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Electrical response of mortar with different degrees of saturation and deicing salt solutions during freezing and thawing

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ABSTRACT

This paper discusses a series of electrical measurements made on cementitious mortars containing water–NaCl solutions (0–23.3% concentration by mass) over temperatures in the range of 23 °C to −35 °C. Electrical impedance spectroscopy, acoustic emission, and thermal measurements were made during cooling and heating to detect phase changes and resulting damage. The influence of the degree of saturation (DOS) and NaCl solution concentrations are examined. Three phase changes were detected: (1) eutectic phase change (∼−24 °C), (2) ice/water phase change (∼−4 °C to −4 °C), and (3) chemical phase change (∼−4.5 °C to −5.5 °C). While the resistivity is highly dependent on changes in temperature, a drastic increase in resistivity is observed during freezing. Additionally, a comparison of specimens above and below the critical DOS (i.e., the DOS required for damage to occur) shows that resistivity measurements may able to be used to quantify damage.

1. Introduction

The electrical testing of cementitious materials is being increasingly performed to assess the quality of concrete. Since its original use by Shimizu in the 1920s [1], many researchers have sought to use electrical measurements to describe hydration [2–6], transport properties [7,8], or cracking [9–15]. Electrical testing has many advantages due to the low cost of testing equipment and short time duration that is needed to perform the test. However, the testing geometry [16,17], the testing temperature [17–20], the degree of saturation of the sample [17,18,21,22], and the pore solution concentration/leaching [17,18,23] must be considered in order to properly interpret the results. While the influence of many of these variables can be minimized through standardization, it is crucial that they are properly considered for.

Many times electrical properties are used to measure the transport properties of concrete, such as the Rapid Chloride Penetrability Test [24]. While several researchers have done excellent works describing changes in the electrical response that occur due to heating [19,25,26] or environmental temperature fluctuations [18,27–29], a relatively small number of researchers have used electrical properties to study freezing concrete [30]. Olson et al. [31] used freezing to develop a measure of conduction through the capillary and gel pores for to better understand the structure of cement paste. Sato and Beaudoin [32] examined the freezing behavior of a Wollastonite micro-fiber reinforced cement paste by using an AC impedance spectroscopy.

Previous freeze–thaw studies [33,34] have shown that the level of damage that develops depends on the degree of saturation (the volume ratio of fluid in the sample as compared to the total volume of fluid that the sample can hold at 7 torr) in a concrete element. It has been suggested that there is a critical degree of saturation (between 80% and 91%) above which freeze–thaw damage can begin to initiate. Fig. 1a [33] illustrates that as the degree of saturation decreases, the level of damage that develops also reduces until the critical degree of saturation is reached. For samples in which the degree of saturation is below the critical degree of saturation, freeze–thaw damage is expected to not occur.

Deicing chemicals can also increase the freeze–thaw damage in concrete by resulting in additional distress due to the osmotic pressure and/or the crystallization pressure [35–39]. Previous studies [40–43] identified that chemical reactions between the cementitious matrix and salt solution can result in a chemical phase change that can cause severe damage in cementitious materials. Fig. 1b illustrates the phase diagram for a NaCl–H_2O solution. Fig. 1b shows that in a cementitious systems exposed to NaCl, a chemical phase change can occur (as indicated by a dashed line) which was observed at a temperature range between −6 °C and 8 °C due to

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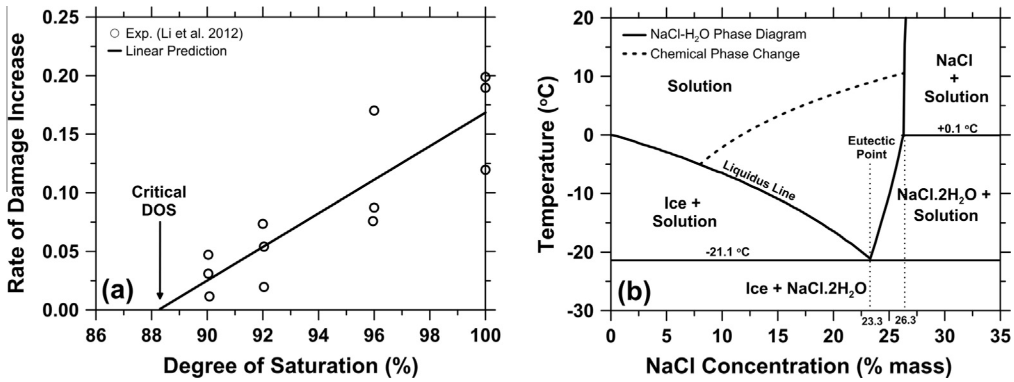

Fig. 1. (a) Rate of decrease of relative dynamic elastic modulus (freeze–thaw damage) as a function of the degree of saturation [33] and (b) comparison of NaCl–H_2O phase diagram with chemical phase transition in NaCl–H_2O–cement system [42].

the presence of aluminate phases in cement. This chemical phase transition was detected using thermal measurement by observing the heat released during phase transition and the damage was assessed using acoustic emission technique [41,42].

2. Research objective

Previous research by Olson et al. [31] examined how the electrical response of a cement paste changed during freezing. This paper builds on that work by examining the electrical response of cementitious mortar with varying degrees of saturation and varying NaCl deicing salt concentrations. The main objective of this paper is to determine whether the electrical resistivity measurement can be used to detect phase changes and to evaluate the damage in mortar samples during freezing and thawing. The effect of the degree of saturation and NaCl deicing salt concentration on the electrical response of the mortar are also studied.

3. Experimental procedures

This paper describes experiments performed on two series of mortars that investigated: (1) the role of the degree of saturation (DOS) and (2) the role of deicing salt solutions. The following section describes the sample preparation, sample conditioning, and basic material properties for the two testing series. The second section describes the experimental testing program including the freeze–thaw device (i.e., the longitudinal guarded comparative calorimeter) and the four measurements made on the mortar for all freeze–thaw tests: (1) electrical impedance spectroscopy during freezing and thawing, (2) passive acoustic emission (AE) during freezing and thawing, (3) ultrasonic pulse velocity before and after test, and (4) thermal analysis.

3.1. Sample preparation, conditioning, and properties

All tests were performed on a mortar mixture with a water-to-cement ratio (w/c) of 0.42 by mass and a sand volume fraction of 55%. The mortar contained ordinary Type I Portland cement (OPC) with a fineness of 375 m^2/kg (C_3S = 60%, C_2S = 10%, C_3A = 9%, C_4AF = 10%, and Na_2O (Equiv) = 0.86%). The aggregate was natural sand with a maximum size of 4.75 mm, specific gravity of 2.61, fineness modulus of 2.89, and an absorption value of 2.2% by mass. ASTM C305-12 [44] was followed during the preparation of the samples in a standard mortar mixer. The mortar samples with dimensions of 25.4 mm × 25.4 mm × 300 mm (1 in × 1 in × 11.81 in) were cast, vibrated, screeded and sealed to cure for 28 d. Following curing, the mortar was cut to 25.4 mm × 2 5.4 mm × 50.8 mm (1 in × 1 in × 2 in) samples using a wet saw. Samples were then placed in a vacuum oven at 65 °C ± 1 °C and a pressure of 20 mmHg ± 5 mmHg for 7 d to remove their moisture. Upon testing, the samples were individually equipped with wires for EIS testing. The wires were affixed to the top and bottom surfaces of the sample when positioned with the longest side vertical, as they appear in the test setup (Fig. 2a). A conductive liquid nickel coating was used to hold each separated wire strand on the cross section surfaces and it was assured that the conductive coating covered the entire top and bottom surfaces.

The sample with attached wires was then placed in a desiccator using two small spacers underneath each sample to provide a small gap between the bottom of the container and the lower surface of the sample (Fig. 2b). The sample was then evacuated to a pressure of 10 mmHg ± 5 mmHg for 3 h. While still under vacuum, de-aerated solution (de-aerated by vacuuming the solution for 15 min) was added to the container until the sample was completely submerged and the sample remained submerged in the designated solution under vacuum for 1 h. Afterwards, the submerged sample in the solution was transferred to a 23 °C ± 0.5 °C chamber and remained in the chamber before testing, approximately 3 d. This condition was considered to be 100% degree of saturation. The deionized (DI) water and NaCl solution (5%, 15%, or 23.3% salt concentration by mass) was used to saturate the mortar samples.

The samples intended for testing at lower degrees of saturation were submerged for 3 d and were then removed from the solution. The samples were allowed to dry and the mass was monitored every 15–30 min until the desired mass (i.e., degree of saturation) was obtained. Samples were then sealed in a double sealed bag for a week before freeze–thaw testing to assure uniform distribution of moisture throughout the sample. Samples with various degrees of saturation (75%, 85%, 95%, and 100%) were prepared. The degree of saturation was determined using the ratio of the volume of absorbed water to the total volume of water that can be absorbed by the sample.

After conditioning and before the freeze–thaw experiment, all sides of the sample not covered in nickel coating were wiped dry and wrapped in a thin plastic layer to prevent moisture exchange with their surrounding environment. A small circular hole was made in one side of the plastic to attach the AE sensor on the surface of the sample to monitor AE activity during freezing and thawing (Fig. 2c).

Since the conductive coating created uneven top and bottom surfaces, separate samples without coating were prepared for every sample conditioning to perform ultrasonic pulse velocity measurement through the length of the mortar sample before and after freeze–thaw test.