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Determining porosity and pore network connectivity of cement-based materials by a modified non-contact electrical resistivity measurement: Experiment and theory

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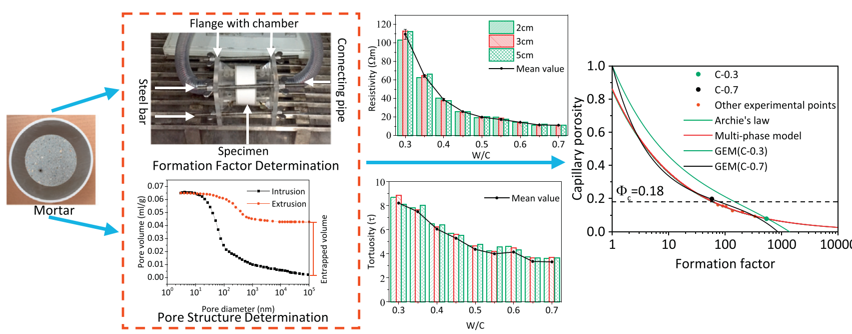
HIGHLIGHTS

- A modified non-contact electrical resistivity measurement can test the resistivity of mature cement-based materials.
- The factor of m in Archie's law and t in General effective model has the quantitative relationship with tortuosity.
- General effective model gives a precise description of pore structure and formation factor contrast to the empirical model.

GRAPHICAL ABSTRACT



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ABSTRACT

A modified non-contact electrical resistivity measurement is proposed to determine porosity and pore network connectivity of cement-based materials. The porosity of mortar with different water-to-cement ratios was tested by mercury intrusion porosimetry (MIP) and the tortuosity was calculated from pore entrapment volume fraction. The relationship between pore structure and resistivity was studied using the three most commonly used theories. It was observed that the multi-phase phenomenological model can indicate the effect of porosity and pore connectivity on the resistivity of cement-based materials. The parameter m in Archie's Law as an index of pore connectivity can be expressed as a variable that varies with the tortuosity. The parameters in the GEM model are closely related to the nature of the material: the parameter M should be related to the composition of the material and the degree of hydration of the cement, and the parameter t is a variable that varies with the pore connectivity. Based on these theories, equations are derived to correlate the porosity and pore network connectivity to the formation factor of cement based materials. The developed approach thus has a potential to be used as a simple and effective tool for pore structure investigation of cement based materials.

1. Introduction

Cement-based materials are the most widely used building materials around the world, so their durability against the aggressive service environment is most worthy of study. In general, cement-based materials are

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considered to be multiphase porous solid materials, and their pore structures play an important role on their durability [1, 2]. The pore structure of cement-based materials determine the ingress of harmful ions [3, 4], moisture [5, 6], gas [7, 8] and other ingredients, which may lead to corrosion of internal steel and thus cause service life reduction. Therefore, the study on the pore structure of mature cement-based materials (28 days) is of great significance to the study of structural degradation mechanisms and the prediction of service life.

Many methods have been proposed for the study of the pore structure of cement-based materials, such as mercury intrusion porosimetry (MIP) [9], nitrogen adsorption/desorption (NAD) [10], backscattered electron (BSE) image analysis [11, 12], small-angle scattering [13] and so on. Since the electrical parameters of porous solid materials depend on the pore structure and the nature, mobility, and concentration of the fluid filling pores, they are very sensitive to the change in the pore structure, if the pore fluid keeps unchanged. Assuming the pore fluid in a mature cement-based material is unchanged, electrical resistivity measurement can also be used to investigate the pore structure based on pre-defined resistivity-pore structure physical models. Compared with other methods, resistivity measurement does not require special pretreatment of the specimen (e.g., different sample preparation methods for MIP lead to different results [14]), enabling in-situ measurement (i.e., other methods require representative small samples or local observations [15]), thus maximizing the integrity of the pore structure [16].

Due to the complexity of the pore structure of cement-based materials, it is difficult, if not impossible, to establish a general theory that can fully reflect the relationship between pore structure and electrical resistivity [17, 18]. It is more practical to represent the pore structure using pore structure characteristic parameters, i.e., porosity and pore network connectivity, and correlate these parameters to the electrical resistivity of cement-based materials. There have been a number of attempts to analyze the relationship between resistivity and pore structure of cement-based materials [17, 19–21], and different test methods have been proposed for resistivity testing. The most commonly used methods include the four-electrode method [15, 22] (or known as Wenner method) and the method specified in ASTM C1202 [23]. The common drawback of these two methods is the presence of metallic electrodes and the use of direct current (DC), which raise two major problems: (1) The DC current applied to a cement-based specimen produces a polarization effect; (2) The contact between the electrode and the material will have a greater impact on the test results [24]. To eliminate the polarization effect, a high-frequency (1000 Hz) alternating current (AC) can be applied for measuring electrical resistivity [25–27]. However, the presence of metallic electrodes still leads to contact problems and potentially a problem of gas release caused by electrolysis of water around the electrodes [28]. In order to overcome these problems, Li et al. developed the non-contact electrical resistivity measurement device, which has be used to study the hydration mechanism and porosity evolution of cementitious materials [29–31], and have been modified to take account the effect of different curing temperatures [17, 32]. This device is based on the principle of transformer and eliminates the presence of electrodes used in the traditional methods [33].

The non-contact electrical resistivity measurement method was designed for study of fresh-state to early age cement-based materials. In the present paper, this method is modified to test the electrical resistivity of mature cement-based materials (after 28 days). Physical models were developed to discuss the possibility of characterizing the pore structure (porosity and connectivity) of mature cement-based materials through the modified non-contact electrical resistivity measurement.

2. Raw materials and sample preparation

2.1. Materials and mixtures

All tests were carried out using ordinary Portland cement (ASTM type I). The chemical composition and physical porperties of cement are presented in Table 1. The fine aggregate was natural river sand with a fineness modulus of 2.64. The details of mixture proportions are given in Table 2. This study is limited to investigate the effect of pore structure of binder phase (altered due to w/c), thus sand/cement ratio is fixed. The effect of sand gradation and coarse aggregate will be addressed in another work. All mixtures with the raw materials was mixed using distilled water. Polycarboxylic superplasticizer (SP) with 30% water-reducing rate was used in the w/c of 0.3, 0.35 and 0.4.

2.2. Sample preparation

Each mixture was mixed for 2 min in a planetary-type mixer at 45 rpm, followed by a high speed (90 rpm) for 1 min. The resultant mortars were cast in cuboid molds of dimensions of 100 mm × 100 mm × 300 mm, and placed in room condition at 23 ± 2 °C. After 24 h, the samples were de-molded and immersed in water at 23 ± 2 °C for 6 months.

Circular cross-section cores with the dimension of diameter 70 mm × height 300 mm were prepared by drilling the original cuboid specimens using a concrete core drilling machine. All of the circular cross-section samples were placed in an oven at 105 °C for 24 h to remove the internal moisture. Each core specimen was then put in a PVC pipe with the dimension of diameter 100 mm × height 300 mm, and the 15 mm wide gap between the specimen and the PVC pipe was filled by epoxy. When the epoxy hardened, slices 20 mm, 30 mm and 50 mm thickness were cut from the middle portion of each core specimen. Thinner slices were not considered, since the air voids inside the specimen can affect the measured transport properties so representative results could not be obtained [34]. The slice specimen used for electrical resistivity measurement in this study is illustrated in Fig. 1. Before measurements, the slice specimens were saturated by 3% NaCl solution: each slice was vacuumed for 3 h in a chamber, which was followed by NaCl solution injection and immersion under vacuum for 18 h [35]. NaCl is selected because it is the most common non-toxic lab reagent, and its aqueous solution has a much higher conductivity than the pore solution (when the concentration is high, limited chemical binding of chloride doesn't lower the conductivity obviously).

The remaining parts of the sliced core specimens were cut into pieces (~5 mm × 2 mm × 1 mm), which were oven dried at 105 °C for 24 h and used as MIP samples. Sufficient samples were prepared for three repeated MIP measurement, for each mixture.

3. Experimental program

3.1. Modified non-contact electrical resistivity measurement

Fig. 2 shows the working scene of the modified non-contact resistivity measurement. The whole system included the specimen platform, mainframe and the computer for data collection. The specimen platform consists of two flanges with chamber and a connecting pipe, both sides of the specimen are clamped by the flanges, and the flanges on both sides are fixed by steel bars. Both ends of the chamber are connected by a connecting pipe through a transformer core and a leakage current meter as shown in Fig. 2.

A wirewound coil acts as primary of the transformer, the connecting pipe filled with 3% NaCl solution together with sample acts as the secondary. When a 1 kHz AC voltage is applied to the primary coil, a toroidal current will be induced in the secondary coil. This current is measured by the leakage current measurement. Meanwhile, the voltage is determined from the transformer principle. The overall resistance of

Table 1
Chemical composition (% by mass) and fineness of the cement.

CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	LOI	Fineness (m^2/kg)
64.47	20.87	4.87	3.59	2.13	2.52	0.65	0.11	0.77	368.9