: how to determine the electrode shape and size, how to ensure close contact between the electrodes and the concrete surface, how to determine cell constant, and the influence of reinforcing bar on impedance testing. These problems will be solved individually.

2. Tester Design

The tester design, especially the electrode selection and arrangement, is the primary problem that must be considered for the field permeability test. Four points should be considered. First is the choice of electrode material. This test does not use the electrode/interface reversible electrochemical reaction. Hence, the electrode can be inert, and an economical stainless steel electrode can be used. Second is the electrode shape and size. Using symmetric electrodes can produce a symmetric charge transfer pathway, which is easy to calculate. Therefore, a circular electrode is used as the inner electrode and an annular electrode is utilized as the outer. The diameter of the inner and outer electrodes should be able to cover one to two times the area of the coarse aggregate. The specific size can also be further determined. Third is that the valid test depth should be roughly equal to the concrete cover. The effective test depth is small if the distance between the inner and outside electrodes is small. The effective test depth will also increase if the distance is increased. The electrode size and arrangement can be mathematically determined if a particular test depth is specified. Fourth is the contact between the electrode and the measured surface. A thin layer of the electrolyte solution with good conductivity can be introduced to ensure a close contact between the two.

The preliminary tester design is presented in Figure 1 by the preceding considerations.

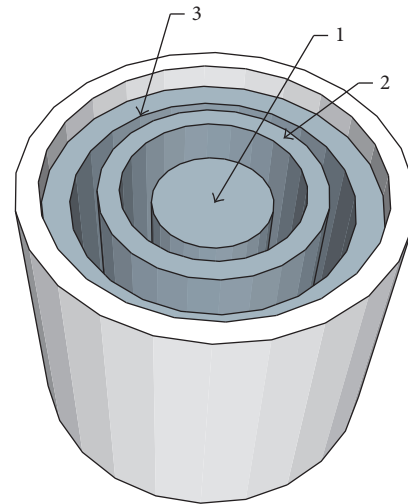


FIGURE 1: Schematic diagram of the tester.

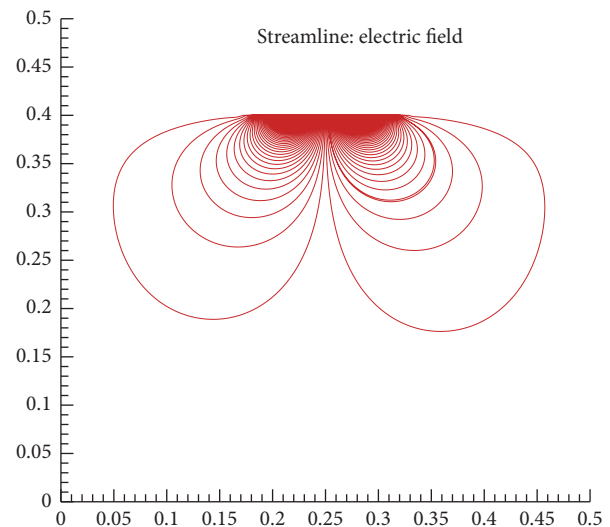


FIGURE 2: Electric field intensity distribution of the near-surface concrete.

The tester chamber is made of polymethyl methacrylate. In the center of the tester is a column (marked as 1) on which the inner circular electrode is fixed. A hollow cylinder marked as 2 serves as an isolator forming an inner chamber and an outer chamber. A hollow cylinder marked as 3 is used to fix the outer annular electrode. An annular notch is located on hollow cylinder 2 to mount a rubber ring for the complete separation of the inner and outer chambers. The outermost annular ring 4 is used to fix the tester onto the concrete surface. The two electrodes are connected to the impedance analyzer by wires. The highly conductive electrolyte solution (1 mol/L NaCl) is injected into the inner and outer chambers after the tester is mounted.

The electrode size and arrangement are calculated using the electric field finite element analysis (Figure 2). The finite element model is axisymmetric. The size of the internal and the external electrodes and their spacing are the same as

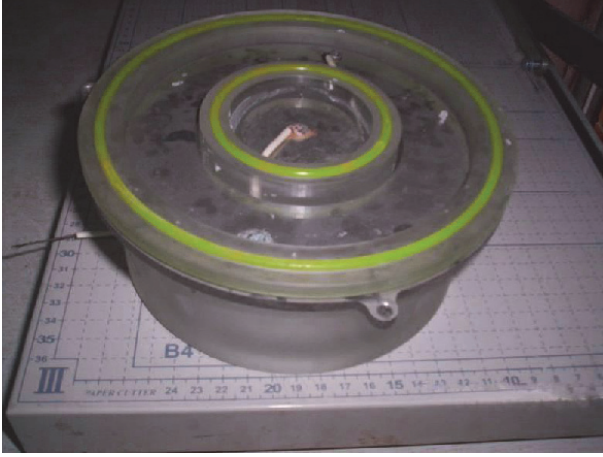


FIGURE 3: Prototype of the AC impedance field testing.

those of the prototype (see Figure 3). The potential difference between the two electrodes is 10 mV, and the relative permittivity is 4.2. The length of the solution domain is 0.5 m and the width is 0.4 m. The element is square, with side length of 0.005 m.

The effective test depth should be approximately equal to 30 mm, which is the typical thickness of the concrete cover. Accordingly, the finite element simulation shows that the diameter of the inner circular electrode can be 50 mm; the internal diameter of the outer electrode is 88 mm; and the external diameter is 142 mm. These values allow the near-surface electric flux to account for more than 90% of the total electric flux. The manufactured tester based on the results of the finite element simulation is presented in Figure 3.

Many holes are drilled in the concrete before the test to fix the tester using bolts and nuts. Subsequently, 1 mol/L NaCl solution similar to that used in the theoretical part of this paper is utilized to conduct vacuum salt saturating. The tester is fixed together, and the highly conductive electrolyte solution is injected into the chambers. The AC impedance test is then performed.

3. Cell Constant Determination

The theoretically deduced chloride ion diffusion coefficient is calculated as follows [12]:

$$D_{Cl^-} = \frac{aRT}{1.64F^2 R_{cp} C_{Cl^-}}, \quad (1)$$

where R is the universal gas constant ($=8.314 \text{ J K}^{-1} \text{ mol}^{-1}$); T is the absolute temperature (K); F is the Faraday constant ($=96485 \text{ C mol}^{-1}$); R_{cp} is the resistance of the connected capillary pores or connected microcracks in concrete (Ohm); and C_{Cl^-} is the concentration of chloride (mol L^{-1}). The cell constant (a) is the ratio of the length of the current conduction pathway to its cross-sectional area. The cell constant is the ratio of the thickness of the test block to the specimen/electrode contact area when two identical electrodes attached to two opposite surfaces of the specimen are used.

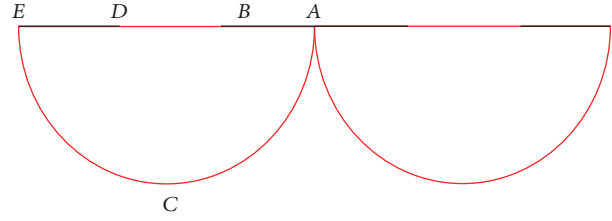


FIGURE 4: Assumption for the electric field lines.

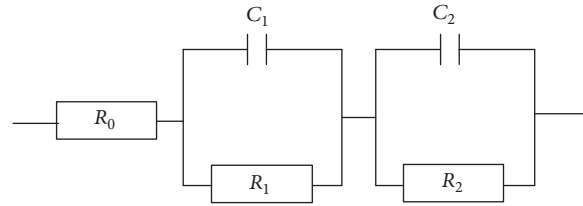


FIGURE 5: Equivalent circuit of the field test.

The current conduction pathway in the field test conditions is curved. The two contact areas of the specimen/electrodes are not equal (Figure 3). Therefore, the cell constant of the field testing needs to be further determined.

The following assumptions are made for simplicity: (1) the current conduction pathway of the concrete surface is a straight line with a length equal to the net distance of the electrodes; (2) the current conduction pathway of the outermost effective measurement range is a circular arc, and the lowest point is the effective test depth; and (3) the length of the current conduction pathway is the average length of the inner and outer conduction pathways, and the cross-sectional area of the current conduction pathway is the average area of the two electrodes (Figure 4).

The length of the outermost arc can be calculated according to the A , C , and E coordinates. The cell constant, a , is finally determined as 9.2 m^{-1} .

4. Determination of Equivalent Circuit for Field Test

A highly conductive electrolyte solution is introduced to reduce the impedance between the concrete surface and the electrodes. Therefore, the equivalent circuit for the field test (Figure 5) is different from that of [12].

The two left units of the equivalent circuit in Figure 5 are similar to those used in [12]. The right unit of the circuit in the same figure comprises a resistor-capacitor in parallel representing the resistance and the interfacial capacitance caused by the introduction of the electrolyte solution. The resistance of the 1 mol/L NaCl solution layer ($R_2 = \rho L' / A'$, where L' is the distance between the concrete surface and the electrode and A' is the electrode area) is actually much smaller than the resistance of the capillary pore solution in

TABLE I: Concrete mix.

Specimen number	Materials used (unit: kg/m ³)						
	Cement	Fly ash	Slag	Silicon fume	Water	Fine aggr.	Coarse aggr.
C0	466.0	0	0	0	186.0	750.0	1125.0
FA1	372.8	92.3	0	0	186.0	735.7	1103.6
FA2	276.6	186.4	0	0	186.0	720.5	1080.9
FA3	186.4	276.6	0	0	186.0	705.3	1058.0
GS1	349.5	0	116.5	0	186.0	747.6	1121.4
GS2	233.0	0	233.0	0	186.0	744.2	1116.3
GS3	116.5	0	349.5	0	186.0	740.8	1111.2
SF1	442.7	0	0	23.3	186.0	747.6	1121.4
SF2	419.4	0	0	46.6	186.0	744.2	1116.3
SF3	396.1	0	0	69.9	186.0	740.8	1111.2

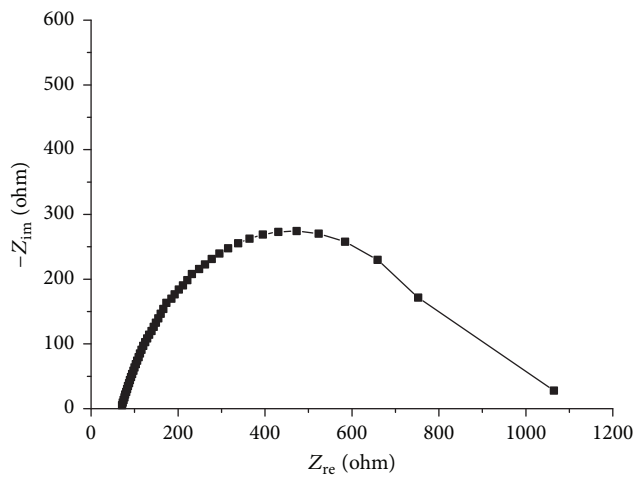


FIGURE 6: Nyquist plot of a typical field AC impedance measurement.

concrete. Accordingly, the right semicircular generated by R_2 and C_2 in parallel is much smaller than that of R_1 and C_1 in parallel. Therefore, the right arc is not visible. The Nyquist plot of the measured impedance data (Figure 6) does not exhibit two semicircular arcs, which can also prove this point.

5. Factors Affecting Field Test

The two main factors affecting the *in situ* determination of the transport properties using the AC impedance technique are water/salt saturating level and the presence of the reinforcing bar. The current carriers in the impedance testing are dissolved ions. A significant increase in the measured impedance, which does not reflect the true permeability-related parameters, is obvious if the saturating level is low. Exploratory tests show that the measured impedance difference could be several hundreds or even thousands of times for the same concrete specimens with different saturating levels. Therefore, the in-field test water/salt saturating level must be guaranteed before the testing.

Commercially available water saturating equipment can be used for laboratory testing. However, related equipment

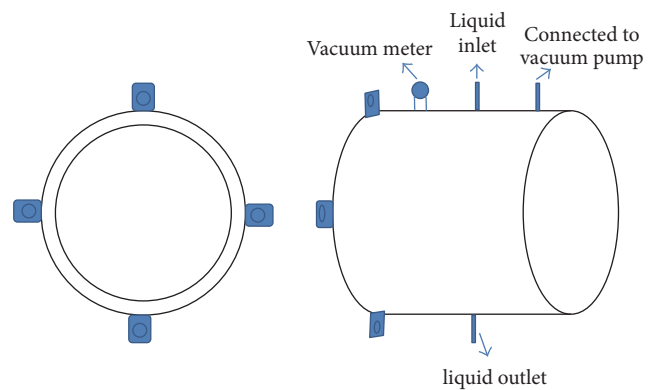


FIGURE 7: Design of the field test saturating equipment.

for the field testing is not yet available. Hence, a prototype is designed (Figure 7).

First, lower the pressure inside the container from normal atmospheric pressure to 0.1 atm or less, then inject saturated calcium hydroxide solution into the container, and continue to maintain the vacuum degree. The saturation process may take up to 6 hours, depending on the density of the cover concrete. Also an alternative approach is recommended. Attach a piece of sponge to the concrete surface and spray calcium hydroxide solution to the sponge and ensure that the sponge is saturated. In general, it may take three days for the concrete surface to be saturated using this method.

The presence of the reinforcing bar as a conductor may comprise part of the current conduction pathway, which will affect the impedance measurement for the near-surface concrete. An experimental study and a theoretical analysis of the influence of the reinforcing bar on the AC impedance test will be presented in this section.

The specimens ($\Phi 300 \text{ mm} \cdot 120 \text{ mm}$) are prepared using the concrete mix mentioned in Table I.

The concrete cover thickness for the reinforced concrete specimen is 30 mm. All the specimens are subsequently immersed in a saturated solution of calcium hydroxide after mold removal. The typical plain and reinforced concrete specimens are illustrated in Figure 8.



(a) Plain concrete blocks



(b) Reinforced concrete blocks

FIGURE 8: Concrete blocks.



FIGURE 9: AC impedance test.

Salt saturating is first performed using the 1 mol/L NaCl solution. The 1 mol/L NaCl solution is injected to reduce the interfacial effect after the tester is fixed onto the concrete surface. An Agilent 4294A precision impedance tester is used to perform the test. The frequency range of the impedance test is 40 Hz–40 MHz, while the amplitude of the applied AC is 0.5 V. Five points (logarithmic spacing) are collected for each order of magnitude. An image of the testing is presented in Figure 9.

The typical Nyquist plots of the impedance test of the plain and reinforced concrete blocks with the similar mix design are illustrated in Figure 10.

The presence of the reinforcement bars has little effect on the AC impedance testing compared with the impedance test result of the plain concrete specimen (Figure 10). The reason lies in the designed tester. The effective test depth is 30 mm. In other words, the location of the reinforcing bar is beyond the designed test depth. The finite element simulation shows that the electric field intensity decreases with the increasing depth. The near-surface part of the concrete decides the impedance

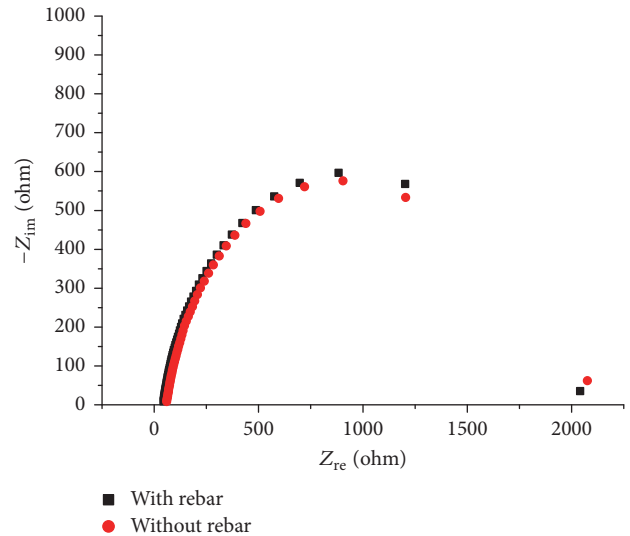


FIGURE 10: Influence of the reinforcing bar on AC impedance testing.

to a great extent. Therefore, the field impedance test is not subject to the influence of the reinforcement presence after a careful design.

6. Experimental Study of the Concrete with Different Cementitious Materials

Table 1 shows the concrete mix used in this research. The shape, size, and curing condition of the prepared specimens and the test parameters used are all similar to those in Section 5. The testing age is 28 d and 90 d.

The typical impedance data obtained are presented in Figures 11 and 12.

The chloride ion diffusion coefficient can be obtained using (1) by numerical fitting between the measured impedance data and the selected equivalent circuit and using the approximate *in situ* cell constant (i.e., $a = 9.2 \text{ m}^{-1}$) after

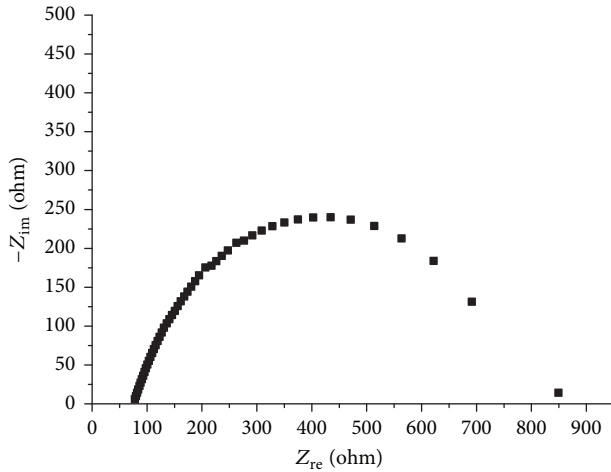


FIGURE 11: Nyquist plot of specimen number SF3 (28 d).

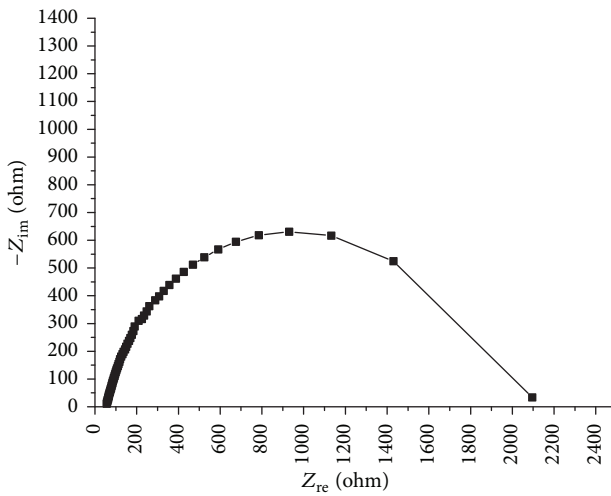


FIGURE 12: Nyquist plot of specimen number SF3 (90 d).

data validation checking. The diffusion coefficient can then be further corrected according to the type of cementitious materials (see [12]). The chloride ion diffusion coefficients for the concrete with different cementitious materials are presented in Table 2.

7. Analysis and Discussion

The proposed *in situ* method for the chloride ion diffusion coefficient determination can discriminate concretes with different cementitious materials (Table 2). The concretes with mineral admixtures will effectively reduce the diffusion coefficient at a long age. As regards the concrete with fly ash, especially on the high replacement level, the anti-intrusion ability will decrease at an early age (i.e., less than 90 d) probably because of the lower fly ash activity. Experimental studies have shown that the reaction degree is very low before 28 d [13]. Moreover, the fly ash reactivity is only able to increase significantly after 90 d. By contrast, the silica fume has a much higher reactivity. The particles of the silica fume

TABLE 2: Test results of the chloride ion diffusion coefficients.

Specimen	Chloride ion diffusion coefficients	
	D (28 d) ^a ($\times 10^{-12} \text{ m}^2 \text{ s}^{-1}$)	D (90 d) ($\times 10^{-12} \text{ m}^2 \text{ s}^{-1}$)
C0	3.60 (3.71)	1.40
FA1	5.82 (5.90)	1.10
FA2	2.70 (2.82)	1.21
FA3	4.73 (4.88)	1.63
GS1	2.85 (2.96)	1.35
GS2	2.15 (2.32)	1.20
GS3	1.42 (1.60)	1.03
SF1	1.20 (1.31)	0.41
SF2	0.83 (0.87)	0.34
SF3	1.03 (1.12)	0.36

^aData in () are obtained from the PERMIT ion migration test; see Section 7 for details.



FIGURE 13: “PERMIT” ion migration test.

are also much smaller than those of the cement particles. Therefore, the pores in the concrete can be effectively filled, which results in a decrease of the interconnected porosity that consequently reduces permeability. However, the chloride ion diffusion coefficient for the high replacement level (i.e., 15%) increases maybe because of the mixture becoming too viscous to compact, which causes the porosity to increase. Compared with the fly ash and the silica fume, slag has an in-between activity. Microscopic observation and mercury injection tests show that the reaction degree of the slag obviously increases with the increasing age, which decreases the permeability [14]. Therefore, the test result of this study reflects the well-recognized understanding.

Besides, the proposed test method was validated in the lab by carrying out a comparative investigation and correlating the results with the migration coefficient from the “PERMIT” test [10, 11]; the procedure to carry out the PERMIT ion migration test can be found in [10]. The concrete mixes are the same as that of Table 1. The test specimen is 600 mm * 300 mm * 150 mm; see Figure 13 for details.

The results in Table 2 also indicate that the proposed method correlates well with the “PERMIT” ion migration test, which has gradually gained recognition worldwide.

Compared with the water and gas permeability tests, the proposed field test method requires full water/salt saturating, which makes it less affected by the moisture in the concrete. The impedance method greatly reduces the testing time compared with the “PERMIT” ion migration method. The measuring time is less than 5 min after the preparatory work is done. However, the concrete must be fully saturated with sodium chloride solution; otherwise, the obtained results are unreliable.

8. Conclusions

The AC impedance spectroscopy technology is less time consuming. The concrete mesostructure can be well revealed through numerical fitting between the preset equivalent circuit and the measured impedance data. The AC impedance technique-based method is applied to the field test using the carefully designed tester.

The proposed method is not subject to the influence of the presence of the reinforcement because the electrode shape, size, and arrangement are considered. The cell constant is approximately calculated based on the finite element analysis of the electric field and through many assumptions.

A highly conductive electrolyte solution fills the space between the electrodes and the concrete surface to guarantee the validity of the test data, ensure good repeatability, and minimize the interface effect. The additional resistance and capacitance caused by introducing the electrolyte solution can be ignored in the Nyquist plot.

The most important factor affecting the measuring accuracy is the saturating degree after the influence of the reinforcing bar is eliminated. The near-surface concrete must be fully saturated before the tests to restrain the unpredictable error effectively.

The proposed method has good theoretical basis and can effectively distinguish the antipenetration ability of different concretes.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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