# MEASURING THE INFLUENCE OF TEMPERATURE ON ELECTRICAL PROPERTIES OF CONCRETE

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**Abstract**
Electrical measurements are becoming a popular method to examine transport properties of concrete due to their ability to be performed rapidly and their ease of use. Previous work has shown that specimen geometry, temperature, and storage conditions influence electrical readings. As such, caution must be taken when correlating such readings with transport properties. It is important to correct these factors so that measurements more accurately describe the material. The work presented in this paper looks specifically at the role temperature of the specimen play in resistivity measurements. This is done by cycling samples through a temperature range and developing corrections characterizing their behavior. A normalized formation factor is proposed to describe transport through the microstructure. This allows for the determination of a transport property that can be used for service life prediction. The work is currently being developed as a part of a nation-wide project aimed at investigating the possibility of moving towards performance based specifications.

## 1. Introduction

Measurement of the electrical properties of concrete is a practical, rapid, nondestructive method used to study transport properties in concrete. Prior research has shown that measurements of resistivity are affected by factors including pore solution [1]–[3], testing temperature [4]–[7], conditioning [3], [6], [8], and degree of saturation [9]–[12]. Resistivity measurements can be used to determine the formation factor, which is a numerical parameter that describes the volumes and connectivity of the pore network [13]–[16] and has been related to ionic transport properties [10], [11], [17].
In general, resistivity measurements of porous material can be described in Equation 1:

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$$\rho = \rho_0 \cdot \frac{1}{\phi\beta} = \rho_o \cdot F \tag{1}$$

where $\rho$ is the total bulk resistivity, $\rho_0$ is the resistivity of pore solution, $\phi$ is the fluid filled porosity, $\beta$ is the connectivity of the pores, and F is the formation factor. The formation factor can be calculated by dividing the bulk resistivity by the resistivity of the pore solution [11].

The measured resistivity, discussed in the next section, is highly dependent on testing temperature, e.g. [4], [6], [18], [19]. This work will discuss an activation energy based correction for resistivity measurements, utilizing a tabletop temperature cycling system [18]. This is an improvement over previous methodologies, as it allows the operator to start and finish the test with the value needed for a correction [20]. This work is conducted as part of a multi-state study, and the data gathered here will be used to inform the development of a standard test method [21].

## 2. Background

Previous literature has shown that an activation energy based relationship can be developed to account for the influence of temperature on the measured resistivity, e.g. [4], [22], shown in Equation 2.

$$\frac{\rho_{T_{ref}}}{\rho} = \exp\left[\frac{-E_{a-cond}}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)\right] \tag{2}$$

Where $\rho_{T_{ref}}$ is the resistivity at temperature $T_{ref}$, $\rho$ is the measured resistivity at temperature T, $E_{a-cond}$ is the activation energy of conduction, R is the universal gas constant, T is the temperature of the measured sample in K, and $T_{ref}$ is the reference temperature, typically 23 °C = 296 K. It should be noted that the activation energy of conduction represents only the influence of the temperature at the time of test and as such, it differs from the activation energy of hydration which describes the role of temperature on the hydration process [6].

Previous research evaluated the $E_{a-cond}$ for a variety of different ages, and found that after approximately 7 days a sufficient extent of hydration has occurred and the value does not change appreciably [8]. This study will evaluate the specimens at an age of 180 days.

## 3. Testing Procedure

The specimens used in this study were standard 4" x 8" (~102 x 204 mm) concrete cylinders made from various pavement and bridge deck mixtures from across the United States. Mixtures in this study include various straight cement, binary, and ternary mixtures, and unless otherwise noted cementitious materials were each batched separately. The water-to-cementitious ratio (w/cm) and cementitious materials content are summarized in Table 1 and unless noted cement and supplementary materials were batched separately.

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Table 1: Summary of the paste portion of the mixture designs, with Mixture ID noting the cement, fly ash, slag, and ternary mixtures.

| Mixture ID | w/cm | Water (kg/m$^3$) | Cement (kg/m$^3$) | Fly Ash (kg/m$^3$) | Slag (kg/m$^3$) |
|---|---|---|---|---|---|
| C-1 | 0.42 | 141 | 335 | - | - |
| C-2 | 0.41 | 185 | 390 | - | - |
| FA-1 | 0.40 | 135 | 335* | - | - |
| FA-2 | 0.37 | 154 | 332 | 82 | - |
| FA-3 | 0.42 | 144 | 273 | 68 | - |
| FA-4 | 0.39 | 122 | 248 | 62 | - |
| FA-5 | 0.45 | 143 | 279 | 43 | - |
| S-1 | 0.43 | 141 | 247 | - | 82 |
| Ter-1 | 0.40 | 125 | 262[†] | 66 | - |

\*     denotes an ASTM C595 IP(25) cement [23]
†     denotes an ASTM C595 IS(20) cement [23]

As part of the multi-laboratory study, the specimens were prepared at different laboratories and shipped to the testing lab at Oregon State University. Specimens were sealed and cured in a sealed condition by placing the concrete into double 6 mil (~0.15mm) thickness plastic bags until the beginning of the test. The testing for temperature correction discussed here was conducted at an age of 180 d.

The procedure described here continuously measures the resistivity of the sample while the temperature of specimen is varied from 5 °C to 30 °C. A diagram of the testing apparatus can be seen in Figure 1, which is a similar setup as that used by [18]. This setup has the ability to use a full standard concrete cylinder.
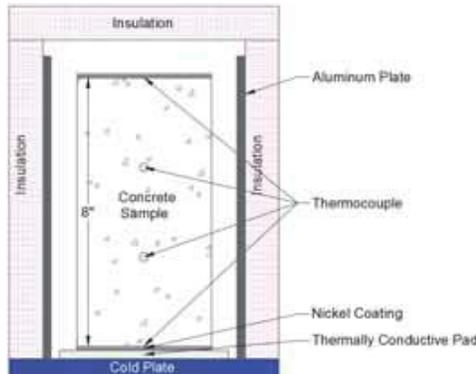


Figure 1: Diagram of Testing Apparatus, not shown is the 18 gauge wire used to connect the nickel coating electrode to the resistivity meter and the plastic wrap placed on the concrete cylinder to prevent excessive drying during the testing period.

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A 4" x 8" (102 mm x 204 mm) sample was prepared by applying a conductive nickel coating at the top and bottom of the sample. Surfaces were scarified with a 20 grit concrete sanding block to ensure proper adhesion between the concrete surfaces and the coating. This nickel coating forms the electrodes as typically seen in uniaxial concrete measurements. The nickel coating electrodes are connected to the resistivity meter using 18 gauge copper wire. The top and bottom surfaces are then covered with aluminum tape to ensure sufficient contact with the wire and electrode as well as to prevent moisture loss from evaporation. Additionally, thin plastic is wrapped around the exposed sides to prevent moisture loss as the test is performed. Type T thermocouples with an accuracy of ±0.713 °C [24] were placed at the bottom, top and middle thirds of the sample to continuously monitor measurements of the temperature.

The temperature of the sample is controlled by a thermoelectric cold plate. A thermally conductive pad (ThermaCool TC3008™) is placed in-between the cold plate and the sample to ensure appropriate thermal connection between the plate and the sample as well as to protect the cold plate from abrasion. The sample is isolated from the exterior environment through the use of aluminum shrouding which is connected to the cold plate. The aluminum shrouding is surrounded by R-10 insulating foam board. This promotes unidirectional cooling and heating. A preliminary test was performed where a hole was drilled and a thermocouple was place inside. It showed that the temperature inside the sample and the temperature at the surface were nearly identical.

Two types of resistivity meters were used to track the resistivity of the specimen. The Giatec RCON2™ applies an AC current at a frequency of 1000 Hz and measures total impedance and phase angle. The Proceq Resipod Resistivity Meter, set in its uniaxial configuration, also applies an AC current but with a frequency of 40 Hz and measures total impedance. Once the testing apparatus is prepared, the data acquisition software measures resistance and temperature at a frequency of 0.1 Hz. Initially, the cold plate is set to allow the sample to equilibrate to 5°C. The sample temperature is then raised at a set rate of 0.6 °C / h.

The average specimen temperature was determined as the average of the top three thermocouples, as the temperature measured by the bottom thermocouple is more representative of the cold plate temperature. The heating rate was chosen to minimize thermal gradients in the specimen. It should be noted that during the test the testing device stays in a temperature controlled environment at 23°C.

Resistivity of the specimen is determined by using the real component of impedance, determined by multiplying the cosine of the phase angle and the measured total impedance, and then multiplied by the geometry factor. The geometry factor for the uniaxial test used in this study is simply the ratio of cross-sectional area to length. [6]

## 4. Results and Discussion

The activation energy of conduction, $E_{a-cond}$, is a single parameter that can be used in an exponential, Arrhenius-type correction for resistivity measurements. The $E_{a-cond}$ can be calculated from the slope of the best fit line for a figure of the natural log of resistivity versus the inverse of the absolute temperature, as shown in Figure 2. The slope of the best fit line is then multiplied by the universal gas constant (R=8.314 J/ (K·mol)) to determine $E_{a-cond}$.

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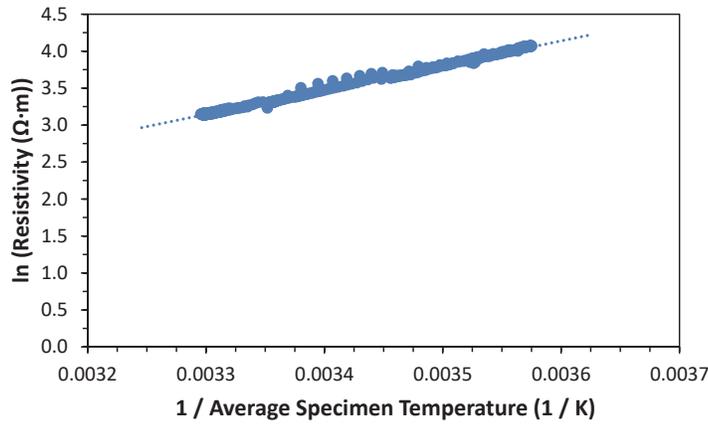
Figure 2: Arrhenius type plot of natural logarithm of resistivity vs. inverse of absolute temperature, the slope of the best fit line used to calculate the activation energy of conduction.

Using the measured resistivity at 23 °C and the $E_{a-cond}$ determined as described above, the resistivity of the concrete sample at any temperature can be estimated. An example is shown in Figure 3 for mixture FA-5, with the other mixtures showing similar results. It can be observed that the correction of the temperature following the activation energy concept provides data that is similar to that which was experimentally measured.
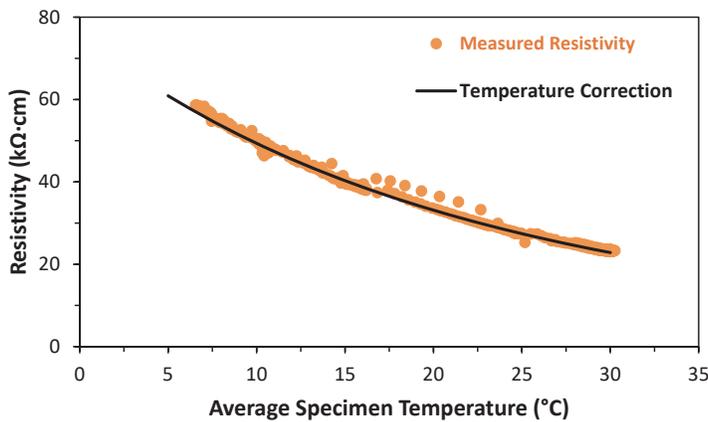


Figure 3: Typical experimental results showing resistivity and average specimen temperature, results for mixture FA-5.

Values for $E_{a-cond}$ were determined for all of the mixtures in this study, and the results are shown in Table 2. Results indicate an average value of 27.4 kJ/mol.

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Table 2: Activation energies of conduction for each of the mixtures in this study.

| Mixture ID | $E_{a-cond}$ (kJ/mol) |
|---|---|
| C-1 | 25.1 |
| C-2 | 25.4 |
| FA-1 | 32.2 |
| FA-2 | 25.9 |
| FA-3 | 31.4 |
| FA-4 | 25.9 |
| FA-5 | 27.5 |
| S-1 | 28.8 |
| Ter-1 | 24.8 |
| AVG | 27.4 |
| SD | 2.8 |

As most of the conduction occurs through the pore solution, a relationship was investigated between $E_{a-cond}$ and the pore solution. The pore solution properties, specifically the ionic strength, was estimated based upon the mixture designs and alkali contents of the cementitious materials, using the pore solution calculator developed by NIST, available at http://concrete.nist.gov/poresolncalc.html [25]. Figure 4 shows that a higher pore solution ionic strength, which corresponds to a higher concentration pore solution or a lower pore solution resistivity, in general shows a lower activation energy. The relationship of higher ionic strength to lower activation energy of conduction has been reported previously for extracted pore solutions as well [26]. A linear fit is shown to help guide the eye, as well as symbols for the different types of mixtures. With a larger data set, this correlation could potentially help to estimate $E_{a-cond}$ based upon the pore solution properties.
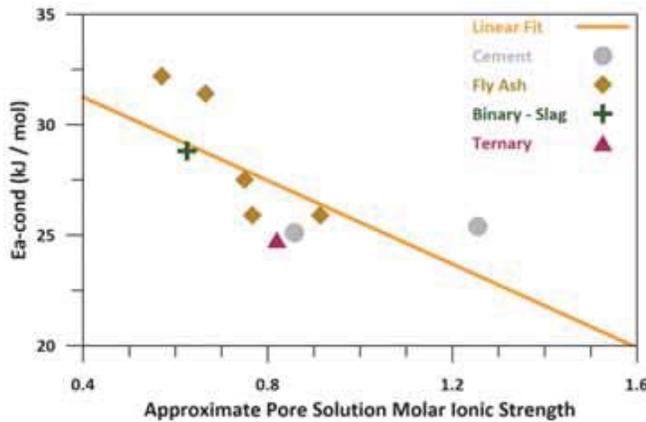


Figure 4: Activation energy of conduction versus pore solution molar strength shows the general trend that mixtures with high concentration pore solutions showed a lower temperature sensitvity, i.e. a lower $E_{a-cond}$.

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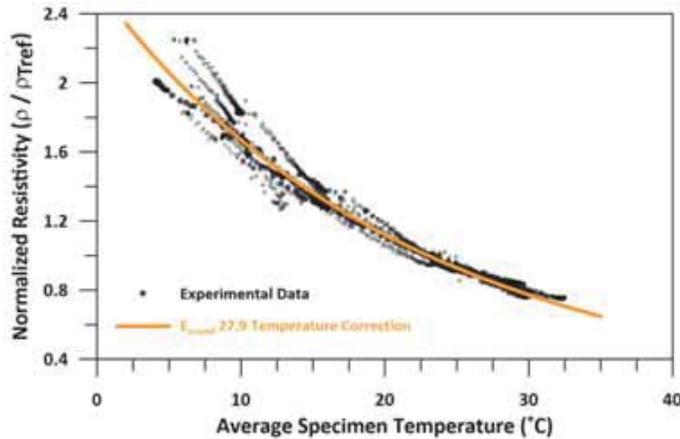22-24 August 2016, Technical University of Denmark, Lyngby, Denmark

Figure 5: Experimental results showing resistivity versus average specimen temperature for the mixtures in this study, overlaid with the temperature correction fit based on the average activation energy of conduction.

One objective of this work is to evaluate the possibly of a single value of $E_{a-cond}$ being used as a temperature correction for a wide range of mixture compositions. While that evaluation is still being conducted, Figure 5 shows data from each of the mixtures used in this study. It can be seen that when the normalized resistivity, $\rho/\rho_{T_{ref}}$, is plotted against temperature the Arrhenius function captures the general trend of the data. The data points are overlaid with a temperature correction based upon the average $E_{a-cond}$ of 27.9. A reasonable agreement is noted, suggesting that a single value of $E_{a-cond}$ could be used for sealed samples using the standard deviation noted in Table 2. Future work may however show estimating $E_{a-cond}$ based on pore solution concentration to be more suitable.

### 5. Ongoing Work

Additional testing will be performed on these samples to determine the role that degree of saturation has in terms of activation energy. For this testing, specimens will be saturated using a 7 torr vacuum level [27], and the $E_{a-cond}$ will be evaluated as the specimen loses moisture. Preliminary analysis of the C-1 samples shows that activation energy can change by approximately 20 % as the specimen moves from a sealed state to a saturated state. This is expected as although the pore solution may become more diluted, the effect of filling in the pores will increase the conductive volume and connectivity enough that resistivity measurements are less affected by temperature. This behavior has been noted by previous researchers [6], [28].

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### 6.  Conclusions

This paper presented a methodology to determine the activation energy of conduction, $E_{a-cond}$, based upon a continuously monitored electrical resistivity setup that incorporates a thermoelectric cold plate. This method demonstrates potential for use in determining a temperature correction for resistivity measurements. The method simply requires a standard test cylinder to be placed into the apparatus, and monitored with a resistivity meter and thermocouple.

The $E_{a-cond}$ was calculated for a wide range of mixtures in their sealed state, and results indicate that an average value of 27.9 provides reasonable agreement with all of the mixtures in this study. These mixtures were made using a wide variety of constituent materials that ranged from w/cm = 0.37 to 0.45 with Type I OPC cements, to ternary blends including fly ash and slag cement. A correlation between $E_{a-cond}$ and pore solution ionic strength was noticed with systems of a higher ionic strength, i.e. higher concentration, typically having lower values of $E_{a-cond}$. Furthermore, ongoing work with these samples demonstrates a value that can vary more than 20 % as the mixture goes from sealed to a saturated state.

### 7.  Acknowledgements

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