Alternate Methods for the Extraction and Measurement of Pore Solution in Fresh Concrete

by

Joseph Biever

### A THESIS

### submitted to

Oregon State University

Honors College

### in partial fulfillment of the requirements for the degree of

Honors Baccalaureate of Science in Civil Engineering (Honors Scholar)

> Presented June 3<sup>rd</sup>, 2021 Commencement June 2021

# AN ABSTRACT OF THE THESIS OF

Joseph Biever for the degree of Honors

<u>Baccalaureate of Science in Civil Engineering</u> presented on June 3, 2021. Title: <u>Alternate</u> Methods for the Extraction and Measurement of Pore Solution in Fresh Concrete

Abstract approved:

W. Jason Weiss

Knowledge of the pore solution composition and pore solution resistivity can be helpful for determining the durability of concrete structures. Most notably, pore solution resistivity can be used in the calculation of the formation factor of concrete, which is a ratio of the electrical resistivities of concrete and its pore solution. Additionally, the formation factor is equal to the inverse of the product of porosity and pore connectivity, which are critical parameters for concrete durability assessment. This paper explores alternative experimental techniques to extract pore solution of fresh cement pastes to be used to measure its composition and resistivity. All test methods were statistically analyzed using t-tests with a significance level of 0.05. First, the centrifuge extraction method was compared to the gas (nitrogen) pressure technique. The results showed that these two methods yielded no statistically significant difference in measured resistivity of the extracted pore solutions. Second, a cell method of measuring resistivity was compared to a probe method. A statistically significant difference was found. Future experimentation will be required to determine the reason for the cell method's higher recorded resistivities. Finally, 30-, 60-, and 90-minute time from initial mixing was compared to find a difference in the resistivity of extracted pore solutions. Pore solutions extracted between 30-90 minutes after initial mixing did not show any statistically significant difference in measured electrical resistivity.

Key Words: Fresh Paste, Fresh Concrete, Pore Solution, Resistivity, Formation Factor

Corresponding e-mail address: bieverj@oregonstate.edu

©Copyright by Joseph Biever June 3, 2021 Alternate Methods for the Extraction and Measurement of Pore Solution in Fresh Concrete

by Joseph Biever

## A THESIS

### submitted to

Oregon State University

Honors College

in partial fulfillment of the requirements for the degree of

Honors Baccalaureate of Science in Civil Engineering (Honors Scholar)

> Presented June 3<sup>rd</sup>, 2021 Commencement June 2021

Honors Baccalaureate of Science in Civil Engineering project of Joseph Biever presented on June 3, 2021.

APPROVED:

W. Jason Weiss, Mentor, representing Civil and Construction Engineering

O. Burkan Isgor, Committee Member, representing Civil and Construction Engineering

Rita Maria Ghantous, Committee Member, representing Civil and Construction Engineering

Toni Doolen, Dean, Oregon State University Honors College

I understand that my project will become part of the permanent collection of Oregon State University, Honors College. My signature below authorizes release of my project to any reader upon request.

Joseph Biever, Author

# **Table of Contents**

1	INT	RODUCTION 1	L
	1.1	BACKGROUND	1
	1.2	FORMATION FACTOR AND PORE SOLUTION	1
	1.3	CURRENT TECHNIQUES	3
	1.3.1	1 Pore Solution Extraction	3
	1.3.2	2 Cell Resistivity Measurement	4
	1.4	ALTERNATIVES	1
	1.5	INVESTIGATION OF TIME OF MEASUREMENT	5
	1.6	RESEARCH OBJECTIVES	5
2	ME	THODOLOGY	7
	2.1	MATERIALS	7
	2.2	SPECIMEN PREPARATION	7
	2.3	EXPERIMENTAL METHODS	7
	2.3.1	l Extraction Methods	3
	2.3.2	2 Resistivity Methods	3
	2.3.3	3 Mixing Time	3
3	RES	SULTS	Э
	3.1	EXTRACTION METHODS ANALYSIS	Э
	3.2	RESISTIVITY METHODS ANALYSIS	Э
	3.3	MIXING TIME ANALYSIS	)
4	CO	NCLUSION11	1
5	ACI	KNOWLEDGEMENTS 12	2
6	REF	FERENCES13	3

# 1 Introduction

### 1.1 Background

When concrete is cast on site, fresh concrete samples are often taken for quality control (QC)/quality assessment (QA) testing. Testing is performed to ensure that the delivered mixture meets engineering requirements, and this often consists of slump (ASTM C-143), air content (ASTM C-231), and unit weight (ASTM C-138) for fresh concrete QC. Compressive strength measurement (ASTM C-39) is typically performed at 28 days for further QC/QA. If the delivered concrete does not meet the standards and specified properties, mitigation actions must be implemented, which might include additional testing, reduced fees to the owner of the project, or the rejection and removal of cast concrete on site. However, since multiple performance tests such as compression testing are assessed several weeks after casting, the mitigation options become severely limited if concrete does not meet the required specifications.

More recent studies on fresh concrete, however, have shown great potential on how concrete is assessed in the field for QC/QA purposes when it is delivered. Formation factor, discussed further in the next section, can provide important durability information on fresh and hardened concrete. This means that all concrete testing can potentially be done on site with immediate results, which are extremely beneficial, as this process allows the assessment of delivered concrete before placement.

### 1.2 Formation Factor and Pore Solution

The formation factor (F) is a measurement of the pore structure of a cement paste or concrete, either in fresh or hardened state. Essentially, every concrete mixture has a characteristic pore system that can evolve over time, which can be characterized by the formation factor. This characterization is helpful in determining a concrete's susceptibility to durability-related problems such as chloride ingress (1-3), sulfate attack, or freeze-thaw damage. The formation factor of fresh concrete might also provide critical information about the performance of hardened concrete. F is defined as the ratio of a concrete bulk resistivity to the pore solution resistivity as shown in Equation 1 (4).

$$F = \frac{\rho_b}{\rho_o} \tag{1}$$

where:

 $\rho_b$  = concrete bulk resistivity in Ohm-m  $\rho_o$  = pore solution electrical resistivity in Ohm-m A large formation factor is better for durability concrete because it might imply that concrete will have low porosity and/or pore connectivity. For example, a large formation factor might result in lower ionic transport rates and water permeability. This means water and ions do not flow as freely through concrete and potentially cause durability issues such as sulfate attack, reinforcement corrosion, etc. While it is not a widely used technique for field measurement, formation factor is starting to become a part of standard measurements. For example, AASHTO PP-84, the *Standard Practice for Developing Performance Engineered Concrete Pavement Mixtures*, accepts formation factor specifications in order to estimate the resistance to chloride ingress in concrete.

Formation factor calculations are typically made on hardened concrete samples. More recently, however, studies have been performed on the effectiveness of formation factor with fresh concrete. Sallehi et al. calculated the formation factor of a variety of cementitious pastes containing OPC and supplementary cementitious materials such as fly ash, silica fume, and slag (5). They found the formation factor of fresh cement pastes to be correlated to the porosity, tortuosity, and water-to-cementitious material ratio (w/c) of concretes (5). This means that formation factor is not just useful for hardened concretes but could potentially be used for fresh concrete for QA/QC purposes at construction sites for same day characterization.

Determining bulk resistivity of fresh concrete is a relatively easy process. Two conductive rods made of stainless steel are placed into a cement or concrete paste. An AC potential is applied, and the AC current is measured. After geometric corrections are made, the resistivity of the bulk solution can be found. Pore solution resistivity, however, is more difficult to obtain as the pore solution must first be removed from the cementitious materials. Pore solution is essentially the fluid in the pores of paste, mortar, or concrete that contains a number of dissolved ionic species. The most important ionic species include sodium (Na<sup>+</sup>), potassium (K<sup>+</sup>), calcium (Ca<sup>+</sup>), hydroxide (OH<sup>-</sup>), and sulfate (SO<sub>4</sub><sup>2-</sup>), which affect the electrical resistivity and durability properties of concrete (6). Once water has been added to a concrete mixture, it is difficult to extract the ionic water in the fresh mixture. Consequently, Snyder et al. determined ways to indirectly compute the electrical resistivity of a pore solution by understanding the chemical composition of the pore solution (7). Chemical composition of pore solution has been determined in past studies through the use of inductively coupled plasma (6), Ion Chromatography (8), atomic absorption spectroscopy (9), and XRF (10). In the interest of affordability and simplicity, this thesis will focus on direct measurement of pore solution resistivity.

In hardened concrete samples, pore solution is obtained by using a pore press device. This device uses a hydraulic press to squeeze pore solution from a cast or extracted cylinder sample (11, 12). Fresh concrete paste pore solutions are typically extracted by applied positive\_pressure or vacuum (5, 13). Extraction of fresh pore solution is further discussed in the next section.

To measure the pore solution resistivity of fresh concrete accurately in the field, there are a number of factors that must be considered. For example, temperature affects the electrical resistivity, and field measurements must be corrected to a common temperature, e.g., 23 °C (14, 15). Additionally, carbonation or other contamination of pore solution can affect measured resistivity. An unknown influence on resistivity of pore solution is the time when the pore solution is extracted. From the initial time of mixing (when cementitious materials are combined with water) to testing, the ionic concentration of the pore solution might change, and this would affect measured resistivity (5).

- 1.3 Current Techniques
- 1.3.1 Pore Solution Extraction

The two most prevalent techniques to extract fresh paste pore solution are the nitrogen pressure device and the vacuum pump (5, 13). Both utilize pressure differences and a filter to extract the pore solution from a cement paste. The control for this study will be a nitrogen pressure extractor, as it is the easier of the two methods to perform and minimizes carbonation. Figure 1 shows a labeled diagram of the apparatus used in experimentation.



Figure 1: Expanded diagram of nitrogen pressure extraction apparatus

The nitrogen pressure method of extraction takes approximately 5 minutes to obtain a sufficient amount of pore solution, which is around 8 mL. Between each measurement, the apparatus must be taken apart, cleaned, rinsed with DI water, and a new filter must be inserted. It is only capable of testing one sample at a time. It also requires a nitrogen gas source, which makes it impractical for field applications. While tested and proven, it is a complex method for extracting pore solution,

especially when multiple samples need to be taken simultaneously. To avoid these concerns, simplified method of pore solution extraction will be studied.

### 1.3.2 Cell Resistivity Measurement

Electrical measurements of pore solution have been performed by using a resistivity meter and running a current through a cell of known dimensions filled with the pore solution (10, 11). Figure 2 shows detail of the resistivity cell and the brass plates.



Figure 2: Resistivity cell with conductive metal plates at both ends for circuit connections. A small syringe is pictured (right) that is used to insert pore solution through the small holes near the top of the cylinder.

To perform a measurement, the acrylic cell is inserted into the indentation on either side of the brass plates. Pore solution is then inserted with the syringe through the holes on the top of the acrylic cell. Alligator clips are attached to the connection points for the resistivity meter, and the resistance in ohms is recorded. This cell method is preferred for measuring pore solution properties of cured concrete as it requires less than 2 mL of pore solution. Many times, only around 2 mL of fluid can be extracted from fully hydrated concrete samples (11, 12, 16, 17). There are a number of steps that must be taken with this method of electrical measurement to ensure accuracy. Error from cross-contamination between pore solution samples, oxidation of the brass plates, and air trapped in the acrylic cell must be minimized. The plates, syringe, and cell are rinsed with DI water between uses to prevent crosscontamination of pore solution samples. Wires need to be securely fastened, and connection points are cleaned to ensure proper current flow. The brass plates are rubbed with a 600 grit and 1000 grit sandpaper to remove the oxidized surface. Finally, precision is used to minimize air bubbles within the acrylic cell. After the test is performed, calculations are made to adjust for the phase angle, geometry of the cell, and to correct resistivity to standard temperature 23°C (14, 15).

### 1.4 Alternatives

An alternative to the nitrogen pressure method of extraction utilizes a centrifuge to extract pore solution. A centrifuge spins at high velocities to separate particles

according to their densities. A centrifuge can separate the pore solution from cement paste because cement particles are denser than the pore solution, causing them to separate. The 50 mL centrifuge tubes can be filled with concrete paste, and 5 minutes later four pore solution samples are separated from the cementitious materials. The pore solution can then be decanted into another vessel for testing. The benefits of a centrifuge are portability, ease of use, and quantity of samples it can run. The process requires very little space and electrical power to operate, and no cleaning is required between uses. The 50 mL centrifuge tubes can be disposed of after decanting the pore solution. It also has the ability to run 4 samples simultaneously for the setup used, with potential to have additional samples tested in more advanced setups, allowing for testing to be performed much faster. The centrifuge method also yields more pore solution from a single cycle. Testing four 50 mL samples of 0.35 w/c paste yields roughly 20 mL of pore solution total. An image of the centrifuge used in this study is pictured in Figure 3a, along with an image of the separated pore solution in Figure 3b



Figure 3a: Picture of centrifuge setup with 50 mL centrifuge tubes. Figure 3b: Image of specimen after cycled in centrifuge. As the material at the bottom is consolidated, the pore solution left atop can be decanted.

An alternative to the cell method of measuring resistivity utilizes a commercial resistivity probe. The probe attaches to a benchtop meter. Probes can also contain a temperature sensor like the one used in this study, and the internal computer adjusts the reading to a standard 23°C reading. The conductivity probe is calibrated before testing with a standard solution. Typically, probes are not used to measure resistivity in pore solution measurement because they require at least 5 mL of pore solution. Therefore, many past studies have used the cell method as it requires only 2 mL of pore solution for a reading. By utilizing smaller plastic test tubes and a thinner probe, this study was able to obtain probed resistivity measurements with only 5 mL of pore solution. An example of the probe and benchtop meter conducting a reading is pictured in Figure 4. A single reading takes a minute to obtain, which is significantly faster than the cell resistivity method.



Figure 4: Benchtop meter with conductivity probe in 10 mL centrifuge tube (right).

# 1.5 Investigation of Time of Measurement

A simplified method of extracting and testing a pore solution would exclude time as a factor in the field-testing methods. Ready-mix trucks typically arrive on site anywhere from 30 to 90 minutes after time of initial concrete mixing. It is worth noting some deliveries may arrive prior to 30 minutes from time of initial mixing, although these are rare cases. 90 minutes cannot be exceeded as it is a violation of ASTM C-94 guidelines. Ideally, pore solution extraction and testing could be performed anywhere within the 30–90-minute from initial time of mixing window with no effect on measurements taken. Past studies have taken multiple measurements within this time window (5). Time from initial mixture will be studied in this paper to explore if there is a statistically significant difference in values with increasing time.

### 1.6 Research Objectives

The first objective of this paper is to compare a nitrogen pressure extraction of pore solution to the alternative centrifuge method. T-tests will be utilized to see if there is a statistically significant difference in the measured data. The second objective will be comparing the cell method of resistivity measurement to the proposed probe alternative. T-test statistical analysis will be used once again to see if there is a difference in data. The third objective is to examine the variation in pore solution resistivity obtained from a cement paste that has sat for 30-90 minutes since initial mixing. ANOVA tests will be run to find if there is any statistically significant difference in a pore solution extracted from a cement paste that is delivered 30-,60-, or 90-minutes after its initial mixing to simulate ready-mix delay to construction sites.

# 2 Methodology

## 2.1 Materials

An ordinary type I portland cement (OPC I) consistent with ASTM C150–19a was used in this study. Specific gravity of this cement paste was 3.15, and the Blaine fineness was found to be 420 m<sup>2</sup>/kg. Water used in all mixtures was deionized (DI) water. Table 1 lists chemical properties of the type I Portland cement used in this study.

Compart Oridaa	Type I cement (OPC I)		
Cement Oxides	Percent by mass (%)		
Silicon Dioxide (SiO <sub>2</sub> )	19.9		
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	4.6		
Ferric Oxide (Fe <sub>2</sub> O <sub>3</sub> )	3.2		
Calcium Oxide (CaO)	62.0		
Magnesium Oxide (MgO)	3.8		
Sulfur Trioxide (SO <sub>3</sub> )	2.8		
Alkalies (Na <sub>2</sub> O+0.658*K <sub>2</sub> O)	0.57		
Loss on Ignition (LOI)	1.6		
Bogue phase composition	Percent by mass (%)		
Tricalcium Silicate (C <sub>3</sub> S)	57.0		
Dicalcium Silicate ( $C_2S$ )	14.0		
Tricalcium Aluminate (C <sub>3</sub> A)	7.0		
Tetracalcium Aluminoferrite	10.0		
(C <sub>4</sub> AF)			

Table 1: Chemical Properties of OPC I used in study

# 2.2 Specimen Preparation

This study examines only cement paste specimens. Before experimentation, fifty 500gram pre-measured OPC batches were vacuum sealed to ensure minimal exposure to oxygen over the duration of experiments. To begin an experiment, a bag was opened and combined with the proper quantity of DI water to obtain a w/c of either 0.35, 0.45, and 0.55. Because mix time was also a variable, a timer was started immediately once water was added to the cementitious materials. The cement paste was mixed in a Renfert 1000 mL tabletop vacuum mixer at 400 RPM and 80% suction to minimize air entrainment and was agitated for one minute and thirty seconds every 15 minutes until it had reached its desired mix time. This continuous agitation is intended to simulate the agitation that occurs in ready-mix truck conditions.

2.3 Experimental Methods

### 2.3.1 Extraction Methods

*Nitrogen Pressure Extraction* - Cement paste was prepared in three w/c variations of 0.35, 0.45, and 0.55 and mixed for one minute and thirty seconds. It was agitated at 15 minutes and at 30 minutes from initial mixing for the same duration. During mixing, the nitrogen pressure extractor was cleaned with DI water and wiped dry with a clean towel. A fresh cellulose filter with an average pore diameter of 0.45 micrometers was placed over the wire mesh filter and screwed onto the main chamber. Once the cement mixture was prepared, it was immediately loaded into the main chamber of the pore solution extraction chamber shown by the diagram in Figure 1. The top was screwed onto the chamber, and 200 kPa of nitrogen gas was used to pressurize the chamber for 5 minutes to extract the fluid. A labeled 10 mL centrifuge tube was placed below the apparatus to catch the pore solution for testing. It was filled to the 5 mL for each sample.

*Centrifuge Extraction* - To extract pore solution using the centrifuge, the same 0.35, 0.45, and 0.55 w/c samples were mixed for one minute and thirty seconds. Samples were agitated at 15 minutes and at 30 minutes from initial mixing. Once prepared, the cement was poured with a funnel into four 50 mL test tubes to the 50 mL marker. The screw on caps were fastened snug, and all four tubes were inserted into a Cole-Parmer 4 x 50mL swing bucket rotor centrifuge. It was spun at 4000 rpm for a time of 5 minutes. The specimens were then removed, and the pore solution was decanted off the top of the 50 mL tubes into smaller centrifuge tubes of 10 mL volume and labeled. The 10 mL testing centrifuge tubes were filled to 5 mL for each sample.

### 2.3.2 Resistivity Methods

*Cell measurement* -The first method utilizes a small cylinder of known dimensions. The apparatus has an electrical current running through it from a resistance meter to measure the resistivity of the circuit. A frequency of 10 kHz was used for all samples to minimize the phase angle adjustment. Cross sectional area of the resistivity cell was measured to be 69.84 mm<sup>2</sup>, and the length was 25.35 mm which gave a geometry factor of 2.755 mm. All resistivity measurements were corrected for temperature with the standard temperature for adjustment to 23 °C.

*Probe Measurement* - Resistivity was also measured using a resistivity probe on a VWR Symphony B40PCID benchtop meter. The measurements were all taken as conductivity in mS/cm, which is inversely related to resistivity. The probe is stored in a KCL solution, used to condition the electrodes, and is calibrated using a single solution of known resistivity. The calibration solution chosen was 0.05 ohm-m as this value is close to the measurements that would be taken. After calibration, the device was lowered directly into the 10 mL test tube containing 5 mL of pore solution. Temperature of the solution was measured by the device and was directly processed and adjusted by the benchtop meter.

### 2.3.3 Mixing Time

To analyze mix time, 0.35, 0.45, and 0.35 w/c samples were prepared, and mixed for one minute and thirty seconds. Each mixture was agitated every 15 minutes for one minute and thirty seconds until they reached a total of 30-, 60-, or 90-minutes. The centrifuge method of extraction was used for all mix times. Each extracted pore solution was prepared for resistivity measurements as before and labeled to avoid misreading. The probe method of resistivity measurement was used for all samples for comparison.

# 3 Results

## 3.1 Extraction Methods Analysis

Table 2 shows the measured resistivity values of pore solution extracted using both the nitrogen pressure and centrifuge extraction methods. Because the calculated probabilities (p-values) across all three w/c tests were above 0.05, a significant difference between the test methods does not exist.

w/c	Extraction	Resistivity	Standard Coefficie		P-Value
	Method	(ohm-m)	Deviation	of	
			(ohm-m)	Variation	
0.35	Pore Press	0.222	0.00419	0.0188	0.241
	Centrifuge	0.225	0.00332	0.0147	
0.45	Pore Press	0.258	0.00432	0.0167	0.145
	Centrifuge	0.262	0.00159	0.0061	
0.55	Pore Press	0.294	0.00224	0.0076	0.372
	Centrifuge	0.295	0.00087	0.0030	

Table 2: Statistical analysis of differences in extraction methods of pore solution

The centrifuge method of extraction was the easier of the two methods to perform in experimentation. Samples were easier to prepare, and cleanup was much faster. It also yielded 3 mL more pore solution in 0.35 w/c samples. There are many added benefits to using the centrifuge method of extraction over the nitrogen pressure method.

### 3.2 Resistivity Methods Analysis

Table 3 shows resistivity measurements made using both the probe and cell methods. The centrifuge method of extraction was used for this comparison for consistency in results. All calculated probability values were lower than the significance interval of 0.05, indicating a statistically significant difference was present between the cell and probe methods of resistivity measurement.

w/c	Resistivity	Resistivity Standard		Coefficient	P-Value
	measurement	measurement	Deviation	of	
	method	(ohm-m)	(ohm-m)	Variation	
0.35	Cell	0.276	0.00439	0.0159	0.00577
	Probe	0.225	0.00332	0.0148	
0.45	Cell	0.317	0.00268	0.0085	0.00160
	Probe	0.262	0.00159	0.0061	
0.55	Cell	0.370	0.00573	0.0155	0.00297
	Probe	0.295	0.00087	0.0029	

Table 3: Statistical analysis of differences in resistivity measurements

Table 3 indicates that the resistivity measured in the cell for the 0.35, 0.45, and 0.55 w/c pastes yielded pore solution resistivities that were 0.051, 0.055, and 0.075 ohm-m larger than the probe method. Thus, the team has reason to believe there are errors with the cell measurement and are currently exploring the cause of the discrepancy. Further investigation of the cell method will include measuring the electrical properties of solutions with known electrical properties to confirm both the geometry correction factors and properties of the hardware being used. After testing, a replacement of wiring and thorough cleaning of the equipment is planned.

# 3.3 Mixing Time Analysis

Table 4 shows pore solution resistivity measurements performed on 0.35, 0.45, and 0.55 w/c cement pastes pore solutions at three different mixture times. For all pore solutions, there is no noticeable trend of resistivity with respect to mixing time. For all three of the experiments, analysis of variation (ANOVA) testing was performed to find if there was a statistically significant difference in the measured resistivity. All three of the ANOVA test results (comparing 30, 60 and 90 minute times) show that there is no statistically significant difference in pore solution resistivity at 30-, 60-, and 90-minutes after initial mixture of cement paste.

w/c	Time From	Resistivity	Standard	Coefficient of	P-Values
	Initial Mixing	(ohm-m)	Deviation	Variation	(ANOVA
	(min.)		(ohm-m)		results)
0.35	30	0.206	0.0179	0.0869	0.604
	60	0.199	0.0127	0.0638	
	90	0.198	0.0124	0.0626	
0.45	30	0.253	0.0099	0.0391	0.621
	60	0.259	0.0014	0.0054	
	90	0.254	0.0028	0.0110	
0.55	30	0.289	0.0122	0.0422	0.606
	60	0.285	0.0041	0.0144	
	90	0.284	0.0032	0.0113	

*Table 4: Statistical analysis of differences between 30-, 60-, and 90-minutes from initial mixing* 

Ready-mix trucks typically arrive on site between 30- and 90-minutes after the mixture is first prepared at a batching plant. Because the statistical analysis shows the pore solution resistivity does not significantly change in this fresh state of concrete, testing can be simplified. All pore solution resistivity measurements taken within the 30-90 minute window from initial mixing will not require time adjustments.

### 4 Conclusion

The first objective of this thesis was to compare a nitrogen pressure extraction of pore solution to the proposed centrifuge method. The experimental results indicate that the there is no statistically significant difference in the pore solution properties when a centrifuge is used for extraction rather than a nitrogen pressure extraction. Additionally, the centrifuge method can perform multiple tests at a time rather than one, is easier to operate, and is more portable than the nitrogen pressure extractor. Therefore, the centrifuge method of pore solution extraction is a viable and efficient field-testing technique.

The second objective of this thesis was to compare two measurement methods for pore solution resistivity to determine whether they are statistically similar. P-test analysis showed a statistically significant difference between these methods at a significance interval of 0.05. Although the probe method of measuring resistivity is a more ideal field test method due to its usability and faster measurement times, further experimentation is required to ensure it is a viable test method. This will be achieved by using multiple resistivity buffer solutions on both the cell and probe resistivity methods, and by replacing and cleaning hardware on the resistivity unit before testing.

The third objective of this thesis was to examine the variation in pore solution resistivity obtained from a cement paste in the first 90 minutes of mixing. Statistical analysis of the measured pore solution resistivity showed that time from initial mixture of 30-, 60-, and 90- minutes could be considered statistically similar. This makes measurements in the field more practical, as it allows pore solution resistivity tests to be calculated without factoring in corrections for time from initial mixing.

# 5 Acknowledgements

I would like to dedicate this work to my uncle, Brian Biever, who I have to thank for my undergraduate college experience here at Oregon State. Without him I may have never been set on this academic path. I am continually inspired to learn and make the most of my academic experience, and I hope this makes the entire Biever family proud.

I would like to thank my family for their support through my academic journey thus far: my parents Robert and Jennifer Biever, and my sister Abby. Whenever I encounter a difficult time or want to spend the weekend home with my family, they are always there for me. They have been my biggest cheer leaders, and I would not have made it this far without them.

I would also like to thank my extremely talented mentors at Oregon State University: Dr. Jason Weiss, Dr. Burkan Isgor, for sharing their extensive knowledge and passion for concrete materials. I am absolutely honored to work for such a talented and respected team of researchers. I would also like to give thanks to Dr. Rita Maria Ghantous for offering me my first job in the concrete materials research lab and setting me on the path I am on now. Dr. Weiss, Dr. Isgor, and Dr Maria Ghantous are all co-authors of this work and assisted with suggestions and editing for the final paper.

Finally, to all my friends and co-workers who have helped me with project details such as coding, gathering supplies, and making concrete jokes, thank you for the support and inspiration.

# 6 References

- Snyder K., "The Relationship Between the Formation Factor and the Diffusion Coefficient of Porous Materials Saturated with Concentrated Electrolytes: Theoretical and Experimental Considerations," Concr Sci Eng., 2010, 3.
- Weiss W, Ley M, Isgor O, Van Dam T., "Toward Performance Specifications for Concrete Durability: Using the Formation Factor for Corrosion and Critical Saturation for Freeze-Thaw," Transportation Research Board 96<sup>th</sup> Annual Meeting, 2017.
- Spragg RP., "Development of Performance Related Specifications that Include Formation Factor," Purdue University dgi, editor: Thesis (Ph. D.)--Purdue University, 2017.
- 4. Archie GE., *"The Electrical Resistivity Log as an Aid in Determining Some Reservoir Characteristics,"* Transactions of the AIME., 1942, 146:54-62.
- Sallehi H, Ghods P, Isgor OB., "Formation Factor of Fresh Cementitious Pastes," Cement and Concrete Composites, 2018, 91:174-88.
- 6. Andersson K, Allard B, Bengtsson M, Magnusson B., "*Chemical Composition of Cement Pore Solutions*," Cement and Concrete Research, 1989, 19(3):327-32.
- 7. Snyder KA, Feng X, Keen BD, Mason TO., "Estimating the Electrical Conductivity of Cement Paste Pore Solutions from OH -, K + and Na + Concentrations," Cement and Concrete Research, 2003, 33(6):793-8.
- Lothenbach B, Matschei T, Mschner Gr, Glasser FP., "Thermodynamic Modelling of the Effect of Temperature on the Hydration and Porosity of Portland Cement," Cement and Concrete Research, 2008, 38(1):1-18.
- 9. Rajabipour F, Sant G, Weiss J., "Interactions Between Shrinkage Reducing Admixtures (SRA) and Cement Paste's Pore Solution," Cement and Concrete Research, 2008, 38(5):606-15.
- Tsui Chang M., "The Evaluation of Cementitious Pore Solution Composition and Electrical Resistivity Using X-ray Fluorescence (XRF)," Masters Thesis, Oregon State University, 2019.
- Barneyback RS, Diamond S., "Expression and Analysis of Pore Fluids from Hardened Cement Pastes and Mortars," Cement and Concrete Research, 1981, 11(2):279-85.

- Longuet P, Burglen L, Zelwer A., "The Liquid Phase of Hydrated Cement," Rev Matér Constr Trav Publics, 1973, 676:35-41.
- Tsui-Chang M, Suraneni P, Montanari L, Munoz JF, Weiss WJ., "Determination of Chemical Composition and Electrical Resistivity of Expressed Cementitious Pore Solutions Using X-Ray Fluorescence," ACI Materials Journal, 2019, 116(1):155-64.
- Coyle AT, Spragg RP, Suraneni P, Amirkhanian AN, Tsui-Chang M, Weiss WJ., "Activation Energy of Conduction for Use in Temperature Corrections on Electrical Measurements of Concrete," Advances in Civil Engineering Materials, 2019, 8(1):158-70.
- Spragg R, Villani C, Snyder K, Bentz D, Bullard JW, Weiss J., "Factors That Influence Electrical Resistivity Measurements in Cementitious Systems," Transportation Research Record, 2013, 2342(2342):90-8.
- Cyr M, Labrecque F, Daidié A., "High-Pressure Device for Fluid Extraction from Porous Materials: Application to Cement-Based Materials," Journal of the American Ceramic Society, 2008, 91:2653-8.
- Spragg R, Bu Y, Snyder K, Bentz D, Weiss W., "Electrical Testing of Cement-Based Materials: Role of Testing Techniques, Sample Conditioning, and Accelerated Curing," Publication FHWA/IN/JTRP, 2013.