

FINAL REPORT ~ FHWA-OK-20-03

THE USE OF RESISTIVITY TESTING FOR QUALITY CONTROL OF CONCRETE MIXTURES

Julie Ann Hartell, Ph.D.

School of Civil and Environmental Engineering
Oklahoma State University
Stillwater, Oklahoma

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THE USE OF RESISTIVITY TESTING FOR QUALITY CONTROL OF CONCRETE MIXTURES

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Julie Ann Hartell, Ph.D.

School of Civil and Environmental Engineering
Oklahoma State University



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| 16. ABSTRACT This study proposes a new quality control and compliance method for concrete mixture design using standard surface resistivity testing. This method helps in determining key mixture parameters such as fly ash content and w/cm of placed concrete. Based on the gain in resistivity over time, it was found that the slope of the surface resistivity versus time curve could be used to differentiate fly ash content. And, the resistivity value obtained at a sample age of 14 and 28 days could be used for identifying the water-to-cementitious material ratio of a concrete mixture containing no fly ash and containing up to 20% fly ash. Several other parameters such as, aggregate type and admixture addition are also evaluated for their effect on the outcome of a resistivity test. The proposed resistivity method could be used as a means for quality acceptance of mixture design during the construction stage. Three methodologies (Procedure A, B and C) for OkDOT Classes A and AA concrete mixtures are developed and trialed as part of a field study. In addition, the influence of laboratory ambient temperature and curing temperature was also investigated. It was found that if resistivity testing is performed in a standard temperature-controlled environment, resistivity variances are negligible. Finally, with all quality control material testing, an alternative test method is investigated in the event the primary lab specimen fails to meet the specification. The secondary compliance testing method targets the adequacy of concrete constructed onsite. In the end, the outcomes of the project can aid a DOT in devising a strategy for implementation of the resistivity method. The new tool enables control of placed concrete with respect to the approved mixture design. | | | |
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SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

| SYMBOL | WHEN YOU KNOW | MULTIPLY BY | TO FIND | SYMBOL |
|-------------------------------------|----------------------------|-------------|-----------------------------|-------------------|
| LENGTH | | | | |
| in | inches | 25.4 | millimeters | mm |
| ft | feet | 0.305 | meters | m |
| yd | yards | 0.914 | meters | m |
| mi | miles | 1.61 | kilometers | km |
| AREA | | | | |
| in ² | square inches | 645.2 | square millimeters | mm ² |
| ft ² | square feet | 0.093 | square meters | m ² |
| yd ² | square yard | 0.836 | square meters | m ² |
| ac | acres | 0.405 | hectares | ha |
| mi ² | square miles | 2.59 | square kilometers | km ² |
| VOLUME | | | | |
| fl oz | fluid ounces | 29.57 | milliliters | mL L |
| gal | gallons | 3.785 | liters | m ³ |
| yd ³ | cubic feet | 0.028 | cubic meters | m ³ |
| | cubic yards | | 0.765 | |
| cubic meters NOTE: volumes | | | | |
| MASS | | | | |
| oz | ounces | 28.35 | grams | g |
| lb | pounds | 0.454 | kilograms | kg |
| T | short tons (2000 lb) | 0.907 | megagrams (or "metric ton") | Mg (or "t") |
| TEMPERATURE (exact degrees) | | | | |
| °F | Fahrenheit | | 5 (F- | °C |
| | 32)/9 | | Celsius or (F-32)/1.8 | |
| ILLUMINATION | | | | |
| fc | foot-candles | 10.76 | lux | lx |
| fl | foot-Lamberts | 3.426 | candela/m ² | cd/m ² |
| FORCE and PRESSURE or STRESS | | | | |
| lbf | poundforce | 4.45 | newtons | N |
| lbf/in ² | poundforce per square inch | 6.89 | kilopascals | kPa |

APPROXIMATE CONVERSIONS FROM SI UNITS

| SYMBOL | WHEN YOU KNOW | MULTIPLY BY | TO FIND | SYMBOL |
|-------------------------------------|-----------------------------|-------------|----------------------------|---------------------|
| LENGTH | | | | |
| mm | millimeters | 0.039 | inches | in |
| m | meters | 3.28 | feet | ft |
| m | meters | 1.09 | yards | yd |
| km | kilometers | 0.621 | miles | mi |
| AREA | | | | |
| mm ² | square millimeters | 0.0016 | square inches | in ² |
| m ² | square meters | 10.764 | square feet | ft ² |
| m ² | square meters | 1.195 | square yards | yd ² |
| ha | hectares | 2.47 | acres | ac |
| km ² | square kilometers | 0.386 | square miles | mi ² |
| VOLUME | | | | |
| mL | milliliters | 0.034 | fluid ounces | fl oz |
| L | liters | 0.264 | gallons | gal |
| m ³ | cubic meters | 35.314 | cubic feet | ft ³ |
| m ³ | cubic meters | 1.307 | cubic yards | yd ³ |
| MASS | | | | |
| g | grams | 0.035 | ounces | oz |
| kg | kilograms | 2.202 | pounds | lb |
| Mg (or "t") | megagrams (or "metric ton") | 1.103 | short tons (2000 lb) | T |
| TEMPERATURE (exact degrees) | | | | |
| °C | Celsius | 1.8C+32 | Fahrenheit | °F |
| ILLUMINATION | | | | |
| lx | lux | 0.0929 | foot-candles | fc |
| cd/m ² | candela/m ² | 0.2919 | foot-Lamberts | fl |
| FORCE and PRESSURE or STRESS | | | | |
| N | newtons | 0.225 | poundforce | lbf |
| kPa | kilopascals | 0.145 | poundforce per square inch | lbf/in ² |

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380. (Revised March 2003)

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Chapter 1 Introduction

The durability of concrete is widely recognized to be controlled by the ingress of detrimental agents. Here, preventing penetration of water, oxygen, carbon dioxide, and salts is key to maximize material performance and longevity (Neithalath and Jain, 2010). Rapid Chloride Permeability Test (RCPT) and now, resistivity testing, are widely used to assess the quality of a concrete mixture based on its performance in resisting ionic flow (Kessler et al., 2005 and 2008, Nadelman and Kurtis, 2014). In fact, many people feel that the permeability of the concrete is more important than its strength. The challenge has been that permeability was not measured nor specified by engineers because there was no good way to measure it.

This has recently changed with the widespread introduction of the surface resistivity meters. These meters are used to measure the flow of electrons through concrete. They take only a few seconds to run and show good correlation to the rapid chloride permeability test (RCPT) and the bulk diffusion test (Layssi, 2015). The test has been standardized by both ASTM (ASTM C1760) and AASHTO (AASHTO T 358) and it is gaining popularity in many states. This work aims to recommend a method for Oklahoma for mixture design quality control and compliance using resistivity testing.

1.1 Scope of Research

The purpose of this project is to investigate the potential of resistivity testing in assessing the performance of typical concrete mixtures used in bridge and pavement infrastructure in Oklahoma. The efforts are concentrated towards the development of guidelines using resistivity as a mean for mixture approval and compliance in addition to ODOT's current specifications. This would allow ODOT to produce high quality and durable concrete. These specifications could be used as a means for quality control and material compliance during the construction stage. This means that strength would no

longer be the only value that is used to accept a concrete mixture and instead a measurement of permeability could be included. A systematic approach using resistivity testing for Classes A and AA concrete mixture design compliance control during construction is developed. Moreover, curing and sample temperature along with laboratory conditions may influence the resistivity measurements and there are no means proposed in literature to correct this inherent deficiency. Therefore, the influence of temperature and the applicability of a correction factor to rectify results of resistivity measurements taken outside of the test method's recommended temperature range is investigated. Within the devised experimental plan, an extensive field trial study is conducted. This will help with the validation process of the resistivity method developed. Finally, with all quality control material testing, an alternative method investigated in the event a sample fails to meet the specification. This secondary compliance testing method targets the adequacy of the material constructed onsite. As such, an alternative secondary resistivity testing procedure, in case of failed material compliance test, is investigated. In the end, the outcomes of the project will aid in devising a strategy for easy implementation of the resistivity method within material quality control and compliance activities of a unit.

Within the scope of this project, the applied experimental research is conducted to develop a resistivity method for ODOT classes A and AA concrete mixtures, utilizing different type I cements and fly ash sources typically used in the state of Oklahoma, as well as common admixtures used by contractors.

1.2 Research Aims and Objectives

The research aims and objectives of the study are as follows:

Specific Aim 1 - Develop a systematic approach using resistivity testing for Classes A and AA concrete mixture design compliance control during construction. Objectives of

the study are to first perform an experimental parametric investigation to model time-resistivity behavior of typical ODOT Class A and Class AA concrete mixtures. This will aid in establishing a time-dependent resistivity model to identify the water-to-cement ratio of a given mixture and the type of cementitious materials present in the mixture. Next, evaluate the efficacy of the resistivity model and its application to compliance control of mixture design via a field study.

Specific Aim 2 -Develop a temperature correction factor to rectify results of resistivity measurements taken outside the test method's specified temperature range. The objectives are to perform an experimental parametric investigation to model temperature-resistivity behavior of typical ODOT Class A and Class AA concrete mixtures. And, establish a temperature correction factor compatible with the developed resistivity method for compliance control.

Specific Aim 3- Investigate alternative secondary testing procedures using resistivity in case of failed material compliance test. The objectives are to establish the probability of detection of mixture design parameters for various resistivity testing procedures performed in-situ and performed in the laboratory on cored samples.

Chapter 2 Literature Review

The hydrated paste matrix of concrete is porous in nature. The material consists of solid and liquid phases. The solid phase is mainly composed of crystallized hydrated calcium silicates and other minor crystalline products. The liquid phase is generally saturated with various ions (e.g., Ca^{2+} , OH^- , K^+ , Na^+ , and SO_4^{2-} ions). With age (i.e., maturity) the cementitious matrix changes, it gains density and strength as solid-solution interactions continue (Samson et al, 2000). In-service, external agents may enter the porous medium and alter its delicate balance. Foreign components in the form of an aqueous solution (e.g., chlorides or sulfates) or gas (e.g., carbon dioxide) ingress into the porous cementitious matrix causing various material durability issues and corrosion of rebar in cases of reinforced concrete. Here, ionic movement through the partially or completely saturated pore system is, in part, responsible for the detrimental effects. The mechanisms that involve ion transport are capillary action, diffusion, migration in electrical field and permeation due to the pressure gradient, to name a few (Neithalath and Jain 2010). Field structures are often subjected to combinations of these transport mechanisms, which makes it difficult to single out the ongoing process. The problem is that the standard methods for measuring these principles are considered time-consuming, variable and impractical. Still, it is well known that resistance against ionic or fluid penetration is the best defense mechanism for concrete against durability issues. Therefore, there is a need for finding an economical and rapid nondestructive method for measuring these processes (Vivas et al, 2007).

The standard method to evaluate such properties has been the rapid chloride permeability testing (RCPT). The results provide an indication on the ability of a mixture to resist ionic flow which is an indication of the movement of fluid and ions. However, the test takes over a day to prepare and several hours to conduct the actual measurement. Moreover, the test method has often been criticized for producing

variable results. Therefore, there is a need for finding alternative methods for measuring these processes. (Vivas et al. 2007)

The physical and chemical nature of concrete makes it particularly sensitive to electrical conductivity. Recently, investigations have demonstrated that electrical methods such as the surface resistivity and bulk resistivity methods are cost effective and accurate means for assessing the quality of a concrete mixture based on its performance in resisting ionic flow established through comparative relationship with RCPT (Figure 2.1.1). (Kessler et al. 2005; Rupnow and Icenogle 2011; Spragg et al. 2013) Procedures and recommendations have been published which led to the developments of standards such as:

- FM5-578 – Florida Method of Test for Concrete Resistivity as an Electrical Indicator of its Permeability,
- AASHTO TP 95-11: Standard Method of Test for Surface Resistivity Indication of Concrete's Ability to Resist Chloride Ion Penetration,
- ASTM C1760 – 12: Standard Test Method for Bulk Electrical Conductivity of Hardened Concrete.

And, since their introduction, resistivity has been used in the industry for the past decade as a viable means to assess the quality of concrete mixtures with respect to durability performance. (Layssi et al. 2015, Nadelman 2014) (Baroghel et al 2011)

| Chloride Ion Permeability | RCP Test Charged Passed (coulombs) | Surface Resistivity Test 28 day test kΩ-cm |
|---------------------------|--|--|
| High | > 4,000 | < 12 |
| Moderate | 2,000-4,000 | 12 - 21 |
| Low | 1,000-2,000 | 21 - 37 |
| Very Low | 100-1,000 | 37 - 254 |
| Negligible | < 100 | > 254 |

Figure 2.1.1: Equivalent Surface Resistivity Values Rounded for Utilization. (FM5-578)

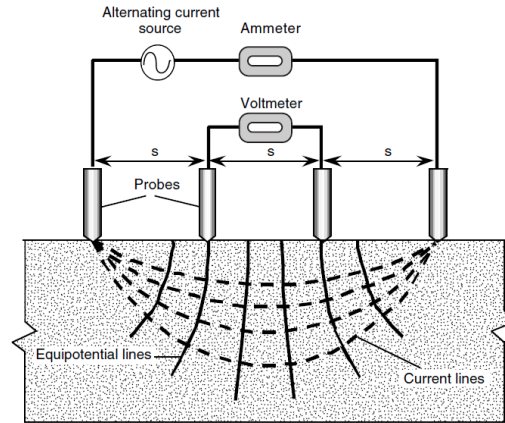


Figure 2.1.2: Test Principle of surface resistivity using four point wenner probe apparatus
(ACI 228-2R 2013)

Resistivity methods were initially used in geotechnical areas to measure the resistivity of soils to provide an indication of their permeability characteristics. The four point Wenner probe which was originally developed for that purpose by Wenner in the early 1900's. It has now gained popularity as a non-destructive surface method to measure the ability of concrete to conduct current. As seen in Figure 2.1.2, the four probes are electrically connected to a concrete surface through adequate contact and the outer probes produce a small alternating current. Meanwhile, the inner two probes connected to a voltmeter measure the response to current flow. (ACI 228 – 2R 2013) Alternatively, the resistance of a concrete cylinder can also be determined using the plate electrodes which are placed on the end of the sample. In both methods, the resistance value obtained can be factorized by specimen geometry by simply applying a ratio of sample cross-sectional area to length. (Morris et al 1996)

Past investigations demonstrate that resistivity measurements are mainly influenced by the microstructure of concrete, pore solution conductivity, saturation condition and temperature of concrete. (Spragg 2013, Bu et al. 2014, Layssi et al. 2015) However, there are many factors which may influence the accuracy of the measured values due to the

test principle itself and the inherent variability of concrete materials. Therefore, many investigations have been conducted to evaluate potential factors which may affect the reliability of the measurements. The following sections provide a summary of main recommendations from previous research activities. Table 2.1.1 provides a summary of main recommendations from previous research activities.

Table 2.1.1: Summary of parameters influencing resistivity testing variability

| Procedure Parameters | Recommendations | References |
|------------------------------|---|--|
| Sample Geometry | $\rho_{real} = \rho_{measure}/K$ Correction factor K must be applied to account for geometrical effects (curvature of samples). | Morris et al. 1996 |
| Edge effect | Resistivity value will increase if measurement is taken near the edge of sample. | Morris et al. 1996 Kessler et al. 2005 |
| Curing Method | FDOT method restrictions to one curing (moist curing) since moist curing produces on average higher values (9.7%) in comparison to limewater curing. Kansas Test Method specifies a result multiplier (1.1) for limewater curing. Storage solution and solution volume to sample ratio seem to have an effect on resistivity values. A solution/sample ratio of 2.0 is recommended. | Kessler et al. 2005 KT-79 Spragg et al. 2013 |
| Curing Temperature | Curing temperature will affect the degree in maturity at a given age therefore should be specified. | Spragg et al. 2013 |
| Sample/Testing Temperature | A relatively narrow range in temperature (e.g., ± 2 °C) should be specified since ion mobility increases with temperature. 3% and 5% change in values for every 1°C in temperature difference for moist and dry concrete respectively. | Spragg et al. 2013 Podler 2001 |
| Surface wetting | A surface resistivity reading is valid only when the surface is wet. If allowed to dry for several minutes, the reading will be lower. | Kessler et al. 2005 |
| Operator Statistical Scatter | For same samples, conditions and apparatus, the results indicate that operator induced variability is minimal and scatter is due to intrinsic properties of samples Bulk Electrical Conductivity ASTM Precision Statement: Single-operator variability 9.2% COV. FM5-578 Surface resistivity Precision Statement: Single-operator variability 8.2% COV. | Kessler et al. 2005 ASTM C 1790 FM5-578 |

Chapter 3 Experimental Program

In order to accomplish the objectives of the research, an experimental program was organized, which include the materials handling and testing, concrete mixing, demolding and curing, and, lastly, the experimental procedures followed in accordance with ASTM and AASHTO standards. The activities performed to complete research tasks are presented in this chapter.

3.1 Materials

The materials required to make concrete mixtures were brought from various sites in Oklahoma. The materials were stocked outside and inside the laboratory, cleaned, and tested as per requirements before mixing the concrete. The details for each material used are given in following sections.

3.1.1 Cement

In all the concrete mixtures, Type-I (ASTM C 150) Central Plains Portland cement was used. Few concrete mixtures were also prepared using Type-I/II Buzzi cement for comparison. The cement bags received were stocked inside the Bert Cooper Engineering Lab at a clean and dry place. The chemical composition of cements is shown in Table 3.1.1

Table 3.1.1 Chemical Compositions of Cement Sources

| Chemical Composition | Cement (% by weight) | Cement (% by weight) |
|--------------------------------|----------------------|----------------------|
| Cement | Central Plains | Buzzi Unicem |
| MgO | 1.9 | 1.86 |
| CaO | 62.9 | 64.25 |
| SO ₃ | 3.3 | 2.63 |
| SiO ₂ | 19.4 | 20.56 |
| Al ₂ O ₃ | 5.1 | 4.41 |
| Fe ₂ O ₃ | 3.4 | 3.28 |

3.1.2 Fly Ash

The concrete mixtures prepared with the replacement of Class-C fly ash (ASTM C 618) content were obtained from Red Rock, Headwaters Hugo, Ray Nixon and Muskogee. In order to establish the baseline criteria, and develop the guidelines for quality control, class-C fly ash from Red Rock and Hugo Headwaters were used as the main SCM. Other fly ash sources were used for the comparative analysis and validation of established criteria. The fly ash received from the various sources were sealed in 5-gallon buckets and stocked inside the Bert Cooper Engineering Lab. The chemical compositions of fly ash sources are shown in Table 3.1.2.

Table 3.1.2: Chemical Compositions (% by weight) of Fly Ash Sources

| Chemical Compositions | Red Rock (% by weight) | Muskogee (% by weight) | Ray Nixon (% by weight) | Headwaters, Hugo (% by weight) |
|--------------------------------|------------------------|------------------------|-------------------------|--------------------------------|
| K ₂ O | 0.58 | 0.41 | 0.46 | 0.39 |
| MgO | 5.55 | 7.46 | 5.87 | 6.70 |
| CaO | 23.12 | 29.74 | 24.41 | 25.84 |
| SO ₃ | 1.27 | 1.89 | 1.07 | 1.91 |
| Na ₂ O | 1.78 | 1.82 | 1.73 | 1.78 |
| SiO ₂ | 38.71 | 32.88 | 36.27 | 36.20 |
| Al ₂ O ₃ | 18.82 | 18.37 | 19.17 | 17.85 |
| Fe ₂ O ₃ | 5.88 | 5.58 | 6.28 | 5.61 |

3.1.3 Coarse Aggregates

The concrete mixtures were prepared with various types and sizes of concrete aggregates as per ASTM C 33. The aggregates were obtained from Richard Spur Limestone (#56, #57 and #67), Quapaw (#57), Coleman Dolomite (#57), and Roosevelt Gabbro (#56). All the mixtures were made with aggregates received from either Richard Spur or Quapaw sources, aggregates from other sources were used for the comparison. The coarse aggregates were stocked outside the Bert Cooper Engineering Lab. The aggregates were tested for sieve analysis (ASTM C136), dry rodded unit weight (ASTM C29), specific gravity and absorption (ASTM C127) for the purpose of quality control and

mixture design. The chemical compositions of coarse aggregates are shown in Table 3.1.3.

Table 3.1.3: Chemical Compositions (% by weight) of Coarse Aggregate Sources

| Chemical Compositions | Richard Spur Limestone (% by weight) | Coleman Dolomite (% by weight) | Roosevelt Gabbro (% by weight) |
|--------------------------------|--------------------------------------|--------------------------------|--------------------------------|
| Ca | 35.93 | 20.67 | 7.24 |
| CaO | 50.27 | 28.92 | 10.13 |
| CaCO ₃ | 89.73 | 51.62 | 18.08 |
| Mg | 1.02 | 9.74 | 1.07 |
| MgO | 1.69 | 16.15 | 1.77 |
| MgCO ₃ | 3.54 | 33.77 | 3.71 |
| Fe ₂ O ₃ | 0.25 | 0.85 | 4.07 |
| Al ₂ O ₃ | 0.6 | 2.08 | 16.91 |
| Si | 3.38 | 4.03 | 24.3 |
| SiO ₂ | 7.24 | 8.63 | 51.99 |
| S | - | - | - |
| SO ₃ | - | - | - |
| Sodium Oxide | - | - | 0.422 |
| Titanium Dioxide | - | - | 0.16 |
| Potassium Oxide | - | - | 0.316 |

3.1.4 Fine Aggregates

In all the concrete mixtures, natural sand from Dover quarry meeting the specifications of ASTM C 33 was used. The sand was stocked outside the Bert Cooper Engineering Lab. The fine aggregates were tested for sieve analysis (ASTM C136), specific gravity and absorption (ASTM C128) to meet up to the standards.

3.1.5 Water

The potable water used in all concrete mixtures was provided by Stillwater Municipal Water System. Mixing water was tempered at laboratory temperature prior to mixing to ensure uniformity in material temperatures and initial hydration.

3.1.6 Chemical Admixtures

For comparative analysis, the concrete mixtures were prepared with the addition of chemical admixtures. The air-entraining admixture (AE) (ASTM C 233), MasterAir AE 90 from BASF, and mid-range water reducer (WR) (ASTM C 494), MasterPolyheed 1020 from BASF were used in the concrete mixtures.

3.1.7 Mixture Designs

A total of 195 concrete mixtures were prepared for this research study. For each concrete batch, slump, unit weight, and pressure air meter tests were performed to maintain the quality of concrete mixtures. The cylindrical concrete samples (Ø100 mm x 200 mm approx.) were prepared (ASTM C 192) to perform the experiments from each concrete mixture. The details of the concrete mixtures produced are as follows:

Thirty concrete mixtures were prepared for parametric investigation to model time-resistivity behavior, having w/cm ratios (0.40, 0.45, 0.50, 0.55 and 0.60) and fly ash content (0%, 5%, 10%, 15%, 20% and 25%). In these concrete mixtures, crushed Limestone (#56), natural sand, type-I Portland cement and class-C fly ash from Red Rock were used. Table 3.1.1 presents the mixture design details. To evaluate variances in mixtures design parameters (e.g. aggregate source, SCM source, admixture addition) several other concrete mixture designs were prepared based on the preliminary designs presented in Table 3.1.4.

Table 3.1.4: Basic concrete mixture design details

| w/cm | Fly Ash (%) | Water (kg/m ³) | Cement (kg/m ³) | Fly Ash (kg/m ³) | Coarse Aggregate (kg/m ³) | Fine Aggregate (kg/m ³) | Paste (%) |
|------|-------------|----------------------------|-----------------------------|------------------------------|---------------------------------------|-------------------------------------|-----------|
| 0.40 | 0% | 145.4 | 362.5 | 0 | 1097.6 | 714.9 | 27.8% |
| 0.40 | 5% | 145.4 | 326.2 | 36.2 | 1097.6 | 714.9 | 27.8% |
| 0.40 | 10% | 145.4 | 309.9 | 52.6 | 1097.6 | 714.9 | 27.8% |
| 0.40 | 15% | 145.4 | 263.4 | 99.1 | 1097.6 | 714.9 | 27.8% |
| 0.40 | 20% | 145.4 | 210.8 | 151.7 | 1097.6 | 714.9 | 27.8% |
| 0.40 | 25% | 145.4 | 158.1 | 204.4 | 1097.6 | 714.9 | 27.8% |
| 0.45 | 0% | 163.2 | 362.5 | 0 | 1097.6 | 714.9 | 29.2% |
| 0.45 | 5% | 163.2 | 326.2 | 36.2 | 1097.6 | 714.9 | 29.2% |
| 0.45 | 10% | 163.2 | 309.9 | 52.6 | 1097.6 | 714.9 | 29.2% |
| 0.45 | 15% | 163.2 | 263.4 | 99.1 | 1097.6 | 714.9 | 29.2% |
| 0.45 | 20% | 163.2 | 210.8 | 151.7 | 1097.6 | 714.9 | 29.2% |
| 0.45 | 25% | 163.2 | 158.1 | 204.4 | 1097.6 | 714.9 | 29.2% |
| 0.50 | 0% | 181.5 | 362.5 | 0 | 1097.6 | 714.9 | 30.5% |
| 0.50 | 5% | 181.5 | 326.2 | 36.2 | 1097.6 | 714.9 | 30.5% |
| 0.50 | 10% | 181.5 | 309.9 | 52.6 | 1097.6 | 714.9 | 30.5% |
| 0.50 | 15% | 181.5 | 263.4 | 99.1 | 1097.6 | 714.9 | 30.5% |
| 0.50 | 20% | 181.5 | 210.8 | 151.7 | 1097.6 | 714.9 | 30.5% |
| 0.50 | 25% | 181.5 | 158.1 | 204.4 | 1097.6 | 714.9 | 30.5% |
| 0.55 | 0% | 199.3 | 362.5 | 0 | 1097.6 | 714.9 | 31.8% |
| 0.55 | 5% | 199.3 | 326.2 | 36.2 | 1097.6 | 714.9 | 31.8% |
| 0.55 | 10% | 199.3 | 309.9 | 52.6 | 1097.6 | 714.9 | 31.8% |
| 0.55 | 15% | 199.3 | 263.4 | 99.1 | 1097.6 | 714.9 | 31.8% |
| 0.55 | 20% | 199.3 | 210.8 | 151.7 | 1097.6 | 714.9 | 31.8% |
| 0.55 | 25% | 199.3 | 158.1 | 204.4 | 1097.6 | 714.9 | 31.8% |
| 0.60 | 0% | 217.7 | 362.5 | 0 | 1097.6 | 714.9 | 33.0% |
| 0.60 | 5% | 217.7 | 326.2 | 36.2 | 1097.6 | 714.9 | 33.0% |
| 0.60 | 10% | 217.7 | 309.9 | 52.6 | 1097.6 | 714.9 | 33.0% |
| 0.60 | 15% | 217.7 | 263.4 | 99.1 | 1097.6 | 714.9 | 33.0% |
| 0.60 | 20% | 217.7 | 210.8 | 151.7 | 1097.6 | 714.9 | 33.0% |
| 0.60 | 25% | 217.7 | 158.1 | 204.4 | 1097.6 | 714.9 | 33.0% |

Seven concrete mixtures were prepared to have 0.45 w/cm ratio, fly ash content (10%, 15%, 20% and 25%) with the addition of AE. In these concrete mixtures, crushed Limestone (#57), natural sand, type-I Portland cement and class-C fly ash from Red Rock were used.

Seven concrete mixtures were prepared to have 0.45 w/cm ratio, fly ash content (10%, 15%, 20% and 25%) with the addition of AE. In these concrete mixtures, crushed Limestone (#57), natural sand, type-I Portland cement and class-C fly ash from Red Rock were used.

Eleven concrete mixtures were made, having w/cm ratios (0.40, 0.45, 0.50, 0.55 and 0.60), fly ash content (0% and 20%) with and without adding AE. In these concrete mixtures, crushed Limestone (#57), natural sand, type-I Portland cement and class-C fly ash from Red Rock were used.

Six concrete mixtures were made, having w/cm ratios (0.40, 0.45 and 0.50) and fly ash content (10% and 20%). In these concrete mixtures, crushed Limestone (#56), natural sand, type-I Portland cement and class-C fly ash sourced from Headwaters, Hugo were used.

Thirty concrete mixtures were prepared, having w/cm ratios (0.40, 0.45, 0.50, 0.55 and 0.60) and fly ash content (0%, 5%, 10%, 15%, 20% and 25%) with the addition of AE and WR. In these concrete mixtures, crushed Limestone (#56), natural sand, type-I Portland cement and class-C fly ash from Red Rock were used.

Six concrete mixtures were made with w/cm ratios (0.40, 0.45 and 0.50) and fly ash content (10% and 20%). In these concrete mixtures, crushed Limestone (#56), natural sand, Type-I/II cement sourced from Buzzi, and class-C fly ash from Red Rock were used.

Thirty concrete mixtures were prepared, having (0.40, 0.45 and 0.50) and fly ash content (0%, 10% and 20%) with paste fractions of 24%, 27%, 30% and 33%. These concrete mixtures were produced with crushed Limestone (#56), natural sand, type-I Portland cement, and class-C fly ash from Red Rock were used.

Nine concrete mixtures were made with crushed Limestone (#67) coarse aggregate sourced from Richard Spur, natural sand, type-I Portland cement, and class-C fly ash from Red Rock, having (0.40, 0.45 and 0.50) and fly ash content (0%, 10%, and 20%).

Six concrete mixtures were made with Muskogee class-C fly ash source, crushed Limestone (#56), natural sand, type-I Portland cement, having (0.40, 0.45 and 0.50) and fly ash content (10%, and 20%).

Six concrete mixtures were made with Ray Nixon class-C fly ash source, crushed Limestone (#56), natural sand, type-I Portland cement, having (0.40, 0.45 and 0.50) and fly ash content (10%, and 20%).

Nine concrete mixtures were made with Dolomite (#56) coarse aggregate sourced from Coleman, natural sand, type-I Portland cement and class-C fly ash from Red Rock, having (0.40, 0.45 and 0.50) and fly ash content (0%, 10%, and 20%).

Nine concrete mixtures were made with Gabbro (#57) coarse aggregate sourced from Roosevelt, natural sand, type-I Portland cement and class-C fly ash from Red Rock, having (0.40, 0.45 and 0.50) and fly ash content (0%, 10%, and 20%).

Thirty-six concrete mixtures were prepared, having w/cm ratios (0.40, 0.45 and 0.50,) and fly ash content (0%, 5%, 10% and 20%) and with the addition of AE and WR. In these concrete mixtures, crushed Limestone (#57), natural sand, type-I Portland cement and class-C fly ash from Hugo Headwaters were used.

All the concrete samples were demolded after 24 hours of casting. After demolding, each concrete sample was marked with a specific identification number (ID), which represents the mixture design of concrete sample. The nomenclature is shown in Table 3.1.5. An example is shown in Figure 3.1.1. In this figure, the ID “50-20-56-0-1-4” represents, 50(0.50 w/cm ratio) – 20 (% Fly ash) – 56 (aggregate size) – 0 (No chemical admixtures) – 1 (Limestone aggregate) – 4 (Ray Nixon fly ash).

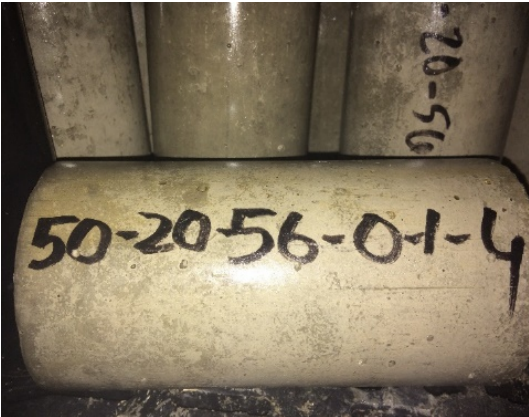


Figure 3.1.1: Example of sample identification

Table 3.1.5: Mixture design nomenclature

| W/C | FA % | Agg. # | Admixtures | Cement Supplier | Fly Ash Supplier | Agg. Type |
|--------------|-------------|--------|--|--|-------------------|----------------|
| 40 (0.40) | 00 (0%) | 56 | OO (No admixtures) | 1 (Central Plains Cement Company) | 0 (No fly ash) | 1 Limestone |
| 45 (0.45) | 05 (5%) | 57 | AO (Air entrainer only) | 2 (Buzzi Unicem) | 1 (Red Rock) | 2 Dolomite |
| 50 (0.50) | 10 (10%) | 67 | OW (Water-reducer only) | | 2 (Headwaters) | 3 Gabbro |
| 55 (0.55) | 15 (15%) | | AW (Air entrainer and Water-reducer) | | 3 (Muskogee) | |
| 60 (0.60) | 20 (20%) | | | | 4 (Nixon) | |

3.2 Specimen Preparation

3.2.1 Curing Methods

The concrete samples were cured according to ASTM C511 "*Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes.*"

3.2.1.1 Limewater Tanks

All the concrete samples were cured in saturated limewater tank storage maintained at 73 ± 2.5 °F temperature, as shown in Figure 3.2.1. This includes samples received from ODOT for the field study. This method of curing was selected (as opposed to moist curing or sealed curing) since it is the current method utilized by ODOT residencies. Most residencies do not have the facilities to conduct moist curing, nor do they have temperature-controlled rooms to maintain sealed curing in a stable environment. Thus, a water tank was deemed easiest to provide temperature control via water heaters or coolers.

A study was completed to determine the effect of variation in curing temperature, a precision temperature-controlled curing tanks was fabricated and set up at pre-determined temperatures by the precision solution heater/cooler.

3.2.1.2 Moist Room

If required, concrete samples were cured in 100% moist room at a controlled temperature of 23 ± 2 °C, as shown in Figure 3.2.2. Care was taken to monitor temperature and prevent undesirable fluctuations.



Figure 3.2.1: Limewater tank at 23°C temperature and precision tank heater



Figure 3.2.2: 100% moist room at 23±2°C temperature

3.3 Test Procedures

3.3.1 Surface Resistivity

The surface resistivity test is becoming a popular method to indicate the quality of concrete, not only due to its ability to access the permeability of concrete mixtures having their own rate of resistivity development due to variable w/cm ratio and cementitious materials but also due to its rapid, user-friendly and low-cost procedure. The investigator found this method to be a simple and easy technique to determine the resistivity of concrete in a controlled environment. The surface resistivity testing was

conducted by following AASHTO T 358, "Standard Test Method for Surface Resistivity of Concrete's Ability to Resist Chloride Ion Penetration" (Figure 3.3.1 and 3.3.2). A set of 6 concrete cylinders were prepared from each concrete mixture to perform resistivity testing, except for a few mixtures where a set of 3 concrete samples were made.

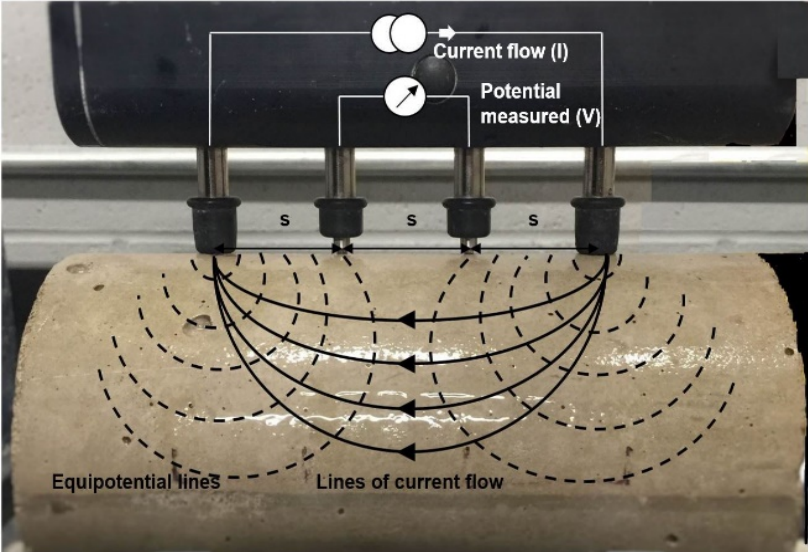


Figure 3.3.1 Illustration of surface resistivity test principle



Figure 3.3.2 Illustration of surface resistivity meter

3.3.2 Sorptivity

The rate of absorption (sorptivity) is one of the important transport mechanisms, which involves ion transport in concrete. This test was chosen for this study because it relates to the ingress of harmful ions (carbon, sulfates, and chlorides) from outside environment breaking into the first barrier (surface) of concrete through capillary action. The sorptivity test was performed by following the ASTM C1585 – 13 *“Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes.”*

3.3.3 Absorption

The total volume of water that can be absorbed by a concrete sample is useful information to relate with the resistivity of the same concrete sample at a given age. The percentage absorption test was conducted by adopting the ASTM C642 – 13 *“Standard Test Method for Density, Absorption, and Voids in Hardened Concrete.”*

3.4 Experimental Procedures

Three different experimental procedure were carried out to meet the objectives of this study. The first being the parametric investigation to determine a *Standard Quality Control and Compliance Test* for mixture design estimation. The second methodology used was to evaluate the *influence of temperature* on resistivity testing. The third procedure was devised to evaluate the potential for a *secondary test method* in the event the result of the primary QC/QA fails to meet the specification.

3.4.1 Standard Quality Control and Compliance Testing

All material batching, concrete mixing, and casting procedures were carried out within a temperature-controlled laboratory to minimize variability in test measurements.

Common material quality control was performed in accordance with relevant ASTM

standardized procedures. The required number of cylindrical specimens (\varnothing 100 mm x 200 mm cylinders) were sampled from a single batch to ensure reproducibility of test results. For the present study, six specimen replicates for each mixture type were prepared for a total of 1170 specimens. They were prepared in three equal layers using rodding and vibration as the method of consolidation. Then, they were demolded after 24 hours of curing in their molds and placed in a temperature-controlled limewater tank, ASTM C 511, for the duration of the test period.

3.4.2 Influence of Temperature

After the concrete specimens were mixed and casted according to ASTM C31, the sealed cylinders were placed into the moist curing room for a period of 24 hours prior to demolding. After demolding, the cylinders were divided into two groups, Group A and Group B, divided based on different tests that were to be done on them. Both groups surface temperature and resistivity test data were recorded. Group A was put back into the curing room that was at standard curing conditions, while group B was put into a temperature-controlled limewater tank that ensured a constant curing water temperature. Five temperature levels were evaluated (67,70,73,76, or 79°F).

Cylinders in both groups were tested according to the following schedule: day 1, 3, 7, 14, 21, 28. Each test day, the group A cylinders underwent 3 different test procedures. The cylinders were removed from the curing room and placed in containers of limewater that were also at standard temperature of 73 degrees Fahrenheit. The tests were conducted in the following order and manner.

The first test was conducted so that the Resipod is at the test trial temperature and the cylinders are at standard conditions. In order to raise or lower the Resipods surface temperature to be within one degree of the testing temperature, the Resipod was placed in an environmental chamber that can adjust the temperature within that of the

desired testing temperature. The Resipod was allowed to rest there, and the surface temperature was periodically checked until it reached the desired testing temperature. Once the surface temperature was within +/- 1 degree of the testing temperature, the temperature was recorded, the surface temperature of the Resipod and cylinder was recorded, and a resistivity test was conducted on the cylinder. Note that at this trial, the Resipod's temperature will be adjusted to testing temperature, while the cylinder temperature was very close to standard conditions (73°F).

The second test conducted was where both the cylinder and the Resipod are brought to the test trial temperature. The Resipod is once again placed in the environmental chamber, while the cylinders are soaked in limewater that is equal to or more extreme than the testing temperature, and the cylinders are allowed to soak and are periodically checked until the cylinder gets within one degree of the trial temperature. For example, if the trial temperature was 79°F, in order to get the cylinders to warm up from 73°F degrees, hot water was used and the cylinders were allowed to soak. The cylinder temperatures were then tested until the cylinder reached 78 to 80 degrees. Once the cylinder reached an acceptable test temperature, the Resipod and cylinders surface temperatures were recorded, and a resistivity test was conducted on the specimen. Note that both surface temperatures were changed until they were sufficiently close to the testing temperature.

The final test for group A is where the Resipod is allowed to reach ambient lab temperature and the cylinders remain at testing temperature. The surface temperature of both the Resipod and the cylinder was recorded, and a resistivity test was conducted.

Note that for Test 2 and for Test 3, the best method to change the surface temperature of the cylinders to match the test trial temperature was to use hot or cold water to temper the cylinders. The temperature of the adjustment water would not be specific,

and would often have to be changed until the cylinders reached a satisfactory testing temperature.

Group B cylinders only underwent one test. The cylinders would be removed from the tempered limewater tank, and their surface temperature would be recorded, and a resistivity test would be conducted. Naturally, the surface temperatures were very close to the trial temperature being tested.

3.4.3 Secondary Compliance Testing

For this study, 9 concrete slabs (20"x16"x 8") of a single mixture design were prepared in a single cast of 3 consecutive batches. 8 slabs were utilized for core extraction and the 9th served as a replicate for surface resistivity testing. For each slab, two 4" x 8" cores were extracted at appropriate days for experimentation. The coring drill was utilized with extreme caution. The primary focus was to ensure that the cores were taken properly. The two cores were taken approximately 3 inches away from any slab edges. The days for coring and testing the concrete were 1, 3, 7, 14, 28, 42, 56 and 91.

The day after mixing the concrete and casting the samples (day 1), the first two cores were extracted from one of the eight slabs. These cores were labeled and then immediately tested for surface resistivity, bulk resistivity and internal temperature. The values for these measurements were recorded as "pre-vacuum saturation". After these values were recorded, the two specimens were placed in a properly sealed chamber where they were placed under vacuum for four hours. After this period, limewater was added to saturate the cores. These specimens were saturated with limewater in this vacuum chamber for twenty-four hours. Once the twenty-four-hour period ended, the specimens were immediately tested again for surface resistivity, bulk resistivity and internal temperature. The values for these measurements were recorded as "post-vacuum saturation". After testing, these specimens were placed in the same limewater

control tank as the control specimens were curing in. This process was repeated for each of the additional slabs in which cores were extracted from for their respected days as listed above.

For each day of coring and testing, the cores from the previous days were taken out of the limewater control tank. Once they were removed from the tank, they were immediately tested for surface resistivity, bulk resistivity and internal temperature. After recording the values, they were placed back in the limewater control tank until the next testing day. For example, on day 7 two cores were extracted from a slab and went through the process as stated earlier. Also, on day 7 the cores taken on days 1 and 3 were tested again for the three measurements but were not placed in the vacuum chamber. They were simply tested and placed back in the limewater control tank. This process was repeated for each testing day through testing on day 91.

Chapter 4 Results and Discussion

4.1 Standard Quality Control and Compliance Testing

The influence of various mixture components and design parameters on the surface resistivity of concrete mixtures is determined via comparative analysis:

- effect of water-to-cement ratio;
- effect of fly ash source and percent replacement;
- effect of cement source;
- effect of admixture type and addition, and;
- effect of aggregate type and gradation.

The results of the laboratory experimental program are presented in the form of surface resistivity versus timeline charts where variation from the mean is expressed as two standard deviations from the mean (95% confidence interval). The resistivity behavior, during the test period of 56 days, is compared for similarities in resistivity gain and trends over time. Next, a comparative analysis was performed for data sets obtained at 7, 28 and 56 days. The latter measurement days are commonly used in the industry to assess early-age, standard-age and long-term (respectively) properties of concrete. The data sets, composed of six cylinder replicates per mixture, were further analyzed using an analysis of variance statistical method (ANOVA) followed by a Student t-test in order to determine whether a change in the above listed parameters along with small variations in mixture design alters the outcome of a surface resistivity test for concrete mixtures of similar binding phase. The results of the statistical analysis are presented in Appendices A to D.

4.1.1 Effect of Water-to-Cementitious Materials Ratio

Several properties of fresh and hardened concrete are routinely tested to verify the quality of the construction material with respect to its approved mixture design. Slump and compressive strength may be indicative of the target water-to-cement ratio; however, there is still a level of uncertainty. The parameter is of importance to attain a required level of durability in accordance with an exposure type (e.g. exposure to sulfate ions, deicing salts or seawater) even if the minimal mechanical properties have been met.

It was found that resistivity may be sensitive to changes in prescribed water content for a given mixture. Figure 4.1.1 compares the results obtained for five mixtures of varying w/c (0.4 w/c to 0.6 w/c). The mixture differences were created by varying the mixture's water content while maintaining the mass in cementitious material constant hence, simulating water addition on a job site.

First, there is a noticeable trend for all mixtures where the rate in resistivity gain is high at an early age (1 to 7 days). This is consistent with hydration theory where the degree in reaction kinetics is high in the first few days of curing and reduces subsequently. During this time, the cementitious matrix is forming accompanied by changes in pore solution chemistry which is reflected in the rapid gain in resistivity. Thereafter, capillary refinement and continuous changes in pore solution chemistry will contribute to the second distinct rate noticed on the graph, i.e., a decrease in the gain in resistivity. This two-step behavior is consistent for all mixtures evaluated in this project and will be further investigated in Section 4.2.

As for the effects of water addition, there is a decrease in resistivity with increase in water-to-cement ratio. This is observable in Figures 4.1.1. A comparative analysis of the means at 7, 28 and 56 days was performed (Tables 4.1.1 to 4.1.3). Results indicate that

there is no difference between mixtures of high water-to-cement ratio (i.e. above 0.55 w/c). This trend was also investigated for other mixture types. It was found that for mixtures above 0.5 w/c containing fly ash or admixtures, results are variable. This may pose a limitation to resistivity testing for its capability in differentiating mixtures of higher water content. However, in the range of interest, between 0.4 w/c and 0.50 w/c, results demonstrate that the surface resistivity method can differentiate these mixtures classes at a 95% confidence interval. (Tables 4.1.1 to 4.1.3). This trend was also noticeable for mixtures containing fly ash (Figure 4.1.2). The results of the statistical analysis are presented in Appendix A. With curing time, the beneficial effects of the pozzolanic reaction are noticeable but, producing slight variations in behavior. This is further investigation in the following section.

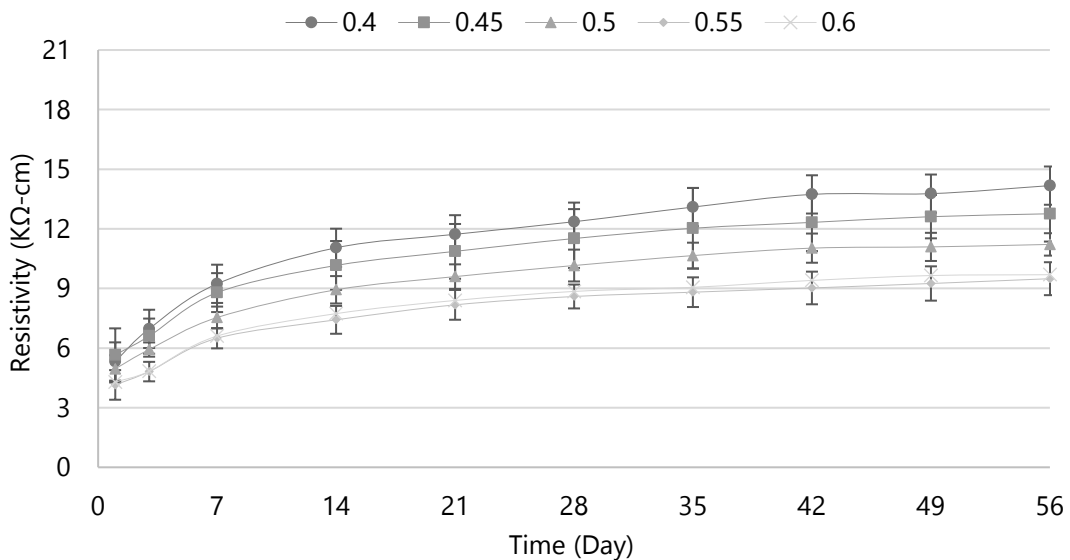


Figure 4.1.1: Time-resistivity behavior of 0.40 w/c, 0.45 w/c, 0.50 w/c, 0.55 w/c and 0.60 w/c concrete mixtures with no fly ash

Table 4.1.1: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying water-to-cement ratio (0.40 to 0.60 w/c), # -00-56-OO-1-0 mixture types, after 7-days of immersion curing.

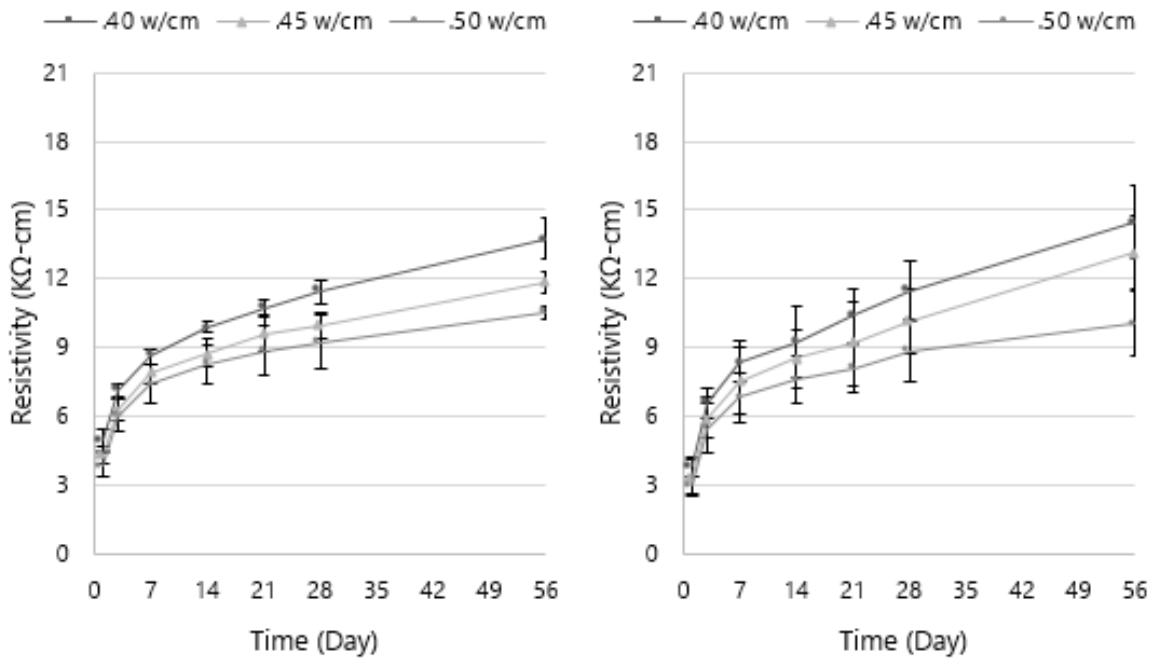
| W/C | 0.40 | 0.45 | 0.50 | 0.55 | 0.60 |
|------|-------------|-------------|-----------------|----------------|-----------------|
| 0.40 | 1.00 | T-Test 0.10 | T-Test 2.56E-06 | T-Test 2.2E-08 | T-Test 1.02E-06 |
| 0.45 | F-Test 0.44 | 1.00 | T-Test 2.74E-04 | T-Test 1.1E-05 | T-Test 1.90E-05 |
| 0.50 | F-Test 0.65 | F-Test 0.23 | 1.00 | T-Test 4.7E-05 | T-Test 2.78E-03 |
| 0.55 | F-Test 0.54 | F-Test 0.00 | F-Test 0.87 | 1.00 | T-Test 0.65 |
| 0.60 | F-Test 0.39 | F-Test 0.93 | F-Test 0.20 | F-Test 0.15 | 1.00 |

Table 4.1.2: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying water-to-cement ratio (0.40 to 0.60 w/c), # -00-56-OO-1-0 mixture types, after 28-days of immersion curing.

| W/C | 0.40 | 0.45 | 0.50 | 0.55 | 0.60 |
|------|-------------|-------------|-----------------|-----------------|-----------------|
| 0.40 | 1.00 | T-Test 0.04 | T-Test 5.22E-06 | T-Test 1.33E-08 | T-Test 2.07E-06 |
| 0.45 | F-Test 0.34 | 1.00 | T-Test 2.72E-03 | T-Test 4.38E-06 | T-Test 1.03E-04 |
| 0.50 | F-Test 0.74 | F-Test 0.20 | 1.00 | T-Test 1.78E-05 | T-Test 1.03E-04 |
| 0.55 | F-Test 0.35 | F-Test 0.07 | F-Test 0.54 | 1.00 | T-Test 0.47 |
| 0.60 | F-Test 0.32 | F-Test 0.98 | F-Test 0.19 | F-Test 0.07 | 1.00 |

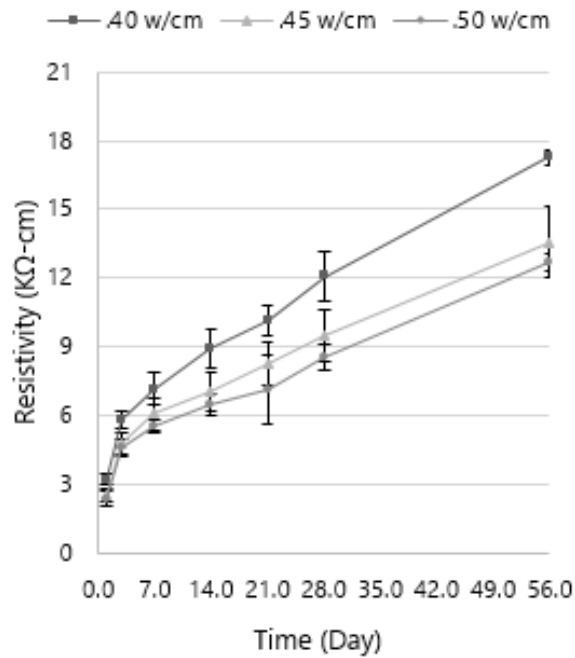
Table 4.1.3: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying water-to-cement ratio (0.40 to 0.60 w/c), # -00-56-OO-1-0 mixture types, after 56-days of immersion curing.

| W/C | 0.40 | 0.45 | 0.50 | 0.55 | 0.60 |
|------|-------------|-----------------|-----------------|-----------------|-----------------|
| 0.40 | 1.00 | T-Test 2.94E-03 | T-Test 3.59E-07 | T-Test 1.26E-08 | T-Test 1.43E-06 |
| 0.45 | F-Test 0.61 | 1.00 | T-Test 5.29E-04 | T-Test 1.83E-06 | T-Test 7.54E-05 |
| 0.50 | F-Test 0.17 | F-Test 0.07 | 1.00 | T-Test 7.40E-06 | T-Test 9.04E-03 |
| 0.55 | F-Test 0.21 | F-Test 0.28 | F-Test 0.42 | 1.00 | T-Test 0.65 |
| 0.60 | F-Test 0.27 | F-Test 0.54 | F-Test 0.02 | F-Test 0.10 | 1.00 |



a)

b)



c)

Figure 4.1.2: Time-resistivity behavior of 0.40 w/c, 0.45 w/c and 0.50 w/c concrete mixtures with varying fly ash replacements: a) 5%), b) 10% and c) 20%

4.1.2 Effect of Supplementary Cementitious Materials

To determine whether the presence of fly ash in a concrete mixture can be detected using resistivity testing, several mixtures containing various levels of percent fly ash replacement were evaluated. Figure 4.1.3 illustrates the influence of fly ash replacement content for 0.45 w/cm mixtures prepared with a Type I cement from Central Plains Cement Company, Class C fly ash from Red Rock and containing no admixtures.

First, it seems that the resistivity value after demolding is greatest for the mixture containing no fly ash followed by 5%, 10% and, so on, up to 25% replacement recording the smallest resistivity value. At the end of the curing period, the opposite trend is noticed where the resistivity value increases with an increase in fly ash replacement. This is due to the change in the rate of resistivity gain during the second phase of curing (section 4.1.1.1). Figure 4.1.3 demonstrates a similar trend to that seen in Figure 4.1.1 where the gain in resistivity is greater during the first week of curing in comparison to later curing age. However, there is a notable difference in the increase in resistivity (slope) during the second stage between mixtures of varying fly ash. It needs to be noted that the same trend is observable for all mixtures evaluated of different water-to-cementitious material ratio and fly ash source. For example, Figure 4.1.4 demonstrates the resistivity behavior of various mixtures prepared with the Hugo fly ash. The results of the statistical analysis are presented in Appendix B. Again, there are no discernable difference near 28-day due to this convergence cause by an increase in resistivity gain (slope). This behavior and how it can be utilized for mixture identification will be further investigated in Section 4.2.

As seen in tables 4.1.4 to 4.1.6, small variations in fly ash content are not discernable for a given sample age; however, as previously mentioned, the rate in resistivity gain may be a more suitable parameter for mixture evaluation. This concept will be further

investigated in the second phase of the project to determine the viability of the method to discern mixtures containing fly ash versus mixtures containing no fly ash along with suitable testing ages.

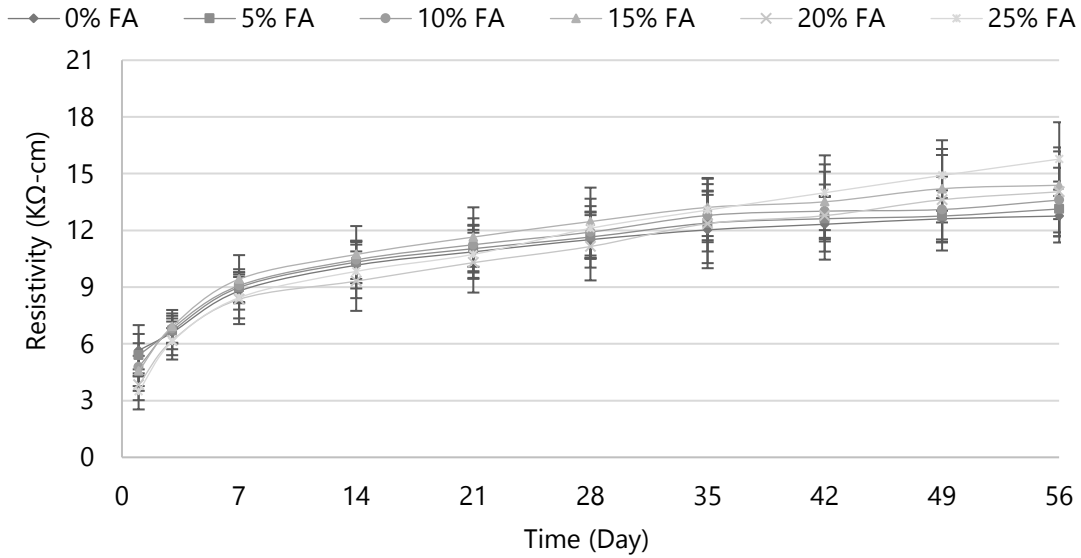


Figure 4.1.3: Resistivity with respect to curing time: comparison between percent fly ash replacement (0% to 25%) for 45-#-56-OO-1-1 mixture types.

Table 4.1.4: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying percent fly ash replacement (0% to 25%) for 45-#-56-OO-1-1 mixture types, after 7-days of immersion curing.

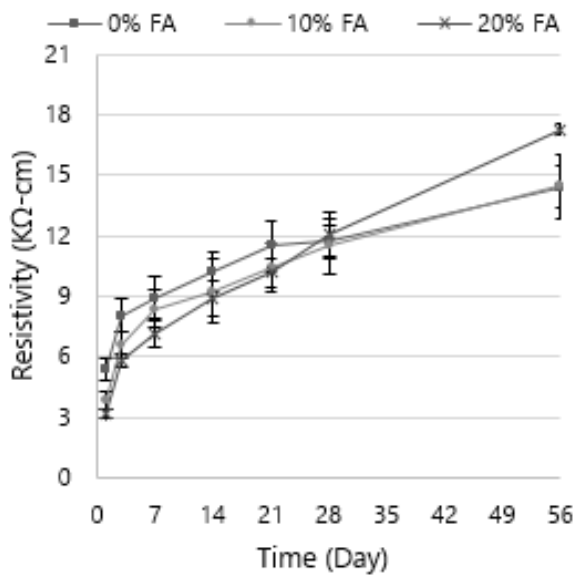
| Fly Ash | 0% | 5% | 10% | 15% | 20% | 25% |
|---------|-------------|-------------|-------------|-------------|-------------|-------------|
| 0% | 1.00 | t-Test 0.48 | t-Test 0.31 | t-Test 0.08 | t-Test 0.22 | t-Test 0.27 |
| 5% | F-Test 0.69 | 1.00 | t-Test 0.70 | t-Test 0.19 | t-Test 0.07 | t-Test 0.08 |
| 10% | F-Test 0.80 | F-Test 0.89 | 1.00 | t-Test 0.30 | t-Test 0.05 | t-Test 0.05 |
| 15% | F-Test 0.58 | F-Test 0.35 | F-Test 0.42 | 1.00 | t-Test 0.02 | t-Test 0.02 |
| 20% | F-Test 0.54 | F-Test 0.32 | F-Test 0.39 | F-Test 0.95 | 1.00 | t-Test 0.81 |
| 25% | F-Test 0.81 | F-Test 0.53 | F-Test 0.62 | F-Test 0.95 | F-Test 0.71 | 1.00 |

Table 4.1.5: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying percent fly ash replacement (0% to 25%) for 45-#-56-OO-1-1 mixture types, after 28-days of immersion curing.

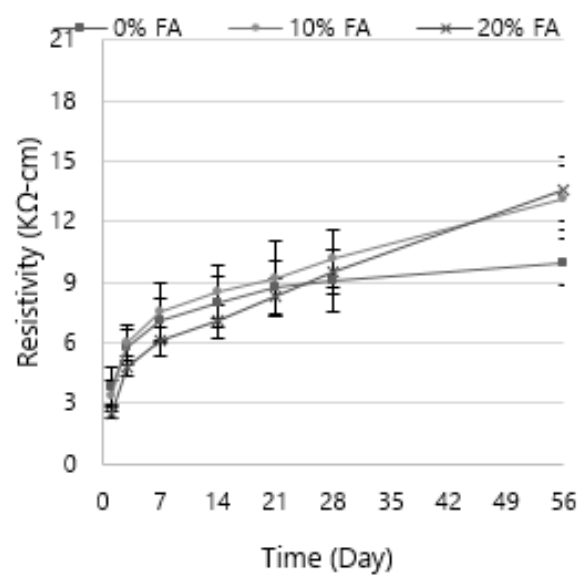
| Fly Ash | 0% | 5% | 10% | 15% | 20% | 25% |
|---------|-------------|-------------|-------------|-------------|-------------|-------------|
| 0% | 1.00 | t-Test 0.73 | t-Test 0.36 | t-Test 0.07 | t-Test 0.46 | t-Test 0.20 |
| 5% | F-Test 0.60 | 1.00 | t-Test 0.50 | t-Test 0.09 | t-Test 0.28 | t-Test 0.27 |
| 10% | F-Test 0.87 | F-Test 0.72 | 1.00 | t-Test 0.25 | t-Test 0.13 | t-Test 0.64 |
| 15% | F-Test 0.68 | F-Test 0.36 | F-Test 0.57 | 1.00 | t-Test | t-Test 0.48 |
| 20% | F-Test 0.68 | F-Test 0.36 | F-Test 0.57 | F-Test 1.00 | 1.00 | t-Test 0.08 |
| 25% | F-Test 0.91 | F-Test 0.53 | F-Test 0.78 | F-Test 0.77 | F-Test 0.77 | 1.00 |

Table 4.1.6: Results of F-test and t-test for verification of equality of sample variances and means for mixtures of varying percent fly ash replacement (0% to 25%) for 45-#-56-OO-1-1 mixture types, after 56-days of immersion curing.

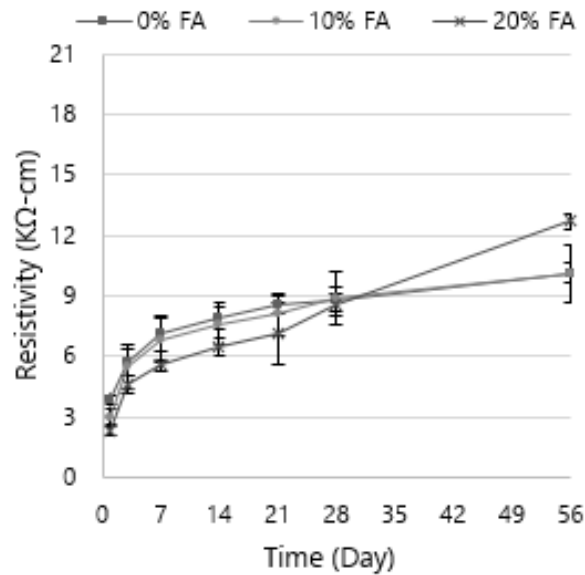
| Fly Ash | 0% | 5% | 10% | 15% | 20% | 25% |
|---------|-------------|-------------|-------------|-------------|-------------|-----------------|
| 0% | 1.00 | t-Test 0.38 | t-Test 0.53 | t-Test 0.01 | t-Test | t-Test 1.08E-04 |
| 5% | F-Test 0.94 | 1.00 | t-Test 0.33 | t-Test 0.02 | t-Test 0.14 | t-Test 3.38E-04 |
| 10% | F-Test 0.67 | F-Test 0.73 | 1.00 | t-Test 0.15 | t-Test 0.48 | t-Test 2.17E-03 |
| 15% | F-Test 0.60 | F-Test 0.66 | F-Test 0.92 | 1.00 | t-Test 0.58 | t-Test 0.03 |
| 20% | F-Test 0.28 | F-Test 0.31 | F-Test 0.50 | F-Test 0.57 | 1.00 | t-Test 0.02 |
| 25% | F-Test 0.48 | F-Test | F-Test 0.78 | F-Test 0.57 | F-Test 0.69 | 1.00 |



a)



b)



c)

Figure 4.1.4: Time-resistivity behavior of 0%, 5%, 10% and 20% fly ash concrete mixtures with varying water-to-cementitious material ratio : a) 0.40 w/c, b) 0.45 w/c, and c) 0.50 w/c

4.1.3 Effects of Various Aggregate Type and Gradation

Herein, the effect of coarse aggregate type and gradation on surface resistivity testing are investigated for mixture designs of three different water-to-cementitious materials ratio (0.40, 0.45 and 0.50 w/cm) along with two different binder compositions (100% type I Portland cement and an 80% cement and 20% class-C fly ash blend). The cement source used was Central Plains and the fly ash source used was Red Rock. All results are presented in Appendix C.

4.1.3.1 Aggregate Type

First, the results from mixtures made with Portland cement only will be discussed. Figure 4.1.5 (a,b,c) demonstrate the time-resistivity curves of 0.40, 0.45, and 0.50 w/cm ratios mixtures made with crushed limestone, dolomite and gabbro rock. Within the variability of the results, there is a similar trend in resistivity gain over time for specimens made with the three aggregate types. Between the three w/cm, there is no clear trend on whether an aggregate type results in a higher or lower resistivity value with respect to the other types. For the 0.40 w/cm mixtures, the mean values obtained for the limestone aggregate is continuously lower than the two other samples; however, the gabbro aggregate mixture records lower values for the 0.45 w/c and 0.50 w/c. Moreover, for the 0.50 w/cm mixtures, limestone recorded the highest values. However, variations in resistivity values through time (peaks and valleys) are noticeable for the gabbro and dolomite concrete curves, especially at 28- and 56-day test ages. These differences are attributed to slight variations in curing temperature and ambient temperature at the time of test, which may be significant when outside standard limits (Gulrez and Hartell 2017). This concept will be considered when assessing the null hypothesis on whether the aggregate type has no influence on the test outcome for similar binders.

For all test ages, there is a significant difference between sample means according to the returned p-values of ANOVA test for the 0.40 w/cm mixtures. Conducting the post hoc tests, it seems that there is a significant difference between the resistivity readings of the limestone mixtures and that of both the gabbro and dolomite mixtures.

Meanwhile, the results indicate differences between limestone and dolomite mixtures only at day-56. As for the gabbro aggregate mixture, the decrease in resistivity due to a decrease in ambient temperature at the time of test may have caused the change in behavior as that seen for the other test ages (Figure 4.1.5). Overall, the coefficient of variation (COV) for mixtures prepared with the limestone and dolomite aggregate were acceptable, however, the COV for the 0.5 w/cm gabbro mixture was consistently higher.

A similar trend is noticeable for 0.45 w/c mixtures (Figure 4.1.5b), at the age of day-7, there is no significant difference found between the means of the samples, but with an increase in age, a significant difference is obtained in results from ANOVA test performed at days 28 and 56. This shows that in the beginning (7 days), the comparative samples attain the same resistive property, and then it disperses with an increase in age. This may be due to the influence of aggregate properties. Post analysis demonstrates a difference between dolomite and gabbro samples at test ages of 28 and 56 days with a mean difference up to 18% approximately and coefficients of variation within the allowable range. It seems like the difference in mean resistivity for the different aggregate types increases with concrete age, which might be due to the influence of aggregate properties on paste medium. However, the effects of temperature at time of test, especially at 56 days may have also played an influential role in the differences observed.

The temperature effect was not as predominant for the 0.50 w/c mixtures. This may have contributed to no observable differences between all of the aggregate types at the three different test ages (Figure 4.1.5c). As such, it would seem that the change in

aggregate type (limestone, dolomite, gabbro) did not affect the outcome of the resistivity test for a portland cement concrete mixture. Conversely, the addition of fly ash to the cementitious blend seemed to have a different outcome.

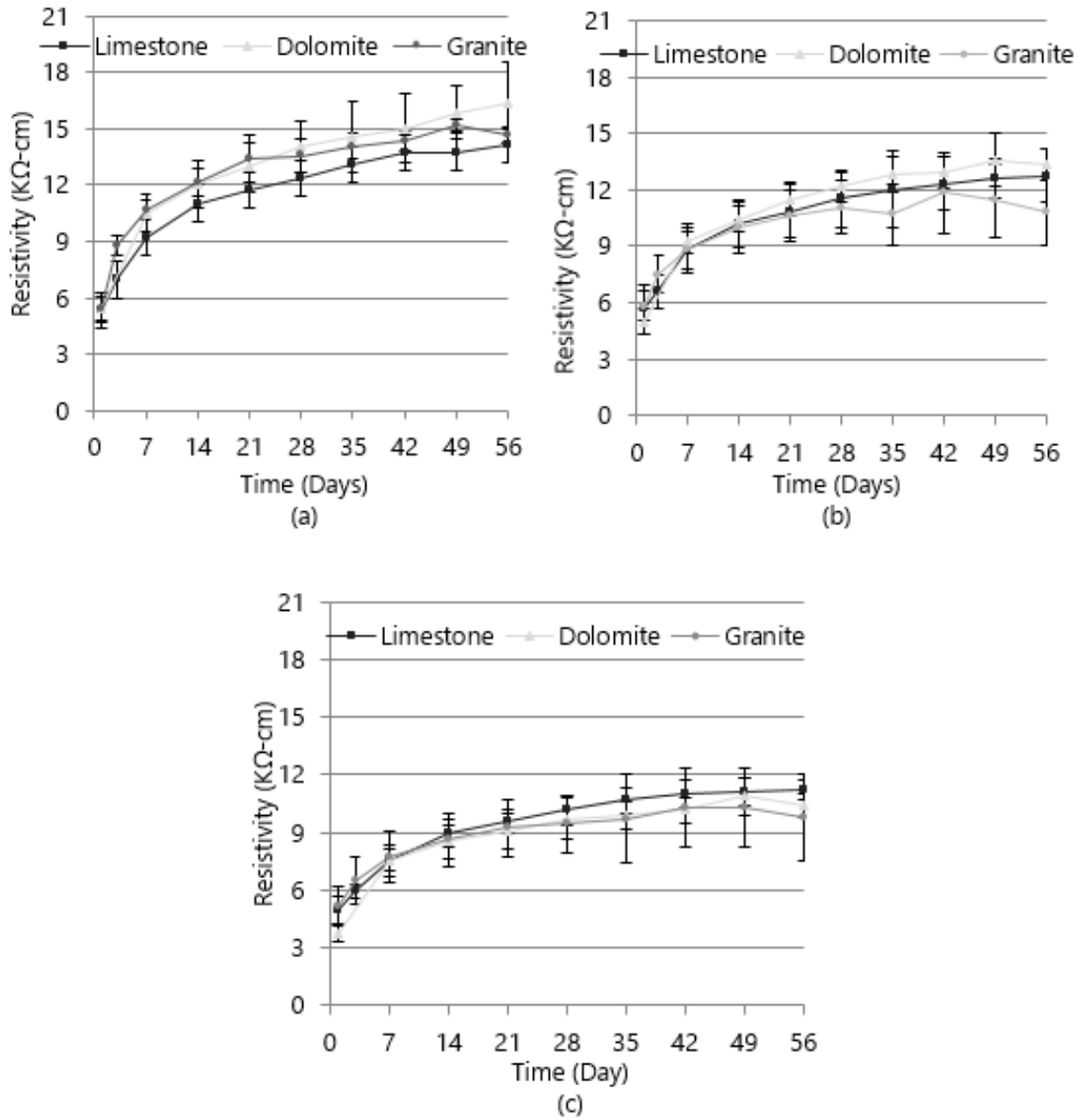


Figure 4.1.5: Time-resistivity behavior of 0% fly ash concrete mixtures (a) 0.40 w/cm (b) 0.45 w/cm (c) 0.50 w/cm with varying aggregate type

The same study was repeated to evaluate whether a change in binder chemistry would yield similar results as that observed for the ordinary portland cement mixtures. The same mixtures were prepared but with a 20% cement replacement with a class-C fly ash. Figure 4.1.6 (a,b,c) displays the time-resistivity curves of mixtures prepared with limestone, dolomite and gabbro aggregates having 0.40, 0.45 and 0.50 w/cm.

These mixtures had a similar trend for all three w/cm ratios investigated. The figures show that the limestone samples gain higher resistivity compared to that of dolomite and gabbro samples at an early age. However, the mixtures containing a dolomite aggregate attain a higher resistivity value due to a higher rate in resistivity gain over time. This behavior is not observed for the concrete prepared with the gabbro aggregate; they maintained a lower resistivity profile than that of dolomite and limestone concrete samples.

The comparative analysis of the three aggregate type mixtures is shown in Appendix C. The results demonstrate that there is a significant difference in resistivity measurements observed based on the ANOVA test between the three aggregates types at days 7, 28 and 56. For concrete prepared with a blend of 20% class-C fly ash and 80% Type I Portland cement, a change in aggregate type may change the outcome of the resistivity test. Likewise, the results of Tukey's test and t-test show significant differences between mean resistivity values for mixtures made with limestone, dolomite, and gabbro aggregates. Except for the test age of 28-days, the recorded percent difference in mean values between the mixtures containing limestone and dolomite aggregate are 4.8%, 9.8% and 5.8% for the 0.40 w/cm, 0.45 w/cm and 0.50 w/cm respectively making them marginally significant to insignificant. This is due to the crossing of both curves near that test age. Based on the profile trends and comparative analysis at 7- and 56-days, the aggregate type may have an effect on the development of resistivity properties over time (Figure 4.1.6).

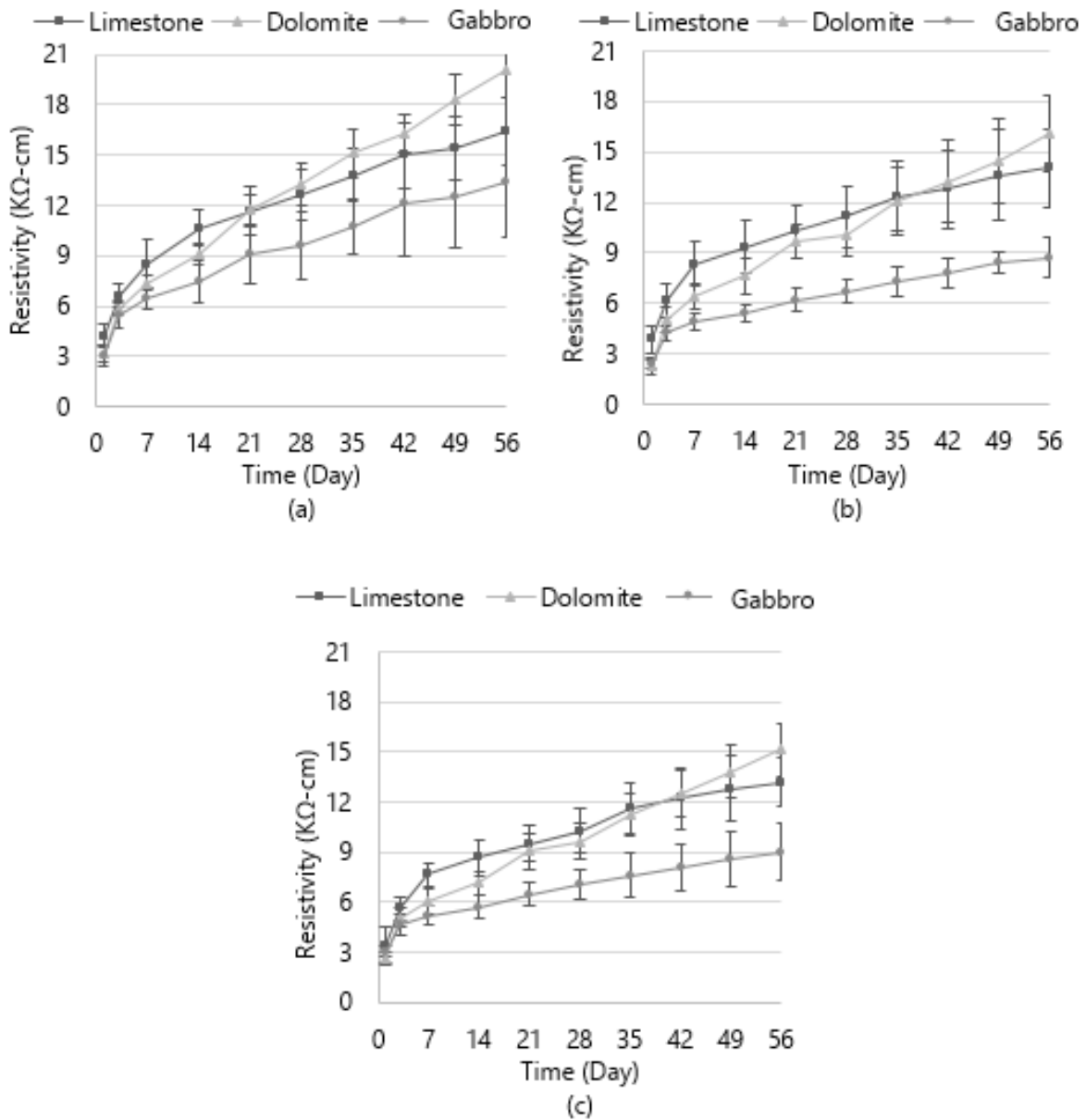


Figure 4.1.6: Time-resistivity behavior of 20% fly ash concrete mixtures (a) 0.40 w/cm (b) 0.45 w/cm (c) 0.50 w/cm with varying aggregate type

Based on the observed results and limited literature on the interaction of aggregate type and cementitious phase on electrical properties, it is difficult to comment on the contribution of each element of the concrete mixture and their role on conductivity properties without further investigation. With the development of this test method and intended applications such as evaluating the durability of a concrete mixture and its

susceptibility to initiating steel corrosion, it is important to understand its limitations. In this case, the concrete mixtures prepared with a gabbro aggregate and a class-C fly ash would be classified as a high risk to chloride ion penetration even at a 0.40 w/cm. However, a mixture containing no fly ash and a gabbro aggregate would be deemed moderate to chloride ion penetration. Further research into this behavior is necessary to understand the phenomena.

4.1.3.2 *Aggregate Gradation*

The effect of aggregate size and gradation on resistivity testing was evaluated using #67, #56 and #57 sizes of crushed limestone aggregates in concrete mixtures. It has been investigated in the past that an increase in the size of aggregates cause an increase in resistivity of concrete. Similarly, a decrease in size of aggregates causes a decrease in resistivity measurements possibly due to the increase in surface area resulting in an increase in the formation of interfacial transition zones (ITZ) (Azarsa and Gupta, 2017).

It is known that the ITZ zones are more permeable than the bulk porous structure. For a same aggregate/paste fraction, the smaller size aggregates produce a larger surface area to interact with mortar, which results in the creation of more ITZ zones that might influence in lower resistivity of concrete samples. However, if larger maximum size aggregates are used in concrete mixtures, the aggregates have less surface area compared to smaller size aggregates and less ITZ zones will be created that may influence in higher resistivity. The larger aggregate size provides increased resistance due to its low porosity compared to the porous hydrated binder matrix. Therefore, based on literature, the size of aggregates and its gradation may have an influence on the outcome of a resistivity test for a given mortar matrix. However, small variations in

aggregate gradations with a maximum aggregate size varying between 1 ½ inches and ¾ inches (commonly used in the transportation industry) has not been investigated.

Here only one mixture design was evaluated as it was deemed, based on the previous results, that the outcome for various w/cm would be similar. Thus only one mix design was investigated to identify the influence of gradation. A crushed limestone aggregate from the same quarry source was used. A standard mixture for bridge decks was selected. 0.45 w/cm and 20% fly ash replacement.

In Figure 4.1.7, the results of surface resistivity testing at days 7, 28 and 56 are shown. It can be seen that the data points of three aggregate sizes are close, and standard deviation bars (95% confidence interval) are overlapping with each other.

The statistical analysis of #67, #56 and #57 mixtures are shown in Appendix C. A significant difference is identified between the three aggregate samples from ANOVA test at day 7; whereas, there is no significant difference found among the aggregate samples at the age of 28 and 56 days. Further analysis shows that there is a significant difference in resistivity between #56 and #67 aggregate samples. The low coefficient of variation obtained for the #67 aggregate mixture (2.4%) may have contributed the rejection of the null hypothesis. Still, the percent difference of 15.1% is considerable leading to the results observed.

Therefore, it may be that an early developmental age, the effect of a developing permeable ITZ zone may be an influential factor; however, the results demonstrate that the difference encountered is moderate and may be an artifact of differences in variance. At a later stage in cement hydration, a small variance in aggregate size and gradation does not seem to change the outcome of a resistivity test for a given mortar matrix.

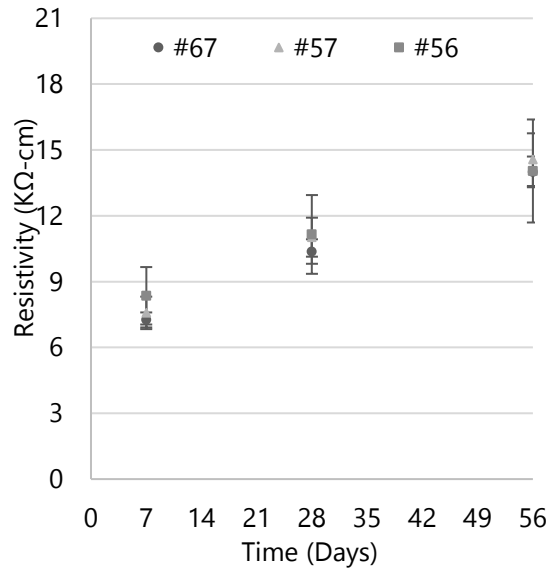


Figure 4.1.7: Time-resistivity behavior of, 0.45 w/cm and 20% fly ash concrete mixtures with varying aggregate gradation: #67, #57, #56.

4.1.4 Effect of Admixtures

To determine the effect of chemical admixtures on resistivity testing, mid-range water-reducer (WR) and air entrainer (AE) were added to the mixture designs previously investigated. The admixture effect on surface resistivity testing was investigated by preparing specimens from 0.40, 0.45, and 0.50 w/cm ratios concrete mixtures with and without replacement of fly ash material (0%, 10%, and 20%). Mixtures were prepared with the Central Plains type I cement, Hugo fly ash source. The paste content of concrete mixtures ranges from 27% to 30% due to water addition and the fine-to-coarse aggregate ratio was kept at 0.40. The time-resistivity behavior of the concrete mixtures, without addition of WR and AE, and with addition of WR and AE having 0%, 10% and 20% fly ash content is shown in Figures 4.1.8 for 0.40 w/cm, Figure 4.1.9 for 0.45 w/cm and Figure 4.1.10 for 0.50 w/cm.

The t-test was conducted to compare the resistivity values between no chemical admixture added concrete mixtures, air entrainment (AO) and water reducer and air entrainment (AW) added to the concrete mixtures. The results of the statistical analysis are presented in appendix D. The results of the analysis for mixtures 0.40 w/cm with 0% fly ash mixtures showed no distinctive differences in resistivity values between no admixture and concrete mixtures containing admixtures (Figure 4.1.8a). The t-test results of 10% and 20% fly ash mixtures showed a significant increase in resistivity values for mixtures containing both WR & AE mixtures. However, the addition of air entrainer alone, did not seem to have a substantial effect. The effect of WR & AE in concrete mixtures with 10% and 20% fly ash content in it can be seen in Figures 4.1.8 (b,c). In Appendices D, the results of t-test have show significant difference in resistivity values for 0.45 w/cm ratio as demonstrated for concrete mixtures in Figure 4.1.9. A similar result is obtained for 0.50 w/cm ratio (Figure 4.1.10).

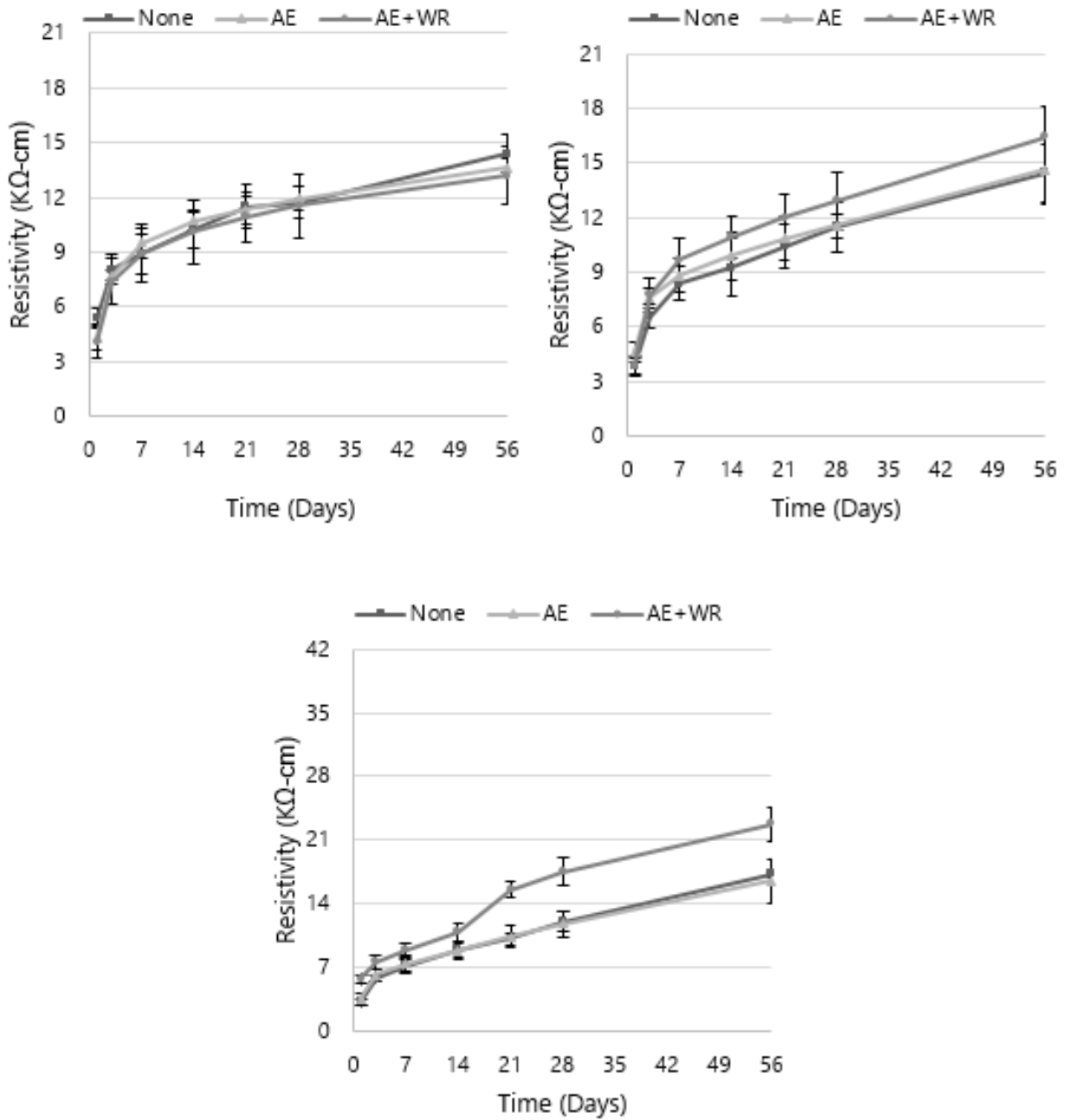


Figure 4.1.8: Time-resistivity behavior of 0.40 w/cm with 0%, 10% and 20% fly ash concrete mixtures with varying admixture addition: a) None (OO), b) AE only (AO) and c) AE+WR (AW)

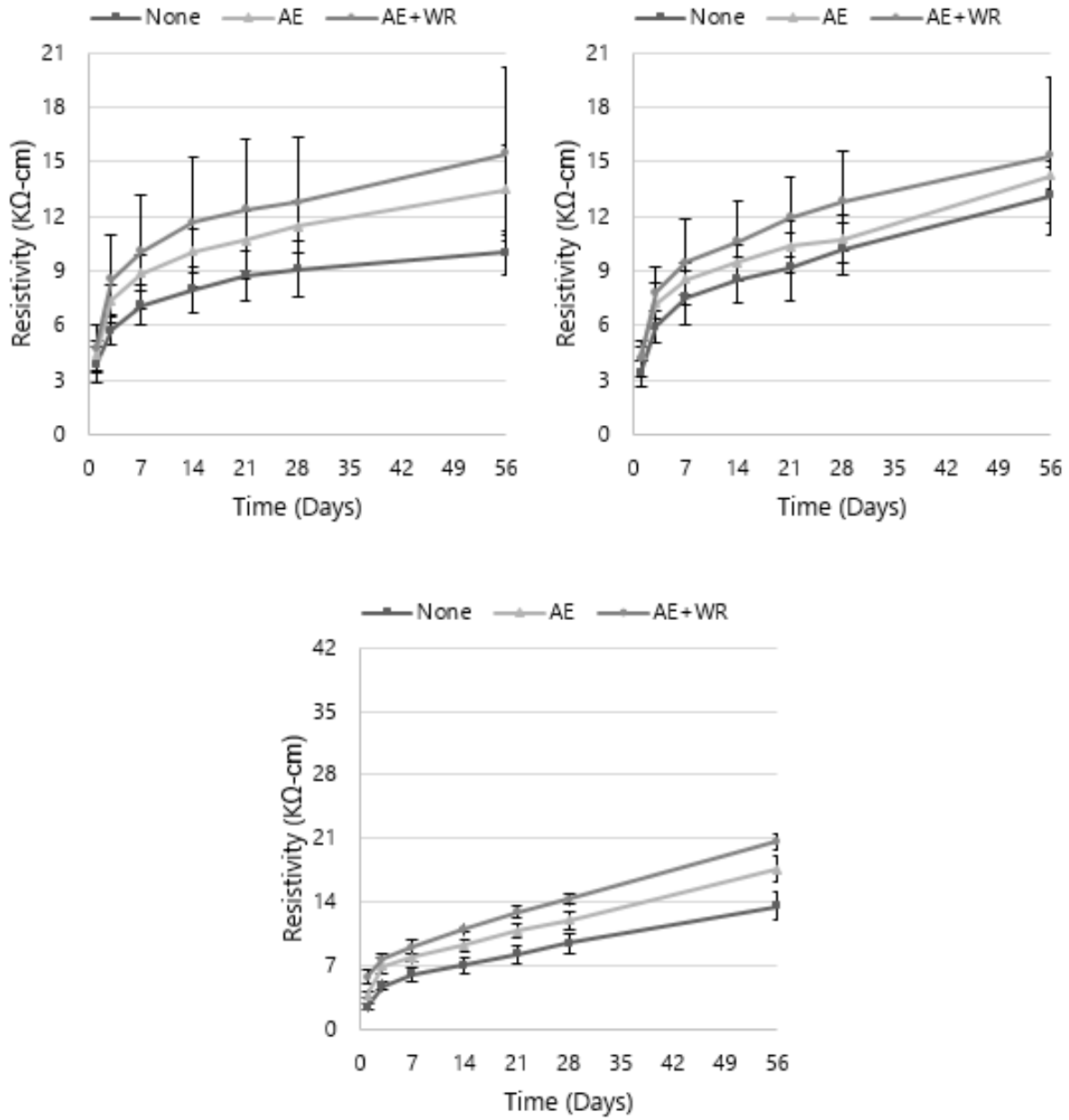


Figure 4.1.9: Time-resistivity behavior of 0.45 w/cm with 0, 10% and 20% fly ash concrete mixtures with varying admixture addition: a) None (OO), b) AE only (AO) and c) AE+WR (AW)

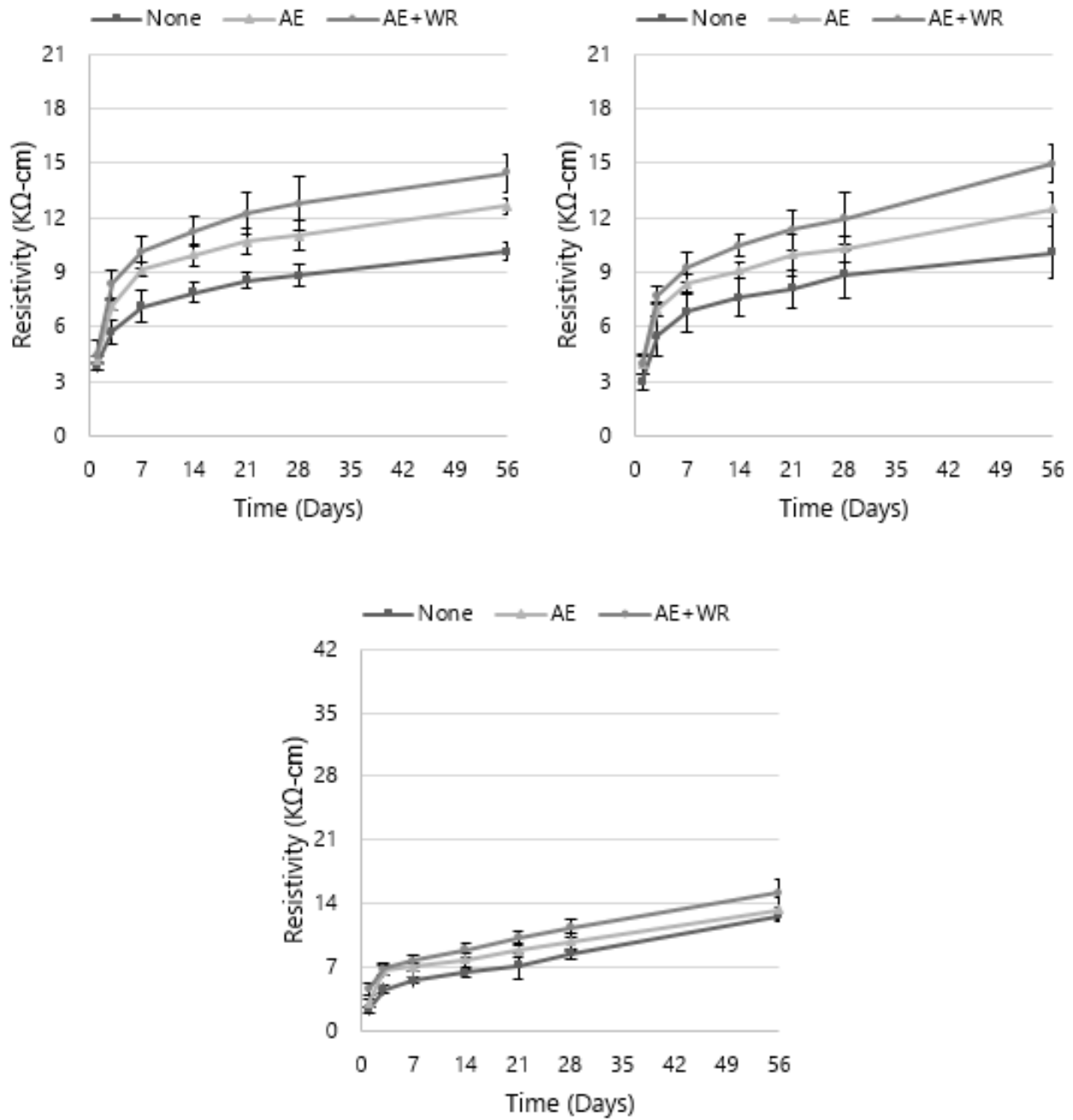


Figure 4.1.10: Time-resistivity behavior of 0.50 w/cm with 0, 10% and 20% fly ash concrete mixtures with varying admixture addition: a) None (OO), b) AE only (AO) and c) AE+WR (AW)

4.1.5 Comparative Study with Other Standard Methods of Testing

The results of the three test procedures; surface resistivity, sorptivity and percentage absorption were statistically analyzed using analysis of variation, ANOVA, followed by Student's t-test. The standard deviation and coefficient of variation (COV) were also calculated for each data set. The null hypothesis (statistical analysis) that proposes there is no significant difference among the data sets, and an alternative hypothesis that determines a significant difference among the data sets (population) is performed, which helps to quantify the effect of a change in tested parameters for each test and comparison with surface resistivity method.

4.1.5.1 Sorptivity

The results obtained for the 28-day resistivity test are compared to that of the sorptivity test where initial and secondary sorptivity are shown for both samples with a cast surface and finished surface. As seen in Figure 4.1.11 resistivity and sorptivity do not correlate well with each other. The reason for poor correlation may be due to the difference in the transport mechanism. The resistivity measurement highly depends on the degree of saturation of the porous matrix and concentration of pore solution as the conductivity of an electrolyte varies with its concentration and ionic content. Whereas sorptivity measures the capacity of the material to absorb water via capillarity. Here, pore solution concentration does not influence the fluid transport mode. The rate of absorption highly depends on capillary size, distribution, shape, tortuosity, and continuity of the pores; it is indifferent to solution type.

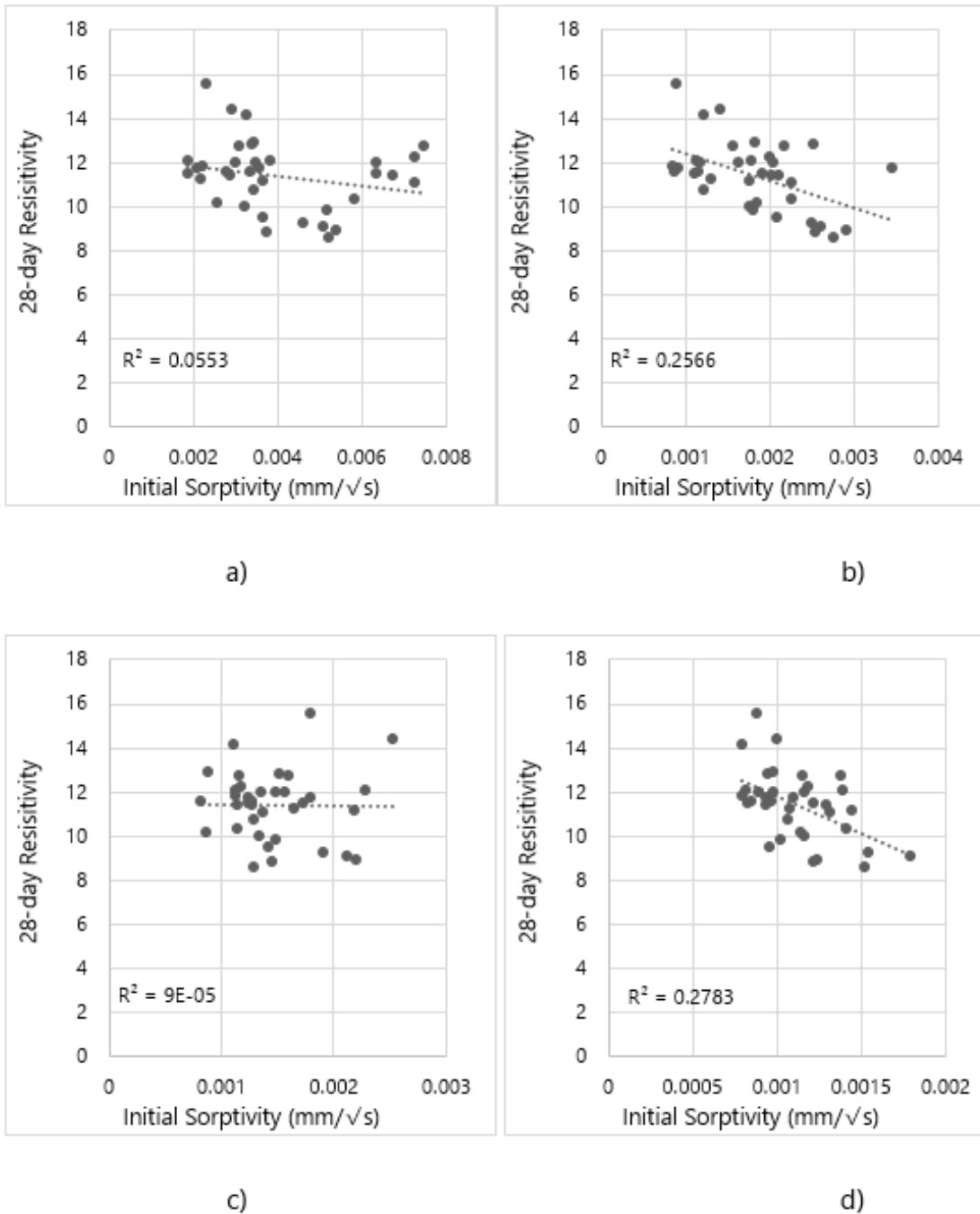


Figure 4.1.11: Comparison of resistivity and sorptivity for all concrete mixtures at 28-days: a) initial sorptivity – finished surface, b) secondary sorptivity – finished surface, c) initial sorptivity – cast surface, and d) secondary sorptivity – cast surface.

4.1.5.2 *Absorption*

Next, the total volume of water that an oven dried concrete sample could absorb (% absorption) was determined, which provides the measure of possible permeable pore space of a given concrete sample. The results of absorption in percentage were compared to the resistivity measurements obtained at 28 days (Figure 4.1.12) Overall, the decrease in w/c and increase in fly ash content resulted in a decrease in porous structure thus total absorption.

From Table E7 available in Appendix E, there is a significant difference in percent absorption results between all three w/c for mixtures containing no fly ash. This is a similar behavior to that encountered for resistivity (section 4.1.1) From the comparative analysis, the benefits of fly ash addition at 28-days are variable. And no clear conclusions can be drawn. In theory, the increase in the volume of pores increases the concrete's ability to absorb a higher quantity of water. The larger porous volume provides an increase in volume of media favorable for electrical transport resulting in the decrease in resistivity. However, the relationship between ionic strength of the conducting solution and pore volume and its impact on resistivity is still not well understood from this study. As such the correlation is not strong. This is in part due the "convergence effect" of resistivity gain near 28 days for mixtures containing various amount of fly ash.

Here, with many of the durability mechanisms driven by sorption, it would be unfair to categorize a mixture through resistivity alone as it does not represent the potential and capacity of a mixture to absorb a solution.

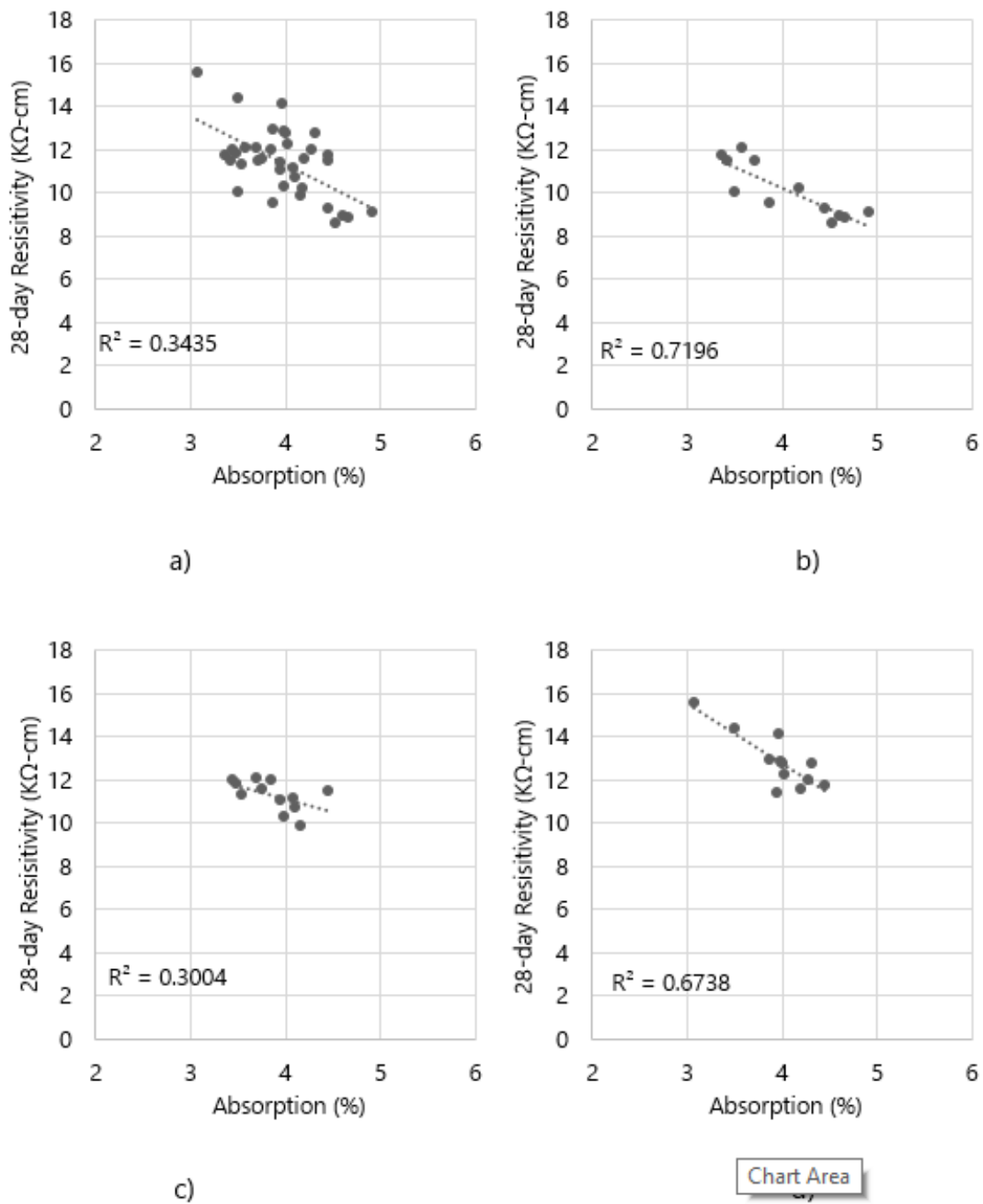


Figure 4.1.12: Comparison of resistivity and absorption for 0.40, 0.45 and 0.50 w/cm ratio with 0%, 5%, 10% and 20% fly ash content concrete mixtures at 28-days: a) no admixture, b) air entrainer, c) air entrainer and water reducer, and d) all mixtures.

It would seem that the addition of an air entraining admixture had a meaningful (but variable) impact on the percent absorption of mixtures. This is opposite to that previously reported for resistivity (section 4.1.1) As such, this resulted in a low R^2 value when isolating mixtures containing an air entrainer alone. On the other hand, the addition of a water-reducer contributed to refining the porous structures has both parameters, percent absorption and resistivity were slightly improved resulting in a slightly better correlation.

4.2 Identification of Mixture Design Parameters

Herein, relevant results are presented along with the methodology used for analysis. The discussion is divided into three sections, which represent three procedures that are recommended for implementation. The advantages and disadvantages for each procedure (A, B and C) are discussed. The procedures proposed are based on the above investigation and interpretation of results. Each procedure also discusses the roles and responsibilities of stakeholders.

4.2.1 Procedure A

For Procedure A, a two-step identification process to identify (step 1) the percentage of class-C fly ash replacement (%FA) and (step 2) the water-to-cementitious material ratio (w/c) is proposed.

For this investigation, two mixture design parameters (w/cm and %FA) were varied incrementally to evaluate their influence on the surface resistivity measurement and determine whether small changes in these important parameters may be distinguishable using resistivity testing. Based on the previous results (section 4.1), there are two noticeable trends. On day-1, the resistivity values recorded for the mixtures containing no fly ash are the highest. This is the case for all mixtures of varying w/cm. However, in

time, the resistivity behavior changes where mixtures containing high amounts of fly ash replacement increase in resistivity thus surpassing their counterparts containing lesser amounts up to none at all. This behavior is due to the increase in resistivity gain over time (slope) because, fly ash replacement slows down the hydration process in the beginning. The alkaline pore solution dissolves the glass content (amorphous aluminosilicate) in fly ash once it reaches a pH of 13.2 due to initiation of cement hydration in the mixture. Then, the products of fly ash start forming which results in a reduction in capillary porosity. As such, the rapid gain in resistivity in comparison to its counterpart containing no supplementary cementitious material was further investigated to determine whether this parameter could be used to distinguish mixtures containing varying amounts in fly ash. It was found that the resistivity gain in time (slope) determined using equation 4.2.1 could differentiate mixtures containing fly ash from mixtures without SCM addition. Appendix F provides details of the statistical analysis performed to achieve the recommended categories and thresholds (Gulrez and Hartell 2018). Results of the ANOVA analysis for all possible slope combinations are presented in Appendix F.

$$s = \frac{y_2 - y_1}{x_2 - x_1} \quad (\text{Eq. 4.2.1})$$

Table 4.2.1 is the outcome of the analysis. It proposes 2 categories for determination whether a mixture contains fly ash or not based on the resistivity measurements taken on day-1 (immediately after demolding) and on day-3. The slope between the two data points can be calculated using Equation 4.2.1 and, using ranges in Table 4.2.1, the presence of fly ash in a mixture could be identified. However, there are two possible result outcomes. First, the slope value falls below the lower limits of "No Fly Ash" concrete, in this scenario the mixture could be considered as inclusive of "No Fly Ash" content however, there is no certainty in this statement as alternative scenarios were not

investigated (other SCMs for example). Second, the slope has a higher value than the upper limit of "Fly Ash" content, in this case the mixture could be considered inclusive of Fly Ash" content; however, there is no certainty in this statement as alternative scenarios were not investigated (other SCMs for example). Further investigations evaluating multiple mix designs would be required to validate both statements. The upper limit of "No Fly Ash" and lower limit of "Fly Ash" mixtures are very close to each other. However, the analysis showed a significant difference between the two categories at a 95% confidence level.

Next, the w/c classification can be determine using Table 4.2.2 or 4.2.3. The range in resistivity values representing a 95% confidence interval from the mean are provided. However, the presence of gaps between categories or the overlap of categories present zones of uncertainty. In addition, in the case of a resistivity value falling below the lower limits of "0.5 w/cm" concrete, in this scenario the mixture could be considered as "> 0.5 w/cm" however, there is no certainty in this statement. Similarly, for resistivity results higher than that of the upper limit of "0.4 w/cm" concrete, the mixture could be considered as "< 0.4 w/cm" however, there is no certainty in this statement.

It is important to mention that statistically the method is uncertain for identification of mixtures above 0.5 w/c. This means that the method cannot discern with a 95% certainty that a mixture is either a w/c of 0.5, 0.55 or 0.6 . Regardless, if a concrete mixture actually falls within this range, ODOT should not be approving the concrete because it would not meet the class A and AA specification. As such, this is a limit state where 0.5w/c could be set as an upper-bound threshold. This concept is also applicable for mixtures of and below 0.4 w/c.

This study did not evaluate mixtures below 0.4 w/c since it is below that specified by ODOT for class A and AA mixtures. As such, mixtures with values that were superior to

the range determined for the 0.4 w/c category must be categorized as uncertain since a lower category was not evaluated for this statistical analysis. Therefore, the 0.4 w/c upper-bound threshold could act as a limit state for this category. Regardless, if a concrete mixture falls within or superior to this category, ODOT should be accepting these mixtures as they meet the Class A and AA criteria.

Table 4.2.1: Range in (1-3) resistivity slope (KΩ-cm/day) combination values for concrete mixtures

| Fly Ash Content | Slope Mean | Lower Limit | Upper Limit |
|-----------------|------------|-------------|-------------|
| No Fly Ash | 0.5 | 0.4 | 0.6 |
| Fly Ash | 1.1 | >0.6 | 1.2 |

Table 4.2.2: Surface resistivity 95% confidence limits at test ages 14 and 28 days for concrete mixtures containing no fly ash

| w/cm ratio | Mean Surface Resistivity (kΩ-cm) Day-14 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-14 | Mean Surface Resistivity (kΩ-cm) Day-28 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-28 |
|------------|---|---|---|---|
| 0.40 | 11.0 | 10.6-11.5 | 12.4 | 11.9-12.8 |
| 0.45 | 10.2 | 9.7-10.6 | 11.5 | 11.0-12.0 |
| 0.50 | 8.9 | 8.5-9.4 | 10.2 | 9.7-10.6 |

Table 4.2.3: Surface resistivity 95% confidence limits at test ages 14 and 28 days for concrete mixtures containing fly ash

| w/cm ratio | Mean Surface Resistivity (kΩ-cm) Day-14 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-14 | Mean Surface Resistivity (kΩ-cm) Day-28 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-28 |
|------------|---|---|---|---|
| 0.40 | 10.7 | 10.2-11.1 | 10.7 | 12.0-13.2 |
| 0.45 | 9.3 | 8.9-9.8 | 9.3 | 10.6-11.7 |
| 0.50 | 8.7 | 8.2-9.1 | 8.7 | 9.7-10.8 |

4.2.2 Procedure B

Procedure B is a simplified version of Procedure A that does not make use of tables and slope calculations to determine a mixture design category. Instead, the user would be required to take several measurements in time to identify the concrete mixture and plot them against a predetermined set of curves.

The recommended testing ages are day 1 (immediately after demolding) and day 3 and weekly thereafter. As previously stated, the slope between day-1 and day-3 data points are statistically significant and aid in identifying the presence of fly ash. Thereafter, weekly measurements can be made (day 7, day 14 and day 28) to identify the w/c category and further aid in the comparative evaluation of the concrete mixture. When performed as part of a quality control and assurance program, the results of the test could be compared to a series of figures to establish the presence of fly ash and w/c category. As seen in Figure 4.2.1., the results of resistivity tests for sample 7 were plotted against a set of 6 curves. It can be seen that Sample 7 best fits with the 0.5 w/c – Fly Ash category. From this example, Sample 7 would be categorized as having a w/c at or above 0.5 w/c and would not satisfy the requirements for both the Class A and Class AA concrete mixture designs.

It needs to be mentioned that the curves were established from a data set composed of over 50 mixture design variations: w/c, fly ash source and content, aggregate type and gradation, cement source, admixture type. The influence of each property previously discussed are captured in these curves to prevent discrimination of an influential parameter. Thus, there is overlapping of each category. Still the established minima and maxima for each category could be used to accept or reject a concrete mixture.

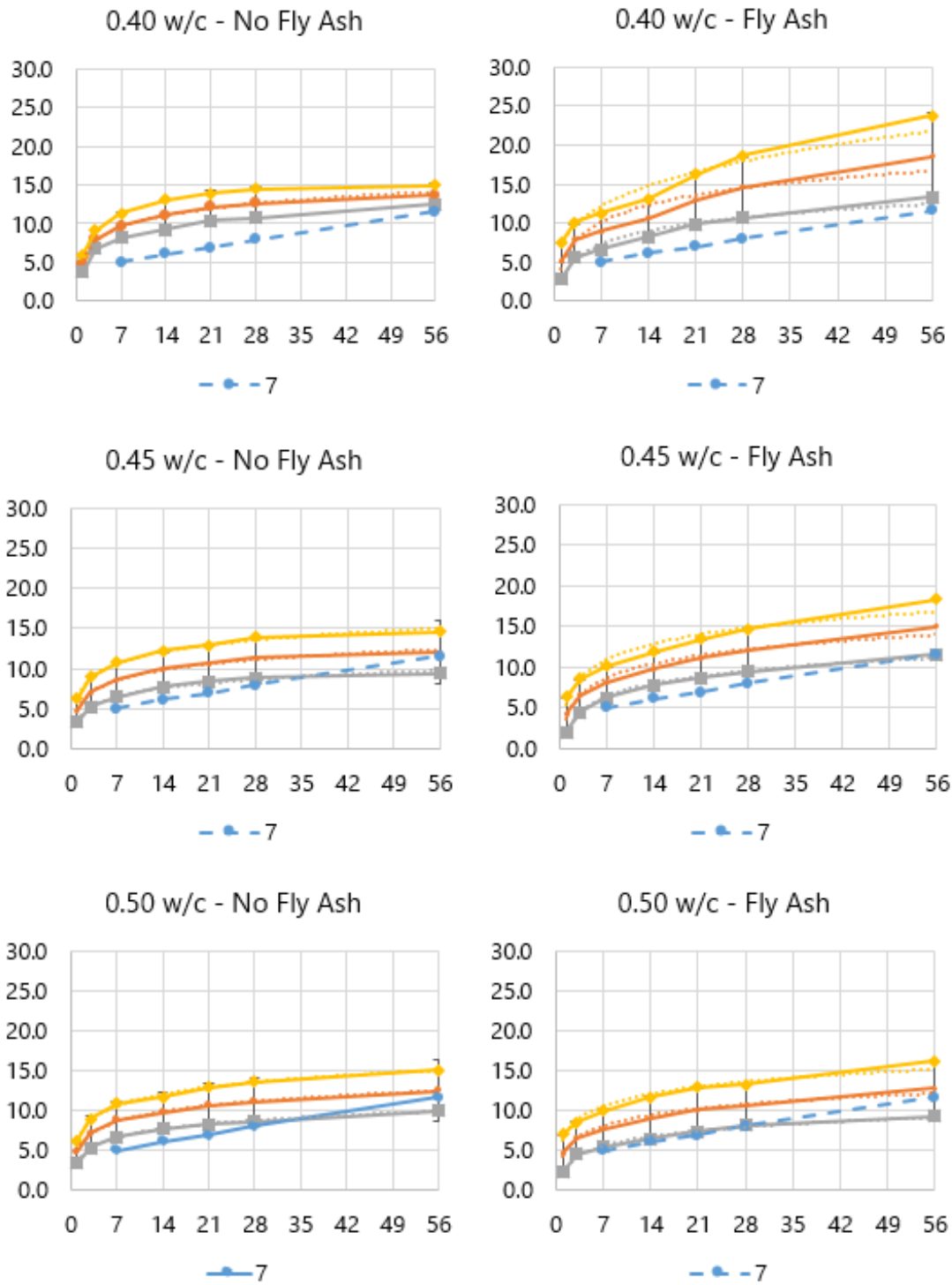


Figure 4.2.1: Example application of Procedure B for determination of mixture design.

This procedure is more intuitive and requires a certain knowledge base of the materials and their resistivity behavior in-time to recognize the appropriate category. Here, additional parameters such as air content and slump, addition of water or water reducing agent at time of casting would be beneficial to aid in the interpretation of results and better categorization of a concrete mixture.

4.2.3 Procedure C

Both Procedures A and B have inherent variability due to the nature of the resistivity test. As previously stated, resistivity is highly sensitive to the cement chemistry and maturity of concrete. In addition, it was determined that the aggregate type could influence the outcome of a test in certain mix design scenarios. As such the concrete mixture, as a whole, must be taken into account when considering its resistivity behavior and not just the binder. During this investigation it was found that for a given mixture design and materials, the results of a resistivity test and associated behavior in time is highly repeatable and distinctive for certain variations in mixture design such as w/c and SCM type and content. It captures the delicate nature of all physicochemical changes that occur in time since electrical conductivity is sensitive to both the physical aspect of the porous concrete and its chemistry. Therefore, a third approach is recommended.

Procedure C involves both the producer and the owner. It would require the concrete producer to submit a resistivity curve of a concrete mixture design (control) along with the latter's design parameters during the mix design approval stage. The owner, or its representative, would then compare its concrete sample results against the control during construction. This process can be incorporated into a QC/QA program for control and acceptance of the concrete mixture design.

Figure 4.2.2 demonstrate the application of Procedure C and its efficacy in distinguishing mixtures. Here, the Lab Sample (Control) resistivity curve is compared against that of

samples taken at a construction site during a field study in 2015. Four site samples with similar mixture design parameters (w/c, FA%, air entrainment, coarse aggregate type) as the control were selected and compared. It can be seen that the curves are similar in behavior. Section 4.3 will further elaborate on this concept and the ability of a producer to reproduce a given mixture. Figure 4.2.2. demonstrates an example of a field sample compared to its control. It can be seen that for one of the samples, the resistivity behavior are entirely different. For the purpose of identification, it is compared to that of a control of lower w/c. Based on this comparison, the mixture approved by ODOT for pavement construction (0.45 w/c, 20% FA, 6% Air) was not delivered. Instead a concrete mixture of lower quality (> 0.5 w/c) was used in the construction of the pavement.

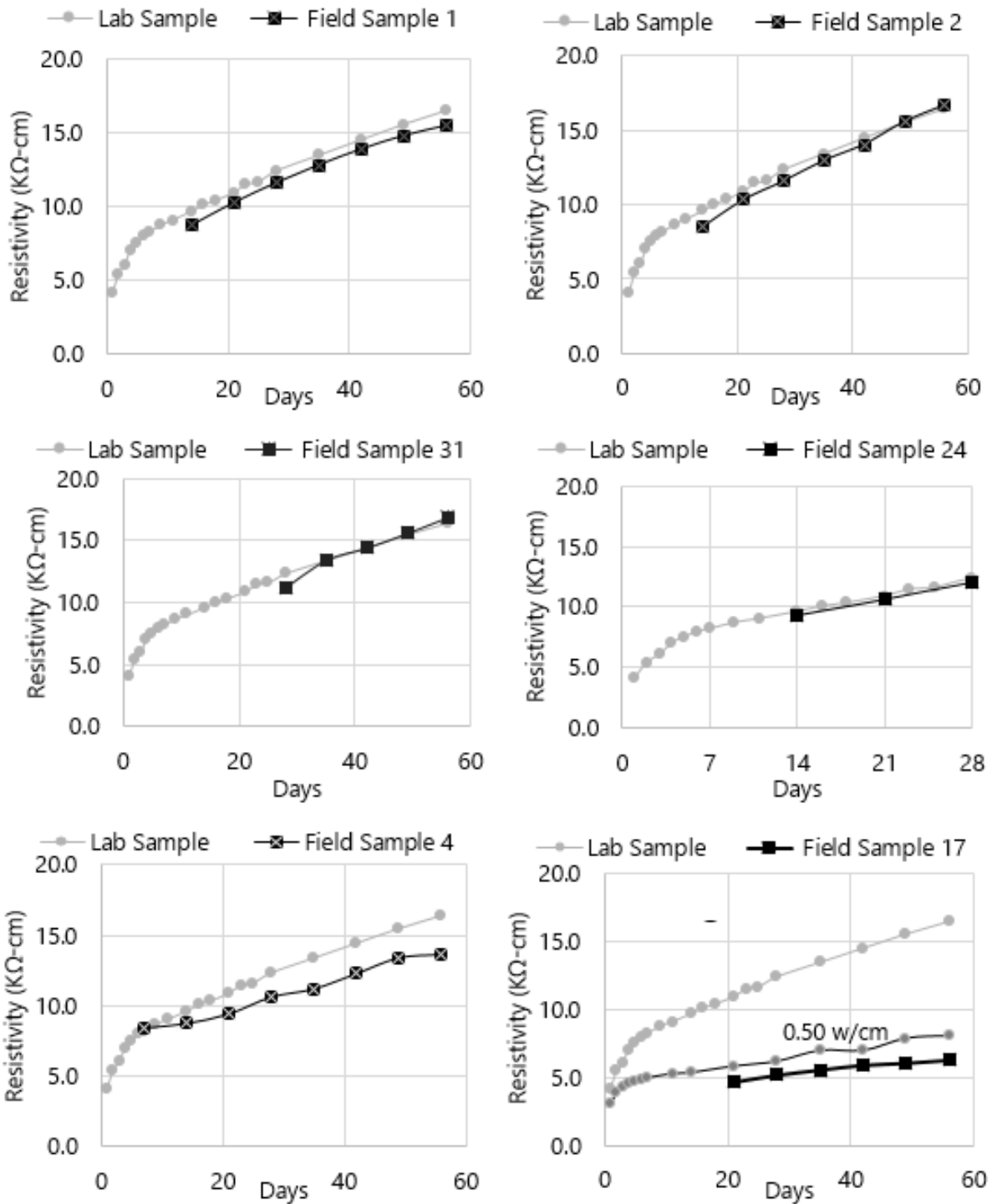


Figure 4.2.2: Example application of Procedure C: comparison of the laboratory prepared Control Sample and six field samples taken during construction of a concrete element.

4.3 Field Study

To help in assessing the effects of mixture and material variability, a state-wide testing regimen began mid-June until the end of August 2015. ODOT samples were delivered at Cooper Lab by ODOT personnel. Each set of concrete samples consists of three (Ø100 x 200 mm) concrete cylinders which represent a concrete mixture design of Class AA & A concretes. The mixture design sheet submitted by the contractor specifying the w/c, fly ash percentage and all quality compliance criteria passed and checked by ODOT were also provided for each set of samples. A summary of mixture design information is presented in Appendix G. The names of both producer and ODOT residency are not provided in this document since it was deemed confidential information. Also, plant provenance is not given. However, mixture design information as well as material supplier is provided to aid in the comparative analysis.

Most of the samples were received within the first seven days of production and were cured in the moist room at BCEL. Surface resistivity testing was performed on each set of samples starting from day-7 up to day-56 on a weekly basis. The AASHTO TP-95 procedure was adopted to conduct the surface resistivity test on field sample. According to chloride ion penetration classification, it was found that most the samples were classified as high to moderately permeable to chloride ions. For these classes of concrete mixtures, steel reinforcement may be susceptible to corrosion. However, the actual corrosion performance of the mixtures was not evaluated.

For the purpose of this study, the results of this field study were utilized to evaluate both procedure B and C proposed in Section 4.2. Procedure A could not be evaluated since it requires data from day-1 and day-3 to perform the analysis. Such was not available due to timely sample delivery. For the other Procedures, the graphical representation of time-resistivity behavior, determined through the surface resistivity

method, was used as a quality control and compliance tool to estimate the water-cement ratio and fly ash content supplied by the contractor in comparison to the mixture design specifications.

For Procedure B, the resistivity curves for each sample set were prepared and compared to the series of six graphs as that demonstrated in Figure 4.2.2. The results for each 67 samples are provided in Appendix G and summarized in table format for each mixture category. It needs to be mentioned, based on overall analysis, that Procedure B's accuracy is variable. As previously explained, the method incorporates a multitude of variates which can be significant in parameter differentiation. Therefore, there is overlap between categories making the method subjective. This point is further demonstrated and discussed in the sections below. A comment on whether the sample would be accepted or rejected according to mix design specifications for bridge deck and pavement construction is also provided (Table 4.3.1).

Table 4.3.1: Specifications for Mixture Design Acceptance

| Class of Concrete | Minimum Cement Content lb/yd ³ | Air Content % | w/c lb/lb | Slump in | Min. 28-day Comp. Str., psi |
|-------------------|---|---------------|-------------|----------|-----------------------------|
| Bridge Deck | 564 | 6.5±1.5 | 0.25 - 0.44 | 2±1 | 4,000 |
| Pavement | 517 | 6±1.5 | 0.25 - 0.48 | 2±1 | 3,000 |

To demonstrate the potential of Procedure C, the resistivity results for all samples are compared to the laboratory curve produced at OSU (Lab Control). The selected Lab Control curve is based on similarities in mix design parameters and material type to provide a near example on how Procedure C could be applied and utilized as part of a QC/QA program.

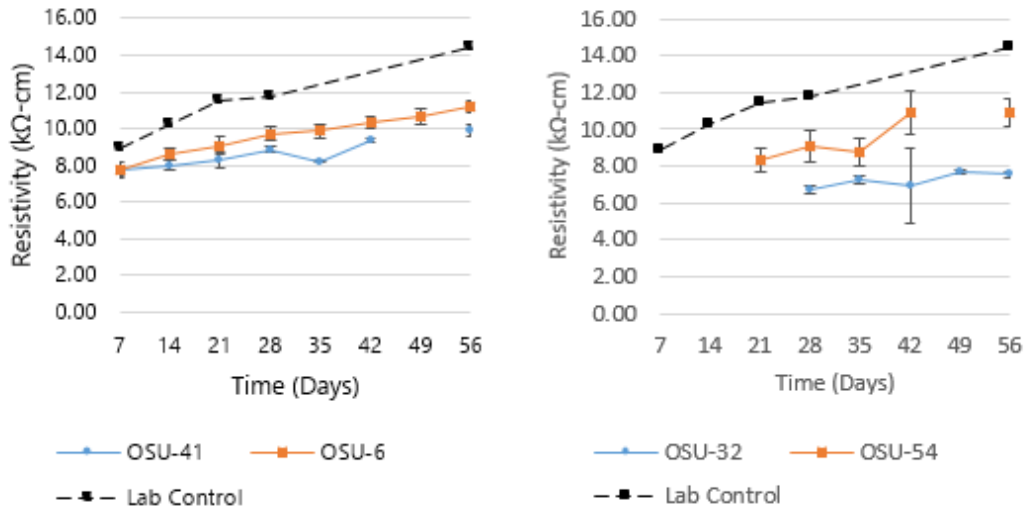
In a first attempt to evaluate Procedures B and C, producer reproducibility of a mixture design is investigated. The results of resistivity testing for the field samples, obtained from various concrete producers across Oklahoma, were categorized with similar

mixture designs; w/cm ratio and fly ash content. This classification is based on the information provided on the producer's mixture design sheet provided to ODOT (Appendix G). A total of 67 concrete mixtures were produced by 16 manufacturers and delivered to 17 different residencies. Out of 16, there were only 10 concrete producers that manufactured more than one concrete mixture with similar mixture design specifications.

Then, the overall behavior of resistivity development over time was quantitatively and qualitatively compared. To determine consistency in concrete production and reproducibility of a mixture design, age-specific statistical comparative analysis is performed using the analysis of variance method, ANOVA, for more than two data sets. A confidence level of 95% ($\alpha = 0.05$) is used to determine the significance. This statistical comparative analysis of resistivity measurements at the age of 28 and 56 days will help to determine the consistency of concrete mixtures made by each concrete producer. The standard deviation and coefficient of variation (COV) were also calculated for each data set. The following sub-sections report the results of the field study per mixture design parameter and producer.

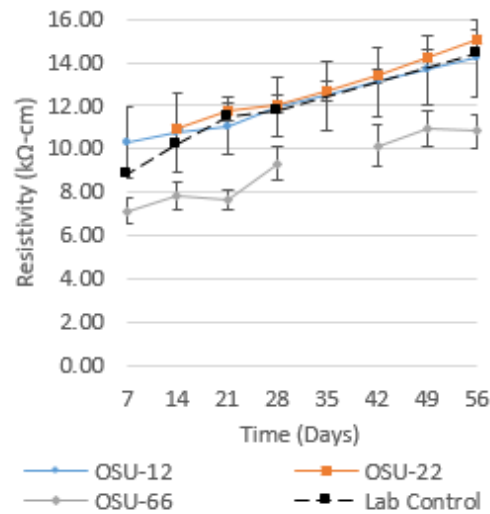
4.3.1 Mixtures with 0.37/0.39 w/cm with 0% Fly Ash

Three concrete producers (M, O, and G) supplied concrete with a w/cm of 0.37-0.39 with no fly ash for a total of 7 different mixes. An ANOVA was performed between each mix for each concrete producer at 28 and 56 days. The results of this test conclude that the null hypothesis is rejected at a significance level, alpha of 5%, meaning that there is a significant difference between the resistivity measurements of the mixtures from each producer (Tables 4.3.2 to 4.3.4). For producers M and O, the resistivity COVs obtained at 28-day and 56-day are quite low in comparison to the acceptable maximum.



a)

b)



c)

Figure 4.3.1: Resistivity-time behavior for concrete mixtures with a 0.37/0.39 w/cm and 0% FA prepared by producers: (a) M, (b) O, (c) G

In addition, peaks and valleys are observable on the resistivity curve (Figure 4.3.1), which is an artifact of temperature fluctuations at time of test. Therefore, in both instance both mixtures could be interpreted has similar in mixture design. However, for producer

M, sample 6 recorded over twice the slump as sample 41 and the resistivity values are still superior. This is indicative of the use of a water reducing agent, but there is no recorded of such. Similarly, results are observed for producer G (Table 4.3.4); however, the difference in resistivity values are more substantial. For both producers, the difference in aggregate gradation is not an influential factor as previously discussed.

Table 4.3.2: Mixture design information and test results for mixtures with a 0.39/0.38 w/cm and 0% FA, Producer M

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 41 | Limestone #57 | 6 | 4 | 8.9/9.9 | 0.06/0.18 | 1/2 | 2.0 E-3 / 5.8 E-4 |
| 6 | Limestone #67 | 6 | 9.5 | 9.7/11.2 | 0.20/0.15 | 2/1 | 2.0 E-3 / 5.8 E-4 |

Table 4.3.3: Mixture design information and test results for mixtures with a 0.39/0.37 w/cm and 0% FA, Producer O

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 32 | Limestone #57 | 5 | 2 | 6.7/7.5 | 0.10/0.08 | 2/1 | 7.9 E-4 / 1.0 E-4 |
| 54 | Limestone | 5 | 4 | 9.0/11.0 | 0.42/0.38 | 5/3 | 7.9 E-4 / 1.0 E-4 |

Table 4.3.4: Mixture design information and test results for mixtures with a 0.38 w/cm and 0% FA, Producer G

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 12 | #57 | 6.1 | 3 | 12.0/14.2 | 0.69/0.90 | 6/6 | 5.7 E-4 / 2.7 E-4 |
| 22 | #57 | 7.1 | 5 | 12.1/15.1 | 0.19/0.25 | 2/2 | 5.7 E-4 / 2.7 E-4 |
| 66 | #57 | 4.5 | 2 | 9.4/10.8 | 0.37/0.39 | 4/4 | 5.7 E-4 / 2.7 E-4 |

In the case of producer G, there is a clear observable difference for Sample 66 (Figure 4.3.1c)). The results of the statistical analysis highlight these differences. As for samples 12 and 22, the producer was consistent in mixture delivery regardless of the difference of 2 inches in slump (Table 4.3.4).

In comparison to the OSU Lab Control curve, which was prepared using similar mixture proportions, only Samples 12 and 22 from producer G are comparable. Although the resistivity gain in time is comparable (indicative of no FA), the overall resistivity values are lower. Based on the influence of w/c on resistivity, the lower resistivities recorded are apparently due to an increase in water content in the concrete mixture.

From Table 4.3.5, Procedure B yielded similar outcome to Procedure C. However, there is apparent uncertainty since the sample curves could be categorized as a 0.45 w/c or 0.50 w/c. therefore, the mixtures prepared by the procedures were not below 0.40 w/c; they were more likely above 0.45 w/c to near and above 0.50 w/c. If these mixtures were used in the fabrication of a bridge deck, they would not meet the ODOT specification. As for sample 32, it would not meet the requirements for pavement construction.

Table 4.3.5: Application of Procedure B for concrete mixtures with a 0.37/0.39 w/cm and 0%, Producers, M, O and G

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| M | 6 | 0.38 | 0% | 0.45-0.50 | N | | Y |
| M | 41 | 0.39 | 0% | 0.45-0.50 | N | Y | Y |
| O | 32 | 0.39 | 0% | >0.50 | N | | N |
| O | 54 | 0.37 | 0% | 0.45-0.50 | M | Y -FA% | Y |
| G | 12 | 0.38 | 0% | 0.45-0.50 | N | | Y |
| G | 22 | 0.38 | 0% | 0.40-0.50 | M | Y | Y |
| G | 66 | 0.38 | 0% | 0.45-0.50 | N | Y | Y |

4.3.2 Mixtures of 0.375/.38 w/cm with 20% Fly Ash

The next set of samples have a 0.375 or 0.38 w/cm with 20% fly ash replacement from two different concrete producers (A and G). According to the ANOVA test, there is significant difference between mixes from each producer at 28 and 56 days.

Starting with producer A, the OSU 56 mix varied in resistivity quite significantly from the other mixes. This behavior was not observed for any of the laboratory mixtures studied herein. It is unclear what could have been the cause of the increase in resistivity behavior. An hypothesis, a change in SCM type or a fly ash replacement superior to 20% content could have resulted in the substantial increase. For another study supervised by Hartell, similar resistivity values and behavior were found for mixtures containing 40% fly ash replacement (Banadkoki and Hartell, 2018). Producer G fabricated similar mixtures, where samples 5, 18 and 28 recorded comparable values to that of sample 56, Producer A. Here, it would be beneficial to have further information on the actual materials used in the fabrication of these concrete mixtures since concrete of superior quality based on resistivity value alone were delivered. The mixture came from three different residencies.

For other samples by producer A, sample 50 produced acceptable results in comparison to the Lab Control Curve; followed by samples 30A and 30B (both from the same mix sample 30) and sample 26. Sample 26 recorded lower resistivity values for the majority of the test period. Based on the resistivity behavior, the lower values are indicative of an increase in water content. Here, both sample 30 and 26 would have suffered from water addition potentially resulting in a concrete of lower quality. Both mixtures 50 and 26 were provided by the same residency as such, another indication in the variability of production of Producer A. On the other hand, producer G delivered mixtures with repeatable resistivity behavior. The range in mixtures were provided by 2 residencies. This is a good demonstration of consistency and that a concrete producer can fabricate

and deliver the same mix over time. In addition, the resistivity behavior of the sampled concrete follows that of the Lab Control. Values are slightly lower (Figure 4.3.3), which could be due to increased w/c, but the mix would still be acceptable for a Class A and Class AA mixtures. This is a good example of the application of Procedure C for the comparison and approval of mixtures produced by a concrete supplier.

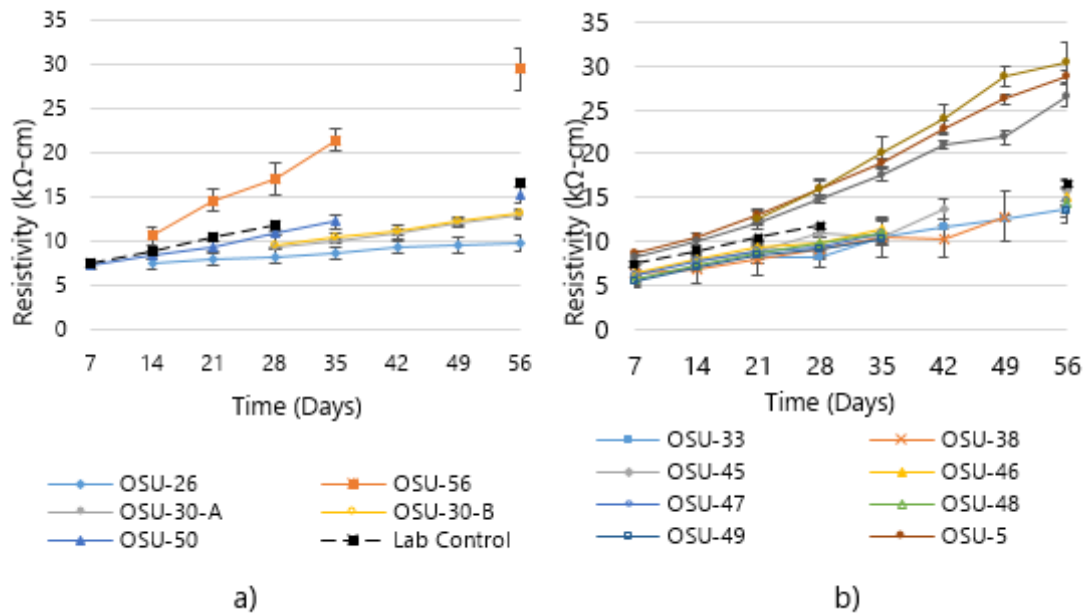


Figure 4.3.2: Resistivity-time behavior for concrete mixtures with a 0.375/0.38 w/cm and 20% FA prepared by: (a) Producer A and (b) Producer G

Table 4.3.6: Mixture design information and test results for mixtures with a 0.375 w/cm and 20% FA, Producer A

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 26 | Limestone | 4.8 | 1.25 | 8.30/9.80 | 0.35/0.45 | 4/5 | 8.6 E-9 / 3.3 E-11 |
| 56 | Limestone | 6 | 1.5 | 17.1/29.5 | 0.86/1.24 | 5/4 | 8.6 E-9 / 3.3 E-11 |
| 30A | Limestone | 6 | 1.5 | 9.3/13.0 | 0.05/0.21 | 1/2 | 8.6 E-9 / 3.3 E-11 |
| 30B | Limestone | 6 | 1.5 | 9.6/13.1 | 0.63/0.23 | 7/2 | 8.6 E-9 / 3.3 E-11 |
| 50 | Limestone | 6 | 1 | 11.0/15.3 | 0.13/0.43 | 1/3 | 8.6 E-9 / 3.3 E-11 |

Table 4.3.7: Mixture design information and test results for mixtures with a 0.38 w/cm and 20% FA, Producer G

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (K Ω -cm) | 28-day / 56-day Std. Dev. Resistivity (K Ω -cm) | 28-day / 56-day COV (%) Resistivity (K Ω -cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|--|--|--|-------------------------|
| 33 | #57 | 6 | 6.5 | 8.2/13.7 | 0.09/0.20 | 1/1 | 1.6 E-15 / 2.4 E-17 |
| 38 | #57 | 5 | 7 | 9.1/- | 0.96/- | 11/- | 1.6 E-15 / 2.4 E-17 |
| 45 | #57 | 5 | 7 | 10.9/15.7 | 0.21/0.56 | 2/4 | 1.6 E-15 / 2.4 E-17 |
| 46 | #57 | 5 | 7 | 9.8/15.1 | 0.33/0.59 | 3/4 | 1.6 E-15 / 2.4 E-17 |
| 47 | #57 | 5 | 5.5 | 9.3/14.6 | 0.57/1.28 | 6/9 | 1.6 E-15 / 2.4 E-17 |
| 48 | #57 | 5 | 5 | 9.8/14.3 | 0.55/0.8 | 6/6 | 1.6 E-15 / 2.4 E-17 |
| 49 | #57 | 5 | 4 | 9.2/13.5 | 0.14/0.34 | 2/3 | 1.6 E-15 / 2.4 E-17 |
| 5 | #67 | 6.25 | 3 | 16.0/28.7 | 0.41/0.36 | 3/1 | 1.6 E-15 / 2.4 E-17 |
| 28 | #67 | 7 | 5 | 14.9/26.6 | 0.22/0.63 | 1/2 | 1.6 E-15 / 2.4 E-17 |
| 18 | #57 | 7 | 3 | 16.0/30.4 | 0.60/1.10 | 4/4 | 1.6 E-15 / 2.4 E-17 |

Table 4.3.8: Application of Procedure B for concrete mixtures with a 0.375/0.38 w/cm and 20%, Producers A and G.

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| A | 26 | 0.375 | 20% | 0.50 | M | Y – FA % | N |
| A | 56 | 0.375 | 20% | 0.40 | Y | | N |
| A | 30 | 0.375 | 20% | 0.45-0.50 | Y | | N |
| A | 50 | 0.375 | 20% | 0.40-0.50 | Y | Y – w/c | N |
| G | 33 | 0.38 | 20% | 0.50 | Y | | N |
| G | 38 | 0.38 | 20% | 0.50 | M | | N |
| G | 45 | 0.38 | 20% | 0.45-0.50 | Y | Y – w/c | N |
| G | 46 | 0.38 | 20% | 0.45-0.50 | Y | Y – w/c | N |
| G | 47 | 0.38 | 20% | 0.45-0.50 | Y | Y – w/c | N |
| G | 48 | 0.38 | 20% | 0.45-0.50 | Y | Y – w/c | N |
| G | 49 | 0.38 | 20% | 0.50 | Y | | N |
| G | 5 | 0.38 | 20% | 0.40 | Y | | Y |
| G | 28 | 0.38 | 20% | 0.40 | Y | | Y |

Table 4.3.8 presents the results of applied Procedure B. The results indicate that only one sample would be rejected, sample 26, as it is clearly categorized as a 0.5 w/c. as for samples 30 and 50, the Procedure C approach would provide a more accurate representation on whether it respects the specifications for a Class A or Class AA. Here the mixtures would satisfy the minima requirement of a 0.4 and 0.45 w/c (Procedure B)

but it is most likely a 0.5 w/c mixture as it is nearest to the average of that category. The same can be interpreted for mixtures fabricated by Producer G. Although the 0.45 w/c minima is satisfied for samples 45 to 48, these are more likely to be of higher w/c. Here, Procedure C could be the distinguishing factor for increased accuracy. Still the method was able to distinguish 4 samples which would not satisfy the requirements for Class A and Class AA mixtures.

4.3.3 Mixtures with 0.41 w/cm with 20% Fly Ash

The results of the statistical analysis performed for Producer F shows that both mixtures are the same (Table 4.3.9). Samples 1 and 2 produced on subsequent days also follows the Lab Control curve produced at OSU (Figure 4.3.3a). This is a great example of how Procedure C could be applied. Looking at the results for Procedure B, the results are variable, as the mixture could be classified as a 0.40 or a 0.45 w/c. Still, the mixtures would be approved for the fabrication of pavements and bridge decks.

In the case of Producer F, delivery in concrete was consistent according to the results of the statistical analysis (Table 4.3.10). This behavior can also be seen on 4.3.3 b). However, it is also noticeable that the resistivity values are significantly lower than the Control. With a similar trend in resistivity gain as that of the control, the loss in resistivity for samples 58 and 59 can be attributed to an increase in w/c. According to the classification using Procedure B (Table 4.3.11), the concrete mixture is classified as a 0.45 w/c to 0.5 w/c. Although it may be acceptable for pavement construction, it would not satisfy the criteria for bridge deck construction.

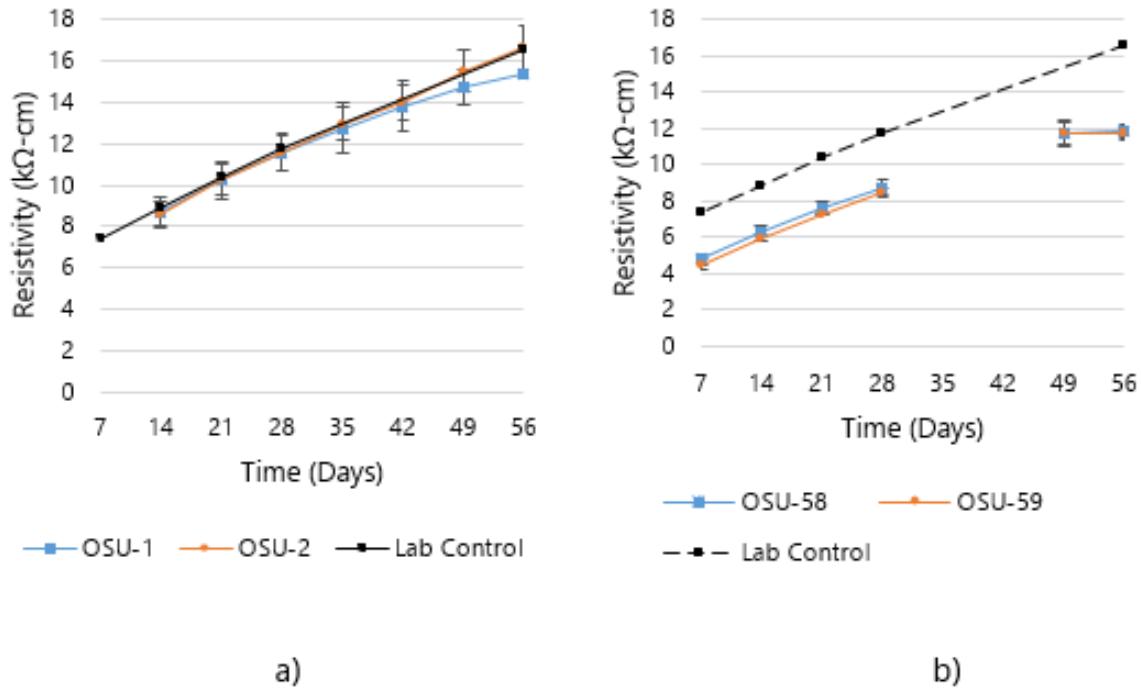


Figure 4.3.3: Resistivity-time behavior for concrete mixtures with a 0.41 w/cm and 20% FA prepared by: (a) Producer K and (b) Producer F

Table 4.3.9: Mixture design information and test results for mixtures with a 0.41 w/cm and 20% FA, Producer K

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 1 | Limestone #57 | 6.8 | 2 | 11.6/15.4 | 0.42/0.03 | 4/0 | 1 / 1.7 E-2 |
| 2 | Limestone #57 | 4.6 | 1.25 | 11.6/16.6 | 0.46/0.53 | 4/3 | 1 / 1.7 E-2 |

Table 4.3.10: Mixture design information and test results for mixtures with a 0.41 w/cm and 20% FA, Producer F

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 58 | #67 | 6 | 5 | 8.8/11.8 | 0.20/0.19 | 2/2 | 9.0 E-2 / 7.4 E-1 |
| 59 | #67 | 6 | 6 | 8.5/11.8 | 0.13/0.15 | 2/1 | 9.0 E-2 / 7.4 E-1 |

Table 4.3.11: Application of Procedure B for concrete mixtures with a 0.41 w/cm and 20% FA, Producers F and K.

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| F | 58 | 0.41 | 20% | 0.45-0.50 | Y | | N |
| F | 59 | 0.41 | 20% | 0.45-0.50 | Y | | N |
| K | 1 | 0.41 | 20% | 0.40-0.45 | Y | | Y |
| K | 2 | 0.41 | 20% | 0.40-0.45 | Y | | Y |

4.3.4 Mixtures with 0.42 w/cm with 15% Fly Ash

Seen on Figure 4.3.4, the five concrete samples taken for Producer J are variable. The observable differences is confirmed by the low p-value obtained from the ANOVA test. Although mixture designs provided for all five samples are the same, samples came from three different residencies. Plant provenance is unknown. Observable similarities between samples 8, 13 and 14 are irrespective of residency.

With respect to the Lab Control, which represents a 0.45w/c with 15% FA content, only sample 13 is comparable to the control. All other samples demonstrate similar gains in resistivity, which is indicative of similarities in FA content, however, the resistivity values are lower. Again, this is an indication of an increase in water content for the mixtures. Critically, Sample 57 recorded some of the lowest resistivity values of for this field study. This is a good demonstration of the efficacy of the resistivity method for discerning concrete that does not meet the specifications and potentially impair the service life of the constructed element.

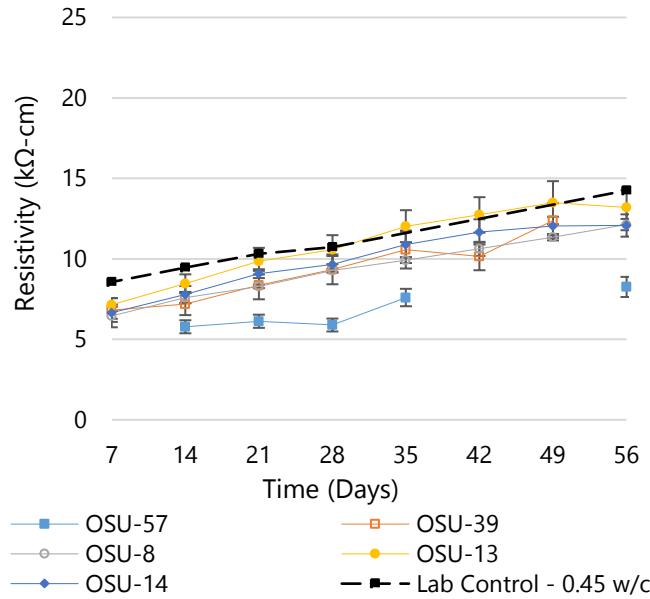


Figure 4.3.4: Resistivity-time behavior for concrete mixtures with a 0.42 w/cm and 15% FA prepared by Producer J

Table 4.3.12: Mixture design information and test results for mixtures with a 0.42 w/cm with 15% FA, Producer J

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 57 | Limestone #67 | 6.0 | 3.75 | 5.9/8.3 | 0.20/0.31 | 3/4 | 8.0 E-8 / 9.3 E-7 |
| 39 | Limestone | 6.0 | 3.5 | 9.4/ - | 0.46/- | 5/- | 8.0 E-8 / 9.3 E-7 |
| 8 | Limestone | 7.4 | 2.5 | 9.3/12.1 | 0.05/0.18 | 1/1 | 8.0 E-8 / 9.3 E-7 |
| 13 | Limestone | 6.2 | 1.75 | 10.6/13.2 | 0.45/0.54 | 4/4 | 8.0 E-8 / 9.3 E-7 |
| 14 | Limestone | 5.7 | 2.0 | 9.7/12.1 | 0.26/0.35 | 3/3 | 8.0 E-8 / 9.3 E-7 |

As for the application of procedure B, the results demonstrate that the samples are in fact of low w/c. Sample 57 is categorized as >0.5w/c. the values obtained are reminiscent of mixtures of 0.55 w/c to 0.6 w/c. This should not be acceptable for the construction of pavement and bridge decks. Sample 39 closely follows, and the rest could be classified as 0.45 to 0.50 w/c. This is in agreement with that observed on Figure 4.3.4.

Table 4.3.13: Application of Procedure B for concrete mixtures with a 0.42 w/cm with 15% FA, Producer J

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| J | 57 | 0.42 | 13% | >0.5 | M | Y-FA% | N |
| J | 39 | 0.42 | 15% | 0.50 | Y | Y-FA% | N |
| J | 8 | 0.42 | 15% | 0.45-0.50 | Y | Y-w/c | N |
| J | 13 | 0.42 | 15% | 0.45-0.50 | Y | Y - w/c | N |
| J | 14 | 0.42 | 15% | 0.45-0.50 | Y | | N |

4.3.5 Mixtures with 0.44 w/cm with 0% Fly Ash

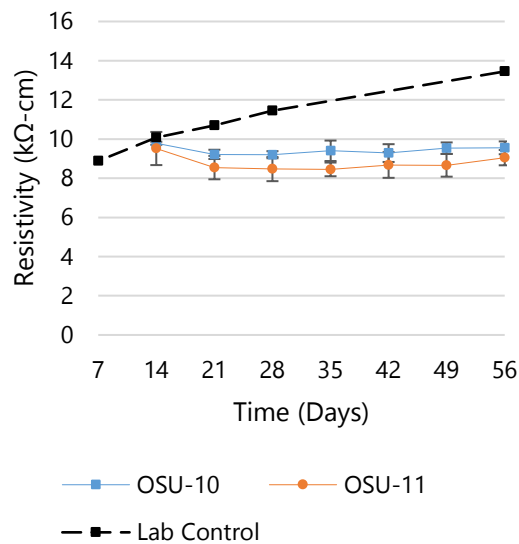


Figure 4.3.5: Resistivity-time behavior for concrete mixtures with a 0.44 w/cm and 0% FA prepared by Producer C

As previously discussed for Producer J, similar conclusion can be made for Producer C. The results of the statistical analysis are moderately significant, making the producer somewhat uniform in the delivery of concrete. However, both samples suffer from low resistivity values. The lack in resistivity gain over time is reminiscent of concrete of high w/c with no SCM. Procedure B confirms this assumption where both mixtures are barely categorized as 0.50 w/c. In both instances, these would not be acceptable in the

construction of bridge decks, and barely meet the requirements for pavement construction.

Table 4.3.14: Mixture design information and test results for mixtures with a 0.44 w/cm with 0% FA, Producer C

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 10 | - | 7.1 | 7.5 | 9.2/9.6 | 0.09/0.16 | 1/2 | 1.7 E-2 / 2.7 E-2 |
| 11 | - | 6.9 | 5.25 | 8.5/9.1 | 0.31/0.16 | 4/2 | 1.7 E-2 / 2.7 E-2 |

Table 4.3.15: Application of Procedure B for concrete mixtures with a 0.44 w/cm with 0% FA, Producer C

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| C | 10 | 0.44 | 0% | 0.5 | N | | N |
| C | 11 | 0.44 | 0% | 0.5 | N | | N |

4.3.6 Mixtures with 0.44 w/cm with 15% Fly Ash

Producer D was not consistent in its delivery of concrete. Figure 4.3.6 shows significant differences between samples, which is also corroborated by the statistical analysis (Table 4.3.16). Sample 24 recorded the highest values; in fact, greater than the Lab Control.

Based on the difference in resistivity gain and slight increase in resistivity, an increase in FA content and decrease in w/c could be the cause for that noticed. This is confirmed in from Procedure B and the mix design sheet indicates an FA content of 19%.

As for the other mixtures, the slopes of the resistivity curve are similar to the control; but there is a decrease in resistivity. Procedure B categorizes samples 25 and 67 in 0.45-0.5 w/c and >0.5 w/c respectively, making sample 67 unacceptable (Table 4.3.17). In this case, both procedures (B and C) differentiated sample mixtures.

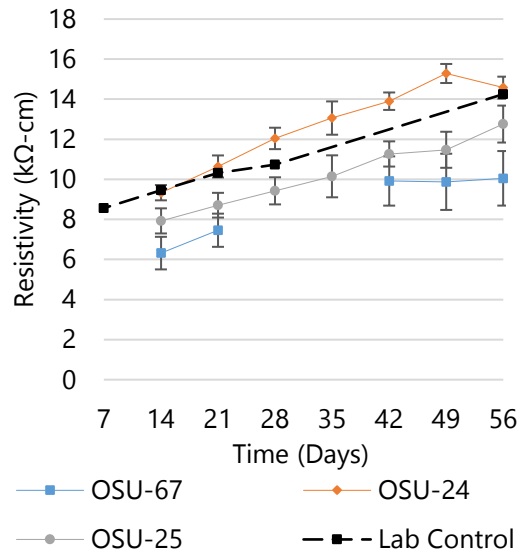


Figure 4.3.6: Resistivity-time behavior for concrete mixtures with a 0.44 w/cm and 15% FA prepared by Producer D

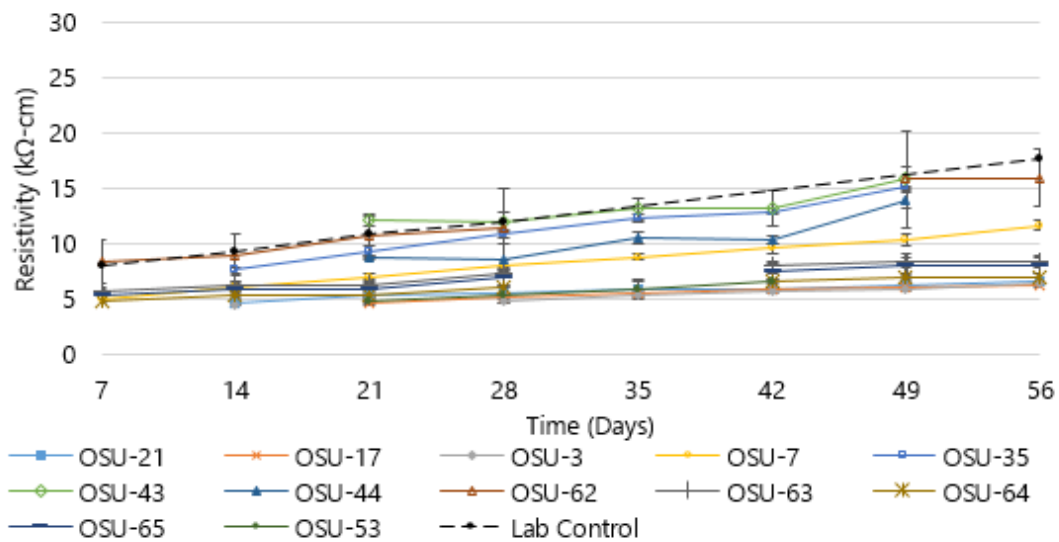
Table 1: Mixture design information and test results for mixtures with a 0.44 w/cm and 15% FA, Producer D

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 67 | Limestone | 6 | 4.5 | -/10.1 | -/0.68 | -/7 | 4.6 E-4 / 9.8 E-5 |
| 24 | #67 | 6.5 | 6.25 | 12.0/14.6 | 0.27/0.28 | 2/2 | 4.6 E-4 / 9.8 E-5 |
| 25 | Limestone | 5 | 6.5 | 9.4/12.8 | 0.34/0.46 | 4/4 | 4.6 E-4 / 9.8 E-5 |

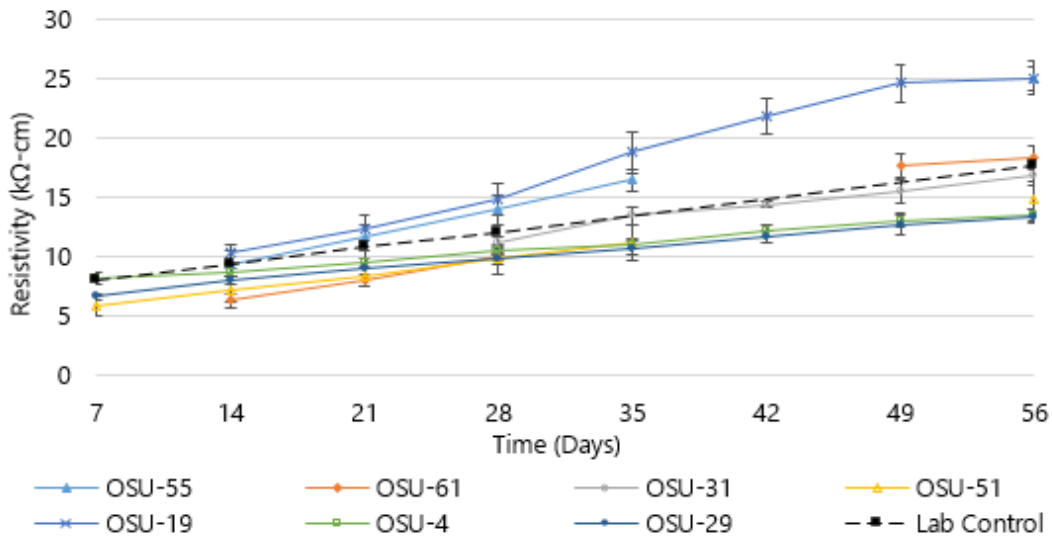
Table 4.3.17: Application of Procedure B for concrete mixtures with a 0.44 w/cm with 15% FA, Producer D

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| D | 67 | 0.44 | 15% | >0.50 | Y | | N |
| D | 24 | 0.43 | 19% | 0.40-0.45 | Y | | Y |
| D | 25 | 0.44 | 15% | 0.45-0.50 | Y | | N |

4.3.7 Mixtures with 0.44 w/cm with 20% Fly Ash



a)



b)

Figure 4.3.7: Resistivity-time behavior for concrete mixtures with a 0.44 w/cm and 20%

FA prepared by: (a) Producer E and (b) Producer G

The greatest number of samples were taken from Producer E construction sites. It can be seen from Figure 4.3.7a), that the production of a standard mixture commonly used in the construction of bridge decks, 0.44 w/c with 20% fly ash replacement, was variable. There are several factors that can contribute to the noticeable inconsistency in concrete production. The samples were provided by 2 residencies, 1 and 4. For residency 1, the provenance of concrete was from 4 concrete plants. And, for residency 4, the provenance is from 2 plants. For each plant there are differences in the source of materials. The some of the sources were evaluated in this study for their effect on resistivity and it was found not to be a contributing factor. Therefore, changes in mixture design are deemed the culprit for the low resistivity values measured for both concrete producers.

Starting with samples from residency 1, there is a perceived difference in 28-day resistivity which may be substantial for samples 7 and 35. In this case, the valley seen at 28-day from the resistivity curve may be an artifact of temperature at time of test and may be misleading for comparative analysis. The results of Procedure B are distinct. Sample 35 would be deemed acceptable while sample 7 is classified superior to 0.50 w/c. As for samples 63 to 65 coming from the same plant, their resistivity behaviors are similar. All three samples recorded resistivity well below the control. Similar to sample 7, the resistivity behavior would classify these mixtures as >0.50 w/c. In addition, the identification of fly ash presence is uncertain due to the low measurements. As such, all three samples would not meet the specification for pavement and deck construction. On-the-other-hand, Sample 43 is deemed acceptable but, the second sample from the same plant (44) is slightly lower. Both samples did not have enough data points to appropriately classify the mixture type.

Table 4.3.18: Mixture design information and test results for mixtures with a 0.44 w/cm and 20% FA, Producer E

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 7 | Limestone #57 | 6 | 8 | 8.0/11.6 | 0.25/0.21 | 3/2 | 2.6 E-14 / 3.7 E-13 |
| 35 | Limestone #67 | 5 | 3 | 10.9/- | 0.15/- | 1/- | 2.6 E-14 / 3.7 E-13 |
| 63 | Limestone #67 | 5 | 4.75 | 7.3/8.3 | 0.22/0.29 | 3/3 | 2.6 E-14 / 3.7 E-13 |
| 64 | Limestone #67 | 5 | 6.5 | 6.1/7.0 | 0.04/0.16 | 1/2 | 2.6 E-14 / 3.7 E-13 |
| 65 | Limestone #67 | 5 | 5.5 | 7.0/8.0 | 0.36/0.35 | 5/4 | 2.6 E-14 / 3.7 E-13 |
| 43 | Limestone #67 | 5 | 3 | 12.0/- | 0.43/- | 4/- | 2.6 E-14 / 3.7 E-13 |
| 44 | Limestone #67 | 5 | 3 | 8.6/- | 0.66/- | 08/- | 2.6 E-14 / 3.7 E-13 |
| 62 | Limestone #67 | 5 | 8 | 11.3/16.0 | 1.79/1.30 | 16/8 | 2.6 E-14 / 3.7 E-13 |
| 21 | Limestone #67 | 8 | 3 | 5.5/6.5 | 0.05/0.02 | 1/0 | 2.6 E-14 / 3.7 E-13 |
| 17 | Limestone #67 | 5.6 | 3 | 5.2/6.3 | 0.03/0.11 | 1/2 | 2.6 E-14 / 3.7 E-13 |
| 3 | Limestone #67 | 5.5 | 6 | 4.9/6.4 | 0.09/0.03 | 2/2 | 2.6 E-14 / 3.7 E-13 |
| 53 | Limestone #67 | 5 | 3 | 5.3/- | 0.33/- | 6/- | 2.6 E-14 / 3.7 E-13 |

Table 4.3.19: Mixture design information and test results for mixtures with a 0.44 w/cm and 20% FA, Producer G

| Mix | Coarse Aggregate | Air (%) | Slump (in) | 28-day / 56-day Average Resistivity (KΩ-cm) | 28-day / 56-day Std. Dev. Resistivity (KΩ-cm) | 28-day / 56-day COV (%) Resistivity (KΩ-cm) | p-value 28-day / 56-day |
|-----|------------------|---------|------------|---|---|---|-------------------------|
| 61 | | 4.5 | 3 | 9.9/18.4 | 0.16/0.50 | 2/3 | 2.0 E-9 / 9.7 E-14 |
| 19 | #57 | 7.4 | 6 | 14.8/25.1 | 0.69/0.75 | 5/3 | 2.0 E-9 / 9.7 E-14 |
| 55 | #57 | 4.5 | 5 | 14.0/25.0 | 0.62/0.49 | 4/2 | 2.0 E-9 / 9.7 E-14 |
| 31 | Dolomite #57 | 5.5 | 3 | 11.2/16.8 | 0.25/0.38 | 2/2 | 2.0 E-9 / 9.7 E-14 |
| 51 | Dolomite #57 | 4.5 | 4.5 | 9.8/14.8 | 0.63/0.75 | 6/5 | 2.0 E-9 / 9.7 E-14 |
| 4 | #57 | 6.2 | 2.5 | 10.5/13.5 | 0.17/0.25 | 2/2 | 2.0 E-9 / 9.7 E-14 |
| 29 | #57 | 6 | 1.75 | 9.8/13.4 | 0.23/0.30 | 2/2 | 2.0 E-9 / 9.7 E-14 |

For residency 4, all four samples are among the lowest resistivity results recorded for this study. Samples 21 and 17 were produced by the same plant; likewise, for samples 3 and 53. None-the-less, all for samples demonstrate resistivity behaviors reminiscent of mixtures of 0.55 w/c and 0.60w/c. At these levels, it is uncertain if fly ash was added to the concrete mixtures. Based on Procedure B and C, only one concrete delivery from

producer E (sample 62) would be classified as acceptable in the construction of bridge deck. The concrete would not even be adequate for pavement construction neither. The consequence of constructing with poor quality concrete is a diminished durability performance. These mixtures would be considered as having a low resistance to chloride ion ingress which can lead to an accelerated rate of reinforcement corrosion among other concrete deterioration issues.

Table 4.3.20: Application of Procedure B for concrete mixtures with a 0.44 w/cm with 20% FA, Producers E and G

| Producer | Samples OSU | Reported w/c | Reported FA (%) | Estimated w/c | Estimated FA (%) | Uncertainty in Analysis | Conformity Mix Des. |
|----------|-------------|--------------|-----------------|---------------|------------------|-------------------------|---------------------|
| E | 7 | 0.44 | 20% | >0.50 | Y | | N |
| E | 35 | 0.44 | 20% | 0.45-0.50 | Y | | N |
| E | 63 | 0.44 | 20% | >0.50 | M | Y – FA% | N |
| E | 64 | 0.44 | 20% | >0.50 | N | Y – FA% | N |
| E | 65 | 0.44 | 20% | >0.50 | M | Y – FA% | N |
| E | 43 | 0.44 | 20% | - | - | | |
| E | 44 | 0.44 | 20% | - | - | | |
| E | 62 | 0.44 | 20% | 0.45 | Y | | Y |
| E | 21 | 0.44 | 20% | >0.50 | M | Y – FA% | N |
| E | 17 | 0.45 | 20% | >0.50 | M | Y – FA% | N |
| E | 3 | 0.44 | 20% | >0.50 | M | Y – FA% | N |
| E | 53 | 0.44 | 20% | >0.50 | M | Y – FA% | N |
| G | 61 | 0.44 | 20% | - | - | | N |
| G | 19 | Y | 20% | 0.40 | Y | | Y |
| G | 55 | 0.44 | 20% | 0.40 | Y | | Y |
| G | 31 | 0.44 | 20% | 0.40-0.45 | Y | Y – w/c | Y |
| G | 51 | 0.44 | 20% | 0.45-0.50 | Y | Y – w/c | N |
| G | 4 | 0.44 | 20% | 0.45-0.50 | Y | | N |
| G | 29 | 0.44 | 20% | >0.5 | Y | | N |

In comparison, producer G was slightly more uniform between production sights but variable overall. However, concrete mixtures were of higher resistivity (Table 4.3.18). It can be seen that samples 19, 55 and 61 present a superior resistivity behavior than the control's. Both 19 and 55 are from the residency 5 and their results are comparable. They would be classified as 0.40-0.45 w/c with FA following Procedure B. Samples 31 and 51 are from residency 15, and both distinct. Sample 51 would be classified at a

higher w/c than that designated by the mixture design. Still both samples would be deemed acceptable with respect to the specifications; likewise, for samples 4. The latter was delivered from residency 17. Its companion, sample 29, also from residency 17, did not perform as well. It would be classified as >0.5 making it the only sample from Producer G rejected according to Procedure B (Table 4.3.20).

4.3.8 Conclusions

Concrete is a composite material, which undergoes health problems mainly due to exposure conditions. The timeline for visible evidence of durability deterioration depends on the quality of the concrete, mainly mixture design. Current practice requires mixture design approval based on submittal of mix design sheet prior to start of construction, and quality assurance testing (air, slump, strength) is commonly conducted during construction. Maintaining a level of quality of concrete, especially the uniformity in concrete mixtures produced, can be challenging. This field study evaluated the consistency of concrete mixtures produced by various concrete producers at over a 2-month period. Using 2 proposed procedures (B and C) as a surface resistivity method, the ability of a producer to deliver a concrete confirming the mix design submitted was evaluated. In addition, the estimated w/c and FA% from the determined resistivity behavior enabled comparison with ODOT specifications for Class A and Class AA concrete mixtures.

From 67 samples tested, only 54 were evaluated due to repeatability in production of the same mixture design. Concrete producer G manufactured 20 concrete mixtures, delivered to 6 residencies in Oklahoma. The time-resistivity curves and statistical analysis have shown that the producer may not be able to maintain uniformity but, according to Procedure B, 11 of the 20 samples would be classified as similar to the mix design submitted. Moreover, only 5 samples would have been rejected according to Procedure

B. This estimation could be improved with the implementation of Procedure C. In this case, it was clear that the majority of the mixtures presented a resistivity behavior below that expected.

As for the other producers, the outcome of the study provides a different picture. Concrete producer E manufactured 12 samples across 6 plants, which were delivered to 2 residencies. From the 12 samples, only samples would qualify as similar to the mix design submitted. 10 of the samples would actually be rejected as they did not meet the maximum w/c specified for pavement and bridge deck construction. Here both Procedure B and Procedure C were helpful in discerning the mixtures or lower performance. For the remaining 22 samples evaluated, only 3 samples would be estimated as representative of the mixture design submitted. 7 samples would be rejected based on Procedure B criteria. The remaining 12 samples presented resistivity behaviors below that expected according to their respective control samples, but they may be deemed acceptable for pavement construction, and bridge deck construction for a few.

The results of this field study showed that there is a need for a better QC/QA tool for the acceptance of concrete during construction. Here, Procedure B and C demonstrated promise, but greater reliability could be achieved using Procedure C. This would require both producer and owner to participate in the implementation process to ensure a successful outcome.

4.4 Secondary Testing for Quality Acceptance of Concrete Mixtures

Surface resistivity testing is a true non-destructive technique which does not alter or damage the surface and integrity of the element tested. This is a great advantage over other means of secondary compliance testing which generally require coring a sample from the structure to be tested in the laboratory. Then, the damaged area requires repair which may not be as performant as the original material. Also, report of findings may take several days which may be an inconvenience when time is constrained. However, variances in the degree of saturation and temperature of the concrete have a large influence on the reliability of the result when a calibrated measurement is required. For these reasons, there is a need for investigating potential procedures which may be deemed acceptable in the event of secondary compliance testing where calibrated measurements are required.

The procedure devised explores two resistivity testing methods and their viability as part of a QC/QC program in the event that the laboratory control failed to meet the acceptance criteria. Both non-destructive evaluation (direct surface measurement) and destructive evaluation (coring) are investigated. The results of the study are presented in Appendix H.

4.4.1 Analysis of Surface Resistivity Testing on Cores

The first method investigated is the possibility of using surface resistivity on cores taken from a concrete element. For the comparative study, a set of slab specimens and companion control cylinders were prepared as that described in Section 3.4.3. First, it needs to be mentioned that for the set of control samples, the results are in good agreement with the resistivity time behaviors previously discussed in Section 4.1. The coefficients of variation for the control cylinders were in acceptable ranges, except for the day-28 control cylinder (10.3%) (Table H1). Figure 4.4.1 demonstrates the results of

the surface resistivity for the control cylinder in comparison to each core set taken from slabs for the respective curing time. For example, the cores taken from a slab that cured for 7 days is compared to the control cured for 7 days. Two resistivity values per core are given: immediately after coring (before vacuum saturation) and the following day, after vacuum saturation in limewater.

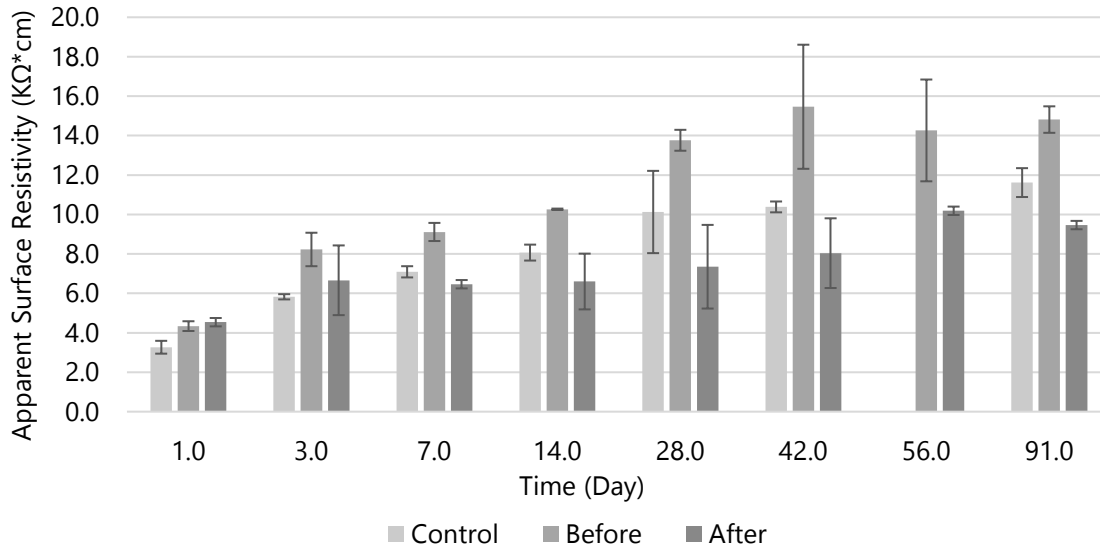


Figure 4.4.1: Comparison in apparent surface resistivity for control cylinders and core before and after vacuum saturation with limewater.

It can be seen that there is a general increasing trend with concrete maturity for all three specimen types; however, it is variable. First, the resistivity recorded for the core sample taken immediately after coring is higher than its two counterparts. This is expected as the degree in saturation of the samples are dissimilar. As previously explained, a partially saturated concrete sample will record a higher resistivity than a saturated one due to the decrease in electrolyte availability. Second, there is a variable behavior in resistivity between the control and core sample after saturation with limewater. On days 1 and 3, the control's surface resistivity is lower than the saturated core; thereafter, the control's resistivity value is larger. Here, differences in ionic strength of the pore

solution due to rapid limewater saturation may be causal to this behavior. Also, the degree in curing may also vary with increased age. The control was receiving continuous immersion curing while the larger slab samples were moist cured; not necessarily in a saturated state towards the bulk of the slab. Based on the statistical analysis (Appendix H, Table H3), these observable differences resulted in significant differences between sample types, where the results of surface resistivity are not comparable to that of the control's resistivity.

Moreover, the increased coefficients of variation obtained for the core samples renders this analysis difficult. The condition of the core surface as well as the exposure of aggregates are at the source of the problem. Careful probe placement was difficult as large aggregate and air voids had to be avoided to ensure a proper reading. The within measurement variability for one cylinder was as high as 15%, which is not acceptable. Consequently, test results are variable for cores and the method may be unreliable as a secondary method for quality acceptance. These effects may be avoided if the probe contact surface is increased, this is the principle of the uniaxial bulk resistivity test.

4.4.2 Analysis of Bulk Resistivity Testing on Cores

Figure 4.4.2 demonstrates the results obtained for bulk resistivity testing performed on the same set of samples as previously discussed. It can be seen that there is a similar behavior in resistivity gain over time as that previously described. This is expected as there is a known linear relationship between surface resistivity and bulk resistivity (Spragg et al. 2013). Figure 4.4.3a illustrates this strong relationship for the control samples ($R^2=0.99$). However, this relationship degrades for cores with a R^2 of 0.89 (Figure 4.4.3b). Again, this is due to the higher variability obtained for surface resistivity conducted on cores. This variability is not seen for the bulk resistivity study conducted on cores. The coefficients of variation for core samples vary between 0.1% and 6.6%

which is deemed acceptable (Table H4, Appendice H). This permitted a conclusive analysis, where the bulk resistivity obtained for the control sample is similar to that of the core sample after vacuum saturation with limewater. This is true for specimens 28 days and older. The difference is marginal at 14 days. It would seem that as the resistivity of concrete stabilizes in time, the difference between the laboratory control and the saturated field core is negligible. Now, it needs to be mentioned that both the control cylinder and the slab sample were cured at the same ambient temperature and in optimized curing conditions. This is not necessarily the case for concrete cured in the field.

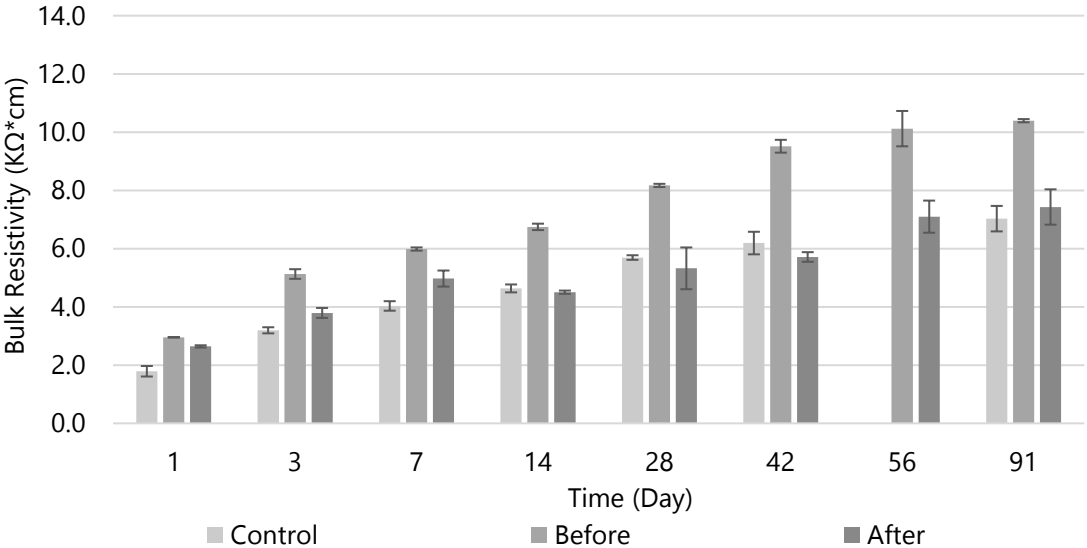


Figure 4.4.2: Comparison in bulk resistivity for control cylinders and core before and after vacuum saturation with limewater.

A change in curing condition will inadvertently affect the concrete maturity. With a change in maturity will come a change in resistivity. Although there is good promise for secondary compliance testing on field cores using bulk resistivity testing, the method requires further analysis to overcome differences in maturity. In absence of knowledge

of concrete maturity for this study, the effects of prolonged immersion curing of cores was investigated.

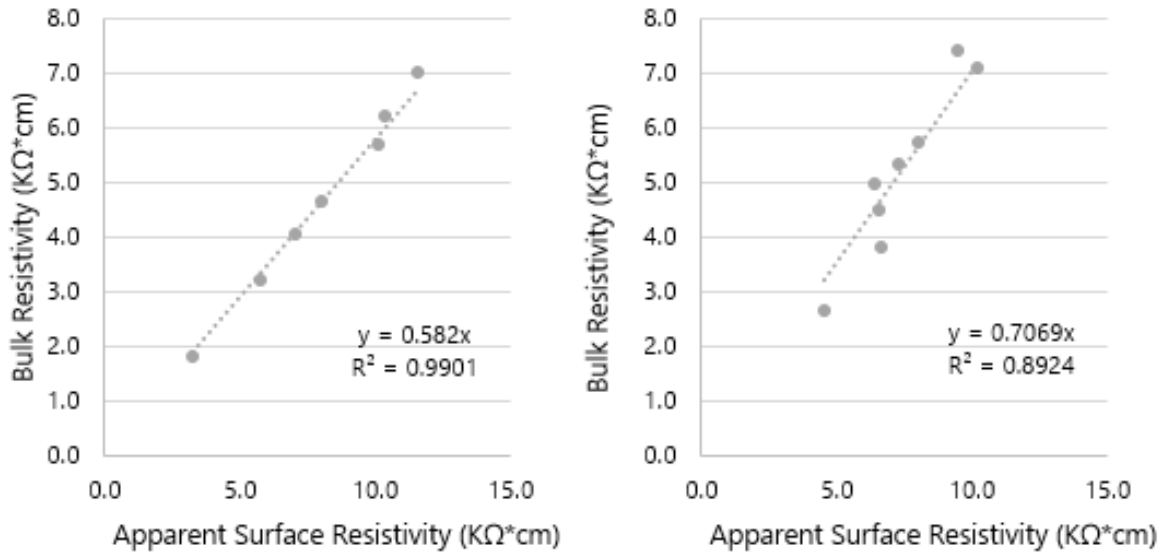


Figure 4.4.3: Linear relationship between bulk resistivity and apparent surface resistivity for a) control cylinder and b) cores after vacuum saturation.

4.4.3 Analysis of Prolonged Curing of Cores

After vacuum saturation, the cores were immersed in limewater for the remaining duration of the test period, up to 91 days. At each test age, the newly vacuum saturated core, the control and the previous cores in immersion curing were tested. The results of surface resistivity and bulk resistivity are shown in Figures 4.4.4 and 4.4.5. The results of the statistical analysis are available in appendix H, Tables H5 to H20.

As seen in Figure 4.4.4, the surface resistivity, up to 56-days of curing, is lower than the control's. Within the first two weeks of curing (days 3 to 14), the returned p-value, from the ANOVA analysis, demonstrate that all cores (both new and immersed) recorded statistically to marginally similar resistivity values. Conversely, as previously discussed, the control is statistically different. At the end of the test period, 91 days, certain core

samples exhibit a resistivity gain surpassing that of the control's. Unexpectedly, the ANOVA analysis returns similarities at days 56 and 91. This is due to the large coefficient of variations recorded, up to 12.7% and 30.6% for 56- and 91-day samples respectively. Thus, the analysis is inconclusive. Moreover, Figure 4.4.6 illustrates the poor linear relationship between surface and bulk resistivity ($R^2=0.71$). Again, this furthers the argument that surface resistivity may not be adequate for core evaluation.

On the other hand, the results for the bulk resistivity survey demonstrated its viability as a method for core resistivity testing. Figure 4.4.5 shows the resistivity gain in time for all core samples in comparison to the control. All core samples continuously immersed in limewater eventually follows the resistivity behavior of the control's. According to an ANOVA analysis, for a given test day, all cores were statistically similar to each other; except for early age behavior, where the resistivity of the new cores are marginally greater.

These preliminary results are quite promising for compliance testing. It demonstrates the ability of a core of different maturity to eventually gain a similar maturity to that of the laboratory control after a certain period of curing. Moreover, the similarities with the lab control resistivity behavior potentially enables the application of one of the recommended procedures (A, B or C). Further investigation with a set of different mixture design parameters is recommended.

The fact that the method gains accuracy at a later age is not detrimental to its implementation. Logistically, according to Procedures A, B or C, the recommended test period is 28 days or greater. Thus, a failed primary compliance test (standard laboratory cured cylinder sample) would be reported after 28 days. If this is the case, field coring should occur immediately afterwards, leaving an additional curing period reaching 56 to 91 days after date of casting. In this investigation, various concrete maturities and slab

curing methods were not investigated. Further investigation on this concept is recommend for implementation of a secondary testing method for acceptance of a concrete mixture design.

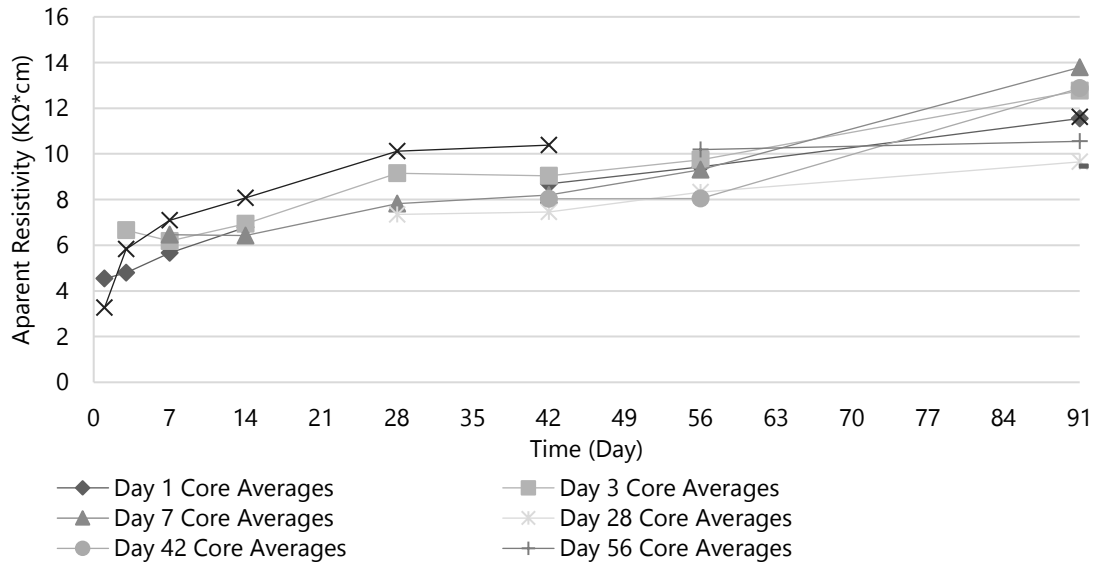


Figure 4.4.4: Apparent surface resistivity - time behavior for cores after vacuum saturation.

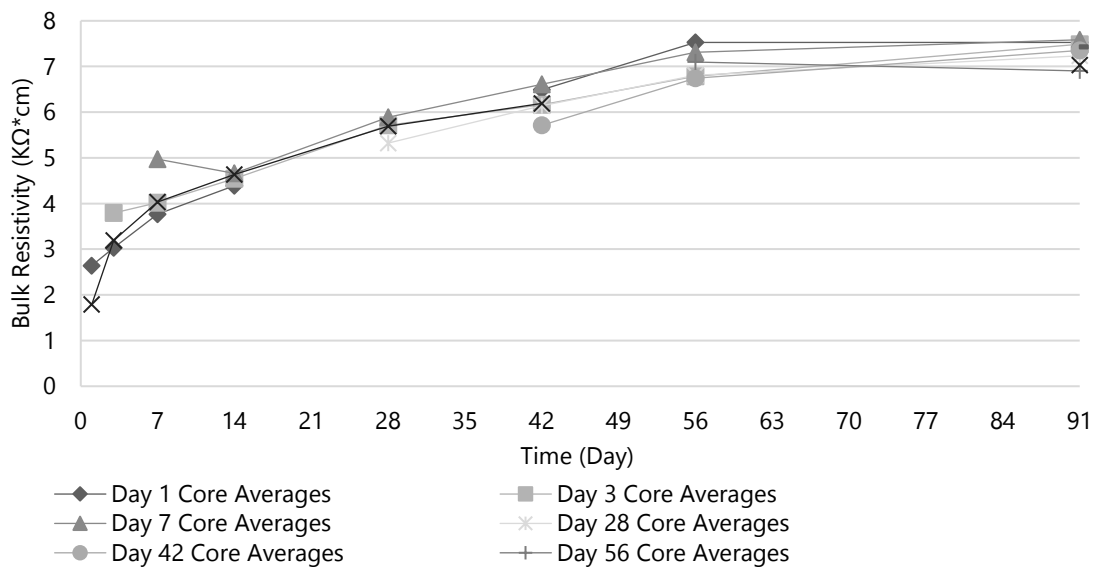


Figure 4.4.5: Bulk surface resistivity - time behavior for cores after vacuum saturation.

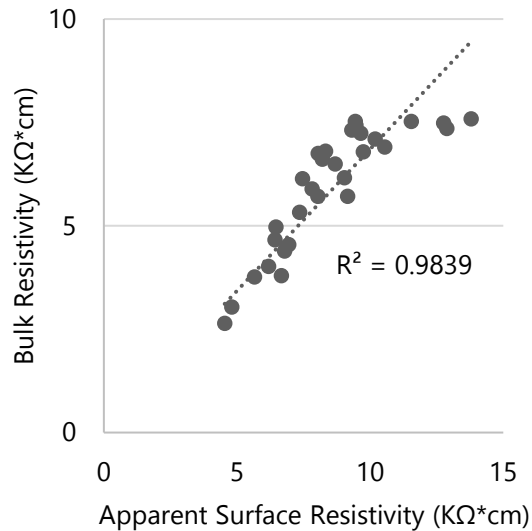


Figure 4.4.6.: Linear relationship between bulk resistivity and apparent surface resistivity for all cores.

4.4.4 Analysis of Surface Resistivity Testing on Slab

Conducting surface resistivity on an “infinite” slab surface is challenging due to the inherent nature of resistivity testing. The test method is sensitive to temperature and the degree in saturation of concrete. These are two of the most difficult parameters to control in the field and difficult to overcome (Bungey et al. 2006).

Several methods of tempering concrete and concrete saturation, from the surface of the slab, were trialed. To do so, slab specimens measuring 12” x 12” x 6” were cast and cured for 28-days in a temperature controlled moist curing room. Thereafter, the specimens were placed in a temperature (73°F) and humidity (50% RH) controlled environment to permit drying of the concrete samples. The specimens were conditioned for a minimum of 56-days. Thereafter, several methods of pooling with known and unknown quantities of potable water, limewater and a solution mimicking pore solution chemistry were trialed. The parameter of interest was to achieve a stable resistivity measurement within a reasonable time period (i.e. 4 hours). None of the

methods produced adequate surface saturation to achieve comparable, repeatable and reliable results within a reasonable time period (1 work day).

Therefore, for the purpose of this study (i.e. determining if a non-destructive approach for secondary compliance testing in the field is viable), evaluation of surface resistivity on slabs was conducted in controlled laboratory conditions only.

Each slab was allowed to cure up to 91-day. At each test age, prior to coring the slab, the surface resistivity test was conducted on that slab's surface along with test replicate on a control slab and the 91-day slab. Therefore, the test results presented herein represent the average surface resistivity for three slab replicates.

Appendix H, Table H21, provides a record of all measurements taken along with sample analysis. It needs to be mentioned that for day-1 the calculated coefficient of variation for a series of 8 measurements is between 10.1% and 13.6%, which is above that acceptable for cylinder testing. Thereafter, the variability diminished with increased maturity. It is unknown whether this is operator error (the operator is more adapt with practice) or if this is due to differences in early age hydration across the concrete slab. Afterwards, the coefficients of variation are between 2.3% and 6.3%, which is deemed acceptable according to standard practice on cylinders. Table H22 provides the results of the test conducted on the three replicates. Here the coefficients of variation are very low, 0.1% to 0.7%. Therefore, the procedure described in section 3.4.3, was considered to be successful and recommended for field use.

Figure 4.4.7 demonstrates the surface resistivity test results in comparison to that obtained for the control cylinders and cores. It can be seen that there is a general trend in resistivity gain over time; however, there is no clear relationship between slab results and that of cores nor the controls. Figure 4.4.8 presents the results of a linear regression analysis to investigate any potential correlations between slab resistivity and that of

other sample types. The obtained R^2 values vary between 0.6 and 0.8. Again, as previously explained, disparities in concrete maturity and degree in saturation may be the cause.

Under controlled ambient conditions, conducting surface resistivity on slab specimens was deemed successful, but the outcome of the comparative study was still variable making this method unpredictable for non-destructive application in the field.

Therefore, it is not recommended for implementation without further investigation into an adequate procedure to overcome the enumerated challenges.

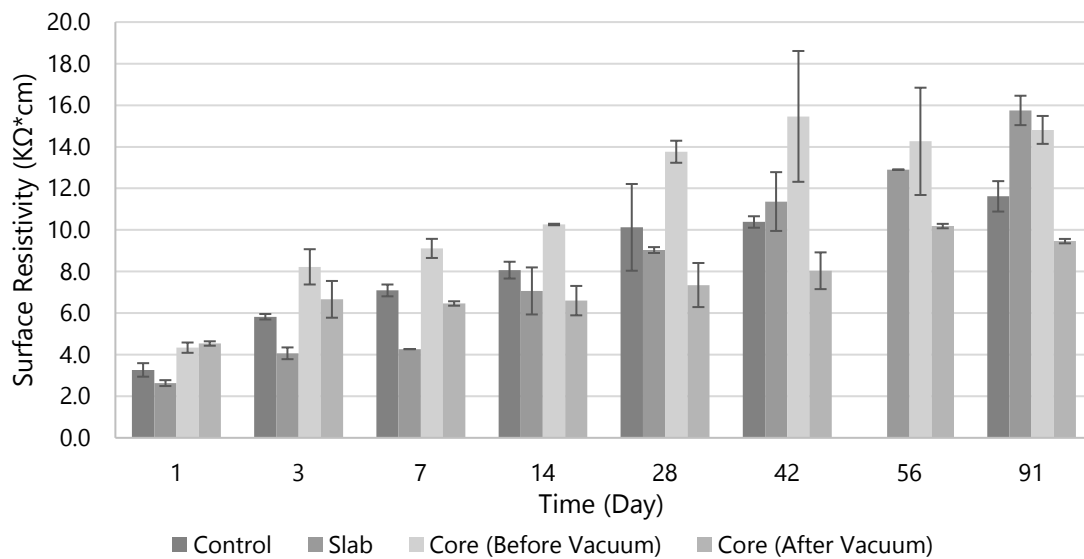


Figure 4.4.7: Comparison in surface resistivity for slabs, control cylinders and core samples.

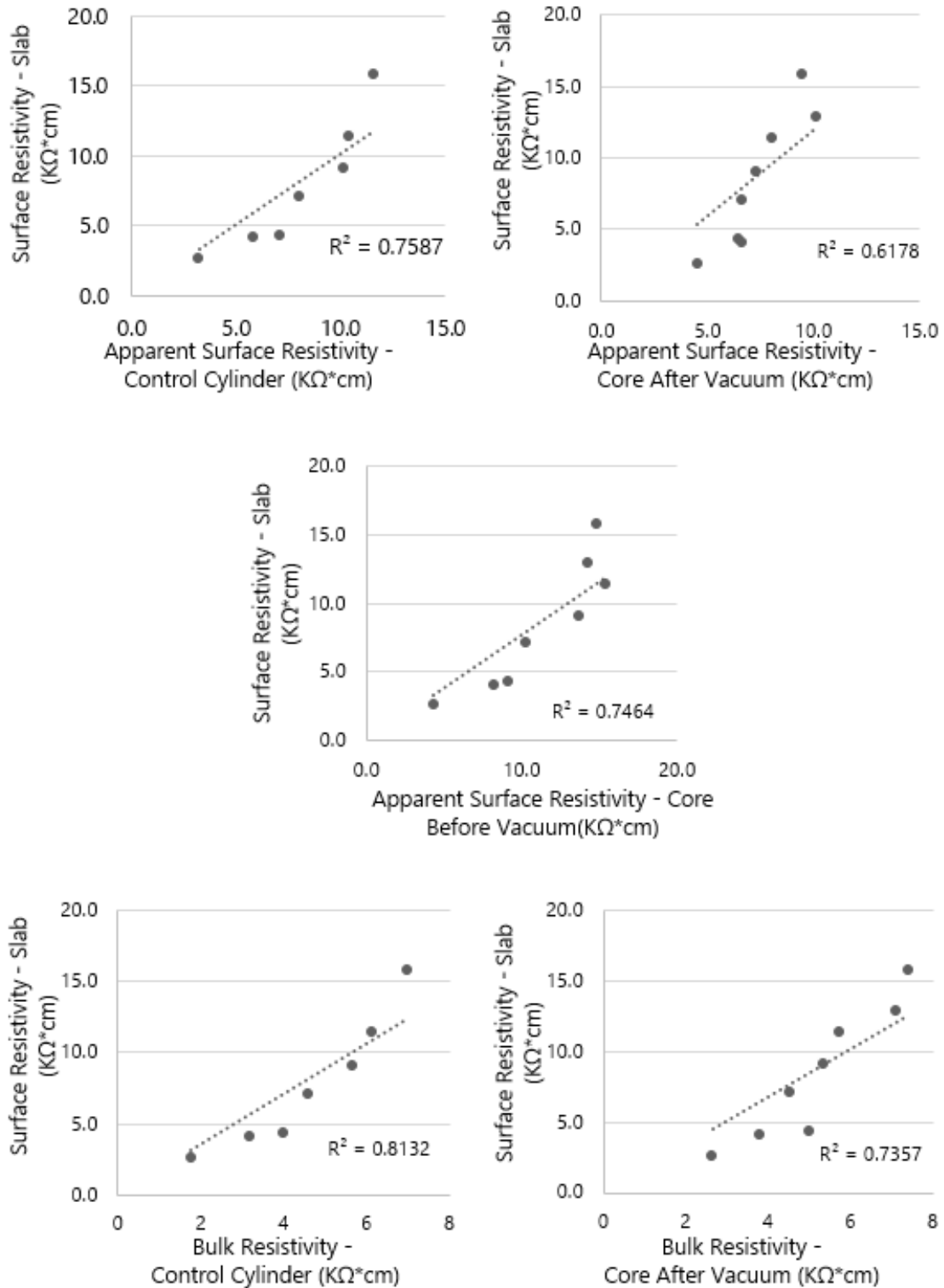


Figure 4.4.8: Linear relationship between slab resistivity and resistivity for other sample types

4.5 Influence of Procedural Variations – Effect of Temperature

There are many factors which may influence the outcome of a test. Whether these are procedural, equipment related or operator factors; a good understanding of how a test result may change in the event the standard procedure is not respected is of importance. For these cases, the results would be due to test/manipulation error as opposed to an actual change in material properties; hence the importance of following a standard procedure using properly functioning and calibrated equipment. There has been extensive investigation on the influence of variations in test procedure which led to the development of the current resistivity testing standards. However, there is no mention of temperature thresholds to conduct the test. It is well known that temperature influences resistivity but how, is still unclear and a subject of much needed research.

As previously explained in the Experimental Procedures section, three different temperature tests were trialed on a given cylinder. Provided in Appendix I is a series of bar charts demonstrating the resistivity results with respect to cylinder curing time (days) and temperature for each trial test condition (Test 1, Test 2, Test 3). The results of a fourth temperature trial (Test 4) are also provided in Appendix I. It was conducted to evaluate the effects of curing temperature on concrete and its influence on resistivity.

Five temperature increments were investigated, 67°F, 70°F, 73°F, 76°F and 79°F. A standard laboratory environment should be 73°F ± 2.5°F, which is also the recommended curing temperature for standard concrete specimens (ASTM C 511). Therefore, the range selected represents small variations from the standard; but still plausible variations in a laboratory environment. The hypothesis is that between 70°F and 76°F (representative of a standard ambient lab temperature) the outcome of a resistivity test will not be affected by temperature. In addition, two temperature

increments, higher and lower than the range, were also evaluated for resistivity result outcome.

Starting with Test 1, the procedure was devised to examine the impact of a change in apparatus temperature while the cylinder remains at ambient conditions. This mimics a procedure where the concrete cylinder is taken from a temperature-controlled immersion curing-tank (tempered at approx. 73°F) and tested in a lab environment, which may be cooler or warmer than 73°F. The equipment would be tempered at room temperature, but the cylinder would be at the standard temperature.

Test 2 simulates both the cylinder and the equipment at the same temperature. This may be the case where cylinders are taken out of the curing tank and left to temper at room temperature prior to testing, which may be warmer or cooler than the standard. By that fact, the equipment would also be at that same room temperature. This event is probable. Generally, several cylinders are taken out at once for testing. Cylinders may be left in the lab (covered with burlap or wrapped to prevent evaporation) for a given time period. Here, all equipment involved in the test would be at that same room temperature. It needs to be mentioned that the temperature change is superficial. Test 2 was devised to change the temperature of the surface. Due to the low thermal conductivity of concrete, it is assumed that the bulk of the cylinder was still near that of the initial temperature (datum).

Test 3 was conducted to measure the change in concrete temperature while the equipment remains at standard ambient temperature. This is to mimic similar situation as Test 2 but the equipment may have been stored in a different room. This situation is potentially the least probable. However, Test 3 was devised to isolate the effects of variations in concrete and equipment temperature.

Test 4 was devised to investigate the influence of curing temperature on resistivity. Often, an immersion curing-tank is stored in a laboratory space which may not be temperature controlled. In addition, the curing tank may not be tempered via tank heaters and/or coolers. As such the curing temperature may vary. The effects of curing temperature are known to affect the degree in maturity of a concrete. With higher curing temperatures, hydration mechanisms are accelerated resulting in a change in the crystal structure. In general, the consequence is an improvement in early-age properties but long-term, the cementitious matrix may be altered to the detriment of beneficial properties such as strength for example. For the study, the effects on resistivity gain over time is analyzed. Moreover, at time of resistivity measurement, the cylinder is at the temperature of the curing tank and the equipment is at the standard controlled room temperature; meaning, a difference in temperature that could add another level of uncertainty. Here, this scenario is reminiscent of Test 3 in terms of concrete and equipment temperature differences. This may aid in isolating the effects due to curing differences alone.

A total of six mix designs were investigated, three different w/c (0.4, 0.45, 0.5) with and without 20% FA content. It needs to be mentioned that the concrete cylinders were made from individual batches for each temperature increment. For example, 12 cylinder replicates per mix design were made from 1 batch. 6 were placed in the standard curing-tank and 6 were placed in the temperature-controlled tank. The first series was cured under standard conditions (73°F) and then subject to temperature tests 1, 2 and 3 consecutively. Only one temperature variance was investigated for that set (e.g. 76°F). As for the 2nd set of 6 replicates, these were placed in the temperature-controlled curing-tank set at the same increment investigated (e.g. 76°F). Due to potential variations between concrete batches, the comparative analysis between temperature

increments may have been slightly overshadowed by the inherent variability due to small variances in mixture parameters. Such seemed to be the case.

Looking at the overall results presented in Appendix I, any noticeable variation in resistivity within that of the standard temperature range (70°F to 76°F) are smaller than the inherent variability of the method. As such they are statistically insignificant. This is beneficial for the implementation of resistivity testing in a laboratory environment as small fluctuations in temperature does not statistically change the outcome of a test result. The need for a temperature correction factor would not be necessary. This is assuming that the laboratory and the curing-tank are kept within controlled temperature conditions.

Now, if one parameter were to be above or below the standard range, the results were difficult to interpret due to variability from mixture design shadowing that of temperature. Therefore, a factored approach was used to normalize resistivity and better isolate the effects of temperature. The results of the analysis are presented in the following subsections.

4.5.1 Effect of Change in Concrete Temperature

In the absence of the datum measurement for all concrete batches (resistivity value of the cylinder immediately after taking it out of the curing-tank, standard curing temperature, with equipment at standard room temperature), an alternative approach was taken to isolate the effects of temperature variance. Here the resistivity results are factored against Test 2 conditions.

Figure 4.5.1 presents an example of factorized resistivity (Equation 4.5.1), where the concrete cylinder and the Resipod are at the same temperature. Since the results are normalize according to itself, all factors are equal to 1. Now, small variances due to

mixture design are eliminated. The error bars illustrate a 95% confidence level (2 standard deviations from the mean) which was calculated from the obtained coefficient of variation. It can be seen that the variability in measurement may be as high as ± 0.2 for a 95% confidence interval. This is deemed over that acceptable. But, a variability below ± 0.16 is within that generally obtained for surface resistivity testing at a 95% confidence level.

$$\frac{\text{Mean Resistivity for Test 2 conditions}}{\text{Mean Resistivity for Test 2 conditions}}$$

(Eq. 4.5.1)

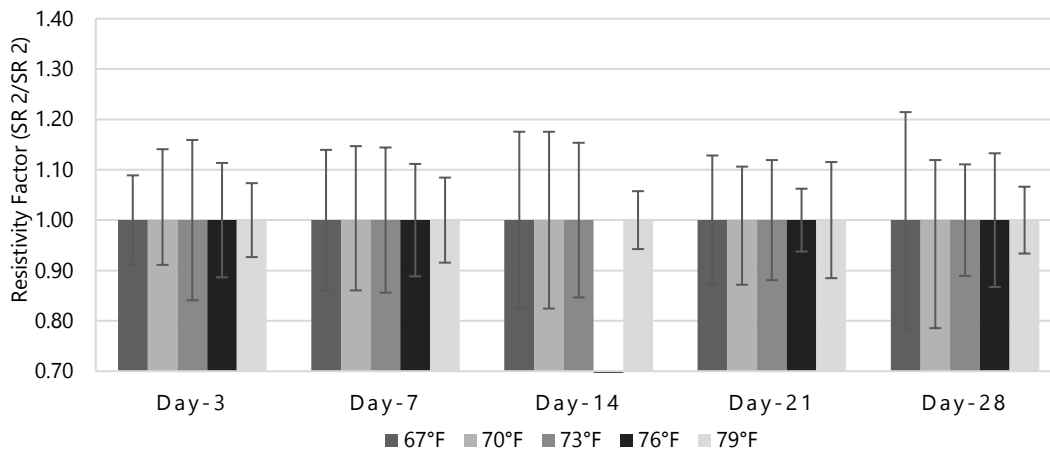


Figure 4.5.1: Resistivity Factor for Test 2 results normalized against Test 2 results – 0.50 w/c, 20% FA

To evaluate the influence of a change in surface temperature for concrete, the resistivity results obtained for Test 1 were factored against that of Test 2 (Eq. 4.5.2). For Test 2 both the cylinder and the Resipod are at the same temperature. For Test 1, the cylinder temperature changed and was approximately 73°F but the Resipod temperature remained the same temperature as Test 2. Therefore, the difference between Test 1 and Test 2 is the difference in temperature between a cylinder at 73°F (datum) and the

investigated temperature. For Example, looking at Day-3@67°F, the Test 1 result recorded was 4.8 KΩ*cm for a cylinder at 73°F and Resipod at 67°F.

$$\frac{\text{Mean Resistivity for Test 1 conditions}}{\text{Mean Resistivity for Test 2 conditions}}$$

(Eq. 4.5.2)

The Test 2 result was 5.5 KΩ*cm for a cylinder at 67°F taken with a Resipod at 67°F. So the influential factor is the increase in temperature between cylinders. From 67°F to 73°F, this resulted in a resistivity factor of 0.87 (4.8 KΩ*cm/5.5 KΩ*cm). Therefore, an increase of 6°F resulted in a 13% decrease in resistivity. Looking at the other temperature extreme, Day-3@79°F, a decrease of 6°F (from 79°F to 73°F) resulted in an 11% increase in resistivity.

In line with theory on the conductivity of an electrolyte, an increase in temperature of the electrolyte will result in an increase in conductivity of the solution. Since resistivity is inversely proportional to conductivity, an increase in temperature of an electrolyte will diminish its resistivity. This principle is well illustrated in Figure 4.5.2. The results at datum (73°F) are approximately 1. Here the variability is due to the that inherent to the test method. Thereafter, an increase of 3 to 6 degrees resulted in a decrease in resistivity factor. And vice-versa for an increase in temperature.

The concept is well illustrated for day-28 results. The factor at datum is actually 1.00. with a change in ±3 degrees both values changed by ±4%. With a change of ±6 degrees, the resistivity values changed by +12% and -7%. However, this average change of ≈10% was not sufficient to make the differences statistically significant with respect to the datum. In general, the results are statistically insignificant. Therefore, a change of 6°F from the datum does not change the outcome of a resistivity test. However, for a larger difference in temperature, the differences may start to be meaningful (example Day-3 67°F is considered to be different from Day-3 79°F). Due to

the missing data, an increase above 6°F cannot be substantiated. But, based on that observed, a difference up to 20% could be expected between extreme temperatures. In some instance across mixture types, meaningful differences are observable between the datum and the extreme. Such is the case for mixtures containing no fly ash after 28 days of curing.

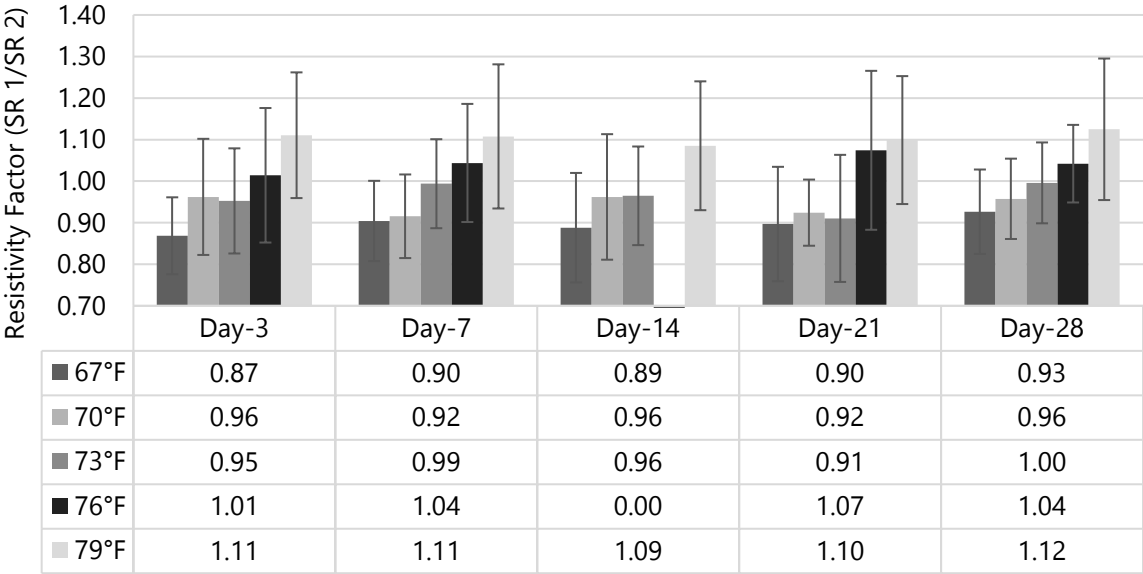


Figure 4.5.2: Resistivity Factor for Test 1 results normalized against Test 2 results – 0.45 w/c, 20% FA.

For this study, the results cannot justify the application of a correction factor or rejection of a resistivity result if the test was conducted at a temperature that borders that of the standard range of 70°F to 76°F. For greater differences, further research would be necessary.

On-the-other hand, for the purpose of using the resistivity method for mixture design identification, a difference in resistivity of 10-15% could lead to misclassification. In this case, a correction factor of 1% to 3% per degree difference from the datum (73°F) could be applicable to correct a time-resistivity curve presenting peaks and valleys. These assumptions are only applicable for mixture designs investigated and temperature

ranges investigated. Further research on the topic would be necessary to expand from laboratory to field applications as well as include other mixtures that are considered to be of high resistivity (concrete containing silica fume addition for example).

4.5.2 Effect of Change in Equipment Temperature

For the application of a temperature correction coefficient to be valid, other potential sources of temperature variance must also be considered. Here the influence of equipment temperature is also studied. To evaluate the influence of a change in equipment (Resipod) temperature, the resistivity results obtained for Test 3 were factored against that of Test 2 (Eq. 4.5.3).

As previously explained, for Test 2 both the cylinder and the Resipod are at the same temperature. For Test 3, the cylinder temperature remained the same temperature as that for Test 2, and the Resipod was tempered at approximately 73°F. Therefore, the difference between Test 3 and Test 2 is the difference in temperature between the Resipod at 73°F (datum) and the investigated temperature for the cylinder.

Seen in Figure 4.5.3, Day-3@67°F, the Test 3 result was 5.2 KΩ*cm for cylinders at 67°F and Resipod at 73°F. The Test 2 result was 5.5 KΩ*cm for cylinders at 67°F taken with a Resipod at 67°F. So the influential factor is the increase in temperature for the equipment. From 67°F to 73°F, this resulted in a resistivity factor of 0.94 (5.2 KΩ*cm/5.5 KΩ*cm). Therefore, an increase of 6°F resulted in a 6% decrease in resistivity. Looking at the other temperature extreme, Day-3@79°F, a decrease of 6°F (from 79°F to 73°F) resulted in an 1% increase in resistivity.

$$\frac{\text{Mean Resistivity for Test 3 conditions}}{\text{Mean Resistivity for Test 2 conditions}} \quad (\text{Eq. 4.5.3})$$

The influence of Resipod temperature at time of test does not seem to be a contributing factor. Figure 4.5.3 demonstrates the results for all resistivity factors calculated for the

concrete mixture 0.45 w/c with 20%FA. The factors vary from 0.94 to 1.02. In addition, there is no clear trend on whether the increase or decrease in equipment temperature leads to a proportionate or inverse correlation. Statistically, all results are within that of the 95% confidence level for all measurements. The same trend is observable for all mixture types (Appendix I). Seen in Appendix I, there are a few factors which would be considered as outliers. These are generally accompanied by a higher than acceptable coefficient of variation; therefore, these are dismissed from analysis.

Based on the observed temperature range, the influence of equipment temperature does not seem to be a contributing parameter and cylinder temperature is the primary parameter affecting the resistivity measurement. Still, for the purpose of control and compliance testing, it is recommended to store and operate the equipment in ambient laboratory conditions and follow the manufacturer's operating guidelines. Moreover, as previously explained in section 4.5, it is important to maintain a laboratory environment within standard temperature limits. Laboratory temperature extremes could lead to changes in the cylinder's surface temperature, which would affect the measurement as previously discussed. Therefore, conducting the test method in a room that is temperature-controlled within a temperature range of 70°F to 76°F is recommended.

Based on the investigator's experiences within this project, changes in ambient laboratory temperatures were a large source of error leading to unusable data and restarting multiple test series. Never underestimate the cool draft of air conditioning, nor the first warm spring breeze coming from an open laboratory door. Now it is demonstrated that such is enough to temper the surface of concrete cylinders waiting to be tested and ultimately affect the resistivity measurement.

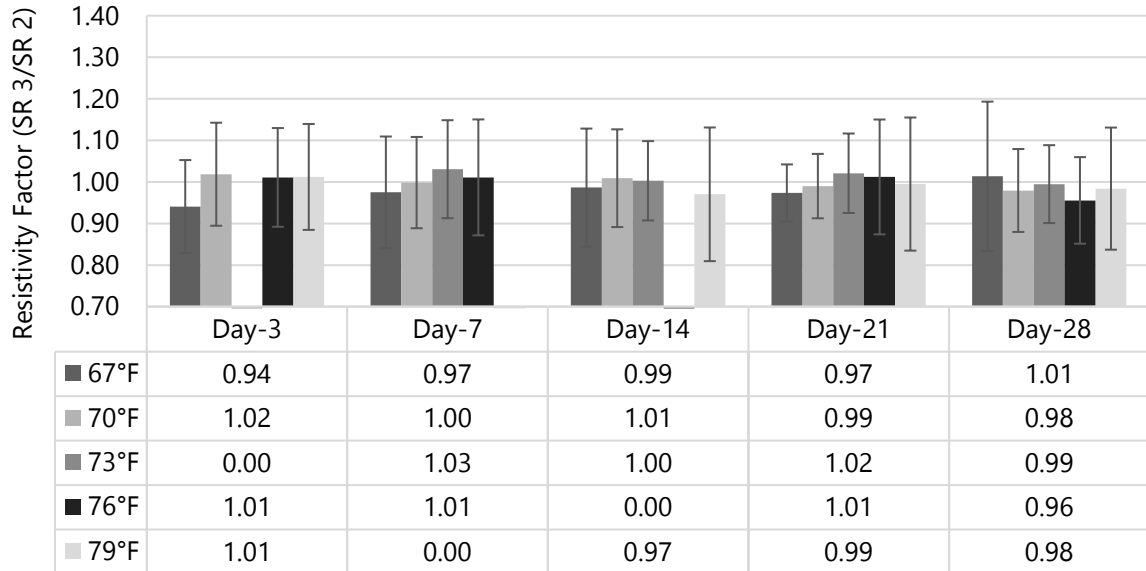


Figure 4.5.3: Resistivity Factor for Test 3 results normalized against Test 2 results – 0.45 w/c, 20% FA.

4.5.3 Effect of Change in Curing Temperature

Another factor which may influence the result of a resistivity test is the temperature of curing. Discussed in section 4.5.1, a change in cylinder temperature will affect the resistivity value. In addition, was explained that the degree in hydration (or maturity) is also influence by temperature. In the latter case, this variance changes, for better or worse, the material properties that may be of interest on a short or long-term basis.

Therefore, in an attempt to isolate the effects of curing alone, the results of Test 4 were factored against that of Test 3. As previously explained, for Test 3 the Resipod is at the datum temperature (73°F) and the cylinder temperature is varied. As for Test 4, at time of testing, the same conditions are met. The Resipod is at lab ambient temperature (approximately 73°F) and the cylinder temperature is at the varied curing temperature. So, the same temperature conditions are met for both Test 3 and Test 4 for both equipment and concrete surface. Therefore, the difference between Test 3 and Test 4 is the difference in hydration causal of the change in curing temperature of the limewater

curing tank. Moreover, the change in the cylinder's core temperature may also be a source of variance. Here it is assumed that for Test 3, the bulk temperature of the cylinder is near that of the datum (curing temperature). For Test 4, the temperature of the cylinder core was monitored and measured at time of test via embedded thermocouples. The later as well as the limewater were monitored throughout the curing period to ensure precise temperature control of the curing-tank.

The factors tabulated and shown in Figure 4.5.4, were calculated using equation 4.5.4. The same calculation as that explained in Sections 4.5.1 and 4.5.2 was followed. The factored results for this comparative study are slightly variable. It needs to be mentioned that the Resipod temperature was not recorded (Appendix I). However, it was previously demonstrated that it should not affect the outcome of the test. Nonetheless, there is an emerging trend.

$$\frac{\text{Mean Resistivity for Test 4 conditions}}{\text{Mean Resistivity for Test 3 conditions}} \quad (\text{Eq. 4.5.4})$$

It can be seen that there is an increase in resistivity with an increase in curing temperature. An increase in curing temperature typically leads to a greater concrete maturity; hence, an increase in the production of hydrated cementitious material improving the concrete's properties. The increasing trend is noticeable. Figure 4.5.4, at 28-days, for curing temperatures of approximately 79°F, there is a significant resistivity gain of 25%. The influence is less prominent at a young age (3% gain at day-3), but over time, the influence of temperature becomes more prominent widening the gap. This behavior is seen for samples containing fly ash, but not for samples other mixture types.

A similar gain in resistivity at 28 days is not seen for mixtures containing no fly ash. Figure 4.5.5 compares the factors for a 0.45 w/c concrete mixture containing no supplementary cementitious material. The change in factor for fall test ages and temperatures, is approximately $\pm 10\%$. Consequently, this is statistically insignificant and the influence in curing temperature may not have a prominent role in affecting the outcome of a test. But the benefits associated with an increased maturity may be overshadowed by the influence of concrete temperature (as that seen in Section 4.5.1)

Here, the curing temperature may not be the sole difference between Test 3 and Test 4. It is assumed that there is also a change in the cylinder's core temperature. For example, the Test 4 sample for Day-28 curing at 79°F as an internal temperature of 79°F. The Test 3 sample is assumed to have an internal temperature near datum. So for this data point, there is an increase in the cylinder's internal temperature. With an increase in temperature, there should be a decrease in resistivity as that explained and demonstrated previously. This behavior is not seen at all for the factored results. There are 2 hypotheses. Either both parameters are at play (increasing and decreasing the resistivity) but the influence of curing and hydration prevails, consequential of the increase in resistivity factor. Or, the internal temperature of the cylinder in Test 2 and 3 is not near datum, but closer to the set temperature. This cannot be validated because the internal temperature of the concrete cylinder was not measured during Tests 2 and 3.

Further investigation on the influence of curing temperature is required. Based on the results of this study, if the curing temperature is maintained within the standard range, the outcome of a resistivity test on standard concrete cylinder should not be affected. This is valid for the test age evaluated (i.e. up to 28 days of curing). For the purpose of the resistivity method within a QC/QA program, 28 days is enough for the control of

mixture design. Thus, the same set of cylinders used for standard compressive strength testing could be used for the nondestructive resistivity method.

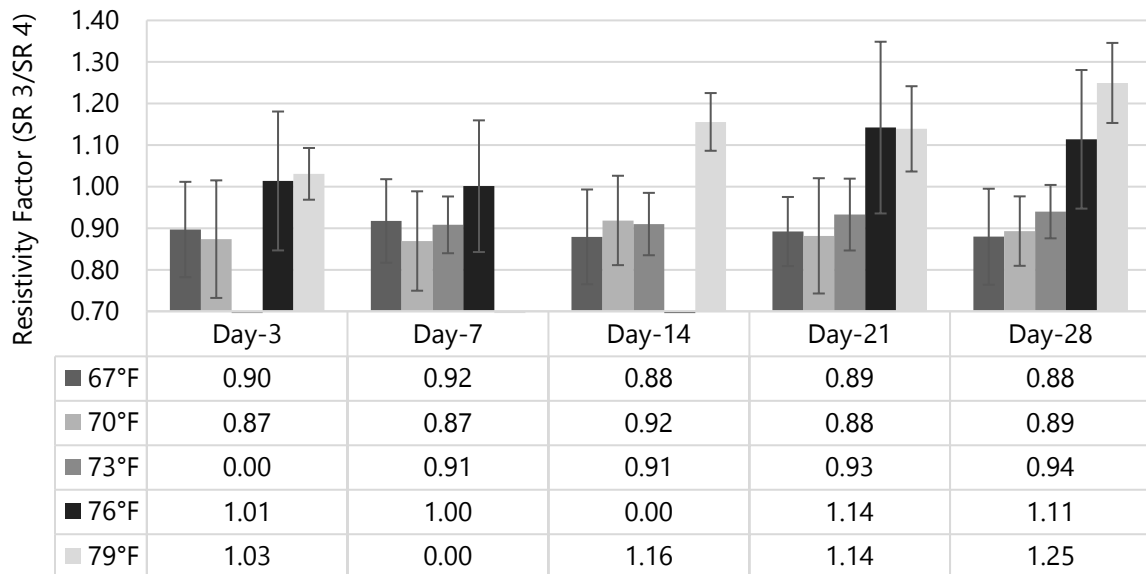


Figure 4.5.4: Resistivity Factor for Test 4 results normalized against Test 3 results – 0.45 w/c, 20% FA.

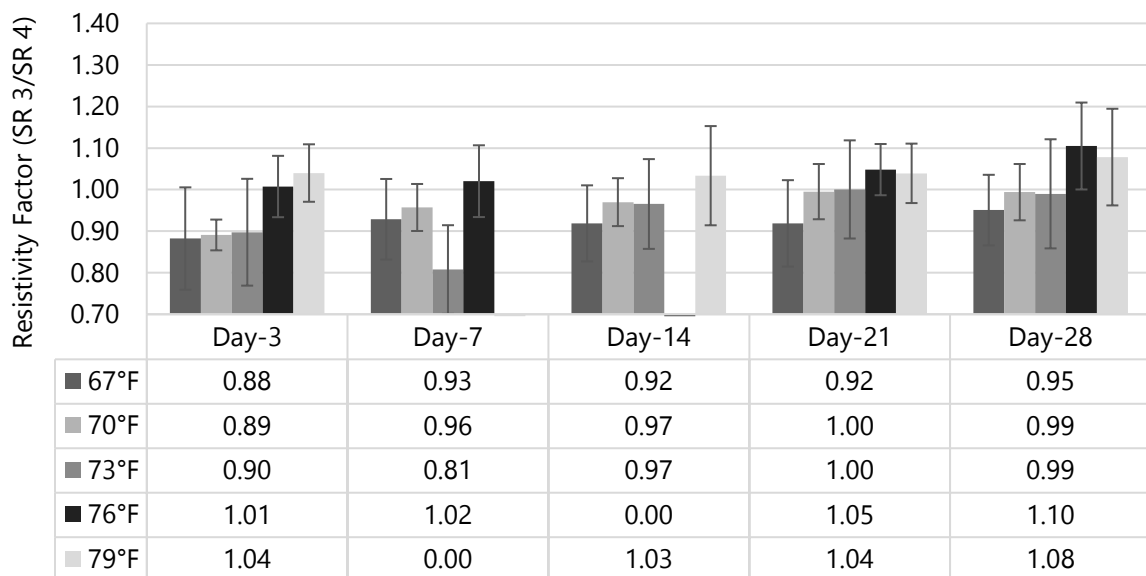


Figure 4.5.5: Resistivity Factor for Test 4 results normalized against Test 3 results – 0.45 w/c, without FA.

Chapter 5 Conclusions

During construction, various standard tests are performed as part of a QC/QA test program. They are performed on concrete in both the plastic and hardened states. These aid in determining the quality of placed concrete and its compliance with mixture designs approved at the beginning of a contract (prior to construction). Generally, slump test, unit weight test, and air pressure test are performed on fresh concrete and compression and/or flexure testing is conducted on hardened concrete. These tests do provide information about consistency, workability and air content as well as specified mechanical strength. Workability may be an indication of water content but with the advent of SCMs and specialty admixtures, workability is a questionable measurement for concrete acceptance. All of these standard tests do not provide an indication of the actual concrete mixture design delivered. It does not ensure that the mixture placed is the same as that submitted. Although strength may be achieved, this parameter alone does not confirm the presence of concrete ingredients that may be beneficial or detrimental to durability performance. The latter is critical for reaching performance and long service life that the concrete mixture was designed for.

The research presented in this report develops a novel resistivity criterion to verify key concrete mixture parameters, w/cm ratio and fly ash content (class-C), which could help to minimize common durability issues derived from excessive water contents and lack of beneficial additives. In turn, the intended service life of the concrete structure can be reached with minimal maintenance costs.

For the development of the resistivity method for the control and acceptance of concrete mixture parameters, the influence of individual concrete parameters had to be isolated and studied. An extensive parametric investigation was conducted to evaluate the influence of water-to-cement ratio, addition of class-C fly ash, aggregate type and

gradation, addition of admixtures. Primarily, w/c was found to have a meaningful influence on the resistivity measurement permitting distinction between concretes of 0.4 w/c, 0.45 w/c and 0.5 w/c, but not between 0.5 w/c and higher w/c concretes. Based on the resistivity gain with time, the presence of fly ash can be confirmed, however, within the test period of 28 days, the percentage of cement replacement cannot be isolated for replacements between 5% and 25%. The source of fly ash and Type I cement did not seem to have an effect but, this is limited to materials evaluated. The presence of an entrained air matrix did not have a significant effect on the measurement. But, the latter combined with a water reducer may be enough to increase the resistivity of concrete. The increase in resistivity seems to be causal of the benefits of water-reducing admixtures on increased hydration of cement and pore refining. Lastly, a change in aggregate mineralogy, small variation in gradation and content, did not affect the measurement with the exception of a gabbro aggregate in the presence of fly ash concrete. The cause is unknown and further investigation is recommended. Based on these discernable effects, recommendations for resistivity implementation as part of a QC/QA program is proposed. Three different Procedures (A, B and C) were devised and trialed.

Procedure A makes use of tables where the change in resistivity between day-1 and day-3 measurements must be calculated to identify the presence of fly ash. Another table is used for w/c categorization. This may be cumbersome for the end-user and the measurements must be performed on day-1, day-3 and day-28 precisely.

Procedures B makes use of charts to categorize a result according to w/c and presence of fly ash. The charts incorporate all variables studied (admixtures, aggregates, cement sources, fly ash sources), thus the range is found to be large as it is based on a 95% confidence level to accurately identify a mixture. As a result, there is overlap between categories.

The Procedure C seems to be the most effective as it requires the development of a resistivity curve specific to a concrete mixture. This burden is placed on the concrete producer and the curve should be submitted along with other documentation at time of mixture design approval. This resistivity curve would be set as the standard during construction. During construction, the standard cylinder samples would be tested at the lab and several measurements in time can be made. Here, there would be flexibility on the day the measurement is performed. A minimum of three data points are recommended (days 3, 7, and 28); however, weekly measurements are recommended for increased accuracy. The procedure could be extended up to 56-days for identification of other SCMs or specialty admixtures.

A three-month field study was conducted to determine the state of production of concrete mixtures of Class A and Class AA in the state of Oklahoma. A total of 67 samples from across the state of Oklahoma was evaluated. The samples were lumped by mixture design and producer. Through comparative analysis of the produced resistivity curves for each sample, it was found that producer can consistently deliver a given concrete mixture over a period of time. Also, changes in mixture design were identifiable. Procedure C and B were applied to validate its suitability. It was found that the majority of concrete mixtures may not have met ODOTs minimal specifications in terms of w/c. This may have consequences on the durability performance of the constructed pavements or bridge decks. From the field study, Procedure C is recommended as it offers an easy and reliable way for the detection of changes in mixture design. The developed thresholds for each w/c and FA category (Procedure B) can be used as a supplementary tool to determine whether a change in resistivity curve is due to an increase in water-to-cement ratio or lack of supplementary cementitious materials for example.

For the method to be applicable, a procedure must be followed. Resistivity testing is known to be sensitive to variances in test procedure. One of the concerns is temperature. Since the topic of the investigation is targeted towards laboratory use, a narrow range of temperature were studied, 67°F to 79°F. The range represents that of recommended ambient temperature for a materials laboratory (73°F \pm 2.5°F) as well as border variances. The influence of the change in cylinder temperature, the change in equipment temperature and the change in curing temperature was evaluated. A factored approach was used to isolate each behavior. Primarily, within the narrow range studied, temperature does not influence the outcome of a resistivity test. However, there are a few exceptions which leads to the recommendation of including the standard temperature tolerances for both curing and laboratory environment in a resistivity procedure. It was found that a change in surface temperature of a concrete cylinder due to ambient laboratory conditions may significantly alter the result. A correction factor is proposed but is limited in its application. Further investigation is required. The influence of a change in resistivity meter is not influential. Finally, a change in curing temperature is influential for fly ash mixtures while not has defining for mixture containing no fly ash. Thus, the importance of maintaining controlled curing conditions as specified in ASTM C 511. To increase the reliability of the resistivity method for mixture design identification, temperature control of the lab environment and immersion curing-tanks should consider in the procedure.

Finally, QC/QA test methods are performed on standard, laboratory cured concrete specimens. Here, the concrete properties are evaluated against specified criteria for the purpose of quality assurance or acceptance. In the event that the primary form of testing fails to meet the specification, a secondary test is generally performed to evaluate whether the companion field concrete meets the specification (or also fails the test requirements). A good example is the compressive strength test. If the standard

cylinder is sub-part, the secondary compliance test is performed on cores taken from the newly constructed concrete element. In this case, resistivity testing would be performed either in the field or in a laboratory environment on cores. Although many attempts, the investigators did not find a reliable method for conditioning the surface of a concrete element to perform a surface resistivity test adequately. Still, a comparative study was performed to evaluate the best approach for secondary compliance testing. It was found that bulk resistivity testing conducted on a core vacuum saturated in limewater yielded viable results. In fact, continued curing of the core afterwards could aid the comparative analysis using Procedure C. Here an adequate correlation equation between surface resistivity and bulk resistivity for the given mixture would aid in improving the accuracy in of the resistivity method. On the other hand, surface resistivity on cores is not recommended as it produces high coefficients of variation.

References

- ACI (American Concrete Institute). (2013). "Report on Nondestructive Test Methods for Evaluation of Concrete in Structures". ACI 228-2R-13, Farmington Hills, MI, 86 pp.
- Samson, E., Marchand, J., and Beaudoin, J., 2000, "Modeling the influence of chemical reactions on the mechanisms of ionic transport in porous materials: an overview," *Cement and Concrete Research*, 30(12), pp. 1895-1902.
- Azarsa, P. and R. Gupta (2017). "Electrical Resistivity of Concrete for Durability Evaluation: A Review." *Advances in Materials Science and Engineering*, 2017, 1-30.
- Bentz, D. P., et al. (2014). "Anticipating the setting time of high-volume fly ash concretes using electrical measurements: feasibility studies using pastes." *Journal of Materials in Civil Engineering*, 27(3).
- Bungey, J. H., Grantham, M. G., and Millard, S. (2006). *Testing of concrete in structures*, Crc Press.
- Castro, J., Weiss, J., Paredes, M., Spragg, R., and Nantung, T. (2012). "Variability analysis of the bulk resistivity measured using concrete cylinders." *Advances in Civil Engineering Materials*, 1(1), 1-17.
- Ferreira, R. M., and Jalali, S. (2010). "NDT measurements for the prediction of 28-day compressive strength." *NDT & E International*, 43(2), 55-61.
- Gowers, K., and Millard, S. (1999). "Measurement of Concrete Resistivity for Assessment of Corrosion." *ACI Materials Journal*, 96(5).
- Gulrez, W., and Hartell, J. (2017). "Effect of Curing Condition and Temperature on Surface Resistivity Measurements." *Proc., 26th ASNT Research Symposium*, 99-107.
- Kessler, R. J., Powers, R. G., and Mario Paredes, M. P. (2005). "Resistivity measurements of water saturated concrete as an indicator of permeability." *CORROSION 2005*.
- Kessler, R. J., Powers, R. G., Vivas, E., Paredes, M. A., and Virmani, Y. P. (2008). "Surface resistivity as an indicator of concrete chloride penetration resistance." *Proc., Concrete Bridge Conference*, St. Louis, Missouri, p20.
- Larsen, C., Sellevold, E., Askeland, F., Østvik, J., and Vennesland, Ø. "Electrical resistivity of concrete part II: Influence of moisture content and temperature." *Proc., 2nd International symposium on advances in concrete through science and engineering*. Quebec, Canada.
- Lastate, J. F. (2010). *Electrical resistivity for evaluation of concrete structures*. *NDT & E International*, 243-275.
- Lastate, J., Sirieix, C., Breyse, D., and Frappa, M. (2003). "Electrical resistivity measurement applied to cracking assessment on reinforced concrete structures in civil engineering." *NDT & E International*, 36(6), 383-394.

- Layssi, H., Ghods, P., Alizadeh, A. R. and Salehi, M. (2015). Electrical resistivity of concrete: Concepts, applications, and measurement techniques. *Concrete International*, 41-46.
- Nadelman, E. I., and Kurtis, K. E. (2014). "A resistivity-based approach to optimizing concrete performance." *Concrete international*, 36(5), 50-54.
- Neithalath, N., and Jain, J., 2010, "Relating rapid chloride transport parameters of concretes to microstructural features extracted from electrical impedance," *Cement and Concrete Research*, 40(7), pp. 1041-1051.
- Morris, W., Moreno, E., and Sagüés, A. (1996). "Practical evaluation of resistivity of concrete in test cylinders using a Wenner array probe." *Cement and Concrete Research*, 26(12), 1779-1787.
- Polder, R. (2000). Test methods for on-site measurement of resistivity of concrete. *Materials and structures*, 33, 603-611.
- Rohne, R. J. (2009). "Effect of Concrete Materials on Permeability of Concrete Mixes Used in Mn/DOT Paving Projects."
- Sengul, O. (2014). "Use of electrical resistivity as an indicator for durability." *Construction and Building Materials*, 73, 434-441.
- Spragg, R., Bu, Y., Snyder, K., Bentz, D., and Weiss, J. (2013). "Electrical testing of cement-based materials: role of testing techniques, sample conditioning, and accelerated curing."
- Rupnow, T., and Icenogle, P. Surface Resistivity Measurements Evaluated as Alternative to Rapid Chloride Permeability Test for Quality Assurance and Acceptance. *Transportation Research Record: Journal of the Transportation Research Board* (2290), 2012, pp. 30-37.
- Vivas, E., Boyd, A. J., Hamilton III, H., and Bergin, M., 2007, "Permeability of Concrete—Comparison of Conductivity and Diffusion Methods."

Appendix A -
Effect of Water Addition & Change in Water-to-Cementation Materials ratio (W/C)

Table A1: Results of Statistical Analysis 5% Fly Ash

| Day | w/c | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0.40 | 8.6 | 0.2 | 1.8 | | 0.40/0.45 | 3.7E-05 | -8.7 |
| 7 | 0.45 | 7.9 | 0.2 | 2.6 | 1.8E-05 | 0.40/0.50 | 9.9E-05 | -13.7 |
| 7 | 0.50 | 7.4 | 0.4 | 5.9 | | 0.45/0.50 | 5.3E-02 | -5.5 |
| 28 | 0.40 | 11.4 | 0.2 | 2.1 | | 0.40/0.45 | 1.9E-06 | -12.7 |
| 28 | 0.45 | 10.0 | 0.3 | 2.7 | 3.2E-07 | 0.40/0.50 | 6.7E-06 | -19.2 |
| 28 | 0.50 | 9.2 | 0.6 | 6.3 | | 0.45/0.50 | 1.8E-02 | -7.5 |
| 56 | 0.40 | 13.8 | 0.4 | 3.2 | | 0.40/0.45 | 2.6E-03 | -14.0 |
| 56 | 0.45 | 11.8 | 0.2 | 1.9 | 3.8E-05 | 0.40/0.50 | 2.9E-04 | -23.2 |
| 56 | 0.50 | 10.6 | 0.1 | 1.3 | | 0.45/0.50 | 1.2E-03 | -10.6 |

Table A2: Results of Statistical Analysis 10% Fly Ash

| Day | w/c | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0.40 | 8.4 | 0.4 | 5.3 | | 0.40/0.45 | 3.3E-02 | -10.27 |
| 7 | 0.45 | 7.5 | 0.7 | 9.7 | 1.3E-03 | 0.40/0.50 | 2.9E-04 | -18.65 |
| 7 | 0.50 | 6.8 | 0.5 | 8.0 | | 0.45/0.50 | 8.8E-02 | -9.34 |
| 28 | 0.40 | 11.5 | 0.7 | 5.8 | | 0.40/0.45 | 8.5E-03 | -11.22 |
| 28 | 0.45 | 10.2 | 0.7 | 6.9 | 3.5E-05 | 0.40/0.50 | 5.0E-05 | -22.62 |
| 28 | 0.50 | 8.9 | 0.7 | 7.5 | | 0.45/0.50 | 8.0E-03 | -12.84 |
| 56 | 0.40 | 14.5 | 0.8 | 5.6 | | 0.40/0.45 | 1.2E-01 | -8.88 |
| 56 | 0.45 | 13.2 | 0.8 | 5.9 | 1.1E-03 | 0.40/0.50 | 2.1E-03 | -30.39 |
| 56 | 0.50 | 10.1 | 0.7 | 7.0 | | 0.45/0.50 | 6.9E-03 | -23.61 |

Table A3: Results of Statistical Analysis 20% Fly Ash

| Day | w/c | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0.40 | 7.2 | 0.4 | 5.1 | | 0.40/0.45 | 3.6E-04 | -15.35 |
| 7 | 0.45 | 6.1 | 0.4 | 5.8 | 5.9E-07 | 0.40/0.50 | 1.6E-06 | -22.42 |
| 7 | 0.50 | 5.6 | 0.1 | 2.5 | | 0.45/0.50 | 8.6E-03 | -8.35 |
| 28 | 0.40 | 12.1 | 0.5 | 4.4 | | 0.40/0.45 | 9.8E-06 | -21.38 |
| 28 | 0.45 | 9.5 | 0.6 | 5.9 | 4.7E-09 | 0.40/0.50 | 5.3E-08 | -29.38 |
| 28 | 0.50 | 8.5 | 0.3 | 3.3 | | 0.45/0.50 | 3.6E-03 | -10.18 |
| 56 | 0.40 | 17.3 | 0.1 | 0.8 | | 0.40/0.45 | 1.4E-03 | -21.29 |
| 56 | 0.45 | 13.6 | 0.8 | 5.9 | 5.4E-05 | 0.40/0.50 | 4.5E-06 | -26.41 |
| 56 | 0.50 | 12.7 | 0.2 | 1.4 | | 0.45/0.50 | 1.3E-01 | -6.50 |

Appendix B - Effect of Fly Ash Source and Addition

Table B1: Results of Statistical Analysis 0.40 w/c

| Day | FA % | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0 | 8.9 | 0.5 | 6.0 | 3.1E-05 | 0/10 | 1.1E-01 | -5.6 |
| 7 | 10 | 8.4 | 0.4 | 5.3 | | 0/20 | 7.3E-05 | -19.2 |
| 7 | 20 | 7.2 | 0.4 | 5.1 | | 10/20 | 4.6E-04 | -14.4 |
| 28 | 0 | 8.9 | 0.5 | 6.0 | 3.1E-05 | 0/10 | 1.1E-01 | -5.6 |
| 28 | 10 | 8.4 | 0.4 | 5.3 | | 0/20 | 7.3E-05 | -19.2 |
| 28 | 20 | 7.2 | 0.4 | 5.1 | | 10/20 | 4.6E-04 | -14.4 |
| 56 | 0 | 14.4 | 0.5 | 3.7 | 1.2E-03 | 0/10 | 9.4E-01 | 0.3 |
| 56 | 10 | 14.5 | 0.8 | 5.6 | | 0/20 | 8.3E-04 | 19.8 |
| 56 | 20 | 17.3 | 0.1 | 0.8 | | 10/20 | 4.0E-03 | 19.4 |

Table B2: Results of Statistical Analysis 0.45 w/c

| Day | FA % | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0 | 7.1 | 0.6 | 7.9 | 1.5E-03 | 0/10 | 2.9E-01 | 5.9 |
| 7 | 10 | 7.5 | 0.7 | 9.7 | | 0/20 | 3.4E-03 | -14.5 |
| 7 | 20 | 6.1 | 0.4 | 5.8 | | 10/20 | 1.4E-03 | -19.2 |
| 28 | 0 | 9.1 | 0.8 | 8.5 | 4.3E-02 | 0/10 | 2.8E-02 | 12.0 |
| 28 | 10 | 10.2 | 0.7 | 6.9 | | 0/20 | 3.2E-01 | 4.4 |
| 28 | 20 | 9.5 | 0.6 | 5.9 | | 10/20 | 9.1E-02 | -6.7 |
| 56 | 0 | 10.0 | 0.6 | 5.9 | 1.8E-03 | 0/10 | 5.0E-03 | 31.6 |
| 56 | 10 | 13.2 | 0.8 | 5.9 | | 0/20 | 3.3E-03 | 35.7 |
| 56 | 20 | 13.6 | 0.8 | 5.9 | | 10/20 | 5.5E-01 | 3.2 |

Table B3: Results of Statistical Analysis 0.50 w/c

| Day | FA % | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | 0 | 7.1 | 0.4 | 6.2 | 2.5E-05 | 0/10 | 3.5E-01 | -3.9 |
| 7 | 10 | 6.8 | 0.5 | 8.0 | | 0/20 | 1.0E-05 | -21.6 |
| 7 | 20 | 5.6 | 0.1 | 2.5 | | 10/20 | 2.9E-04 | -18.4 |
| 28 | 0 | 8.8 | 0.3 | 3.4 | 3.8E-01 | 0/10 | 8.9E-01 | 0.5 |
| 28 | 10 | 8.9 | 0.7 | 7.5 | | 0/20 | 9.9E-02 | -3.4 |
| 28 | 20 | 8.5 | 0.3 | 3.3 | | 10/20 | 2.7E-01 | -3.9 |
| 56 | 0 | 10.1 | 0.3 | 2.5 | 5.2E-04 | 0/10 | 8.7E-01 | -0.7 |
| 56 | 10 | 10.1 | 0.7 | 7.0 | | 0/20 | 1.3E-04 | 25.3 |
| 56 | 20 | 12.7 | 0.2 | 1.4 | | 10/20 | 3.3E-03 | 26.3 |

Appendix C - Effect of Aggregate Type and Gradation

Note: Aggregate Type: Limestone (L), Dolomite (D), Gabbro (G)

Table C1: Results of Statistical Analysis at Day-7 with 0% Fly Ash Content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|---------|-----------|
| 0.40 | L | 9.2 | 0.34 | 3.7 | 1 E-5 | D/G | 0.343 | 1.8 |
| 0.40 | D | 10.5 | 0.50 | 4.7 | | L/G | 5.5 E-6 | 15.0 |
| 0.40 | G | 10.7 | 0.24 | 3.0 | | L/D | 4 E-4 | 13.1 |
| 0.45 | L | 8.8 | 0.49 | 5.6 | 0.210 | D/G | 0.187 | 4.4 |
| 0.45 | D | 9.3 | 0.34 | 3.7 | | L/G | 0.796 | 1.1 |
| 0.45 | G | 8.9 | 0.64 | 6.7 | | L/D | 0.061 | 5.5 |
| 0.50 | L | 7.5 | 0.28 | 3.6 | 0.702 | D/G | 0.530 | 2.6 |
| 0.50 | D | 7.5 | 0.41 | 5.5 | | L/G | 0.513 | 2.6 |
| 0.50 | G | 7.7 | 0.67 | 9.6 | | L/D | 0.968 | 0.0 |

Table C2: Results of Statistical Analysis at Day-28 with 0% Fly Ash Content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|---------|------------|--------|-----------|
| 0.40 | L | 12.4 | 0.47 | 3.8 | 2.1 E-4 | D/G | 0.167 | 3.5 |
| 0.40 | D | 14.1 | 0.67 | 4.8 | | L/G | 0.001 | 9.7 |
| 0.40 | G | 13.6 | 0.46 | 3.4 | | L/D | 5 E-4 | 13.7 |
| 0.45 | L | 11.5 | 0.74 | 6.4 | 0.024 | D/G | 0.006 | 9.0 |
| 0.45 | D | 12.2 | 0.39 | 3.2 | | L/G | 0.293 | 3.5 |
| 0.45 | G | 11.1 | 0.70 | 6.4 | | L/D | 0.081 | 6.1 |
| 0.50 | L | 10.2 | 0.40 | 3.9 | 0.128 | D/G | 0.451 | 3.1 |
| 0.50 | D | 9.7 | 0.54 | 5.6 | | L/G | 0.064 | 7.8 |
| 0.50 | G | 9.4 | 0.76 | 8.1 | | L/D | 0.146 | 4.9 |

Table C3: Results of Statistical Analysis at Day-56 with 0% Fly Ash Content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.40 | L | 14.2 | 0.55 | 3.9 | 0.016 | D/G | 0.048 | 11.0 |
| 0.40 | D | 16.4 | 1.07 | 6.5 | | L/G | 0.230 | 2.8 |
| 0.40 | G | 14.6 | 0.19 | 1.3 | | L/D | 0.004 | 15.5 |
| 0.45 | L | 12.8 | 0.70 | 5.5 | 0.013 | D/G | 0.009 | 18.8 |
| 0.45 | D | 13.3 | 0.43 | 3.2 | | L/G | 0.008 | 15.6 |
| 0.45 | G | 10.8 | 0.85 | 7.9 | | L/D | 0.106 | 3.9 |
| 0.50 | L | 11.2 | 0.28 | 2.5 | 0.165 | D/G | 0.383 | 5.8 |
| 0.50 | D | 10.4 | 0.32 | 3.1 | | L/G | 0.149 | 12.5 |
| 0.50 | G | 9.8 | 1.12 | 11.4 | | L/D | 0.006 | 7.1 |

Table C4: Results of Statistical Analysis at Day-7 with 20% Fly Ash Content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.40 | L | 8.5 | 0.73 | 8.6 | 9 E-5 | D/G | 0.002 | 12.9 |
| 0.40 | D | 7.4 | 0.19 | 2.6 | | L/G | 9 E-5 | 26.6 |
| 0.40 | G | 6.5 | 0.33 | 5.1 | | L/D | 0.011 | 13.8 |
| 0.45 | L | 8.4 | 0.65 | 7.8 | 5 E-9 | D/G | 8 E-5 | 26.5 |
| 0.45 | D | 6.4 | 0.36 | 5.6 | | L/G | 3 E-6 | 52.6 |
| 0.45 | G | 4.9 | 0.25 | 5.0 | | L/D | 7 E-5 | 27.0 |
| 0.50 | L | 7.7 | 0.35 | 4.5 | 8 E-8 | D/G | 0.001 | 14.2 |
| 0.50 | D | 6.0 | 0.37 | 6.1 | | L/G | 1 E-7 | 38.8 |
| 0.50 | G | 5.2 | 0.29 | 5.5 | | L/D | 1 E-5 | 24.8 |

Table C5: Results of Statistical Analysis at Day-28 with 20% Fly Ash content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.40 | L | 12.6 | 0.80 | 6.1 | 3 E-6 | D/G | 2 E-5 | 27.3 |
| 0.40 | D | 13.2 | 0.60 | 4.8 | | L/G | 2 E-4 | 23.8 |
| 0.40 | G | 9.6 | 1.00 | 10.4 | | L/D | 0.167 | 4.8 |
| 0.45 | L | 11.2 | 0.90 | 8 | 1 E-8 | D/G | 3 E-7 | 33.7 |
| 0.45 | D | 10.1 | 0.60 | 6 | | L/G | 2 E-5 | 40.2 |
| 0.45 | G | 6.7 | 0.30 | 4.9 | | L/D | 0.033 | 9.8 |
| 0.50 | L | 10.3 | 0.70 | 6.5 | 1 E-7 | D/G | 4 E-6 | 26.8 |
| 0.50 | D | 9.7 | 0.50 | 5.6 | | L/G | 2 E-6 | 31.1 |
| 0.50 | G | 7.1 | 0.40 | 6.3 | | L/D | 0.109 | 5.8 |

Table C6: Results of Statistical Analysis at Day-56 with 20% Fly Ash content

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.40 | L | 16.4 | 0.99 | 6 | 0.002 | D/G | 0.003 | 33.0 |
| 0.40 | D | 20 | 0.82 | 4.1 | | L/G | 0.008 | 18.3 |
| 0.40 | G | 13.4 | 1.59 | 11.9 | | L/D | 0.001 | 22.0 |
| 0.45 | L | 14 | 1.20 | 8.4 | 3 E-4 | D/G | 6 E-4 | 46.0 |
| 0.45 | D | 16.1 | 1.10 | 7.1 | | L/G | 2 E-4 | 37.9 |
| 0.45 | G | 8.7 | 0.60 | 6.7 | | L/D | 0.043 | 15.0 |
| 0.50 | L | 13.2 | 0.70 | 5.4 | 0.003 | D/G | 8 E-4 | 40.8 |
| 0.50 | D | 15.2 | 0.80 | 5.3 | | L/G | 1 E-4 | 31.8 |
| 0.50 | G | 9 | 0.90 | 9.5 | | L/D | 0.007 | 15.2 |

Table C7: Results of Statistical Analysis at Day-7, 0.45 w/cm and 20% FA, Limestone

Aggregate

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|---------|------------|--------|-----------|
| 0.45 | 56 | 8.4 | 0.65 | 7.8 | 4.4 E-3 | 57/56 | 0.103 | 9.5 |
| 0.45 | 57 | 7.6 | 0.37 | 4.9 | | 56/67 | 0.008 | 15.1 |
| 0.45 | 67 | 7.3 | 0.17 | 2.4 | | 57/67 | 0.106 | 4.1 |

Table C8: Results of Statistical Analysis at Day-28, 0.45 w/cm and 20% FA, Limestone

Aggregate

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.45 | 56 | 11.2 | 0.90 | 8.0 | 0.125 | 57/56 | 0.834 | 1.8 |
| 0.45 | 57 | 11.0 | 0.44 | 4.0 | | 56/67 | 0.028 | 7.7 |
| 0.45 | 67 | 10.4 | 0.28 | 2.7 | | 57/67 | 0.190 | 5.8 |

Table C9: Results of Statistical Analysis at Day-56, 0.45 w/cm and 20% FA, Limestone

Aggregate

| w/c | Agg. Type | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|-----------|--------------------------|-------------------------------|-----------------------------|-------|------------|--------|-----------|
| 0.45 | 56 | 14.0 | 1.17 | 8.3 | 0.205 | 57/56 | 0.655 | 4.3 |
| 0.45 | 57 | 14.6 | 0.60 | 4.1 | | 56/67 | 0.236 | 0.0 |
| 0.45 | 67 | 14.0 | 0.35 | 2.5 | | 57/67 | 0.298 | 4.3 |

Appendix D - Effect of Admixture Addition

Table D1: Results of Statistical Analysis 0.40 w/cm with 0% Fly Ash

| Day | Admix | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 7 | OO | 8.9 | 0.5 | 6.0 | | OO/AO | 5.2E-02 | 6.8 |
| 7 | AO | 9.5 | 0.4 | 4.4 | 1.6E-01 | OO/AW | 1.0E+00 | 0.0 |
| 7 | AW | 8.9 | 0.8 | 8.9 | | AO/AW | 1.3E-01 | (6.4) |
| 28 | OO | 11.7 | 0.4 | 3.6 | | OO/AO | 3.5E-01 | 1.8 |
| 28 | AO | 11.9 | 0.3 | 2.6 | 5.5E-01 | OO/AW | 6.8E-01 | (1.5) |
| 28 | AW | 11.6 | 0.9 | 7.6 | | AO/AW | 3.4E-01 | (3.2) |
| 56 | OO | 14.4 | 0.5 | 3.7 | | OO/AO | 8.5E-02 | (5.3) |
| 56 | AO | 13.6 | 0.2 | 1.8 | 9.2E-02 | OO/AW | 8.7E-02 | (8.6) |
| 56 | AW | 13.2 | 0.8 | 6.0 | | AO/AW | 3.8E-01 | (3.4) |

Table D2: Results of Statistical Analysis 0.40 w/cm with 10% Fly Ash

| Day | Admix | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 7 | OO | 8.4 | 0.4 | 5.3 | | OO/AO | 1.4E-01 | 5.2 |
| 7 | AO | 8.8 | 0.5 | 5.5 | 2.1E-03 | OO/AW | 1.8E-03 | 15.1 |
| 7 | AW | 9.7 | 0.6 | 6.1 | | AO/AW | 2.3E-02 | 9.4 |
| 28 | OO | 11.5 | 0.7 | 5.8 | | OO/AO | 7.9E-01 | 0.7 |
| 28 | AO | 11.6 | 0.3 | 2.9 | 2.1E-03 | OO/AW | 7.5E-03 | 12.5 |
| 28 | AW | 12.9 | 0.8 | 6.3 | | AO/AW | 3.8E-03 | 11.7 |
| 56 | OO | 14.5 | 0.8 | 5.6 | | OO/AO | 8.2E-01 | 1.2 |
| 56 | AO | 14.6 | 0.9 | 6.4 | 5.4E-02 | OO/AW | 4.2E-02 | 13.8 |
| 56 | AW | 16.5 | 0.8 | 5.2 | | AO/AW | 6.6E-02 | 12.5 |

Table D3: Results of Statistical Analysis 0.40 w/cm with 20% Fly Ash

| Day | Admix | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 7 | OO | 7.2 | 0.4 | 5.1 | | OO/AO | 2.7E-01 | 3.5 |
| 7 | AO | 7.4 | 0.4 | 5.0 | 7.4E-07 | OO/AW | 6.5E-06 | 24.4 |
| 7 | AW | 9.0 | 0.3 | 3.8 | | AO/AW | 2.5E-05 | 20.3 |
| 28 | OO | 12.1 | 0.5 | 4.4 | | OO/AO | 3.9E-01 | (2.6) |
| 28 | AO | 11.8 | 0.7 | 5.7 | 7.3E-09 | OO/AW | 2.6E-07 | 28.6 |
| 28 | AW | 15.5 | 0.4 | 2.9 | | AO/AW | 4.6E-07 | 32.0 |
| 56 | OO | 17.3 | 0.1 | 0.8 | | OO/AO | 3.7E-01 | (4.1) |
| 56 | AO | 16.6 | 1.2 | 7.4 | 2.7E-04 | OO/AW | 5.7E-04 | 31.8 |
| 56 | AW | 22.7 | 0.9 | 4.1 | | AO/AW | 2.2E-03 | 37.4 |

Table D4: Results of Statistical Analysis 0.45 w/cm with 0% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 7.1 | 0.6 | 7.9 | | OO/AO | 1.7E-04 | 24.8 |
| 7 | AO | 8.9 | 0.5 | 5.6 | 4.8E-04 | OO/AW | 1.4E-03 | 41.7 |
| 7 | AW | 10.1 | 1.6 | 15.6 | | AO/AW | 1.0E-01 | 13.5 |
| 28 | OO | 9.1 | 0.8 | 8.5 | | OO/AO | 2.7E-04 | 25.9 |
| 28 | AO | 11.5 | 0.7 | 6.2 | 4.1E-04 | OO/AW | 1.2E-03 | 40.0 |
| 28 | AW | 12.7 | 1.8 | 14.4 | | AO/AW | 1.4E-01 | 11.2 |
| 56 | OO | 10.0 | 0.6 | 5.9 | | OO/AO | 1.3E-02 | 34.4 |
| 56 | AO | 13.5 | 1.3 | 9.3 | 1.6E-02 | OO/AW | 1.9E-02 | 54.2 |
| 56 | AW | 15.4 | 2.4 | 15.5 | | AO/AW | 2.7E-01 | 14.7 |

Table D5: Results of Statistical Analysis 0.45 w/cm with 10% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 7.5 | 0.7 | 9.7 | | OO/AO | 1.6E-02 | 13.5 |
| 7 | AO | 8.6 | 0.5 | 5.3 | 4.6E-03 | OO/AW | 6.7E-03 | 25.5 |
| 7 | AW | 9.5 | 1.2 | 12.4 | | AO/AW | 1.1E-01 | 10.5 |
| 28 | OO | 10.2 | 0.7 | 6.9 | | OO/AO | 2.0E-01 | 5.4 |
| 28 | AO | 10.7 | 0.7 | 6.2 | 6.2E-04 | OO/AW | 1.7E-03 | 25.9 |
| 28 | AW | 12.8 | 1.4 | 10.6 | | AO/AW | 6.9E-03 | 19.5 |
| 56 | OO | 13.2 | 0.8 | 5.9 | | OO/AO | 9.8E-02 | 8.2 |
| 56 | AO | 14.3 | 0.4 | 2.8 | 2.3E-01 | OO/AW | 1.8E-01 | 16.3 |
| 56 | AW | 15.3 | 2.2 | 14.0 | | AO/AW | 4.5E-01 | 7.5 |

Table D6: Results of Statistical Analysis 0.45 w/cm with 20% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 6.1 | 0.4 | 5.8 | | OO/AO | 5.9E-07 | 31.0 |
| 7 | AO | 8.0 | 0.2 | 2.7 | 2.6E-10 | OO/AW | 5.2E-08 | 49.9 |
| 7 | AW | 9.1 | 0.4 | 4.1 | | AO/AW | 6.8E-05 | 14.4 |
| 28 | OO | 9.5 | 0.6 | 5.9 | | OO/AO | 1.2E-05 | 26.4 |
| 28 | AO | 12.0 | 0.5 | 4.4 | 7.0E-11 | OO/AW | 2.9E-09 | 51.1 |
| 28 | AW | 14.4 | 0.3 | 1.8 | | AO/AW | 1.8E-06 | 19.6 |
| 56 | OO | 13.6 | 0.8 | 5.9 | | OO/AO | 2.9E-03 | 29.7 |
| 56 | AO | 17.6 | 0.7 | 4.1 | 4.1E-05 | OO/AW | 1.8E-04 | 52.1 |
| 56 | AW | 20.7 | 0.5 | 2.2 | | AO/AW | 3.4E-03 | 17.3 |

Table D7: Results of Statistical Analysis 0.50 w/cm with 0% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 7.1 | 0.4 | 6.2 | | OO/AO | 1.1E-06 | 28.6 |
| 7 | AO | 9.1 | 0.2 | 2.1 | 1.9E-09 | OO/AW | 3.0E-07 | 42.6 |
| 7 | AW | 10.1 | 0.4 | 4.3 | | AO/AW | 4.5E-04 | 10.9 |
| 28 | OO | 8.8 | 0.3 | 3.4 | | OO/AO | 6.2E-07 | 24.8 |
| 28 | AO | 11.0 | 0.4 | 3.5 | 4.9E-09 | OO/AW | 2.9E-07 | 44.4 |
| 28 | AW | 12.8 | 0.7 | 5.8 | | AO/AW | 4.8E-04 | 15.7 |
| 56 | OO | 10.1 | 0.3 | 2.5 | | OO/AO | 2.0E-04 | 24.6 |
| 56 | AO | 12.6 | 0.2 | 1.7 | 1.9E-05 | OO/AW | 2.1E-04 | 42.6 |
| 56 | AW | 14.5 | 0.5 | 3.6 | | AO/AW | 5.2E-03 | 14.5 |

Table D8: Results of Statistical Analysis 0.50 w/cm with 10% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 6.8 | 0.5 | 8.0 | | OO/AO | 1.3E-04 | 22.2 |
| 7 | AO | 8.4 | 0.3 | 3.4 | 2.6E-07 | OO/AW | 5.9E-06 | 35.3 |
| 7 | AW | 9.2 | 0.4 | 4.4 | | AO/AW | 1.3E-03 | 10.7 |
| 28 | OO | 8.9 | 0.7 | 7.5 | | OO/AO | 1.1E-03 | 15.8 |
| 28 | AO | 10.3 | 0.4 | 3.5 | 1.3E-06 | OO/AW | 1.8E-05 | 34.7 |
| 28 | AW | 12.0 | 0.7 | 6.1 | | AO/AW | 5.2E-04 | 16.3 |
| 56 | OO | 10.1 | 0.7 | 7.0 | | OO/AO | 7.5E-03 | 24.4 |
| 56 | AO | 12.5 | 0.5 | 3.7 | 1.4E-04 | OO/AW | 6.4E-04 | 48.9 |
| 56 | AW | 15.0 | 0.5 | 3.5 | | AO/AW | 3.7E-03 | 19.7 |

Table D9: Results of Statistical Analysis 0.50 w/cm with 20% Fly Ash

| Day | Admix | Mean Resistivity (KΩ-cm) | Std. Dev. Resistivity (KΩ-cm) | COV (%) Resistivity (KΩ-cm) | ANOVA | Comparison | T-test | Diff. (%) |
|-----|-------|--------------------------|-------------------------------|-----------------------------|---------|------------|---------|-----------|
| 7 | OO | 5.6 | 0.3 | 2.5 | | OO/AO | 4.0E-08 | 27.6 |
| 7 | AO | 7.1 | 0.4 | 3.0 | 2.7E-10 | OO/AW | 3.2E-08 | 39.8 |
| 7 | AW | 7.8 | 0.7 | 4.2 | | AO/AW | 1.7E-03 | 9.6 |
| 28 | OO | 8.5 | 0.6 | 3.3 | | OO/AO | 2.4E-04 | 15.4 |
| 28 | AO | 9.9 | 1.0 | 5.1 | 4.0E-08 | OO/AW | 1.8E-07 | 33.4 |
| 28 | AW | 11.4 | 1.0 | 4.2 | | AO/AW | 3.0E-04 | 15.6 |
| 56 | OO | 12.7 | 0.4 | 1.4 | | OO/AO | 1.7E-01 | 5.2 |
| 56 | AO | 13.4 | 1.3 | 4.9 | 4.8E-03 | OO/AW | 4.8E-03 | 19.7 |
| 56 | AW | 15.2 | 1.5 | 4.9 | | AO/AW | 3.3E-02 | 13.8 |

Appendix E - Results for Absorption Test

Table E1: Results of Statistical Analysis for Effect of w/cm Ratio on Percent Absorption

| w/c | FA % | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 0.40 | 0 | 3.4 | 0.0 | 0.2 | | 0.40/0.45 | 9.5E-06 | 45.5 |
| 0.45 | 0 | 4.9 | 0.1 | 1.9 | 9.5E-08 | 0.40/0.50 | 5.3E-08 | 38.4 |
| 0.50 | 0 | 4.7 | 0.0 | 0.4 | | 0.45/0.50 | 1.3E-02 | -4.9 |

Table E2: Results of Statistical Analysis for Effect of Fly Ash Ratio on Percent Absorption

| w/c | FA % | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 0.40 | 0 | 3.4 | 0.0 | 0.2 | | 0/10 | 2.2E-01 | 1.7 |
| 0.40 | 10 | 3.4 | 0.1 | 2.0 | 2.1E-02 | 0/20 | 1.8E-02 | 6.3 |
| 0.40 | 20 | 3.6 | 0.1 | 2.7 | | 10/20 | 8.3E-02 | 4.5 |
| 0.45 | 0 | 4.9 | 0.1 | 1.9 | | 0/10 | 2.0E-03 | -14.9 |
| 0.45 | 10 | 4.2 | 0.2 | 3.6 | 3.9E-04 | 0/20 | 1.0E-03 | -21.1 |
| 0.45 | 20 | 3.9 | 0.2 | 4.9 | | 10/20 | 9.6E-02 | -7.3 |
| 0.50 | 0 | 3.4 | 0.1 | 2.0 | | 0/10 | 2.7E-04 | 34.2 |
| 0.50 | 10 | 4.6 | 0.2 | 3.3 | 1.3E-04 | 0/20 | 8.6E-04 | 32.2 |
| 0.50 | 20 | 4.5 | 0.2 | 4.5 | | 10/20 | 6.7E-01 | -1.5 |

Table E3: Results of Statistical Analysis for Effect of Admixture on Percent Absorption

| w/c | FA % | Mean Resistivity (K Ω -cm) | Std. Dev. Resistivity (K Ω -cm) | COV (%) Resistivity (K Ω -cm) | ANOVA | Comparison | T-test | Diff. (%) |
|------|------|-----------------------------------|--|--------------------------------------|---------|------------|---------|-----------|
| 0.40 | OO | 3.4 | 0.0 | 0.2 | | OO/AO | 4.2E-03 | 14.5 |
| 0.40 | OA | 3.9 | 0.1 | 3.7 | 3.2E-03 | OO/AW | 5.4E-03 | 24.3 |
| 0.40 | AW | 4.2 | 0.3 | 6.2 | | AO/AW | 1.2E-01 | 8.6 |
| 0.45 | OO | 4.9 | 0.1 | 1.9 | | OO/AO | 3.2E-01 | -4.8 |
| 0.45 | OA | 4.7 | 0.3 | 6.2 | 3.7E-01 | OO/AW | 1.3E-01 | -23.7 |
| 0.45 | AW | 3.7 | 0.3 | 7.1 | | AO/AW | 7.7E-01 | -19.9 |
| 0.50 | OO | 4.7 | 0.0 | 0.4 | | OO/AO | 8.6E-05 | -15.3 |
| 0.50 | OA | 4.0 | 0.1 | 1.9 | 4.2E-02 | OO/AW | 2.3E-01 | -7.7 |
| 0.50 | AW | 4.3 | 0.4 | 10.3 | | AO/AW | 2.5E-01 | 8.9 |

Appendix F - Elaboration of Procedure A

The ANOVA statistical method was used to analyze the variation in the mean gain in resistivity as per level of %FA and w/c. The first hypothesis test performed compared the percentages of fly ash replacement to determine if there is a significant difference among the mean resistivity gain values between the five contents of fly ash (0%, 5%, 10%, 15%, 20%, and 25%). First, the concrete mixtures were categorized into groups (levels) with respect to their fly ash content (0%, 5%, 10%, 15%, 20% and 25% replacement). The resistivity data were analyzed to determine if there is a significant difference among the levels based on different slope combinations. The possible slope combinations between test days are (1-3), (3-7), (7-14), (7-21), (7-28), (14-21), (14-28) & (21-28). To determine the slope at a given age range, Equation F1 was used to calculate the change in resistivity over time.

$$s = \frac{y_2 - y_1}{x_2 - x_1} \quad (\text{Eq. F1})$$

The surface resistivity measurements were determined at days 1, 3, 7, 14, 21 & 28, which implies that a single concrete cylinder has six resistivity values throughout the testing period; therefore, there is a violation of independency. Although the observations are dependent, the approach used herein considers data obtained for a given day or slope combination as an individual data set. Second, as will be shown later, the errors or residuals are assumed to be normally distributed. This was determined by normally predicted plots, which is the difference between real values and determined values. Third, the Levene's test was performed to determine if the variances in results are equal or significantly different. Levene's test is defined as an inferential statistic used to assess the equality of variances for a variable calculated for two or more groups. If the variance is found equal, ANOVA was performed. ANOVA is the analysis of variations between more than two groups. If at least one variance is significantly different, then Welch's test

is used. Welch's test is a two-sample location test, which is used to test the hypothesis that two populations have equal means and unequal variances.

After fulfilling the assumptions of ANOVA, the null hypothesis was verified to determine whether a slope combination can differentiate mixtures of different fly ash content. Results of the ANOVA analysis for all possible slope combinations are presented in Table F1. First, Levene's test was performed to analyze if the hypothesis for equal variance is accepted or rejected. It was found that for slope combinations (1-3), (3-7), (7-14) and (7-21) the results showed equal variances. Whereas, for slopes (7-28), (14-21), (14-28) and (21-28), Levene's test results showed unequal variances and hypothesis was rejected.

Subsequently, ANOVA was used for sets of equal variances, and Welch's test was used for sets of unequal variances. If there is no significant difference found among the mean slopes combination, then that slope combination is rejected. It was established that slope combinations (1-3) and (3-7) rejected the null hypothesis meaning there is a significant difference in the resistivity slopes for the fly ash percentages (levels). On the other hand, the slope combinations (7-14) and (7-21) failed to reject the null hypothesis; thus, these slope combinations are not suitable to identify the presence of fly ash content in a concrete mixture. For slope combinations evaluated using Welch's Test, (7-28) accepted the hypothesis meaning that there is no significant difference between the percentages of fly ash. Whereas, the slope combinations (14-21), (14-28) & (21-28) rejected the hypothesis; thus, there is a significant difference between the percentages of fly ash (levels).

Finally, for slope combinations rejecting the Null hypothesis, Tukey's test was used to identify the differences between the three %FA groups. It was found that no slope combinations except for slope (1-3) could differentiate between the 0% fly ash (No fly

ash concrete mixture) and the 5%, 10%, 15%, 20% or 25% fly ash replacement mixture (with fly ash mixtures). Hence, the slope combination (1-3) is the only option that can differentiate between mixtures with "No fly ash" and mixtures containing "Fly ash," as shown in Table F1.

This enabled the development of the first parameter to distinguish mixtures containing fly ash replacement from mixtures containing no supplementary cementitious materials. A range of resistivity values was determined for slope combination (1-3) representing a 95% confidence interval. Presented in Table F2, lower and upper limits were calculated for both "No Fly Ash" mixtures and mixtures containing "Fly Ash."

Subsequently, the potential w/c ratio used in the mixture could be determined knowing whether a mixture contains fly ash or not. Based on previous observation, it was found that the mean resistivity values of mixtures of 0.40, 0.45, and 0.50 w/cm with no fly ash are distinct from each other at a 95% confidence level after 14 days of continuous immersion curing. Therefore, testing days 14, 21 and 28 are viable candidates for w/cm identification. As for the 0.55 w/cm and 0.6 w/cm mixtures, they are not significantly different from each other; however, their combined range in values are distinct from that of the 0.50 w/cm. Thus, w/c identification categories were established for mixtures of 0.40, 0.45 and 0.50 w/cm ratios. The range in resistivity values were calculated based on a 95% confidence interval from the mean. Results are shown in F3 Practically, day-14 was selected to provide a user with an early estimate and day-28 was selected since other quality control tests such as compression strength are commonly performed on this day. This would permit both test to be performed sequentially and on the same sample.

Similarly, the 95% confidence limits were calculated for concrete mixtures containing 5%, 10%, 15%, 20% and 25% class-C fly ash. The w/c identification categories were

established for mixtures of 0.40, 0.45 and 0.50 w/cm ratios containing a minimum of 5% FA and a maximum of 25% FA. The range in resistivity values representing a 95% confidence interval from the mean are shown in Table F4. As seen in the latter, there is a slight overlap of 0.2 k Ω -cm at the upper boundary of the 0.50 w/cm mixture and lower boundary of the 0.45 w/cm. Therefore, from the result of the surface resistivity test performed on day-14 or day-28, using ranges in Table F6, the w/cm of a mixture could be estimated.

However, the presence of gaps between categories or the overlap of categories present zones of uncertainty. In addition, in the case of a resistivity value falling below the lower limits of "0.5 w/cm" concrete, in this scenario the mixture could be considered as "> 0.5 w/cm" however, there is no certainty in this statement. Similarly, for resistivity results higher than that of the upper limit of "0.4 w/cm" concrete, the mixture could be considered as "< 0.4 w/cm" however, there is no certainty in this statement.

The criteria developed was then trialed in a laboratory setting to determine the validity of the method. Several mixtures were prepared for the trial varying %FA replacement and w/cm along with varying paste content. The paste volume of the concrete mixtures ranged from 27% to 31%. Moreover, admixtures such as an air entrainment agent (AE) and a mid-range water reducer were also added to some of the mixtures (in accordance with recommended manufacturer dosage) to determine their effect on the resulting outcome.

Table F5 presents the results obtained for the first step of the method, the calculated slope of resistivity between days 1 and 3. The values were compared with the limits listed in Table F2. Out of the 32 concrete mixtures, 28 concrete mixtures were correctly identified (96% success rate) with respect to containing fly ash as a supplementary cementitious material. Some mixtures that did not meet the criteria did not contain any

fly ash. As for the other mixture that failed the validation, the calculated slope for the mixtures containing 10% fly ash with Limestone aggregate, and 10% and 20% fly ash with Dolomite aggregates were superior to the upper boundary of the "Fly Ash" category; therefore, the validation is deemed uncertain.

After successful validation of identification of fly ash content in concrete mixtures, the w/cm of concrete mixtures were validated with respect to their "No Fly Ash" or "Fly Ash" concrete category. The mean resistivity values that fall under the gap between the limits of two concrete mixtures would be categorized as uncertain. In Table F6, the concrete mixtures with no fly ash content, at days 14 and 28 were validated for statistical criteria to determine the possible w/c ratios.

Starting with the day-14 assessment, out of 7 concrete mixtures, 5 concrete mixtures were correctly identified (71% success rate); however, the success rate increased when evaluating the mixtures on day-28 (86%). In Table F7, the mixtures containing fly ash content are validated at days 14 and 28. At day 14, 13 out of 21 mixtures with 62% success rate are either correctly identified or classified as uncertain; whereas, at day 28, the success rate increased at 86%. The possibility of correct identification of w/cm ratio in concrete mixtures is more successfully achieved at day 28 than day 14 due to overlapping of confidence limits. However, variation in curing temperature outside the ASTM specified limits (Gulrez and Hartell 2017 a, b) were observed in 0.45 w/cm ratio with 10% and 20% was observed.

The identification of concrete mixtures with "No Fly Ash" or "Fly Ash" content from Table F2, and possible w/cm having "No fly ash" and "Fly Ash" content from Tables F3 and F4, are successfully validated.

Table F1: Results of Levene’s Test, ANOVA and Tukey’s Test for slope combinations

| Slope Combination | Mean of Slope | Equal Variances | ANOVA Test | Tukey’s Test Group-I | Tukey’s Test Group-II |
|-------------------|---------------|----------------------|----------------------|----------------------|-----------------------|
| 1-3 | 0.89 | p-val = 0.1419 – Ho | p-val < 0.001 (Ho X) | 0% | 5% - 25% |
| 3-7 | 0.49 | p-val = 0.2722 – Ho | p-val = 0.027 (Ho X) | 10% – 20% | 0% - 20% |
| 7-14 | 0.17 | p-val = 0.1056 – Ho | p-val = 0.770 – Ho | No difference | |
| 7-21 | 0.14 | p-val = 0.0600 – Ho | p-val = 0.556 – Ho | No difference | |
| 7-28 | 0.12 | p-val = 0.049 (Ho X) | p-val = 0.274 – Ho | No difference | |
| 14-21 | 0.12 | p-val = 0.002 (Ho X) | p-val < 0.001 (Ho X) | 10% – 20% | 0% - 10% |
| 14-28 | 0.10 | p-val = 0.006 (Ho X) | p-val < 0.001 (Ho X) | 20% | 0% - 10% |
| 21-28 | 0.09 | p-val < 0.001 (Ho X) | p-val = 0.044 (Ho X) | 0% – 20% | 0% - 10% |

Note: Ho: Null hypothesis, meaning it is correct. HoX: the Null hypothesis is rejected; p-val is the P-value.

Table F2: Range in (1-3) resistivity slope (KΩ-cm/day) combination values for concrete mixtures

| Fly Ash Content | Slope Mean | Lower Limit | Upper Limit |
|-----------------|------------|-------------|-------------|
| No Fly Ash | 0.5 | 0.4 | 0.6 |
| Fly Ash | 1.1 | >0.6 | 1.2 |

Table F3: Surface resistivity 95% confidence limits at test ages 14 and 28 days for concrete mixtures containing no fly ash

| w/cm ratio | Mean Surface Resistivity (kΩ-cm) Day-14 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-14 | Mean Surface Resistivity (kΩ-cm) Day-28 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-28 |
|------------|---|---|---|---|
| 0.40 | 11.0 | 10.6-11.5 | 12.4 | 11.9-12.8 |
| 0.45 | 10.2 | 9.7-10.6 | 11.5 | 11.0-12.0 |
| 0.50 | 8.9 | 8.5-9.4 | 10.2 | 9.7-10.6 |

Table F4: Surface resistivity 95% confidence limits at test ages 14 and 28 days for concrete mixtures containing fly ash

| w/cm ratio | Mean Surface Resistivity (kΩ-cm) Day-14 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-14 | Mean Surface Resistivity (kΩ-cm) Day-28 | 95% Conf. Limits Surface Resistivity (kΩ-cm) Day-28 |
|------------|---|---|---|---|
| 0.40 | 10.7 | 10.2-11.1 | 10.7 | 12.0-13.2 |
| 0.45 | 9.3 | 8.9-9.8 | 9.3 | 10.6-11.7 |
| 0.50 | 8.7 | 8.2-9.1 | 8.7 | 9.7-10.8 |

Table F5: Validation of fly ash content in concrete mixtures

| Mixture Description | Slope Combination (1-3) | Validated |
|---------------------|-------------------------|-----------|
| 40-00-AW-1-0-1 | 0.4 | Yes |
| 45-00-AW-1-0-1 | 0.6 | Yes |
| 50-00-AW-1-0-1 | 0.6 | Yes |
| 45-10-AO-1-1-1 | 0.9 | Yes |
| 45-20-AO-1-1-1 | 1 | Yes |
| 40-00-OO-1-0-1 | 0.6 | Yes |
| 45-00-OO-1-0-1 | 1.2 | No |
| 50-00-OO-1-0-1 | 0.5 | Yes |
| 40-10-OO-1-1-1 | 1.4 | Uncertain |
| 45-10-OO-1-1-1 | 1.1 | Yes |
| 50-10-OO-1-1-1 | 1.1 | Yes |
| 40-20-OO-1-1-1 | 1.2 | Yes |
| 45-20-OO-1-1-1 | 1.2 | Yes |
| 50-20-OO-1-1-1 | 1.1 | Yes |
| 40-00-OO-1-0-1 | 0.8 | No |
| 50-00-OO-1-0-1 | 0.1 | Yes |
| 45-10-OO-1-3-1 | 1.2 | Yes |
| 50-20-OO-1-3-1 | 0.7 | Yes |
| 40-10-OO-1-4-1 | 0.8 | Yes |
| 40-20-OO-1-4-1 | 0.9 | Yes |
| 45-10-OO-1-4-1 | 0.7 | Yes |
| 45-20-OO-1-4-1 | 0.7 | Yes |
| 50-10-OO-1-4-1 | 0.8 | Yes |
| 40-10-OO-1-1-2 | 1.3 | Uncertain |
| 40-20-OO-1-1-2 | 1.3 | Uncertain |
| 45-10-OO-1-1-2 | 1.4 | Uncertain |
| 45-20-OO-1-1-2 | 1.4 | Uncertain |
| 50-10-OO-1-1-2 | 1.5 | Uncertain |
| 50-20-OO-1-1-2 | 1.2 | Yes |
| 40-00-OO-1-0-3 | 1.7 | No |
| 45-00-OO-1-0-3 | 0.8 | No |
| 50-00-OO-1-0-3 | 0.6 | Yes |

Table F6: Validation of w/cm ratios with no fly ash content concrete at days 14 and 28

| Day | Mixture Description | Resistivity Mean | Determined w/cm | Validated |
|-----|---------------------|------------------|-----------------|-----------|
| 14 | 40-00-AW-1-0-1 | 10.6 | 0.4 | Yes |
| 14 | 45-00-AW-1-0-1 | 10 | 0.45 | Yes |
| 14 | 50-00-AW-1-0-1 | 8.8 | 0.5 | Yes |
| 14 | 50-00-OO-1-0-1 | 9.9 | 0.45 | No |
| 14 | 40-00-OO-1-0-1 | 7.6 | >0.50 | No |
| 14 | 50-00-OO-1-0-1 | 8.1 | >0.50 | Uncertain |
| 14 | 50-00-OO-1-0-3 | 8.6 | 0.5 | Yes |
| 28 | 40-00-AW-1-0-1 | 11.4 | 0.45 | No |
| 28 | 45-00-AW-1-0-1 | 11.1 | 0.45 | Yes |
| 28 | 50-00-AW-1-0-1 | 10.2 | 0.5 | Yes |
| 28 | 50-00-OO-1-0-1 | 9.6 | >0.50 | Uncertain |
| 28 | 40-00-OO-1-0-1 | 14.7 | < 0.40 | Uncertain |
| 28 | 50-00-OO-1-0-1 | 10.2 | 0.5 | Yes |
| 28 | 50-00-OO-1-0-3 | 9.4 | >0.50 | Uncertain |

Table F7: Validation of w/cm ratios with fly ash content concrete at days 14 & 28

| Day | Mixture Description | Resistivity Mean | Determined w/cm | Validated |
|-----|---------------------|------------------|-----------------|-----------|
| 14 | 45-10-AO-1-1-1 | 10.2 | 0.4 | No |
| 14 | 45-20-AO-1-1-1 | 9 | 0.45 | Yes |
| 14 | 40-10-OO-1-1-1 | 12.7 | <0.4 | Uncertain |
| 14 | 45-10-OO-1-1-1 | 11 | 0.4 | No |
| 14 | 50-10-OO-1-1-1 | 7.9 | >0.5 | Uncertain |
| 14 | 40-20-OO-1-1-1 | 12.3 | <0.4 | Uncertain |
| 14 | 45-20-OO-1-1-1 | 10.3 | 0.4 | No |
| 14 | 50-20-OO-1-1-1 | 8.7 | 0.5 | Yes |
| 14 | 45-10-OO-1-3-1 | 10 | 0.5 | No |
| 14 | 50-20-OO-1-3-1 | 8.7 | 0.5 | Yes |
| 14 | 40-10-OO-1-4-1 | 12.2 | 0.4 | Yes |
| 14 | 40-20-OO-1-4-1 | 10.8 | 0.4 | Yes |
| 14 | 45-10-OO-1-4-1 | 9.6 | 0.45 | Yes |
| 14 | 45-20-OO-1-4-1 | 8.3 | 0.5 | No |
| 14 | 50-10-OO-1-4-1 | 8.8 | 0.5 | Yes |
| 14 | 40-10-OO-1-1-2 | 10.5 | 0.4 | Yes |
| 14 | 40-20-OO-1-1-2 | 9.1 | 0.45 | No |
| 14 | 45-10-OO-1-1-2 | 8.8 | 0.5 | No |
| 14 | 45-20-OO-1-1-2 | 7.7 | >0.5 | No |
| 14 | 50-10-OO-1-1-2 | 8.6 | 0.5 | Yes |
| 14 | 50-20-OO-1-1-2 | 7.2 | >0.5 | Uncertain |
| 28 | 45-10-AO-1-1-1 | 12 | 0.4 | No |
| 28 | 45-20-AO-1-1-1 | 11 | 0.45 | Yes |
| 28 | 40-10-OO-1-1-1 | 15.7 | <0.4 | Uncertain |
| 28 | 45-10-OO-1-1-1 | 11.7 | 0.45 | Yes |
| 28 | 50-10-OO-1-1-1 | 9 | >0.5 | Uncertain |
| 28 | 40-20-OO-1-1-1 | 15.6 | <0.4 | Uncertain |
| 28 | 45-20-OO-1-1-1 | 13.2 | 0.4 | No |
| 28 | 50-20-OO-1-1-1 | 10.3 | 0.5 | Yes |
| 28 | 45-10-OO-1-3-1 | 11.5 | 0.45 | Yes |
| 28 | 50-20-OO-1-3-1 | 10.6 | 0.5 | Yes |
| 28 | 40-10-OO-1-4-1 | 14.2 | <0.4 | Uncertain |
| 28 | 40-20-OO-1-4-1 | 13.7 | <0.4 | Uncertain |
| 28 | 45-10-OO-1-4-1 | 11.2 | 0.45 | Yes |
| 28 | 45-20-OO-1-4-1 | 11.3 | 0.45 | Yes |
| 28 | 50-10-OO-1-4-1 | 10 | 0.5 | Yes |
| 28 | 40-10-OO-1-1-2 | 12.9 | 0.4 | Yes |
| 28 | 40-20-OO-1-1-2 | 13.2 | 0.4 | Yes |
| 28 | 45-10-OO-1-1-2 | 10.6 | 0.45 | Yes |
| 28 | 45-20-OO-1-1-2 | 10.1 | 0.5 | No |
| 28 | 50-10-OO-1-1-2 | 10.3 | 0.5 | Yes |
| 28 | 50-20-OO-1-1-2 | 9.7 | 0.5 | Yes |

Appendix G-

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Mixture Design Identification and Information for Field Study From June 2015 to August 2015

| OSU # | ODOT # | Residency | Producer | Casting Date | W/C | Fly Ash (%) | Cement | Fly Ash | Coarse | Sand | Air Entrainer | Water Reducer | Other | Slump (in) | Air (%) |
|-------|--------|-----------|----------|--------------|------|-------------|-------------------|---------------------|-----------------------|---------------------|------------------|------------------------|--|------------|---------|
| 1 | 6-3 | 6 | K | 6/15/2015 | 0.41 | 20% | Type II, Holicim | - | Dolese Cooperton #57/ | Eagle Sand | Euclid AEA-92S | - | - | 2 | 6.8 |
| 2 | 7-2 | 6 | K | 6/16/2015 | 0.41 | 20% | Type II, Holicim | - | Dolese Cooperton #57/ | Eagle Sand | Euclid AEA-92S | - | - | 1.25 | 4.6 |
| 3 | 20 | 4 | E | 6/17/2015 | 0.44 | 20% | Type I, Holicim | Headwaters Class C | Martin Marietta #57 | Martin Marietta C33 | BASF MB AE-90 | BASF Polyheed 1020 | - | 6 | 5.5 |
| 4 | 318 | 17 | G | 6/23/2015 | 0.44 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 2.5 | 6.2 |
| 5 | 8 | 1 | G | 6/23/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 3 | 6.3 |
| 6 | 27 | 13 | M | 6/24/2015 | 0.38 | 0% | Type I/II Holicim | Lafarge | Dolese Cooperton #57 | Dolese Cooperton | Master Builders | Master Builders | Master Builders | 9.5 | 6 |
| 7 | 1 | 1 | E | 6/24/2015 | 0.44 | 20% | Type I, Holicim | Headwaters Class C | Vulcan Materials | Aztec Company | Master Air AE-90 | - | Master Polyheed 1020 | 8 | 6 |
| 8 | 65 | 18 | J | 6/24/2015 | 0.42 | 15% | Ash Grove I/II | Lafarage - Red Rock | Dolese | Dolese | BASF MB AE-90 | BASF Polyheed 997 | BASF DELVO Stabalizer/B ASF GLENIUM-7500 | 2.5 | 7.4 |
| 9 | 11 | 9 | F | 6/25/2015 | 0.44 | 20% | Ash Grove I/II | Lafarage - Red Rock | Western Aggregates | Arbuckle Materials | Chryso - Air 260 | Chryso - EnviroMix 300 | - | 5.5 | 6.5 |
| 10 | 5 | 11 | C | 6/18/2015 | 0.44 | 0% | Buzzi Unicem | - | Stratford | Frisco Sand | Darex II | Daratard | Adva | 7.5 | 7.1 |
| 11 | 7 | 11 | C | 6/19/2015 | 0.44 | 0% | Buzzi Unicem | - | Stratford | Frisco Sand | Darex II | Daratard | Adva | 5.25 | 6.9 |
| 12 | 46 | 5 | G | 6/25/2015 | 0.38 | 0% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 3 | 6.1 |
| 13 | 76 | 18 | J | 6/30/2015 | 0.42 | 15% | Ash Grove I/II | Lafarage - Red Rock | Dolese | Dolese | BASF MB AE-90 | BASF Polyheed 997 | BASF DELVO Stabalizer/B ASF GLENIUM-7500 | 1.75 | 6.2 |

| OSU # | ODOT # | Residency | Producer | Casting Date | W/C | Fly Ash (%) | Cement | Fly Ash | Coarse | Sand | Air Entrainer | Water Reducer | Other | Slump (in) | Air (%) |
|-------|--------|-----------|----------|--------------|-----------|-------------|----------------|--------------------|---------------------|---------------------|------------------|---------------------------|--|------------|---------|
| 14 | 76 | 18 | J | 6/30/2015 | 0.42 | 15% | Ash Grove I/II | Lafarge - Red Rock | Dolese | Dolese | BASF MB AE-91 | BASF Polyheed 998 | BASF DELVO Stabalizer/B ASF GLENIUM-7501 | 2 | 5.7 |
| 15 | 12 | 16 | D | 6/30/2015 | 0.44 | 20% | Central Plains | Lafarge | Pryor Stone | Anchor Stone | GRACE | GRACE | - | 5.25 | 6.8 |
| 16 | 14 | 7 | J | 6/30/2015 | 0.32 | 10% | Monarch Cement | Lafarge | Martin Marietta #57 | Hoiliday Sand | Master Builders | Master Builders | Master Builders | 9 | 7.2 |
| 17 | 5 | 4 | E | 6/26/2015 | 0.45 | 20% | Ash Grove | Headwaters Class C | Martin Marietta #58 | Martin Marietta #59 | BASF MB AE-90 | BASF Polyheed 1020 | - | 3 | 5.6 |
| 18 | 10 | 1 | G | 6/29/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 3 | 7 |
| 19 | 20 | 5 | G | 6/30/2015 | 0.44 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 6 | 7.4 |
| 20 | 484 | 13 | | 6/30/2015 | - | - | - | - | - | - | - | - | - | 3 | 7.2 |
| 21 | 7 | 4 | E | 7/1/2015 | 0.44 | 20% | Ash Grove | Headwaters Class C | Martin Marietta #58 | Martin Marietta #59 | BASF MB AE-90 | BASF Polyheed 1020 | - | 3 | 8 |
| 22 | 48 | 5 | G | 7/1/2015 | 0.38 | 0% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5 | 7.1 |
| 23 | 4 | 3 | | 7/2/2015 | 0.48 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5 | 5 |
| 24 | 1 | 12 | D | 7/2/2015 | 0.43 | 19% | Type I/II | - | - | - | - | - | - | 6.25 | 6.4 |
| 25 | 51 | 14 | D | 7/6/2015 | 0.44 | 15% | Central Plains | Lafarge | Apac-Dewey | Sober-Brothers | GRACE | GRACE | GRACE | 6.5 | 5 |
| 26 | 15 | 16 | A | 7/6/2015 | 0.37 4 | 20% | Central Plains | Lafarge & PSO | Pryor Stone | Anchor Stone | Chryso - Air 260 | - | - | 1.25 | 4.8 |
| 27 | 12 | 7 | Neo | 7/7/2015 | 0.48 | 15% | Buzzi Unicem | Sooner - Red Rock | Kemp Stone | Muskogee Sand | Master Air AE-90 | Master Builders poly 1020 | - | 3.5 | 6 |
| 28 | 1 | 5 | G | 7/7/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5 | 7 |

| OSU # | ODOT # | Residency | Producer | Casting Date | W/C | Fly Ash (%) | Cement | Fly Ash | Coarse | Sand | Air Entrainer | Water Reducer | Other | Slump (in) | Air (%) |
|-------|--------|-----------|----------|--------------|-----------|-------------|-----------------|----------------------------|-----------------------|------------------------|----------------------|--------------------------------------|-----------------------|------------|---------|
| 29 | 331 | 17 | G | 7/8/2015 | 0.44 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 1.75 | 6 |
| 30 | 21 | 15 | A | 6/16/2015 | 0.37 5 | 20% | Type IL(10) | Lafarge - Redrock | Boral, Davis | Duit, Dover | Chryso Air 260 | - | - | 1.5 | 6 |
| 31 | 22 | 15 | G | 6/16/2015 | 0.44 | 20% | Type I/II | Lafarge N.A | The Quapaw Company | Dolese Brothers | MB-AE-90 | Glenium 7500 | Pozzalith 80 | 3 | 5.5 |
| 32 | 75 | 2 | O | 6/30/2015 | 0.39 | 0% | Type I, Holicim | Fly Ash Direct, OKLA Union | Dolese Cooperton #57/ | T&G Sand Plant | - | - | - | 2 | 5 |
| 33 | 1-6 | 8 | G | 7/8/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 6.5 | 6 |
| 34 | 7 | 6 | H | 7/9/2015 | 0.43 | 15% | Type I/II | lafarge class C | Dolese Limestone | laverne natural sand | - | WR Grace | - | 7 | 6.5 |
| 35 | 1 | 1 | E | 7/14/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Dolese #67 | Alan Ritchey Materials | Master Air AE-90 | MasterPoly heed 1020 | - | 3 | 5 |
| 36 | 7 | 5 | G | 7/14/2015 | 0.38 | 20% | Type I/II | Class F | - | - | - | - | GLENIUM-7500-MRWRA | 6 | 5 |
| 37 | 56 | 2 | L | 7/15/2015 | 0.39 | 0% | Type I, Holicim | Lafarge | Dolese Cooperton #57 | Kline Materials | BASF Master Builders | - | - | - | 5 |
| 38 | 14-145 | 8 | G | 7/16/2015 | 0.38 | 20% | Type I/II | FA | - | - | - | - | GLENIUM-7500-MRWRA | 7 | 5 |
| 39 | 108 | 18 | J | 7/16/2015 | 0.42 | 15% | Ash Grove I/II | Lafrage Class C | Dolese Davis | Dolese-Guthrie | BASF MB AE 90 | BASF Polyheed 997/ BASF Gelnium 7500 | BASF DELVO Stabilizer | 3.5 | 6 |
| 40 | 7 | 12 | D | 7/16/2015 | 0.46 | 20% | Central Plains | Lafarge | APAC-ZEB | APAC-Muskogee | GRACE | GRACE | - | 2 | 6 |
| 41 | 73(4) | 13 | M | 7/23/2015 | 0.39 | 0% | Type I/II | - | - | - | AE 90 | BASF POZ 80 | - | 4 | 6 |
| 42 | 6 | 16 | N | 7/23/2015 | 0.37 | 20% | Type I/II | Class C | - | - | MBAE90 | POLYHEED 997 | - | 6 | 6 |
| 43 | 2 | 1 | E | 7/14/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Dolese #67 | Alan Ritchey Materials | MasterAir AE-90 | MasterPoly heed 1020 | - | 3 | 5 |

| OSU # | ODOT # | Residency | Producer | Casting Date | W/C | Fly Ash (%) | Cement | Fly Ash | Coarse | Sand | Air Entrainer | Water Reducer | Other | Slump (in) | Air (%) |
|-------|--------|-----------|----------|--------------|-----------|-------------|----------------|--------------------|---------------------|------------------------|-----------------|--|-----------------------|------------|---------|
| 44 | 3 | 1 | E | 7/17/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Dolese #67 | Alan Ritchey Materials | MasterAir AE-90 | MasterPoly heed 1020 | - | 3 | 5 |
| 45 | 7 | 8 | G | 7/23/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 7 | 5 |
| 46 | 15 | 8 | G | 7/28/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 7 | 5 |
| 47 | 16 | 8 | G | 7/28/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5.5 | 5 |
| 48 | 31 | 15 | G | 7/29/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5 | 5 |
| 49 | 30 | 15 | G | 7/29/2015 | 0.38 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 4 | 5 |
| 50 | 40 | 16 | A | 7/29/2015 | 0.37 5 | 20% | Type IL(10) | Lafrage Oologah | - | - | Chryso Air 260 | - | - | 1 | 6 |
| 51 | 32 | 15 | G | 7/29/2015 | 0.44 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 4.5 | 4.5 |
| 52 | 3 | 10 | I | 7/16/2015 | 0.42 | 20% | Type I/II | Red Rock Class C | Dolese Richards | Lightle sand | MB AE 90 | MB Poly 900 Type A | - | 5.5 | 6.5 |
| 53 | 30 | 4 | E | 7/22/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Martin Marietta #67 | Martin Marietta c33 | MB AE 90 | BASF Polyheed 1020 | - | 3 | 5 |
| 54 | 79 | 2 | O | 7/24/2015 | 0.37 | 0 | Holcium | - | Dolese | T & G Sandplant | Chryso Air 260 | Chryso - EnviroMix I-40 | CHRYSO NUTAL SET | 4 | 5 |
| 55 | 3 | 5 | G | 7/27/2015 | 0.44 | 20% | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 5 | 4.5 |
| 56 | 30 | 2 | A | 7/28/2015 | 0.37 5 | 20% | Type IL(10) | Lafarge Harrington | Dolese | Duit, Dover | Chryso Air 260 | - | - | 1.5 | 6 |
| 57 | 28 | 7 | J | 7/31/2015 | 0.42 | 13% | - | Lafrage Class C | - | - | BASF MB AE 90 | BASF MB POLYHEED 997 / BASF GLENIUM 7500 | BASF DELVO Stabilizer | 3.75 | 6 |
| 58 | 112 | 9 | F | 8/5/2015 | 0.41 | 20% | Type I/II | Fly Ash C | - | - | CHRYSO | Enviromix 300 | - | 5 | 6 |

| OSU # | ODOT # | Residency | Producer | Casting Date | W/C | Fly Ash (%) | Cement | Fly Ash | Coarse | Sand | Air Entrainer | Water Reducer | Other | Slump (in) | Air (%) |
|-------|--------|-----------|----------|--------------|------|-------------|----------------|---------------------|---------------------|----------------|-----------------|--|--------------------|------------|---------|
| 59 | 111 | 9 | F | 8/5/2015 | 0.41 | 20% | Type I/II | Fly Ash C | - | - | CHRYSO | Enviromix 300 | - | 6 | 6 |
| 60 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| 61 | 3 | 13 | G | 8/3/2015 | 0.44 | 20% | Type I/II | Class F Flyash | - | - | - | - | GLENIUM-7500-MRWRA | 3 | 4.5 |
| 62 | 1 | 1 | E | 8/5/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Dolese | Alan Ritchey | MasterAir AE 90 | Master Polyheed 1020 / Master Glenium 7500 | Masterset Delvo | 8 | 5 |
| 63 | 12 | 1 | E | 8/6/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Vulcan Material #67 | - | MasterAir AE 90 | Master Polyheed 1020 | - | 4.75 | 5 |
| 64 | 10 | 1 | E | 8/6/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Vulcan Material #67 | - | MasterAir AE 90 | Master Polyheed 1020 | - | 6.5 | 5 |
| 65 | 11 | 1 | E | 8/6/2015 | 0.44 | 20% | Holcium Type I | Headwaters Class C | Vulcan Material #67 | - | MasterAir AE 90 | Master Polyheed 1020 | - | 5.5 | 5 |
| 66 | 35 | 15 | G | 8/6/2015 | 0.38 | 0 | Type I/II | - | - | - | - | - | GLENIUM-7500-MRWRA | 2 | 4.5 |
| 67 | 60 | 14 | D | 8/10/2015 | 0.44 | 15% | Central Plains | Lafarage - Red Rock | Apac-Dewey | Sober-Brothers | GRACE | GRACE | GRACE | 4.44 | 6 |

Appendix H -
Results for Secondary Compliance Testing on Cores and Slab Surface

Table H1: Results of Statistical Analysis Apparent Surface Resistivity Control Cylinder

| Day | Average Surface Resistivity (KΩ*cm) | COV (%)Surface Resistivity (KΩ*cm) | Standard Deviation Surface Resistivity (KΩ*cm) |
|-----|-------------------------------------|------------------------------------|--|
| 1 | 3.3 | 5.0 | 0.2 |
| 3 | 5.8 | 1.1 | 0.1 |
| 7 | 7.1 | 2.0 | 0.1 |
| 14 | 8.1 | 2.5 | 0.2 |
| 28 | 10.1 | 10.3 | 1.0 |
| 42 | 10.4 | 1.3 | 0.1 |
| 56 | - | - | - |
| 91 | 11.6 | 3.2 | 0.4 |

Table H2: Results of Statistical Analysis Bulk Resistivity Control Cylinder

| Day | Average Surface Resistivity (KΩ*cm) | COV (%)Surface Resistivity (KΩ*cm) | Standard Deviation Surface Resistivity (KΩ*cm) |
|-----|-------------------------------------|------------------------------------|--|
| 1 | Day | Avg. | COV |
| 3 | 1 | 1.8 | 5.0 |
| 7 | 3 | 3.2 | 1.6 |
| 14 | 7 | 4.0 | 2.0 |
| 28 | 14 | 4.6 | 1.5 |
| 42 | 28 | 5.7 | 0.7 |
| 56 | - | - | - |
| 91 | 42 | 6.2 | 3.1 |

Table H3: Results of Statistical Analysis Bulk Resistivity Cores Day-1

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 2.6 | 2.7 | 2.6 | 0.0 | 0.7 | N/A | N/A |

Table H4: Results of Statistical Analysis Bulk Resistivity Cores Day-3

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 3.1 | 2.9 | 3.0 | 0.1 | 4.9 | 2.47E-02 | N/A |
| Day-3 | 3.9 | 3.7 | 3.8 | 0.1 | 2.3 | | |

Table H5: Results of Statistical Analysis Bulk Resistivity Cores Day-7

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 3.8 | 3.7 | 3.8 | 0.1 | 1.6 | 3.11E-01 | 5.94E-01 |
| Day-3 | 4.1 | 3.9 | 4.0 | 0.2 | 4.1 | | |
| Day-7 | 4.9 | 5.1 | 5.0 | 0.1 | 2.8 | | |

Table H6: Results of Statistical Analysis Bulk Resistivity Cores Day-14

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 4.4 | 4.4 | 4.4 | 0.0 | 0.6 | - | 2.29E-01 |
| Day-3 | 4.7 | 4.4 | 4.5 | 0.2 | 4.2 | | |
| Day-7 | 4.7 | 4.6 | 4.7 | 0.1 | 1.8 | | |
| Day-14 | - | - | - | - | - | | |

Note: Day-14 Data was rejected due to faulty core surface.

Table H7: Results of Statistical Analysis Bulk Resistivity Cores Day-28

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | - | - | - | - | - | 3.11E-01 | 5.94E-01 |
| Day-3 | 6.0 | 5.5 | 5.7 | 0.4 | 6.3 | | |
| Day-7 | 5.8 | 6.0 | 5.9 | 0.2 | 2.8 | | |
| Day-14 | - | - | - | - | - | | |
| Day-28 | 5.6 | 5.1 | 5.3 | 0.4 | 6.7 | | |

Note: Day-14 Data was rejected due to faulty core surface. Day-1 Data was rejected due to manipulation error.

Table H8: Results of Statistical Analysis Bulk Resistivity Cores Day-42

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 6.7 | 6.2 | 6.5 | 0.4 | 5.5 | 1.54E-01 | 4.85E-01 |
| Day-3 | 6.3 | 6.0 | 6.2 | 0.2 | 3.6 | | |
| Day-7 | 6.6 | 6.6 | 6.6 | 0.0 | 0.4 | | |
| Day-14 | - | - | - | - | - | | |
| Day-28 | 6.5 | 5.8 | 6.1 | 0.5 | 8.5 | | |
| Day-42 | 5.8 | 5.7 | 5.7 | 0.1 | 1.4 | | |

Note: Day-14 Data was rejected due to faulty core surface.

Table H9: Results of Statistical Analysis Bulk Resistivity Cores Day-56

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 8.0 | 7.1 | 7.5 | 0.7 | 8.8 | 5.98E-01 | 3.56E-01 |
| Day-3 | 8.8 | 6.8 | 7.8 | 1.4 | 18.4 | | |
| Day-7 | 7.0 | 7.6 | 7.3 | 0.4 | 5.7 | | |
| Day-14 | - | - | - | - | - | | |
| Day-28 | 7.0 | 6.6 | 6.8 | 0.2 | 3.6 | | |
| Day-42 | 6.6 | 6.9 | 6.7 | 0.2 | 3.3 | | |
| Day-56 | 6.9 | 7.3 | 7.1 | 0.3 | 3.9 | | |

Note: Day-14 Data was rejected due to faulty core surface.

Table H10: Results of Statistical Analysis Bulk Resistivity Cores Day-91

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 7.7 | 7.4 | 7.5 | 0.2 | 2.9 | 6.07E-02 | 2.19E-02 |
| Day-3 | 7.6 | 7.4 | 7.5 | 0.1 | 1.5 | | |
| Day-7 | 7.6 | 7.6 | 7.6 | 0.0 | 0.4 | | |
| Day-14 | - | - | - | - | - | | |
| Day-28 | 7.3 | 7.1 | 7.2 | 0.1 | 1.9 | | |
| Day-42 | 7.2 | 7.5 | 7.4 | 0.2 | 2.6 | | |
| Day-56 | 6.9 | 6.9 | 6.9 | 0.1 | 0.8 | | |
| Day-91 | 7.6 | 7.2 | 7.4 | 0.3 | 4.1 | | |

Note: Day-14 Data was rejected due to faulty core surface.

Table H11: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-1

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 4.6 | 4.5 | 4.5 | 0.1 | 1.2 | N/A | N/A |

Table H12: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-3

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 5.0 | 4.7 | 4.8 | 0.2 | 4.4 | 3.29E-02 | N/A |
| Day-3 | 7.0 | 6.4 | 6.7 | 0.4 | 6.6 | | |

Table H13: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-7

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|-------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 5.7 | 5.7 | 5.7 | 0.0 | 0.3 | 4.36E-02 | 1.32E-01 |
| Day-3 | 6.4 | 6.0 | 6.2 | 0.3 | 4.9 | | |
| Day-7 | 6.4 | 6.5 | 6.5 | 0.1 | 0.8 | | |

Table H14: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-14

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 6.7 | 6.9 | 6.8 | 0.1 | 1.8 | 2.01E-01 | 3.83E-02 |
| Day-3 | 7.0 | 6.9 | 6.9 | 0.0 | 0.3 | | |
| Day-7 | 6.3 | 6.5 | 6.4 | 0.1 | 2.2 | | |
| Day-14 | 6.9 | 6.4 | 6.6 | 0.4 | 5.4 | | |

Note: Day-14 Data was rejected due to faulty coring.

Table H15: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-28

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 12.1 | 12.9 | 12.5 | 0.5 | 4.4 | 1.00E-03 | 2.53E-03 |
| Day-3 | 8.6 | 9.7 | 9.2 | 0.8 | 8.5 | | |
| Day-7 | 8.0 | 7.6 | 7.8 | 0.3 | 3.6 | | |
| Day-14 | 7.7 | 7.0 | 7.4 | 0.5 | 6.7 | | |
| Day-28 | 7.7 | 7.0 | 7.4 | 0.5 | 7.2 | | |

Table H16: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-42

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 8.8 | 8.6 | 8.7 | 0.2 | 2.2 | 3.07E-02 | 2.40E-02 |
| Day-3 | 8.7 | 9.4 | 9.0 | 0.5 | 5.3 | | |
| Day-7 | 8.4 | 8.0 | 8.2 | 0.3 | 3.9 | | |
| Day-14 | 7.8 | 7.7 | 7.7 | 0.1 | 1.1 | | |
| Day-28 | 7.8 | 7.2 | 7.5 | 0.4 | 5.9 | | |
| Day-42 | 7.7 | 8.4 | 8.0 | 0.4 | 5.5 | | |

Table H17: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-56

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 9.4 | 9.5 | 9.4 | 0.1 | 0.6 | 2.98E-02 | 1.03E-01 |
| Day-3 | 9.4 | 10.1 | 9.7 | 0.5 | 4.9 | | |
| Day-7 | 9.5 | 9.1 | 9.3 | 0.3 | 3.2 | | |
| Day-14 | 8.5 | 8.6 | 8.6 | 0.1 | 0.8 | | |
| Day-28 | 8.8 | 7.9 | 8.3 | 0.7 | 8.1 | | |
| Day-42 | 7.3 | 8.8 | 8.1 | 1.0 | 12.7 | | |
| Day-56 | 10.2 | 10.2 | 10.2 | 0.1 | 0.5 | | |

Table H18: Results of Statistical Analysis Apparent Surface Resistivity Cores Day-91

| Cores | Sample1 Bulk Resistivity (KΩ*cm) | Sample 2 Bulk Resistivity (KΩ*cm) | Average Bulk Resistivity (KΩ*cm) | Std. Dev. Bulk Resistivity (KΩ*cm) | COV (%) Bulk Resistivity (KΩ*cm) | ANOVA p-Value All Cores | ANOVA p-Value Not Including Day-3 |
|--------|----------------------------------|-----------------------------------|----------------------------------|------------------------------------|----------------------------------|-------------------------|-----------------------------------|
| Day-1 | 10.8 | 12.4 | 11.6 | 1.1 | 9.8 | 4.08E-01 | 5.72E-01 |
| Day-3 | 10.0 | 15.5 | 12.8 | 3.9 | 30.6 | | |
| Day-7 | 13.2 | 14.4 | 13.8 | 0.9 | 6.3 | | |
| Day-14 | 15.8 | 11.1 | 13.5 | 3.3 | 24.7 | | |
| Day-28 | 9.8 | 9.6 | 9.7 | 0.1 | 1.5 | | |
| Day-42 | 11.0 | 14.7 | 12.9 | 2.6 | 20.3 | | |
| Day-56 | 9.0 | 12.1 | 10.6 | 2.2 | 20.4 | | |
| Day-91 | 9.5 | 9.4 | 9.5 | 0.1 | 0.6 | | |

Appendix I -
Effect of Temperature on Surface Resistivity Testing

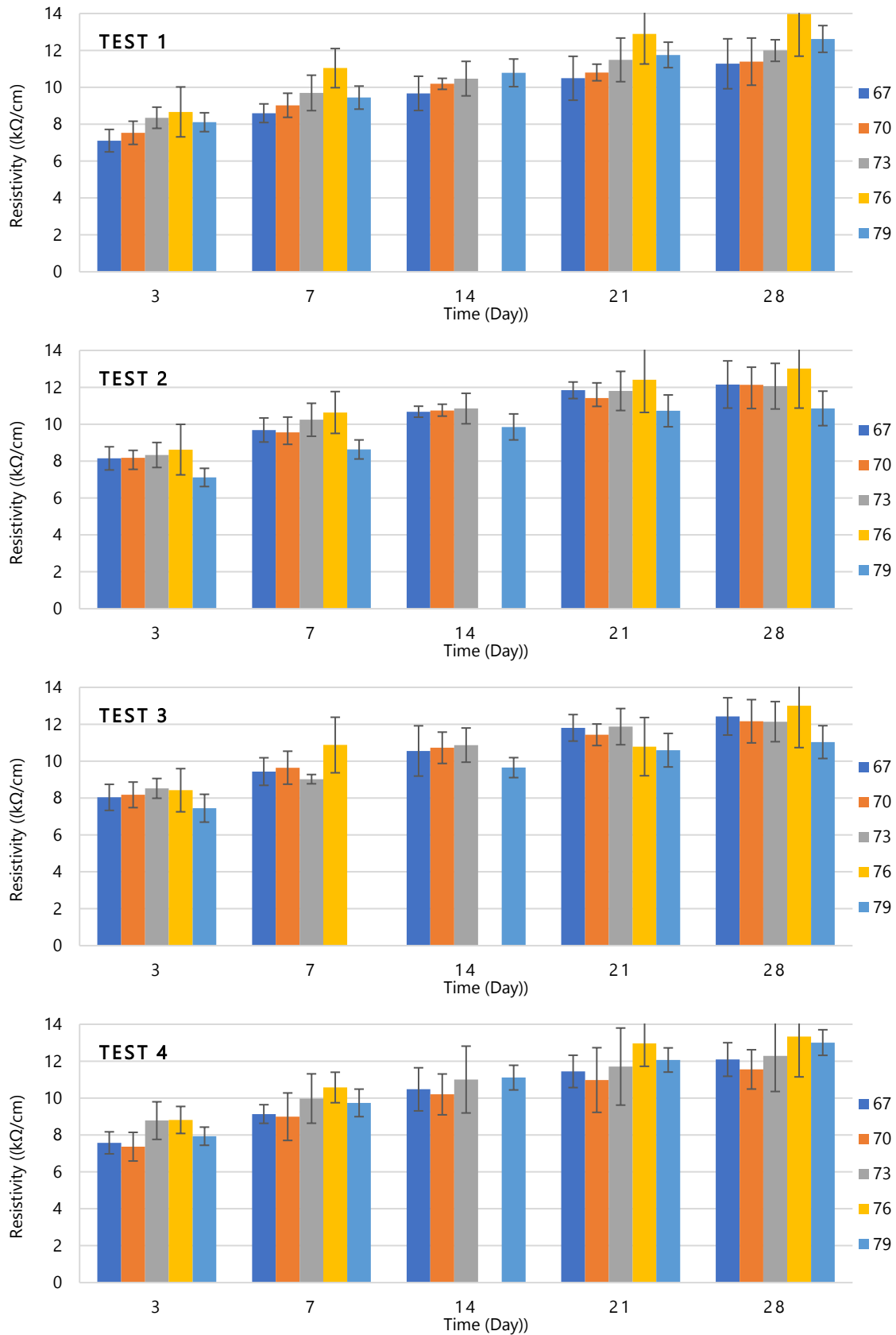


Figure 11: Surface resistivity per temperature test for mixture 40-00-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp.

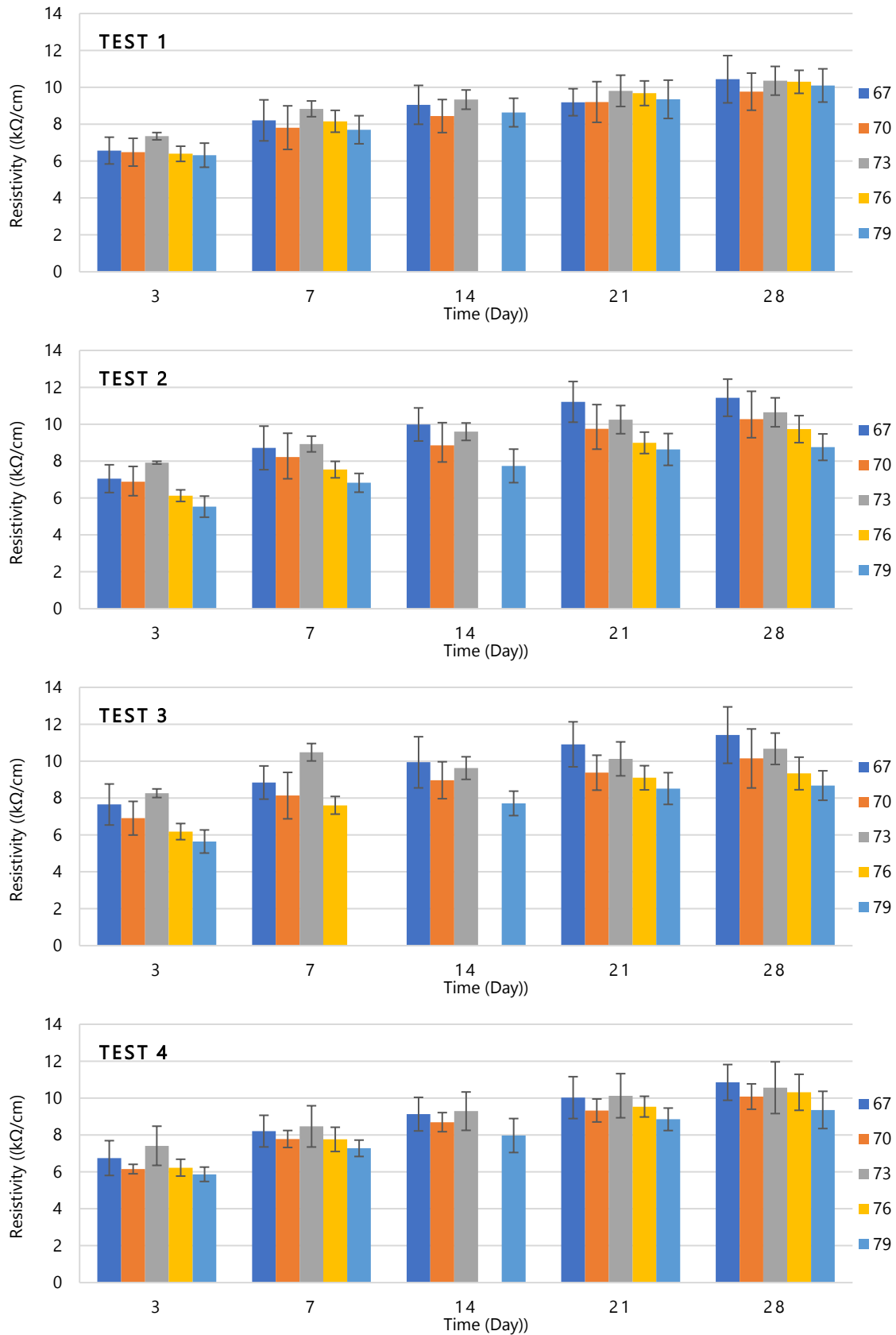


Figure I2: Surface resistivity per temperature test for mixture 45-00-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp

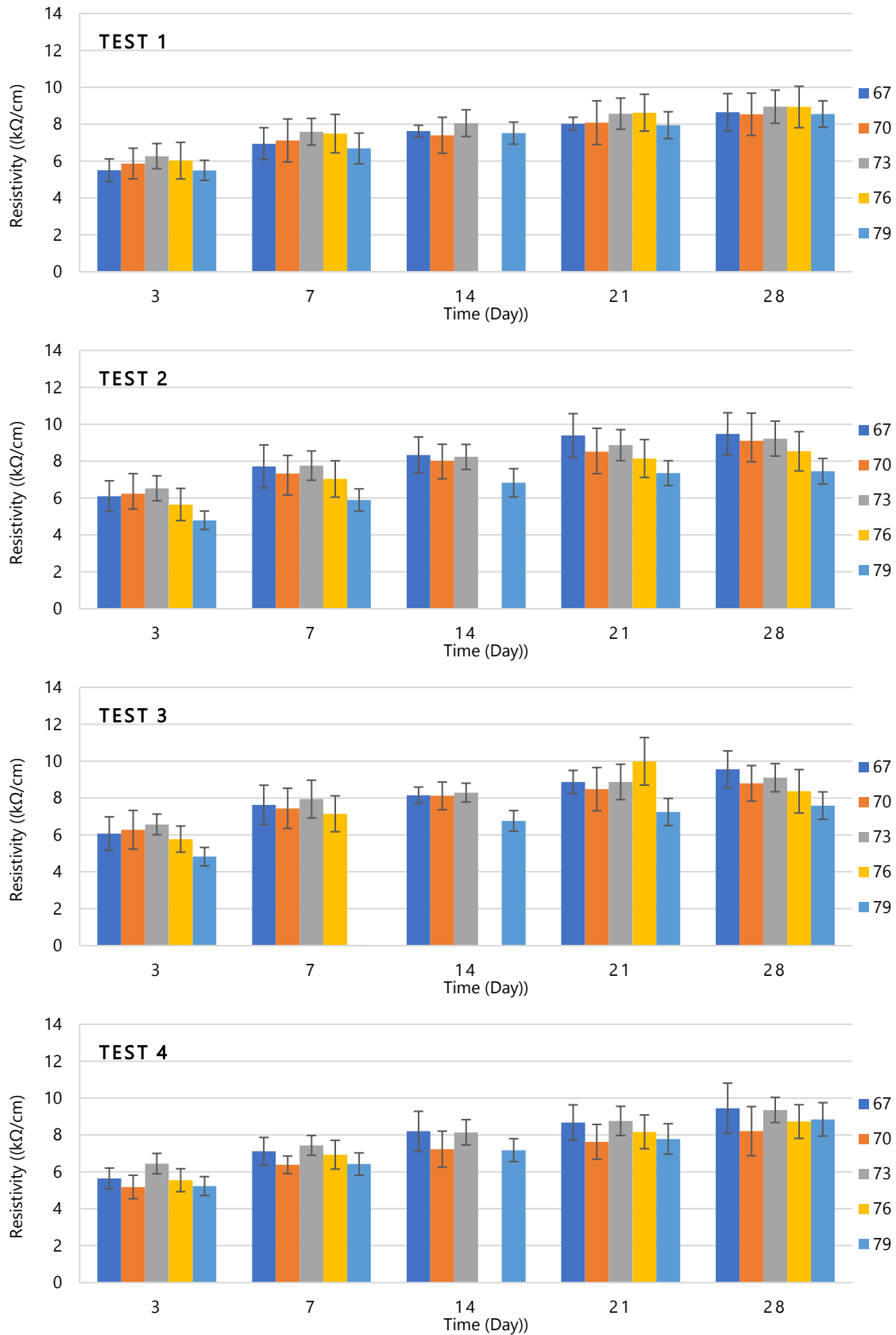


Figure I3: Surface resistivity per temperature test for mixture 50-00-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp

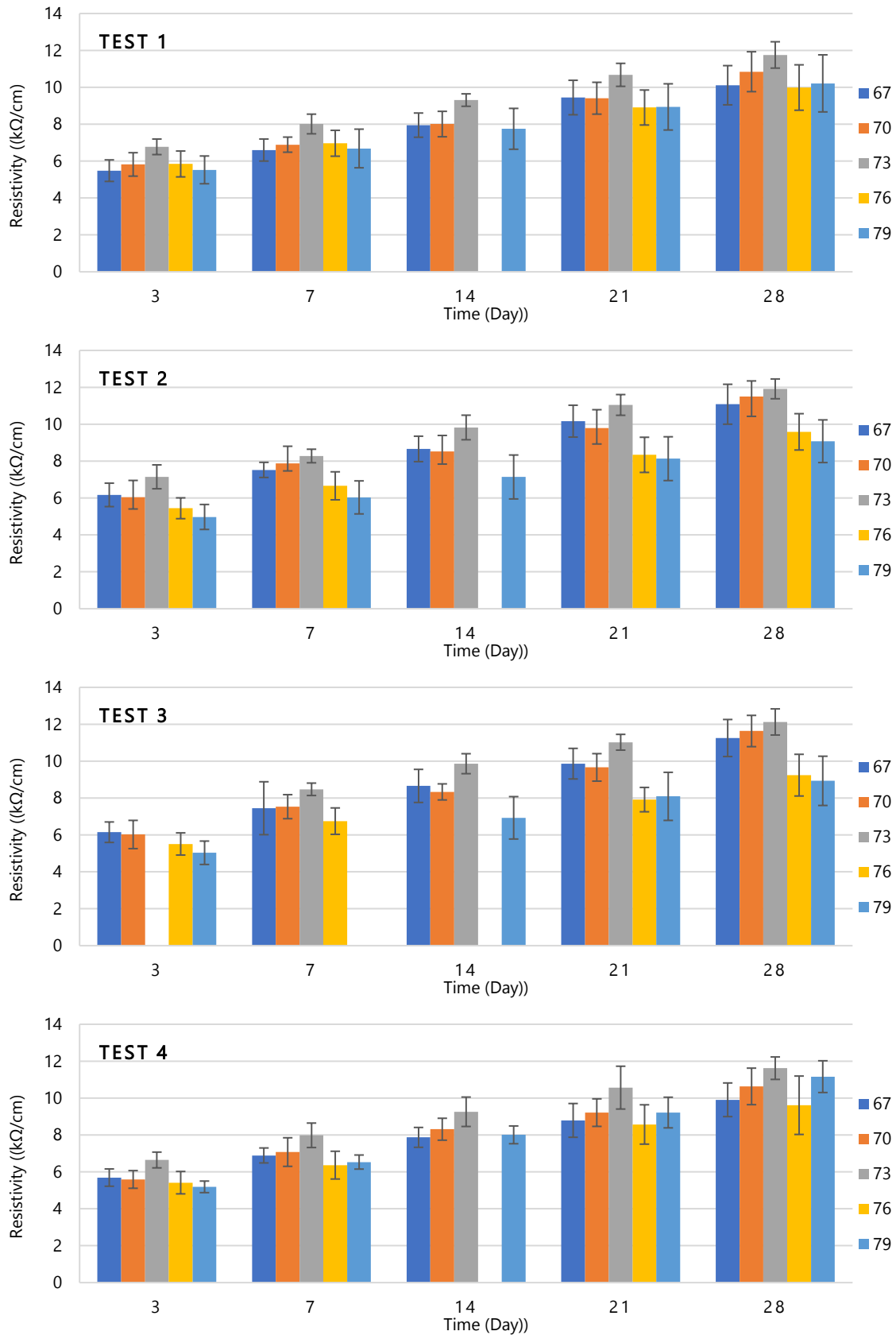


Figure I4: Surface resistivity per temperature test for mixture 40-20-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp

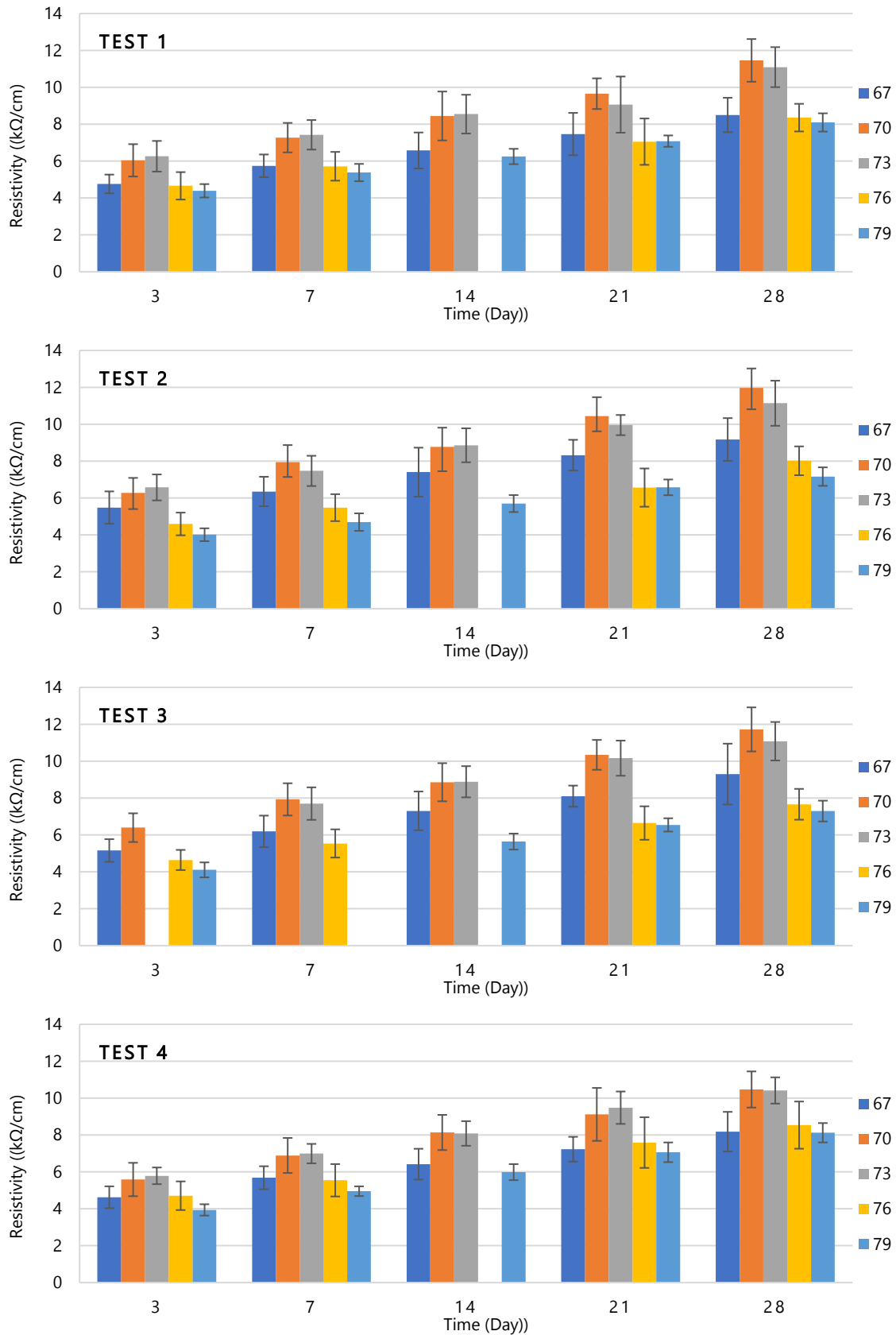


Figure 15: Surface resistivity per temperature test for mixture 45-20-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp

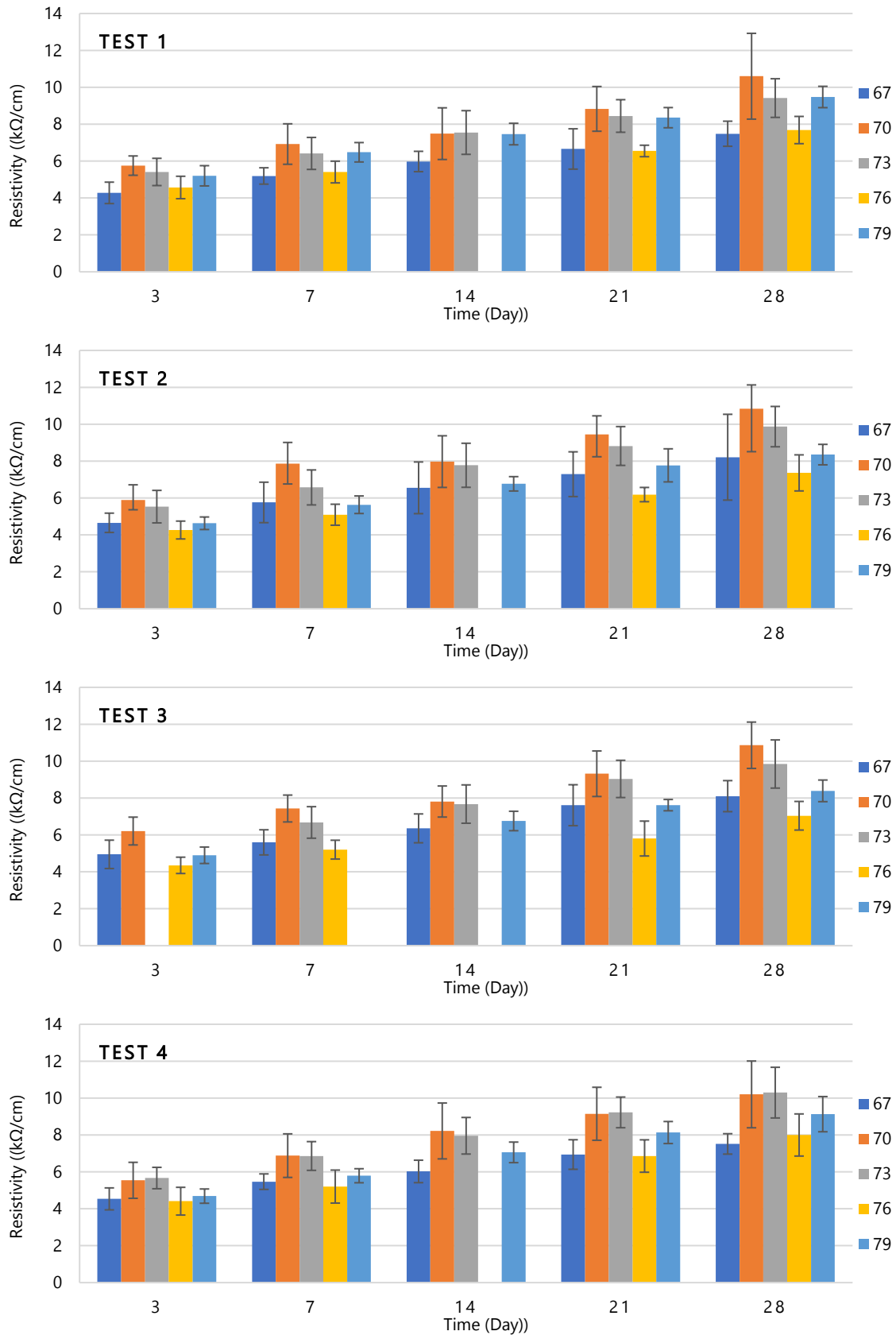


Figure I6: Surface resistivity per temperature test for mixture 50-20-57-00-1-0-1
 Test 1: Influence of Resipod Temp. - Cylinder at Ambient Temp
 Test 2: Influence of Resipod Temp. and Cylinder Temp.
 Test 3: Influence of Cylinder Temp. - Resipod at Ambient Temp.
 Test 4: Influence of Curing Temp. - Resipod at Ambient Temp

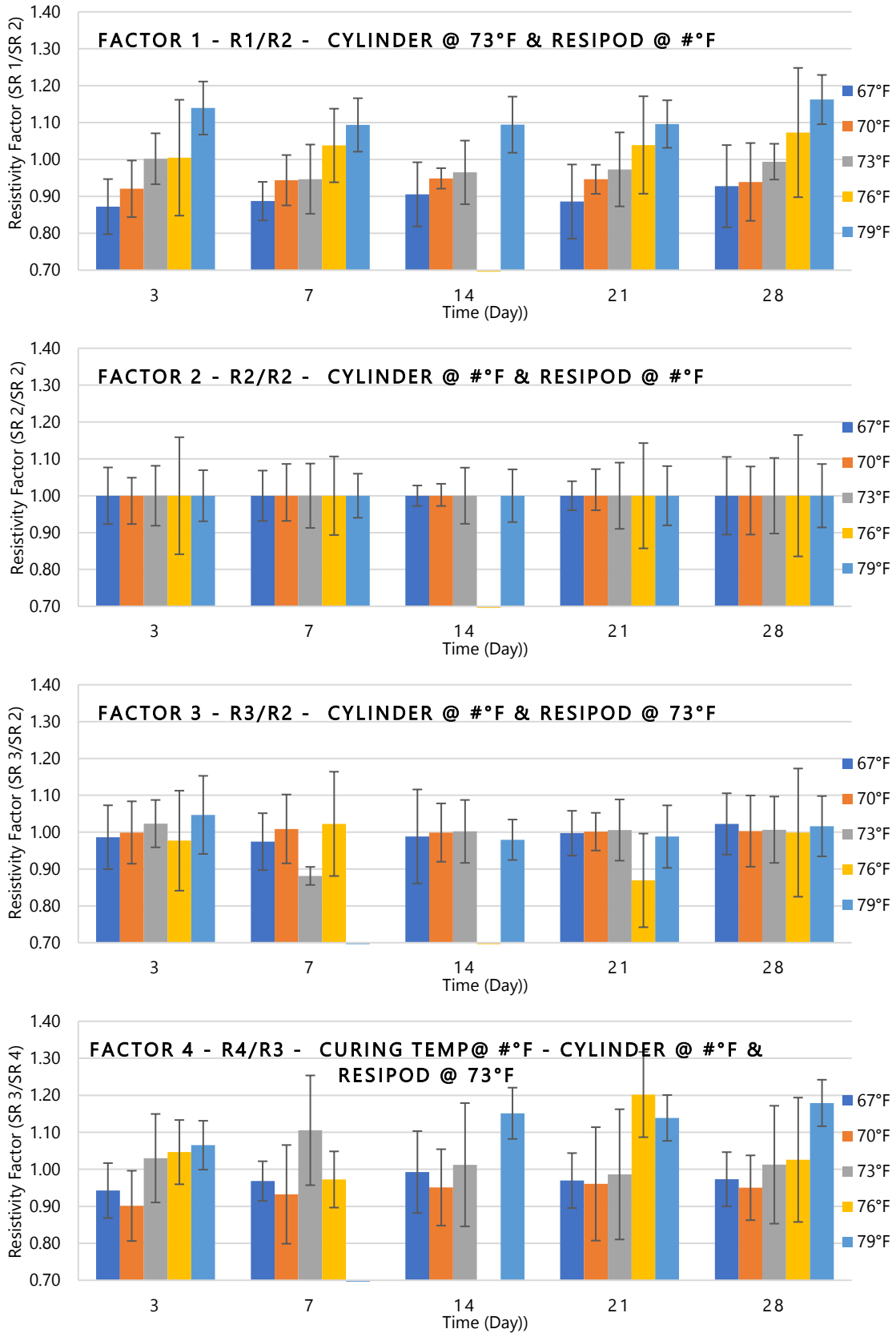


Figure I7: Resistivity Factors for mixture 40-00-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature

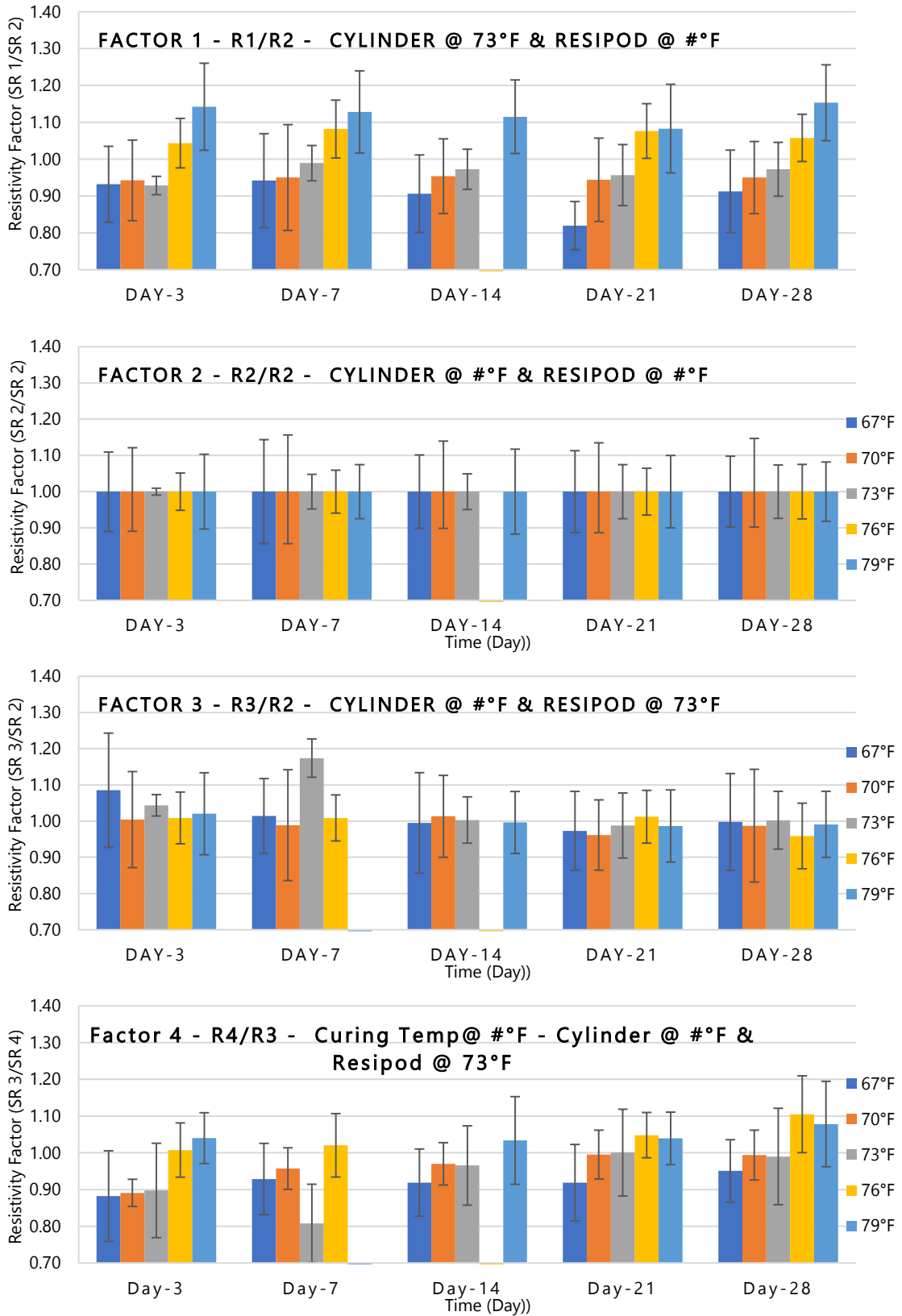


Figure I8: Resistivity Factors for mixture 45-00-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature

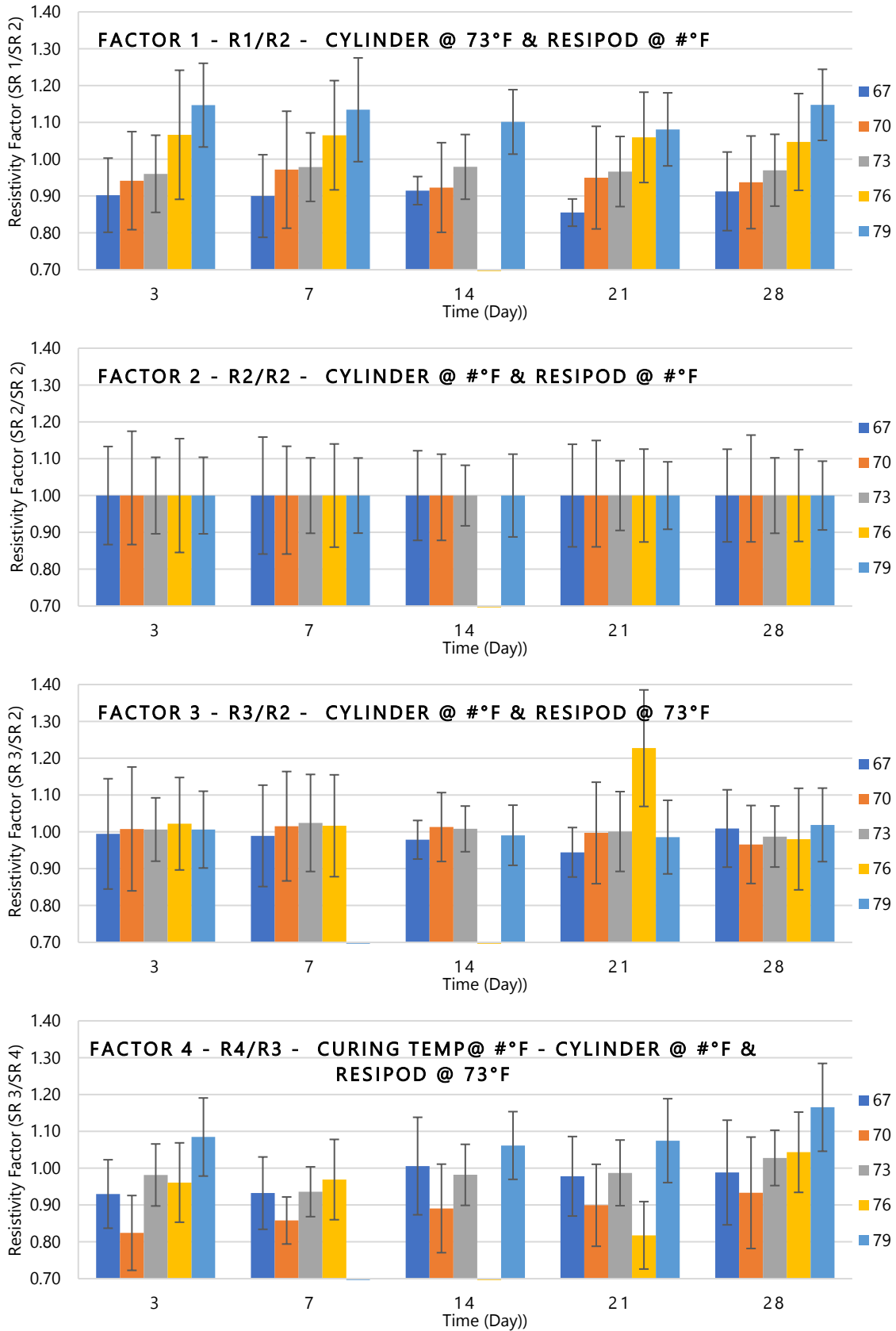


Figure I9: Resistivity Factors for mixture 50-00-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature

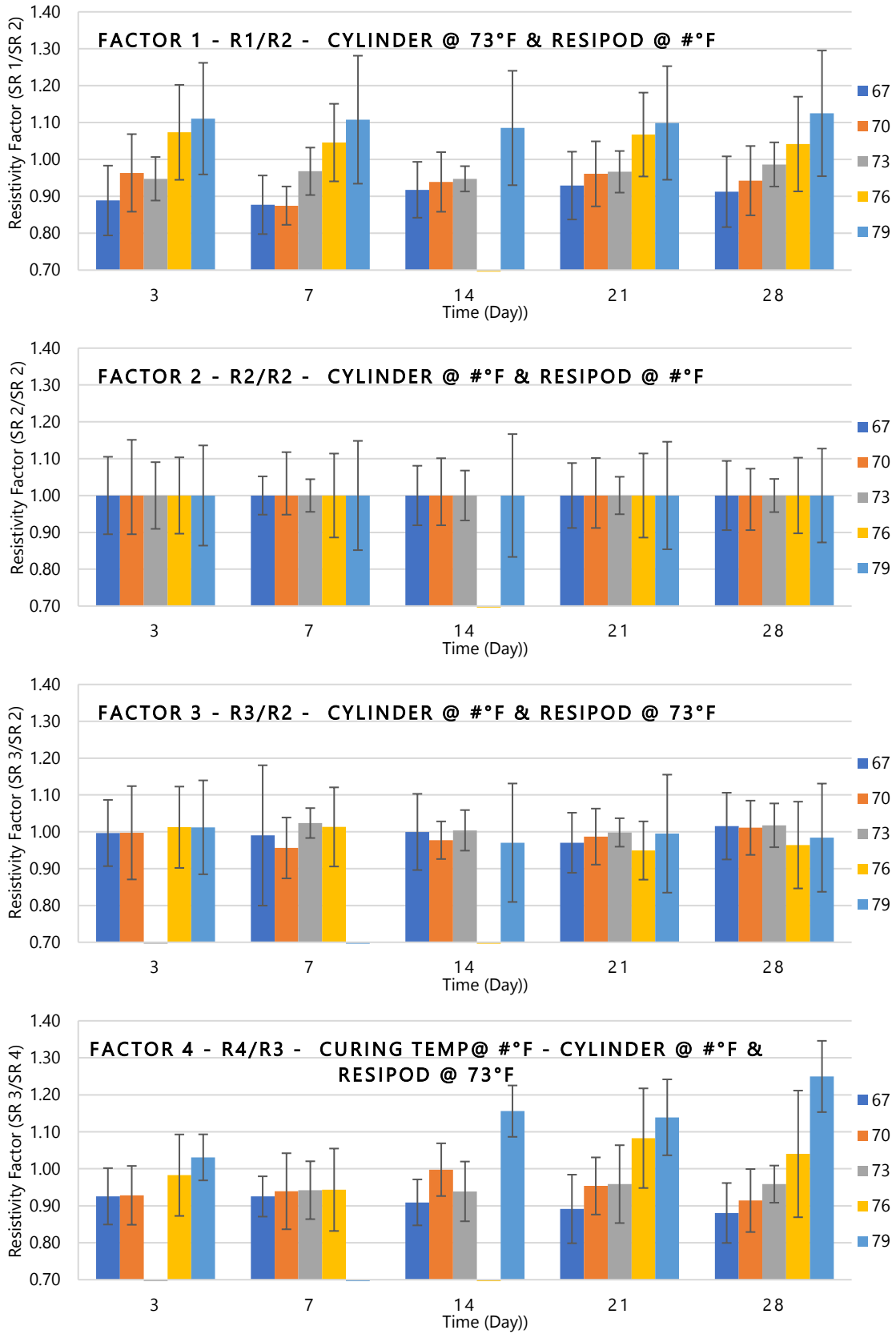


Figure I10: Resistivity Factors for mixture 40-20-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature

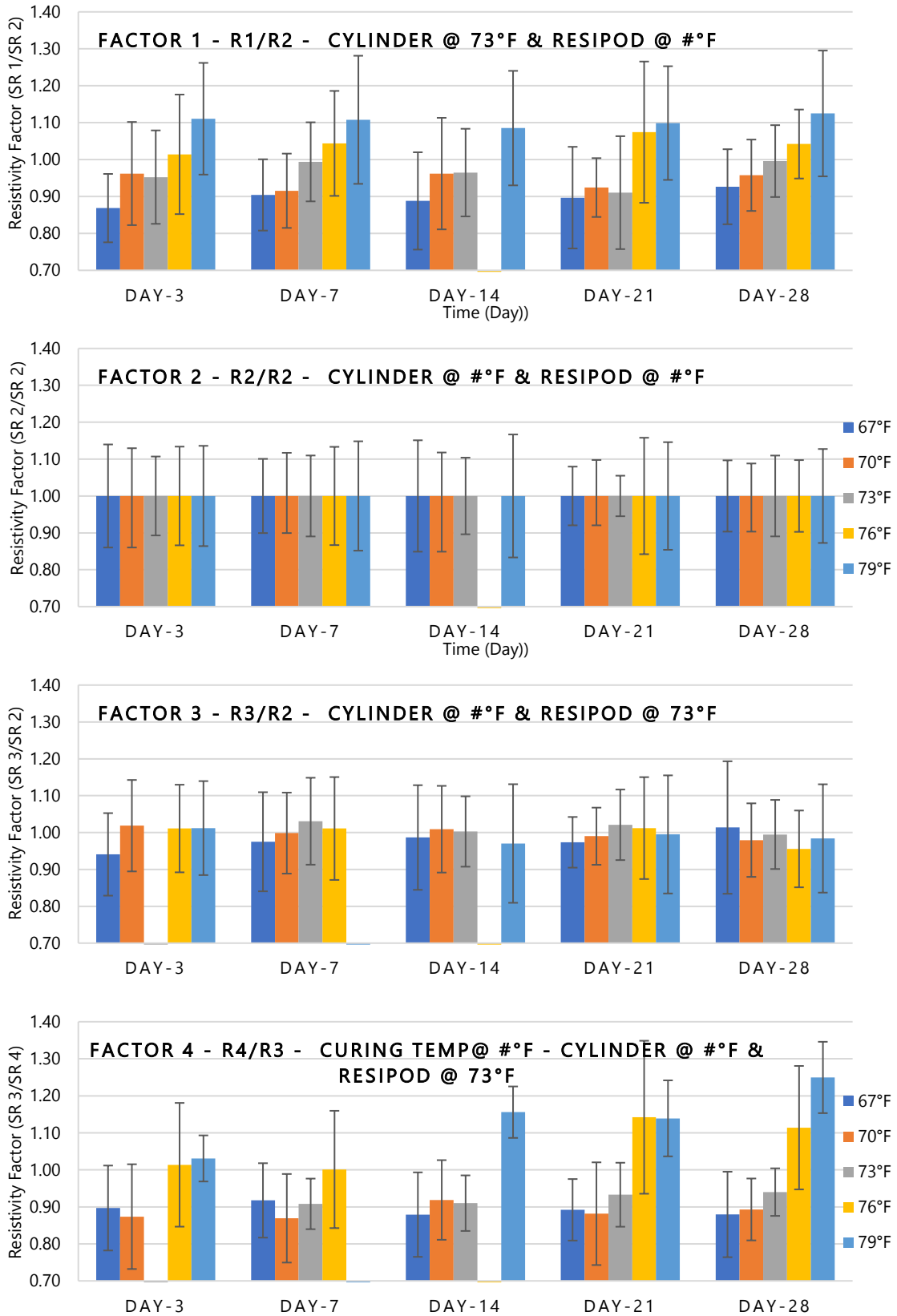


Figure I11: Resistivity Factors for mixture 45-20-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature

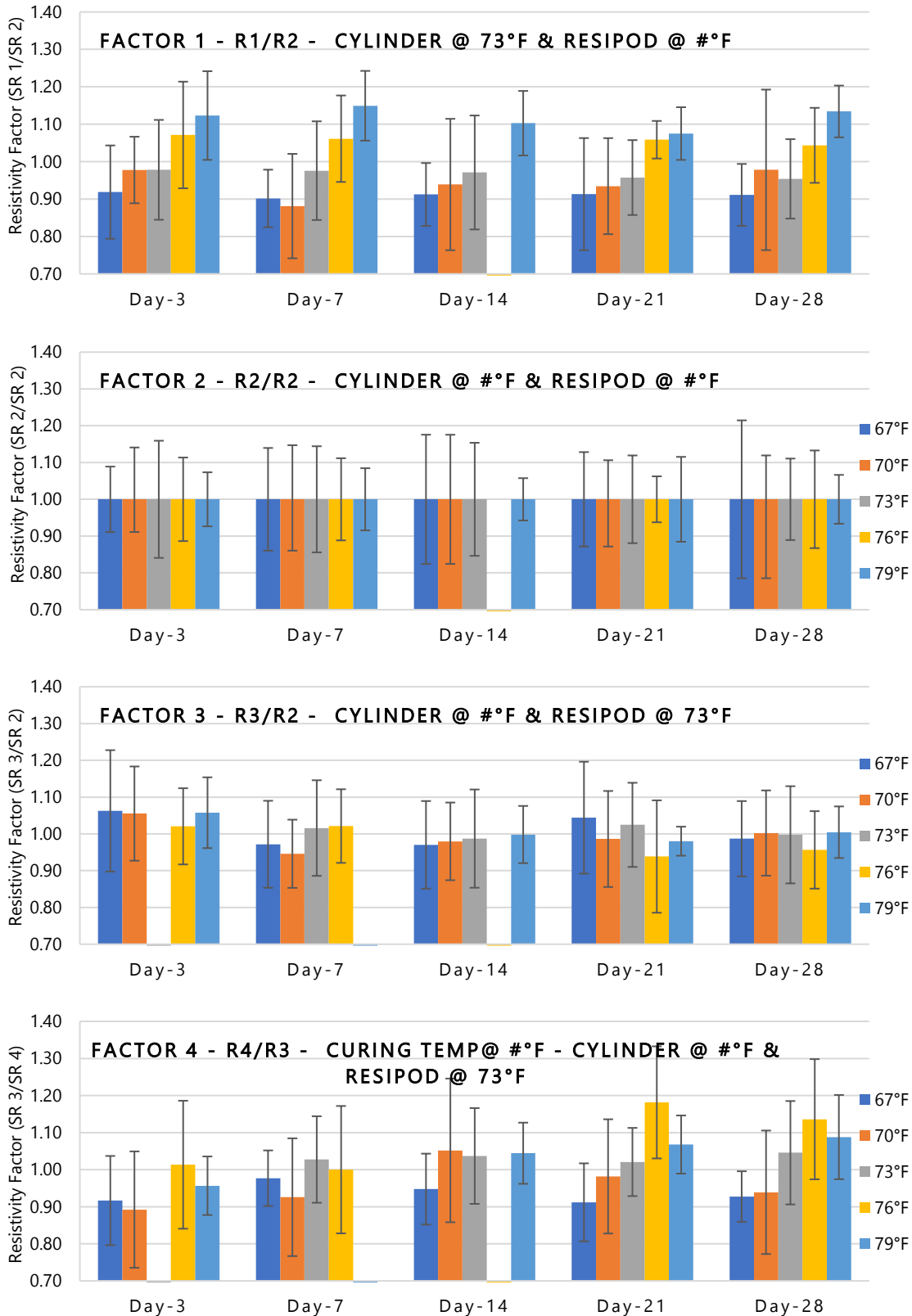


Figure I12: Resistivity Factors for mixture 50-20-57-00-1-0-1
 Factor 1 (Surf. Res. Test 1/Surf Res. Test 2) - Influence of Change in Cylinder Temperature
 Factor 2 (Surf. Res. Test 2/ surf. Res. Test 2) - Control
 Factor 3 (Surf. Res. Test 3/Surf. Res. Test 2) - Influence of Change in Resipod Temperature
 Factor 4 (Surf. Res. Test 4/Surf. Res. Test 3) - Influence of Curing Temperature