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Electrical Conductivity Testing

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Electrical Conductivity Testing

A prequalification and quality assurance tool

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Performance-based durability criteria are becoming more prevalent in specifications for concrete structures. Although several standardized and nonstandardized methods are currently used as index tests, or indicators of potential durability, most require a significant lag time between placing the concrete and obtaining the test results. With the goal of providing the concrete industry with more rapid and easy to use index tests, numerous electrical methods have been developed. Some of these test methods have been standardized,[1,2] and maximum values for these test results are commonly stipulated in performance specifications.

Electrochemical methods, including measurements of conductivity and its inverse, resistivity, have been proposed as methods for assessment of transport properties as well as changes in the pore solution and microstructure in cement-based materials.[3-5] In addition to being rapid, these methods allow testing of the same specimen over time without disturbing its structure. Standardized test procedures, however, need to be developed so that researchers can more readily compare test results and practitioners can adopt them in their specifications. This article describes research carried out to develop a test method for prequalification and quality control of concrete based on electrical conductivity.

BACKGROUND

To better understand how conductivity, a measure of the ability of electrons to be transmitted through a material, can be used to quantify permeability of concrete, some background is necessary. The material we generically refer to as hardened concrete comprises a paste of cement hydrates and discrete aggregate particles. Although cement hydrates are solids, their structures are porous, and the pores can be (at least partially) filled with water that contains ions (pore solution). This pore solution is orders of magnitude more conductive than the solid phases of cement and aggregates in the concrete, so the conductivity of the bulk material (be it paste, mortar, or concrete) is a function of the pore solution composition, the volume and connectivity of the pore system, and the degree of saturation of the bulk material.[6,7]

In general, two competing effects occur as the concrete ages: 1) the conductivity of the pore solution increases—producing an increase in bulk conductivity, and 2) the connectivity and volume of the pores decreases—producing a decrease in bulk conductivity (of course, in the field, the degree of saturation also changes, but we will focus the rest of this discussion on fully-saturated specimens in the laboratory). Due to the dissolution of calcium and alkali ions from the cement as it reacts with water, the pore solution conductivity generally increases over time (with the exception of some mixtures with certain mineral and chemical admixtures). The water originally between the cement particles gradually becomes a highly conductive pore fluid, and after about 24 hours, sodium, potassium, and hydroxyl ions are the dominant species found in the pore solution.[8] Although pore solution chemistry plays a role in the overall or bulk conductivity, obtaining pore solution from cement pastes older than about 6 hours requires high-pressure presses.[9] This limits characterization of pore solution composition to specialized laboratories or to the use of values reported in published literature.

With the exception of mixtures containing silica fume and cases where alkali-silica reaction or some form of external ion penetration occurs, the ionic concentration in the pore solution approaches an asymptotic value after the first few days.[8] Because silica fume reacts with the hydroxides in the pore solution to form secondary hydrates, it decreases bulk conductivity in two ways. First, by reacting with the hydroxides, the ionic concentration of the pore solution decreases; and second, the secondary hydrates formed decrease the volume and connectivity of the pore system.

The presence of chemical and mineral admixtures also influences both pore solution and bulk conductivity. The use of accelerating, retarding, or water-reducing admixtures alters the rate of reaction of cement and the resulting pore structure. Increased resistivity associated with water-reducing admixtures is attributed to improved microstructure, as evidenced by decreased pore volume and size and improved compressive strength.

As cement hydrates, the volume and size of the pores decrease, reducing the volume fraction of the conductive path. Changes in electrical properties of the bulk concrete can therefore indicate the formation of discontinuous pore structure and hence the resultant high penetration resistance.

Only those material properties with a firm basis in materials science give a realistic representation of the nature and behavior of a porous solid such as concrete. As a material property, conductivity can be related to other properties of concrete. For example, conductivity can be related to diffusivity, the rate at which the ions are transported through the concrete, using the Nernst-Einstein equation

$$\sigma_o/\sigma = D_o/D \qquad (1)$$

where σ_o/σ is the ratio of pore solution to bulk conductivity, D_o is the diffusion of ions in the solution (m^2/s), and D is the diffusion of ions in the specimen (m^2/s). Although determining the ionic diffusion coefficient (D in Eq. (1)) through concrete can involve weeks of testing, the relationship in Eq. (1) can be used to rapidly estimate the diffusivity of concrete specimens by measuring the electrical conductivity (σ in Eq. (1)), or resistivity, of the bulk specimen. This is a fairly easily and rapidly measured material property. The diffusion of dilute concentrations of ions in solution and the conductivity of solutions (D_o and σ_o in Eq. (1), respectively) can be readily found in handbooks; however, handbook values reported for the diffusion of ions in solution are typically at infinite dilution, which is not the same as that for the ionic concentrations generally used in diffusion experiments.[10] In addition, the ion under consideration will interact with the pore solution and any other ions in solution. It must be noted, however, that additional difficulty in applying the Nernst-Einstein equation arises from the determination of the pore solution conductivity.

Conductivity and resistivity can be measured using a number of techniques, but the most widespread involve placing a sample between two electrodes. The electrodes may contact the sample directly or through electrolytic contact as in the ASTM C 1202[1] test that is perhaps the most common standard test method using this technique. ASTM C 1202 is often termed the rapid chloride permeability test, as it was called in the original AASHTO T 277[2] version. Although the test method implies a relationship to both chloride movement and permeability in concrete, it actually measures neither. The test determines the total electrical charge passed through a sample placed between conductive solutions of sodium chloride (NaCl) and sodium hydroxide (NaOH) over a period of 6 hours at an applied DC voltage of 60 V. The results are the integral of, or area under, the current versus time curve expressed in coulombs, which have base units of current (in amperes) multiplied by time (in seconds). Therefore, total charge passed is not a fundamental material property. The test measures the electrical conduction of all ions, not just chloride ions and has been criticized for not being indicative of chloride penetration.[4,11] In general, for concrete with low water-cementitious material ratio (*w/cm*) or moderate *w/cm* with the addition of supplementary cementitious materials, the total charge passed correlates well with chloride diffusion (or permeability) coefficients.[12-14] As *w/cm* increases, however, heat is generated by ion-ion and ion-solid collisions. The resulting increase in temperature increases the conductivity of the pore solution (and therefore the current flow) and may also change the microstructure, producing erroneously high coulomb values.

A proposed ASTM standard, "Indication of Concrete's Ionic Conductivity," has been put forward to counteract criticism regarding the heating effects. The test uses the same apparatus as ASTM C 1202, but measures the current passed through the sample after 1 minute under an applied DC voltage of 60 V. This conductivity method, although more awkward than some, is based in materials science and can therefore be used with confidence in durability prediction models, where values determined using the ASTM C 1202 method cannot.

In some limited cases, the material property of conductivity can be related to the results of ASTM C 1202 tests. A linear relationship between initial current and total coulombs has been observed[15] and found to be valid at values less than 3000 coulombs.[16] At greater values, heating effects caused significant deviation from the relationship. A modification that involves multiplying the coulombs during the first 30 minutes of the test by 12 has been proposed as a way to obtain a value equivalent to a 6-hour ASTM C 1202 test, but is less affected by heating.[12] Given that performance-based specifications typically require low coulomb values, where the linear relationship with initial current is still valid, the initial measured current would seem a useful and more rapid indication of durability that could be used to directly and accurately determine the specimen conductivity. When the concrete contains calcium nitrite corrosion inhibitors, however, the initial current results must be interpreted with care.

The use of measured current has the potential to be used as a prequalification or quality control tool. Changes in conductivity over time can be used to determine the time required to meet specified durability levels that are defined by fluid penetration resistance, as opposed to those that are defined by chemical resistance such as in sulfate exposures. The following sections describe an application where the material property of conductivity can be used as early as 1 day after casting.

MATERIALS AND PROCEDURES

The six concrete mixtures shown in Table 1 were investigated in the research program. The mixtures were selected to characterize a wide array of concrete mixtures ranging from high-performance concrete (HPC) to that approaching what might be used in a residential application. The high-performance mixture (0.31 HPC) had a *w/cm* of 0.31, was steam cured, and had 27% of the silica fume blended cement used in the mixture replaced with slag cement. The residential mixture (0.69 PC) had a high *w/cm* of 0.69 and used only portland cement. The other four mixtures had a *w/cm* of 0.40 and contained regular portland cement (0.40 PC), blended silica fume cement (0.40 SF), or portland cement combined with Class C fly ash (0.40 FA) or slag cement (0.40 SG). Details of the materials, mixture proportions, and casting procedures are published elsewhere.[17]

The concrete was allowed to cure in 100 x 200 mm (4 x 8 in.) cylinder molds for 18 to 24 hours and was then cut into 50 mm (2 in.) slices. Two specimens for each concrete mixture were vacuum saturated under tap water for 3 hours. With the specimens in a saturated, surface-dry condition, they were then tightly wrapped with vinyl electrical tape. The samples were kept in the ASTM C 1202 test apparatus for the first 7 days and then stored in lime-saturated water between subsequent weekly measurements. The same two specimens continued to be tested at all ages.

Conductivity was measured using equipment normally used in ASTM C 1202 tests, but several deviations from the standard test method were employed for this application. Due to high conductivity at early ages and the maximum allowable current of 500 mA for the equipment used, 30 V was applied rather than the usual 60 V. A 0.3 N sodium hydroxide solution was used in both chambers of the test cell as shown in Fig. 1. This solution was selected to approximate a typical pore solution and minimize leaching from the specimens. The standard ASTM C 1202 method uses one chamber filled with sodium chloride solution that would change the conductivity of the specimen over time due to chloride penetration combined with the differences in conductivity between chloride and hydroxyl ions. By placing sodium hydroxide in both chambers, the measured changes in conductivity would be primarily caused by changes in the pore structure, and thus the permeability of the concrete, rather than changes in the pore solution.

An automated technique was developed to collect data at 3-hour intervals for the first week and then at weekly intervals until the specimens reached an age of 28 days. At each testing interval, 30 V was applied across the specimen for 15 minutes prior to recording the current measurement used to calculate conductivity. The automated technique allowed the user to select the duration of application of voltage for each test, the time interval between cycles (3 hours minus 15 minutes in this case), the number of cycles, and the number of specimens to be tested (one to eight).

This research was carried out prior to the development of a recently proposed ASTM conductivity method that uses 60 V for a 1 minute exposure. As mentioned previously, however, use of the standard 60 V was not suitable for the specimens and the equipment used. Measurements taken at 5 and 15 minutes were not significantly different. Therefore, it would be expected that the same results would have been measured at 1 minute. In these authors' opinions, the 1 minute proposed in the ASTM standard is appropriate. The temperature rise of the solution after 15 minutes was not significant (≤ 1 °C [1.8 °F]), even for concretes at an early age.

These tests were carried out at a temperature of 23 °C (73 °F), but tests could be carried out at other temperatures to investigate the sensitivity of penetration resistance development to temperature. The authors are currently initiating an investigation of the effects of curing temperature on early-age conductivity results.

RESULTS AND DISCUSSION

The term bulk conductivity is used to clarify that the current flows through the composite concrete sample (the solid material and the pore solution). Bulk conductivity, in S/cm, was calculated using the formula

$$\text{bulk conductivity} = \frac{I \cdot L}{V \cdot A} \qquad (2)$$

TABLE 1:
Concrete mixture proportions

Mixture	Water, kg/m³ (lb/yd³)	Cement, kg/m³ (lb/yd³)	Slag, kg/m³ (lb/yd³)	Fly ash, kg/m³ (lb/yd³)	Silica fume, kg/m³ (lb/yd³)	Fine aggregate, kg/m³ (lb/yd³)	Coarse aggregate, kg/m³ (lb/yd³)
0.31 HPC	130 (219)	305 (514)	115 (194)	—	*	872 (1470)	1042 (1756)
0.40 PC	150 (253)	375 (632)	—	—	—	787 (1327)	1100 (1854)
0.40 SG	150 (253)	244 (411)	131 (221)	—	—	782 (1318)	1025 (1728)
0.40 FA	150 (253)	300 (506)	—	75 (126)	—	767 (1293)	1025 (1728)
0.40 SF	150 (253)	375 (632)	—	—	*	784 (1321)	1100 (1854)
0.69 PC	200 (337)	290 (489)	—	—	—	1205 (2031)	684 (1153)

*CSA Type 10SF blended cement with approximately 7% silica fume

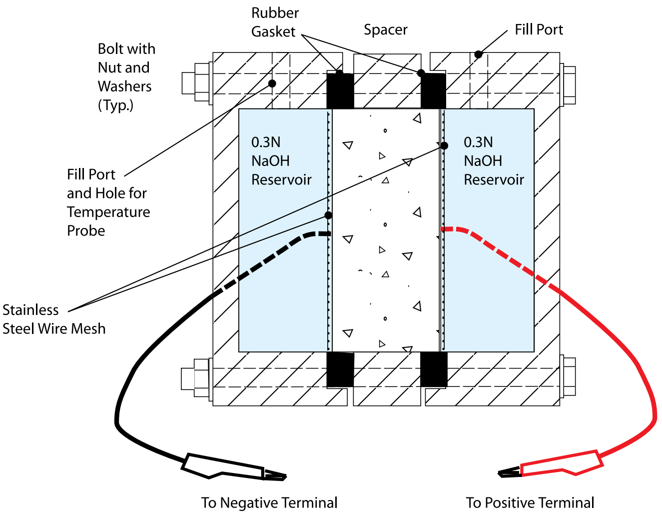

Fig. 1: Schematic of conductivity testing apparatus

where I is the current measured 15 minutes after the voltage is applied (amps), L is the thickness of the specimen (cm), V is the applied voltage (30 V), and A is the cross-sectional area of the specimen (cm^2). The measured conductivities (average of two replicates) of the six concrete mixtures for the first 7 days are shown in Fig. 2. There was less than a 10% difference in measured current between replicate samples. Tabulated results for these conductivity tests were given in previous papers.[19,20] The influences of *w/cm* and supplementary cementitious materials can be seen in the results. Although the 0.40 PC mixture initially had a lower conductivity than all other mixtures, those containing supplementary cementitious materials developed lower conductivities by the time the specimens reached 28 days of age (Fig. 3). This shows that it's not only *w/cm* that controls the rate of fluid transport. From the conductivity results for the first 28 days shown in Fig. 3, it can be seen that most of the decrease in conductivity occurred during the first week after casting.

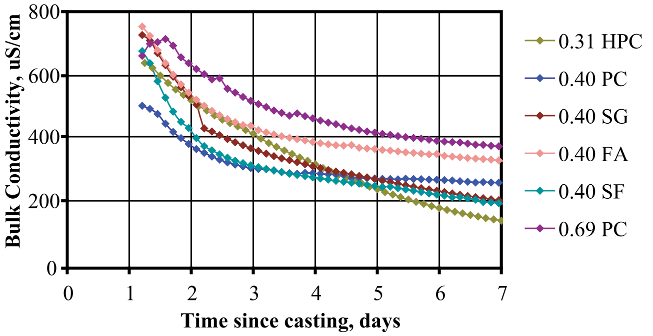

Fig. 2: Bulk conductivity (at 3-hour intervals) versus time after casting to 7 days

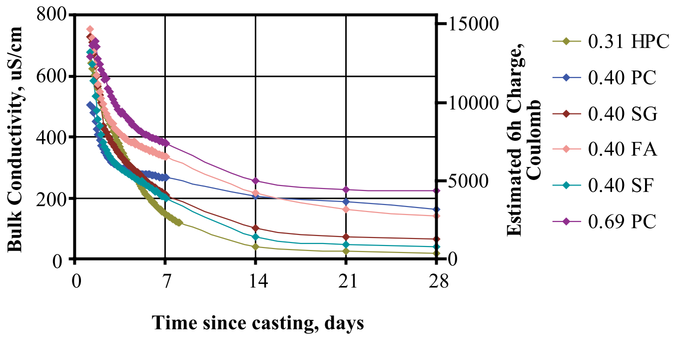

Fig. 3: Bulk conductivity versus time after casting to 28 days

The conductivity results can be used to estimate the total coulombs passed in the standard 6-hour ASTM C 1202 test. To estimate total coulombs, the current measured during the test at 28 days of age is assumed to remain constant and multiplied by the test time (21,600 seconds) and the ratio of the standard test voltage to the applied voltage (60 V/30 V). This value is then adjusted for the difference in diameter by multiplying by the ratio of the area of the standard specimen to the actual specimen. The estimated total coulombs and the actual measured coulombs determined from standard 6-hour tests on specimens from the same concrete mixture are shown in Table 2 (each value is an average of two specimens). Generally, as the total coulombs increase, the difference between the estimated and measured values increase, likely indicating the effect of heating during the standard tests. Except for one case, the variation between the estimated and actual coulombs was within the precision of the standard (42% for an average of three samples). Had the same specimen been used for both tests, it would be expected that the results would have been closer. When permeability is a concern, concrete specifications often require maximum allowable total coulombs in the range of 1000 to 1500. Only three of the concrete mixtures investigated attained values lower than 1500 coulombs at 28 days of age.

PROPOSED APPLICATIONS

The proposed electrical conductivity method provides several benefits over the traditional ASTM C 1202 test. First, the short duration of exposure to applied voltage does not appreciably heat the sample, avoiding erroneously high measured conductivity results. Second, and perhaps more importantly, the method can be used for concrete mixture prequalification and quality assurance. As mentioned previously, the test is nondestructive in the sense that the same specimens can be continuously or intermittently tested over time. The conductivity values presented here were found to correlate to saturated water permeability and other properties.[17,18] Concrete producers can test several possible mixtures to determine the optimal proportions to meet durability specifications by a certain age, as well as providing an estimate of the length of curing required by the contractor to achieve the potential of the mixtures. As most of the changes occur over the first 7 to 14 days after casting, concrete producers can readily determine at that stage if a proposed concrete mixture has the potential to meet a "permeability" specification.

The method could also be used for quality control purposes. Typically, the standard ASTM C 1202 test is carried out at 28 days, and in cases where significant quantities of supplementary cementitious materials are used, it may be delayed until 56 days (the Canadian CSA A23.1 standard[19] allows 56 days to achieve specified coulomb limits). A standard conductivity versus time curve for a specific mixture could be constructed at the project outset. Rather than testing the concrete at 28 or 56 days using the ASTM C 1202 test, results obtained at earlier times (such as 3 or 7 days) could be compared with the standard curve to determine acceptance or rejection of the concrete. This method would result in significantly earlier conclusions regarding the need for changes to mixture proportions and even curing procedures if the tests were performed on field cores or samples that matched lab curing to field temperatures. Contract bonuses or penalties based on adherence to end result specifications, such as those used by several U.S. and Canadian highway agencies, could potentially be awarded earlier.

MANY ADVANTAGES

The test method presented in this article is based on electrical conductivity of concrete, is inherently nondestructive, and can be carried out using ASTM C 1202 equipment currently available in many North American concrete labs with minor modifications. The method can be automated to measure the changes in conductivity of concrete over time during the first 28 days. These results can then be used for mixture prequalification, quality control, and potentially for the determination of required curing time and temperature. Because the method is based on materials science, it forms a firm basis for use in specifications and predictive models for durability. Comparison of measured conductivity development using cores taken from in-place concrete to that of cylinders taken from standard mixtures can also enable earlier determination of specification compliance.

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TABLE 2:
ESTIMATED AND MEASURED TOTAL COULOMBS AT 28 DAYS

	0.31 HPC	0.40 PC	0.40 SG	0.40 FA	0.40 SF	0.69 PC
Estimated coulombs	349	2942	971	2335	526	4864
Measured coulombs	352	4512	978	3419	846	8594
Percent difference	1	35	1	32	38	43

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