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Monitoring the evolution of material structure in cement pastes and concretes using electrical property measurements

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HIGHLIGHTS

- Electrical impedance used to monitor early age characteristics of cement paste incorporating admixtures.
- Conductivity measurements on cement paste and concrete used to study the evolution of porosity.
- The study shows the importance of considering the solid phase conductivity in addition to the pore solution conductivity.

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ABSTRACT

Electrical conductivity is an effective parameter to monitor the microstructural features of cement-based materials. Monitoring the early age hydration process in cementitious materials containing mineral and chemical admixtures, and non-invasive determination of the pore structure features, through the use of electrical response, is discussed in this paper. The initial and final setting times of cement pastes predicted using electrical property based methods are very close to the measured values, showing that conductivity is an effective tool to monitor the setting process in plain and modified systems. The evolution of total porosity in the selected cement paste and concrete systems is predicted using electrical property based models derived from effective medium and percolation theories, and is found to match well with the experimental values.

1. Introduction

It is essential to monitor the changes occurring in concrete at early ages, since many of the later age properties of the material depend on cement hydration during early ages. With the increasing emphasis on characterizing concrete durability, a more detailed understanding of the evolution of pore structure features (porosity, pore sizes, and pore connectivity) of cement based materials is being sought. While destructive techniques such as mercury intrusion porosimetry and solvent replacement methods can provide accurate indications of the porosity in cementitious systems, there is a need for non-destructive/non-invasive methods to continuously monitor the porosity to characterize concrete as-placed. In this context, electrical conductivity measurements emerge as an effective strategy [1]. Electrical conductivity of cement based materials is dependent on the porosity, pore connectivity and the conductivity of the pore solution, all of which are significant in dictating the durability of the material. Numerous studies have taken advantage of this dependence to show that electrical conductivity measurements can be an effective tool to monitor the time-dependent behavior of cement based materials [2–13]. Measurement systems are available which make it possible to apply these methods in situ also, which is more relevant when compared to controlled laboratory conditions.

Extensive research on the application of electrical methods on cement based materials show that many important parameters of concrete can be analyzed effectively using these methods. A wide variety of applications of electrical methods on cementitious materials are reported, ranging from monitoring changes in pore solution and pore connectivity with time [2,3], effects of mineral and chemical admixtures [4–6], diffusion, permeability and chloride conductivity [13–15], and determination of setting time [16]. This paper describes an experimental study on monitoring the evolution of porosity in cementitious pastes and concretes using electrical conductivity. The setting times of these systems are also predicted using the conductivity data. While reference [1] deals with the generalized effective medium theory for the prediction of porosity of plain cements, its adequacy for pastes containing admixtures, as well as the use of other chosen multi-phase models

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for electrical conductivity towards predicting the porosity of cementitious systems are discussed in this paper.

2. Experimental programme

2.1. Materials and mixture proportions

The experimental work is divided into two phases. In Phase I, Type I ordinary Portland cement conforming to ASTM C 150 [17] was used to proportion the cement pastes. Commercially available limestone powder (LS), Class F fly ash (FA) conforming to ASTM C 618 [18], and silica fume (SF) conforming to ASTM C 1240 [19] were used as partial cement replacement materials. The chemical compositions of these materials are provided in Table 1. A commercially available polycarboxylic ether (PCE) based superplasticizer (SP), supplied by BASF chemicals, conforming to ASTM C 494 [20] was used to reduce the water demand and increase the workability of cement pastes.

In Phase II, the properties of materials used were different from that used in Phase I. A commercially available 53 grade ordinary Portland cement (OPC), conforming to IS 12269 [21] was used. Limestone powder, Class F fly ash conforming to IS 3812 [22], and silica fume conforming to IS 15388 [23] were used as mineral admixtures. Chemical compositions of these materials are also given in Table 1. It can be noticed that the chemical compositions of the materials used for both the phases are very similar. Crushed granite as coarse aggregate and river sand as fine aggregate were used for concrete mixtures. The maximum size of the coarse aggregate was 10 mm and the fine aggregate had a fineness modulus of 2.13. A commercially available PCE based superplasticizer was used for concrete mixtures.

For Phase I studies, seven mixes were proportioned with different dosages of mineral and chemical admixtures. A water-to-binder ratio (w/b) of 0.30 was maintained throughout the phase I study. A plain cement paste with a water-to-cement ratio (w/c) of 0.30 was used as the reference mixture. In three mixtures, partial cement replacement was adopted: one with 10% of cement by mass replaced by limestone powder (LS), the second with 20% of cement by mass replaced by fly ash (FA), and the third with 10% of cement by mass replaced by silica fume (SF). In three other plain cement mixtures, 0.20%, 0.30% and 0.40% of SP respectively by mass of cement was used. In the figures and tables, these mixtures are represented by the letter "H" followed by the dosage of the SP. For Phase II studies, seven different concrete mixtures were prepared with the same levels of cement replacement materials as in the Phase I experiments. In the Phase II study, a w/b of 0.45 was used for the plain concrete and those modified with mineral admixtures. For the plain concrete mixtures where SP is used, a w/c ratio of 0.35 was chosen. Three dosages of the superplasticizer – 0.15%, 0.20% and 0.25% – were selected. A binder content of 420 kg/m^3 with 676 kg/m^3 of fine aggregate and fine to coarse aggregate mass ratio of 1:1.5 were used for all the mixtures.

2.2. Test procedures

In the Phase I studies, the plain and modified cement pastes were filled in acrylic molds (100 mm × 40 mm × 40 mm), immediately after mixing. Two stainless steel plates were placed at the ends of the molds to be used as electrodes. Alligator plugs from the impedance analyzer were attached to the electrodes. The impedance measurements were carried out using a Solartron 1260™ gain phase analyzer over a frequency range of 0.1 Hz–10 MHz using an AC signal of 250 mV amplitude. The test setup is shown in Fig. 1a. From the bulk resistance (R_b) of the sample, which is represented by the real coordinate of the meeting point of bulk and electrode arcs in a Nyquist plot [15,24], the effective conductivity, σ_{eff} was calculated as [24]:

$$\sigma_{eff} = \frac{l}{R_b \cdot A} \tag{1}$$

where l and A are the spacing between electrodes and cross sectional area respectively. Data was collected at an interval of 10 min until 3 h after mixing. Thereafter, readings were taken at an interval of 15 min until the age of 6 h, and 2 h until the age of 12 h. Beyond 12 h, measurements were done at 1, 3, 7, 14 and 28 days.

The non-evaporable water content was determined using the loss on ignition method. Test was conducted after 6 h, 12 h, 1, 3, 7, 14 and 28 days of hydration. Thin strips of plain and modified cement pastes were cured in saturated limewater. At the specified ages, small pieces from the samples were removed, pulverized, and soaked in acetone to stop further hydration. The pulverized samples were placed in crucibles and heated in an oven at 105 °C for 24 h. The samples were weighed after removing them from the oven, and the mass reported (w_{105}). This was followed by heating in a muffle furnace at 1050 °C for 3 h. After removing from the furnace, the samples were weighed again and the mass noted (w_{1050}). The non-evaporable water content per gram of binder was calculated as:

$$w_n = \frac{w_{105} - w_{1050}}{w_{1050}} \tag{2}$$

This data was used to determine the degree of hydration of the pastes. Setting times of plain and modified pastes were found out using a Vicat apparatus in accordance with ASTM C 191 [25].

Table 1
Chemical compositions of cement and mineral admixtures used in phases I and II.

Compound (%)	Cement		Limestone powder		Fly ash		Silica fume	
	Phase I	Phase II	Phase I	Phase II	Phase I	Phase II	Phase I	Phase II
SiO_2	20.20	20.80	0.80	0.68	50.24	58.83	93.40	82.16
Al_2O_3	4.70	4.76	0.17	1.06	28.78	30.08	0.42	2.60
Fe_2O_3	3.00	3.96	0.10	0.32	5.72	4.62	0.52	4.09
CaO	61.90	62.04	53.68	51.57	5.86	1.75	1.91	2.34
MgO	2.60	1.88	0.50	1.67	1.74	0.08	_	0.91
Na_2O	0.19	0.28	_	_	0.96	0.76	0.25	0.58
K_2O	0.82	0.20	_	_	0.84	0.36	0.79	1.20
SO_3	3.90	2.21	0.05	0.36	0.51	0.19	0.34	0.75
Loss on ignition	1.90	3.93	43.40	42.20	2.80	0.60	2.30	1.19

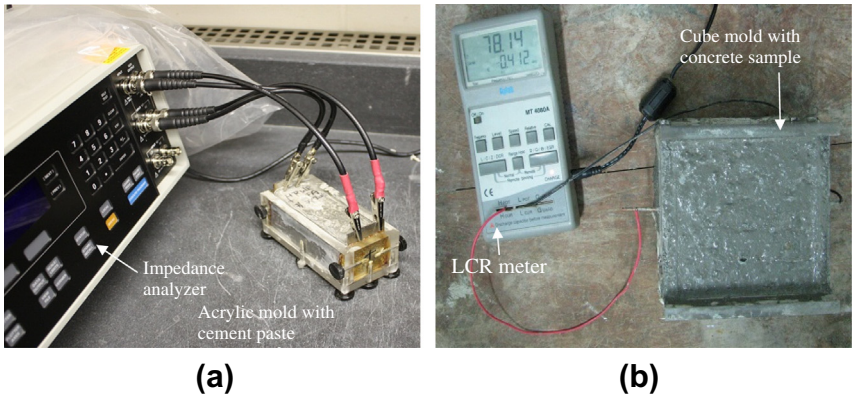

Fig. 1. Measurement of electrical conductivity of (a) cementitious pastes using an impedance analyzer, and (b) concretes using a LCR meter.