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Evaluation of mortar setting time by using electrical resistivity measurements

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HIGHLIGHTS

- Electrical resistivity of freshly mixed mortar was measured to monitor its setting process.
- Increasing electrical resistivity with hydration is affected by mix proportion and chemical admixtures.
- Measured electrical parameters were correlated with the setting time.
- The setting time of mortar can be estimated using the estimating equations.

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ABSTRACT

Cement hydration induces microstructural evolution in cement-based materials, causing setting and hardening. Various experimental techniques were proposed to evaluate the time of setting, which includes the Vicat needle test and ultrasound inspections. This study aims to measure electrical resistivity in order to monitor microstructural evolution. For this purpose, an optimized experimental setup is proposed by performing a series of pre-test analyses. A total of 51 mortar mixes are prepared to study the effects of various mix-proportions and the use of various chemical admixtures. As a result, two parameters that represent a change in electrical resistivity are identified: the rising time, which indicates the onset of an increase in electrical resistivity, and the increasing ratio over time. Both parameters are discussed with conventional test results obtained from the penetration resistance method. Their correlation allows us to estimate the setting time of mortar.

1. Introduction

Improved strength and durability of hardened mortar and concrete can be obtained by utilizing a thorough setting and hardening process. Cement hydration causes microstructural development in cement-based materials, and it is generally accompanied by an increase in hydration products and a decrease in porosity over time [1]. Various test methods were proposed to evaluate the microstructural development, which include isothermal calorimetry, X-ray diffraction, scanning electron microscopy image analysis, mercury intrusion porosimetry, and infrared spectroscopy [2,3].

The setting time is one of the most critical parameters for representing the degree of microstructural development. It indicates the onset of the transition from fluid-like suspension to porous solid, at which point the material becomes unworkable. The setting process is mainly influenced by the mix proportion, the fineness of cement, the incorporated admixtures, and environmental conditions [4–6]. The setting time is generally determined using a Vicat needle test for cement paste [7], or by measuring the penetration resistance for mortar and concrete [8]. Nevertheless, the conventional test methods for concrete are prone to errors in the field, and their reliability depends on the dimensions of the penetration needles and the sample container.

The setting of cement-based materials is related to the percolation of solid grains and the development of their network [5]. The setting process is accompanied by water-phase depercolation. The development of the solid network was explained by the percolation theory and its characteristics were correlated with the elastic modulus of cement-based materials [16–19]. Several nondestructive test methods were proposed to monitor microstructural evolution. In particular, methods that utilize ultrasonic response, such as wave velocity, reflection energy, and wave diffusion [9–15] were especially relevant.

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The measurement of electrical conductivity or resistivity is a promising method for monitoring the setting and hardening of cement-based materials. Ion transport through water-filled porosity determines electrical conductivity, and so cement hydration was experimentally monitored by its measurement [20–23]. The effect of admixtures on cement pastes was also studied using the measurements of electrical resistivity [24,25]. Dried and hardened cement paste and mortar have an electrical resistivity of approximately 10^3–10^4 Ω·m (corresponding to an insulating material) [27]. A saturated sample shows a lower resistivity [26]. Initially, the electrical resistivity was much lower (less than 10^2 Ω·m), but it increased as the setting and hardening process progressed. Accordingly, the electrical resistivity of the fresh concrete mixes was correlated with the water-to-cement ratio (*w/cm*) [28]. A delay in setting time and strength development in fresh concrete with low hydration heat was also investigated by monitoring the electrical resistivity [29]. The use of a non-contact electrical resistivity apparatus allowed for the investigation of the porosity changes in early-age mortar, which allowed for the estimation of the mortar's compressive strength [2,30].

Advancements in electrical test methods used on cement-based materials have brought improvements in the measurement of setting time. In this study, a stable measurement of the setting time of freshly mixed mortar is investigated using the four-electrode method. For this purpose, various types of mortar samples were prepared with different mix-proportions of water, aggregate content, and chemical admixtures. The measured electrical resistivity was compared with the measured penetration resistance. As a result, the estimating equations to determine the setting time were proposed based on the measured electrical parameters.

2. Experimental details

2.1. Sample preparation

The mix proportions of the mortar samples considered various cases in the field. Their *w/cm* was 0.45 or 0.5 by mass, and the fine aggregate-to-cement ratios ranged between 0 and 3 by mass. The samples were mixed for 10 min using a planetary mixer. The mix proportion of each sample is reported in Table 1. All sample cases having 17 different mix proportions were prepared as three duplicated samples for repeatability test. As a result, total of 51 mortar samples were prepared for experiments. In addition, cement paste and kaolinite paste were also tested to confirm the effects of cement hydration.

Portland type I cement with a specific gravity of 3.15 and a Blaine number of 344 m^2/kg was used. The oxide composition of the cement powder is shown in Table 2. River sand was used for the surface-dry saturated condition to minimize the water absorption effects on the test results. The maximum size of the river sand was 5 mm and its specific gravity was 2.31 in the surface-dry saturated condition. Air-entraining (AE) admixture and high-range water-reducing (HRWR) admixture were incorporated to investigate their effects on the setting time.

2.2. Penetration resistance measurement

In accordance with ASTM C 403 [8], penetration resistance measurement is generally used to measure the setting time of freshly mixed mortar or mortar wet-sieved from concrete. The method measures mechanical resistance for 25 mm-deep penetration using various needles. This study used a thin needle with a diameter of 4.5 mm and a cross-sectional area of 16 mm^2. The sample for the penetration resistance measurement was placed in a 500-mL beaker (100 mm diameter by 100 mm height). As previously stated in the introduction, the measurement of the setting time would be different when a different needle or sample container is used. The test standard defines the initial and final setting times that comply with the measured penetration resistances of 3.5 MPa and 27.6 MPa, respectively. The final setting time indicates that the green strength of a freshly mixed mortar reaches 0.7 MPa [8,31].

This study measured the penetration resistance of the samples until it exceeded 30 MPa in an interval of 30 min. A total of five duplicated samples were tested for the measurement of penetration resistance in each mixture. For example, Fig. 1 shows an average of the exponential trend in Sample A5. The R^2 value of correlated relationship is 0.97. The evolving penetration resistance indicates the initial and final setting times. Table 3 reports the measured setting times of all samples. Both setting times increased with higher water content or lower aggregate content. In addition, incorporating chemical admixtures delayed both setting times, as expected.

Table 1
Mix proportions of the prepared samples.

Label	Water (kg/m^3)	Cement (kg/m^3)	Sand (kg/m^3)	*w/cm* (%)	AE[a] (kg/m^3)	ETHER[b] (kg/m^3)	ESTER[b] (kg/m^3)
A1	587	1303	–	45	–	–	–
A2	470	1043	520	45	–	–	–
A3	390	867	867	45	–	–	–
A4	293	651	1301	45	–	–	–
A5	234	520	1561	45	–	–	–
B1	612	1223	–	50	–	–	–
B2	495	990	495	50	–	–	–
B3	416	832	832	50	–	–	–
B4	315	630	630	50	–	–	–
B5	254	507	1522	50	–	–	–
C1	293	651	1301	45	0.088	–	–
C2	293	651	1301	45	0.293	–	–
C3	293	651	1301	45	0.587	–	–
D1	293	651	1301	45	–	0.293	–
D2	293	651	1301	45	–	0.587	–
E1	293	651	1301	45	–	–	0.293
E2	293	651	1301	45	–	–	0.587

[a] AE refers to an air-entraining admixture.
[b] ETHER and ESTER refer to ether-based polyethylene oxide (PEO) vinyl and carboxylate-based methoxy PEO admixtures, respectively.