The Thesis Committee for Anna Kate Miller
Certifies that this is the approved version of the following Thesis:

Towards Better Characterization and Understanding of Internal Stability of UltraFine Grouts

APPROVED BY
SUPERVISING COMMITTEE:

Chadi S. El Mohtar, Supervisor

Maria Juenger

Towards Better Characterization and Understanding of Internal Stability of UltraFine Grouts

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Anna Kate Miller

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Dedication

This thesis is dedicated to Dr. Chadi S. El Mohtar. Ever since the beginning of my graduate career, Dr. El Mohtar has been a huge influence. As an undergraduate, Dr. El Mohtar provided me with the opportunity to assist his graduate students in their research and eventually to begin research of my own. At a time when I was unsure about my future, his support encouraged me to continue on to graduate school. Throughout the entire time I've known him, he has continuously pushed me to achieve and has seen my potential even when I was unsure of it myself. He always encouraged me to explore new avenues of research and has always provided guidance and insight when I needed it. I believe with 100% certainty I would not be where I am today without him.

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Finally, I need to thank Aaron. Though I may have driven him crazy, he kept me sane. He has been supportive above and beyond what was expected or asked for.

Abstract

Towards Better Characterization and Understanding of Internal Stability of UltraFine Grouts

Anna Kate Miller, M.S.E

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Supervisor: Chadi S. El Mohtar

Internal stability of a grout is critical in determining whether a grouting job is successful or not. A highly stable grout would be able to permeate through formations/fractures without losing any significant percentage of its solids fraction to filtration. Having an unstable grout would result in non-uniform distribution of the cement along the grouted distance, with a higher concentration of cement near the injection point (above the design value based on the grout concentration) and a much weaker cementation at the end of the grouted zone. Formations grouted with unstable grouts end up with non-uniformly enhanced properties and increase the local heterogeneity. Due to the many problems associated with unstable grout use, it is undesirable in most cement grouting operations (Rosquoët et al. 2003, Naudts et al. 2004, Tan et al. 2004, Bremen 1997). This Thesis presents a comprehensive study on internal stability of a micro-fine cement (UltraFine) and its impact on grouted sand through an experimental investigation.

The hydration and particle size distribution of the Ultrafine cement is determined through a physio-chemical characterization to help understand the observed behavior during stability tests. Grouts with a wide range of w/c ratios are then tested using the

traditional internal stability characterization methods (Column Bleed Test and API Filter press test). These tests are index tests and do not represent the state of the grout during its application in the field, among other limitation. Additionally, image analysis is used to better understand grout stability and the movement of solids within a standing column. The digital analysis approach allows for characterization of the bleed test beyond measuring the height of free standing water on top by accounting for the change in grout concentration with depth.

A new testing procedure (Dynamic Stability Test) is developed to investigate the impact of continuous shearing on the internal stability of the grouts. All the current methods for determining the stability of a grout are performed under static condition. However, for its first few hours after mixing in the field, the grout is never under static. The new method measures the change in the rheology of a grout using Physica MCR 301 rheometer (Anton Paar, Graz, Austria). The rheological measurements are performed under different shearing conditions between measurements to evaluate impact of shearing/flow on internal stability of the grouts and the results showed that grouts are more stable when continuously sheared, implying that the static tests can underestimate internal stability of grouts.

Last, a new Grout Filtration Test is proposed in this study that is a modification of the filter press to better model the grout performance in the field. Two testing procedures and analysis methods (Simplified field and Laboratory) are presented to quantify the internal stability of grouts. Results from the current and newly proposed testing are used to better understand the internal stability of the grout and identify the most efficient way to measure grout internal stability.

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Chapter 1: Introduction

1.1 Problem Statement

Permeation grouting has been used for decades as a cost-effective low disturbance ground improvement method that has allowed geotechnical engineers to strengthen soils and provide the necessary water flow. While permeation grouting can be a very useful tool on any construction site, as adequate design can be the difference between a success and an expensive, inefficient venture. Current grout design relies heavily on "rules-of-thumb" and outdated charts, and final grouting decisions are made based on index measurements, such as fines content or soil classification, at most. "*A major reason behind the lack of proper design guidelines in permeation grouting is the complexity of the mechanisms governing flow of suspensions through porous media. Particularly, the diversity of possible flow stoppage mechanisms involved and lack of fundamental understanding of how to relate the properties of the grout to these stoppage mechanisms. For some cases, flow stoppage occurs due to rheological blocking especially in highly viscous grouts; however, in other cases, permeation depth is controlled by filtration and it is not uncommon to have both mechanisms contributing to the stoppage of flow*" (El Mohtar et al. 2017).

Filtration is the process through which some of the suspension particles are trapped by the porous medium it is flowing through. This process results in changes in the properties of porous media (decrease in porosity) as well as the rheological properties of the suspension progressing forward (a decrease in the suspension concentration leads to lower yield stress and viscosity). This filtration process depends on the porous medium being permeated as well as the properties of the grout, particularly, its internal stability. Currently, the characterization of the grout material is mostly done using crude measurements that cannot capture the complex shear and time dependent properties of the grout.

Internal stability of a grout is critical in determining whether a grouting job is successful or not. A highly stable grout would be able to permeate through formations/fractures without losing any significant percentage of its solids fraction to filtration. Having an unstable grout would result in non-uniform distribution of cement along the grouted distance, with a higher concentration of cement near the injection point (above the design value based on grout concentration) and a much weaker cementation at the end of the grouted zone. Formations grouted with unstable grouts end up with non-uniformly enhanced properties and an increase in local heterogeneity. Due to the many problems associated with unstable grout use, it is undesirable in most cement grouting operations (Rosquoët et al. 2003, Naudts et al. 2004, Tan et al. 2004, Bremen 1997). This Thesis presents a comprehensive study on internal stability of a micro-fine cement (UltraFine) and its impact on grouted sand through an experimental investigation.

The hydration and particle size distribution of the Ultrafine cement is determined through a physio-chemical characterization to help understand the observed behavior during stability tests. Grouts with a wide range of w/c ratios are then tested using the traditional internal stability characterization methods (Column Bleed Test and API Filter press test). These tests are index tests and do not represent the state of the grout during its application in the field, among other limitations. Additionally, image analysis is used to better understand grout stability and the movement of solids within a standing column. The digital analysis approach allows for characterization of the bleed test beyond measuring the height of free standing water on top by accounting for the change in grout concentration with depth.

A new testing procedure (Dynamic Stability Test) is developed to investigate the impact of continuous shearing on the internal stability of the grouts. All the current methods for determining the stability of a grout (Column Bleed Test, API Filter Press as well as the newly proposed Grout Filtration Test) are performed under static condition. However, for the first few hours after the grout is mixed in the field, the grout is never under static state (this includes mixing, storage in agitation tanks, pumping through the grouting lines up until its permeation through the target formation). The new method measures the change in the rheology of a grout (at a given location) using Physica MCR 301 rheometer (Anton Paar, Graz, Austria). The rheological measurements are performed under different shearing conditions between measurements to evaluate impact of shearing/flow on internal stability of the grouts. Results show that grouts are more stable when continuously sheared, which implies that static tests can underestimate the internal stability of grouts.

Last, a new Grout Filtration Test is proposed in this study that is a modification of the filter press to better model the grout performance in the field. Two testing procedures and analysis methods (Simplified field and Laboratory) are presented to quantify the internal stability of grouts. Results from the current and the newly proposed testing are used to better understand the internal stability of the grout and to identify the most efficient way to measure grout internal stability.

Considering the limited testing methodologies and understanding of internal stability of grout, despite its significant impact on the success of a grouting job, the primary objectives of this research program were to:

1) evaluate the current existing methods for grout stability measurements,

2) evaluate their limitations and the resulting impact on grout stability evaluation,

3) develop necessary testing methods to achieve more insight on internal stability of grouts, and

4) develop a more representative testing procedure to evaluate grout internal stability.

1.2 Thesis Organization

This thesis is organized into seven main chapters. Following this introduction, the physio-chemical characterization of the Ultrafine cement is presented in Chapter 2. Several tests were run to characterize the cement blend used in this study. The results obtained in this chapter are useful for obtaining a more comprehensive understanding of the grout properties investigated in subsequent chapters. The results from particle size distribution and hydration tests run on cement blend are presented and discussed.

Chapter 3 focuses on the mechanical characterization of a wide range of w/c ratio Ultrafine cement grouts. Rheology and internal stability of grouts are two of the most important properties of a cement grout. The rheology of grout has long been used to determine the groutability and grout penetration depth of grouts into soils and rock fractures. Both yield stress and apparent viscosity values are determined in this Chapter for grouts at different w/c ratios with and without superplasticizers. Additionally, the internal stability of a grout can be the difference between a uniformly grouted formation and a non-uniformly distributed cement content leading to a very heterogeneous site. The results from standing column bleed test, a current standard test of grout stability, are presented in this chapter for a range of grouts.

Chapter 4 presents a new approach to evaluate grout stability in a standing column using image analysis. In current practice, the stability of grout is determined by the amount of bleed, which is a measure of the amount of clear water that appears at the top of a grout column after a waiting time of two hours. This method is used to categorize grouts as either “unstable” or “stable”, based on a limiting bleed percentage. A major limitation of this test method is that it does not describe changes over time. Although these can be recorded, the process is time consuming and plots of change over time are not usually reported. If changes over time are reported, only changes observed at the top of the column are reported (height of clear water) and all changes in the grout throughout the rest of the column height are ignored. This chapter presents a new, more comprehensive method of analyzing changes in grout concentration over the height of a standing column over time using image analysis techniques. The image analysis tests allow for the collection of significantly more data with little to no added effort. Manual pipette tests are used to verify the image analysis results.

Chapter 5 addresses the other major limitation in current internal stability testing of grouts: tests are all performed under static conditions. Currently, the methods of determining the stability of a grout (Column Bleed test, API filter press, etc.) only look at grout in a static condition, whereas, in the field, the grout will mostly be flowing during the same period as that of the testing. To get a better understanding of how a grout behaves as it is flowing in the field, a Dynamic Stability Testing protocol is introduced in this Chapter. The newly proposed testing employs a Physica MCR 301 rheometer (Anton Paar, Graz, Austria) and measures the change in modulus of the grout over time under various shearing rate conditions.

A New Grout Filtration Test is presented and discussed in Chapter 6. The new proposed testing can be performed on site or in the lab to evaluate the performance of a grout mix under field conditions. This test allows the engineer in the field to evaluate the penetrability of a grout, mixed using field materials and mixing equipment, through soil samples representative of field conditions. The results from testing a wide range of w/c ratios are presented.

Chapter 7 contains a short summary of the work completed for this study, the conclusions made regarding the current and newly proposed grout stability testing, and recommendations for future research.

Chapter 2: Physio-Chemical Characterization of Cement

2.1 INTRODUCTION

Several tests were run to characterize the cement blend used in this study. The results obtained in this chapter are useful for obtaining a more comprehensive understanding of the grout properties investigated in subsequent chapters. The results from particle size distribution and hydration the tests run on cement blend are presented and discussed in this Chapter.

2.2 MATERIALS USED

The cement blend used throughout this study is a blast furnace slag (BFS) based ultrafine cement. It is a proprietary mix and therefore the exact proportions of ordinary Portland cement (OPC) to BFS are not specified. Blast furnace slag is a very popular supplementary cementitious material (SCM) because it is a waste product of other industrial processes. It also provides reduced risk of alkali-silica reaction (ASR) and provides resistance to sulfate attack (Heaton et al., 2012). Furthermore, BFS lowers the heat and slows the rate of hydration, which is often desirable in grouting projects.

All grout mixes throughout this study are made with de-aired, de-ionized water to eliminate behavior changes due to varied ionic strengths in water. The high range water reducer/superplasticizer (SP) used in this study is a naphthalene sulfonate. This specific product is designed to work well with blended cement, such as BFS or fly ash blended cement. It has a specific gravity of 1.22 and a pH of 9.0. The recommended dosage by the manufacturer is 0.6%-2.4% by weight of cement. However, it is important to note that these recommendations are based on water reducing in concrete mixes, not grout mixes, and therefore should only be interpreted as loose guidelines.

2.3 PARTICLE SIZE DISTRIBUTION

2.3.1 Background

Understanding the particle size distribution of a cement is important; it not only affects the properties of the grout, but also helps to determine if the grout will be useable for a permeation grouting application (i.e. helps determine the "groutability" of a site). In general, particle size affects the hydration rate, workability, bleed, freeze-thaw resistance and gypsum addition required in a grout. As particle sizes decrease, the total surface area increases, which leads to faster reaction (hydration), increased need for water and/or water reducers to maintain workability, less bleeding, less resistance to freeze/thaw cycles and more gypsum required to prevent flash set. Additionally, knowing the particle size distribution of the cement as compared to the void sizes in the formation that is to be grouted can help indicate how successful the grouting program will be. Though the success of a permeation grouting program depends on a multitude of factors, some of which will be discussed later on in this study, the particle size distributions of the cement and the soil are a good place to start. Burwell (1958) suggested that the groutability of a soil be determined by the simple equation:

$$N = \frac{d_{15,soil}}{d_{85,grout}} \qquad \text{(Equation 1)}$$

where N greater than 25 indicated a "groutable" soil and N less than 11 indicated an "ungroutable" soil. A similar, slightly more complex relationship was proposed by Akabulut and Saglamar (2002). Additionally, a "rule of thumb" in industry is that the largest particle size of the grout should be 1/3 the size of the aperture to be grouted. Thus, knowing the particle size distribution of the cement can help determine whether it can be used at a particular site or if spending more to achieve a finer grind or use a chemical grout is justified.

The particle size distribution (PSD) of the cement blend used in this study was determined using laser diffraction (LAS). Laser diffraction is a method of determining PSD by measuring and analyzing the distribution of laser-produced light passing through a suspension of particles. This method is useful because it can analyze different kinds of particles, unlike Blaine, which can only be used for ordinary Portland cement. Additionally, it provides information about the entire particle size distribution, which can be useful in understanding the behavior of the grout. However, this method does come with a few challenges. First, the size distribution of cement/SCMs can be broad (from 100 µm to less than 1 µm). Therefore, it is important to make sure that the equipment being used can handle the range of expected sizes in the cement being analyzed. Second, cement particles are agglomerated. The particles have to be thoroughly dispersed before measuring or the distribution will be skewed. Therefore, using several dispersion methods to make sure particles are properly dispersed is important. Additionally, cement particles are irregular in shape. These irregularities can cause inconsistencies in particle size measurements due to the fact that most available analysis methods assume spherical geometry. However, although cement particles are not spherical, they are generally equi-axial. Therefore, the assumption that they are spherical should not lead to significant losses in accuracy, unlike for plate-like or needle-like particles. Finally, cement mixes have inhomogeneous composition, especially if SCMs are used, and heterogeneous composition affects laser scattering. In a blended cement, not only do the different materials have different refractive indices, but the phases within OPC have different refractive indices as well. This causes additional difficulties in analysis because a single refractive index must be specified to run laser diffraction. Given that the refractive index for OPC is approximately 1.7 and the refractive index for BFS is approximately 1.6, the input refractive index value should not cause significant error.

2.3.2 Procedure

The particle size distribution (PSD) of the cement blend used in this study was determined using the Mastersizer 2000 (Malvern Instruments Ltd.). Standard measurement range for this unit ranges from 0.02 to 2000 µm, which is appropriate for the material in this study. The Hydro 2000MU dispersion unit (Malvern Instruments Ltd.) was used in conjunction with the Mastersizer 2000 to prepare the sample and then deliver it to the optical bench. The test procedure is as follows: First, input the sample material name (cement), density (3.15 g/cm^3), refractive index (1.68) and absorption (0.8) and the dispersant name (isopropyl alcohol) and refractive index (1.39) into the Mastercizer 2000 software. Next, fill a 1000 mL beaker with isopropyl alcohol and place under the mixing apparatus. Note that isopropyl alcohol is used as the dispersant fluid instead of water so that the cement will not hydrate as the test is run. Turn on Hydro 2000MU dispersion unit and optical bench. Set pump speed to 1500 rpm and wait 10 minutes to clear system of bubbles. Increase the pump speed to 2300 rpm.

This speed was chosen because it provides good dispersion without creating air bubbles. Before the sample is added, the background is measured to clean the data of background electrical noise and to prevent scattering of the data from dust on the optics and contaminants floating in the “clean” dispersant. Now the setup is ready for the sample to be added. As previously mentioned, good sample preparation is critical for achieving accurate results. Dry powders, such as the cement blend used, tend to separate out if stored for a long time or vibrated. When this happens, larger particles tend to rise to the top, while the smaller particles collect at the bottom. Because the amount of sample needed for testing is quite small, getting an unrepresentative sample can greatly affect the results. Therefore, care was taken to obtain a representative sample for testing. Once a representative sample is obtained, the sample is slowly added until the desired obscuration is reached. The Mastersizer 2000 software reports exactly what level of signal the sample generates and whether this is ideal, too high, too low, etc. The sample concentration is controlled by monitoring the obscuration of the laser beam caused by the sample. The obscuration is simply the fraction of light "lost" from the main beam when the sample is introduced. The obscuration of the incident laser beam measured during the "measure background" step is removed from the data so that the obscuration measured solely represents the concentration of the sample. For the tests run in this study a goal obscuration of 10% was used with actual obscurations ranging from 9.2% to 11.1%. After a sufficient amount of sample is added, it is subjected to 30 seconds of ultrasonics with ultrasonic displacement equal to 10 µm (highest level) to further disperse the sample. Note: the higher the ultrasonic displacement value, the more powerful the ultrasonic action. This step should not only further disperse the sample but also prevent re-agglomeration. Finally, the test is started. Five trials are run and combined to obtain an average PSD for the sample.

2.3.3 Results

Figure 2-1 (a) presents a histogram of the results of the LAS testing shows a narrow range of common particle sizes as well as an additional range of smaller, less common particle sizes. Blast furnace slag is more difficult to grind than Portland cement clinker; however, it is known to react slower, and therefore a finer grind is desirable (Heaton et. al, 2012). Thus, the small "bump" in the lower particle size ranges may be due to the presence of blast furnace slag in the cement. An additional note is that air bubbles in the system will show a peak at around 100 µm, and therefore a peak at this particle size should not be interpreted as a property of the material. If this particle size is of particular interest, further testing should be run with particular attention to removing air bubbles from the system. Figure 2-1 (b) shows the same results converted into the "percent-passing" form more conventionally used in geotechnical particle size analysis. The cement has d_{10}, d_{50} (mean) and d_{90} particle sizes of 0.95 µm, 4.48 µm and 9.46 µm, respectively. The mode is higher than the mean at 6.10 µm.

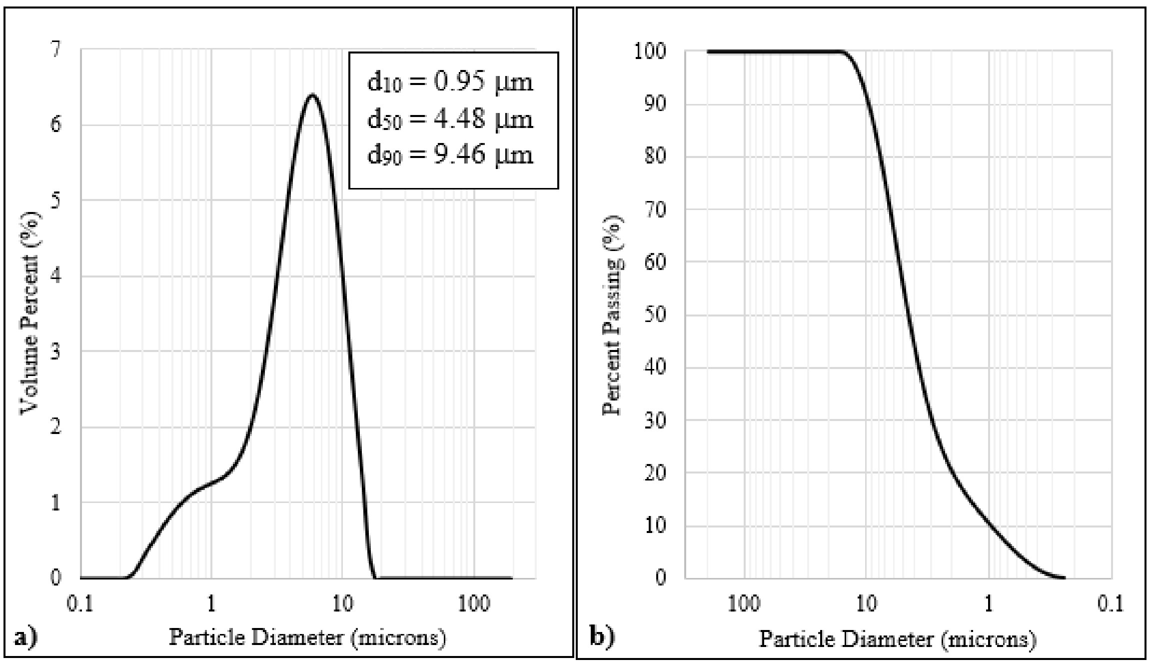

Figure 2-1: LAS test Results

2.4 CALORIMETRY

2.4.1 Isothermal Calorimetry

Isothermal, or constant temperature, calorimetry is a test method useful for investigating cement hydration kinetics as well as comparing the hydration of different cements, SCMs and chemical admixtures. Understanding the hydration kinetics of a cement is important for determining how long the grout will remain workable, the rate at which its viscosity will increase, when it will behave plastically (initial set) and when it will behave rigidly (final set). Calorimetry tests can also be used to obtain the cumulative heat of hydration (the area under the rate of heat evolution curve), which in turn can be used to estimate the degree of hydration and can be correlated to the compressive strength of the cement paste. Though the strength of the cement paste may not be directly useful in grouting applications, it can be used to compare different grouts and estimate in-situ strength increases.

2.4.2 Cement Hydration Process

The hydration process of ordinary Portland cement has been thoroughly studied and well documented in literature. The process is often split into five phases, plus a "phase zero". Though description of the entire hydration process is not necessary for the purpose of this study, a few key elements are necessary for understanding the behavior of the cement blend used in this study. Figure 2-2 shows a typical hydration curve and Figure 2-3 shows a typical heat evolution curve for ordinary Portland cement.

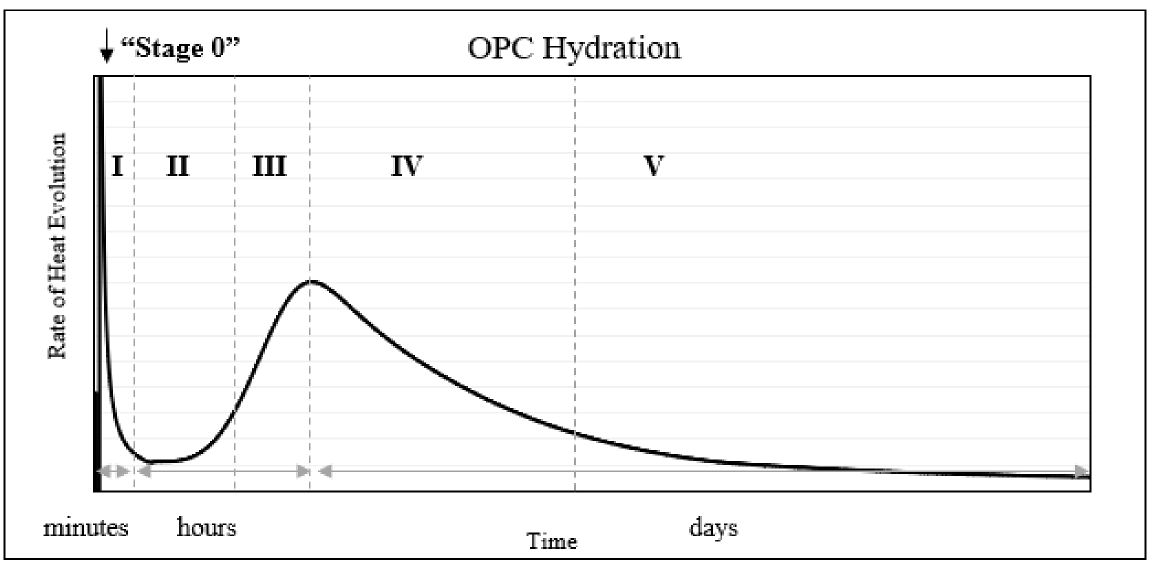

Figure 2-2: Typical Ordinary Portland Cement Hydration Curve

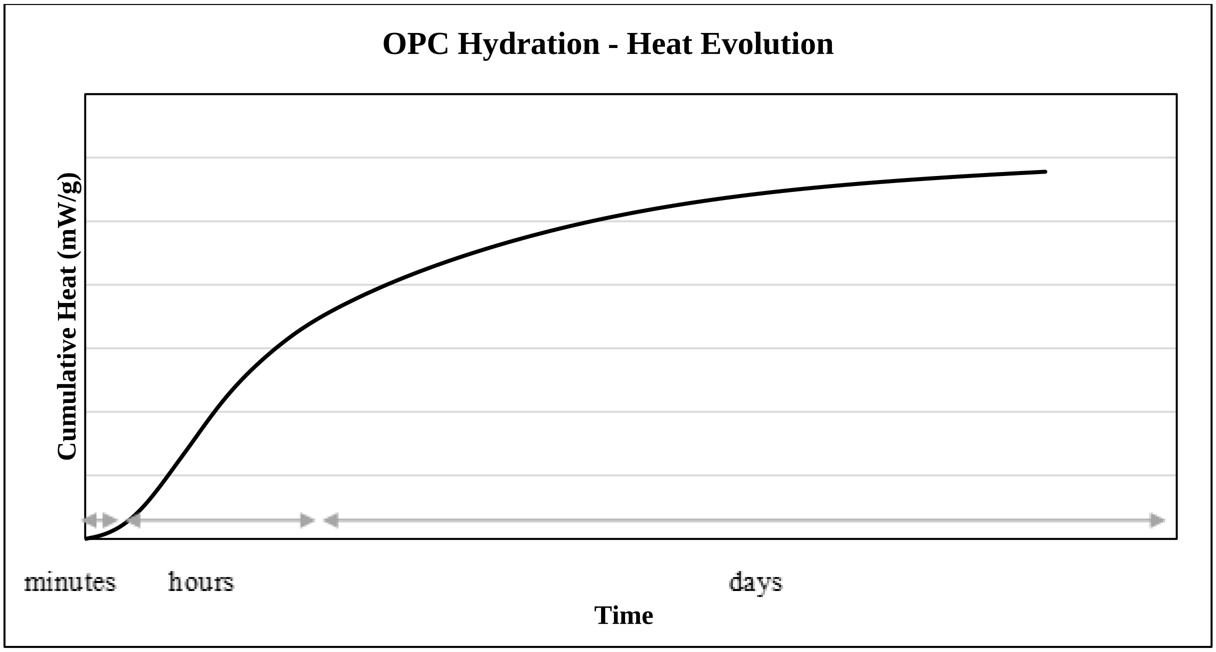

Figure 2-3: Cumulative Heat Evolution for ordinary Portland cement (OPC)

"Stage 0" describes the initial, rapid changes that occur as soon as the dry cement comes into contact with water. In this stage, heat is rapidly released. This heat comes mainly from the dissolution aluminate phases and of free lime. Calcium and aluminate ions go into solution and ettringite forms and precipitates within seconds. The precipitation of ettringite is what causes the mixture to thicken and become "sticky". This stage is very rapid and hard to accurately capture using isothermal calorimetry and therefore is rarely shown. "Stages I and II" correspond to a low rate of heat evolution. In these stages calcium hydroxide ions become super saturated and slow formation of calcium silicate hydrate (CSH) and ettringite leads to continuous increase in viscosity and decrease in workability. The early formation of ettringite and monosulfate is what causes the cement to appear "thixotropic" at this time because mixing the cement will break the temporary structure created by these hydrates. However, cement is not a thixotropic material because the structure is due to irreversible chemical changes in the cement. Though ettringite and monosulfate increase the viscosity of the mixture, the formation of CSH is what leads to the onset of normal initial set at the end of Stage II. Note that Stage I may or may not be shown in isothermal calorimetry results because this stage may also be affected by initial calibrations in the temperature control module. “Stage III” describes where the formation of CSH and CH accelerates and reaches a maximum. In this stage, rapid formation of hydration products leads to solidification and decrease in porosity. The mix changes from plastic to rigid consistency as it reaches final set at the end of Stage III. “Stages IV and V” describe the deceleration of the rate of formation of CSH and CH. In this stage, hydroxyl ions (OH-) increase and cause the pH of the mix to decrease. Decreases in porosity and increases in strength continue with time. Depending on the cement blend used, full hydration may occur in years, or the components may never fully hydrate.

2.4.3 Blast Furnace Slag Hydration

The reaction of blast furnace slag relies on the release of hydroxyl ions (OH-) by calcium hydroxide formation and/or alkali-activation, which mainly occurs in Stage IV of the Portland cement hydration process. This is because blast furnace slag needs the aqueous phase environment in the mixture to reach a pH above approximately 11.5 to 12 in order to react. Once this pH is reached, blast furnace slag reacts with calcium hydroxide, a hydration product mainly formed in Stages III and IV of the cement hydration process. Because it reacts with a hydration product and requires a pH that is achieved during later stages in the clinker hydration process, the hydration of BFS is delayed. Depending on the properties of the slag, this delayed peak has been reported to either be separate or attached to the C_3S hydration peak.

2.4.4 Procedure

Calorimetry testing was carried out using a TAM AIR isothermal calorimeter (TA Instruments) at a temperature of 293 K (20°C). A base case test and two variations were carried out as a part of this study. The base case consisted of a neat grout mix with water-to-binder ratio (hereafter referred to water-to-cement ratio, w/c) of 1.5:1. The three variations are as follows: (1) Lowering the w/c ratio to 0.45:1 and (2) Adding 2.0% superplasticizer (SP) by weight of cement. The ampules in the TAM AIR isothermal calorimeter require 15g of material; however, larger amounts were used to prepare the grout mixes to reduce errors in proportioning the components. Due to the relatively small amounts of grout used and the desire to capture the hydration effects as early as possible, a mix time of 2 minutes in a high shear mixer was deemed sufficient. If superplasticizer was used, it was measured out and mixed into the water first, and then the cement was added. In the field, SP is added after the cement and water are mixed; however, due to the difficulty of delivering precise dosages without pre-weighing the SP, this is impractical in the lab. After the grout is mixed, it is placed in the calorimeter and measurements are taken for 75 hours.

2.4.5 Results

Figure 2-4 presents the results of the base-case test (w/c=1.5, no additives). It can be seen that the both the ultrafine grind of the cement clinker and the inclusion of blast furnace slag significantly affect the hydration of the grout, which causes the hydration curve to appear much different than that of OPC-only. Though the finer grind would be expected to increase the overall heat and the speed of the reaction, the addition of the BFS actually causes a reduction in the overall heat released and a delay in the reaction. As previously mentioned, slag is a latently hydraulic material. Its reactivity is significantly influenced by the pH of the aqueous phase as well as the formation of calcium hydroxide

(CH), which acts as an activator (Luan et. al, 2012). This causes the appearance of a third peak, which, for this cement blend, appears noticeably later than the second peak[1]. Additionally, it is important to note that the results of isothermal calorimetry tests are temperature-dependent. Higher curing temperatures result in increased rate of heat evolution and earlier set times. It has been observed that at high temperatures the third hump disappears, which indicates the hydration of the BFS is happening almost simultaneously with the OPC (Han et. al, 2015). However, in permeation grouting applications, the temperatures are not likely to get high enough to cause these peaks to combine due to the relatively constant temperature of the ground. The effects of high temperatures are much more relevant in concrete applications. In fact, further investigation of lower temperatures, such as would be expected underground, might be useful for understanding in-situ behavior of this material. Finally, whereas the hydration curve for OPC seems to return to very low rates of heat evolution after days of running the test, the OPC-BFS blend retains a higher rate of heat evolution after the same time period. This is also due to the dependence of the BFS on the reaction products of the OPC clinker. Therefore, though cement mixed with slag gains strength slower, it continues gaining strength longer than OPC. However, unlike OPC, which given sufficient water and long-time curing will reach near 100% hydration, blast furnace slag blended cement has been recorded to be far from fully hydrated even if given sufficient water and long-time curing. Furthermore, the slag ratio influences hydration significantly, with high slag ratios leading to decreased hydration overall (Luan et. al, 2012).

[1] This peak is presumed to be the slag hydration peak. However, this peak may be caused by other factors. It is possible that the addition of slag changes the gypsum balance of the system and that this is a gypsum depletion peak.

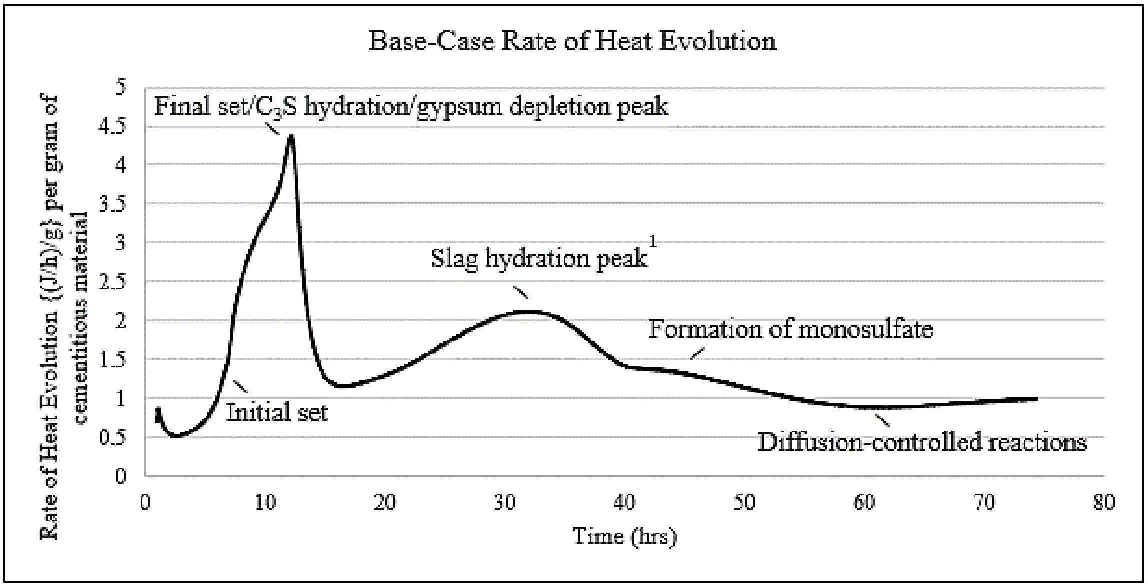

Figure 2-4: Heat of hydration for 1.5:1 water-to-cement ratio grout without additives

Figure 2-5 shows the effect of the variations made to the base case on the rate of heat evolution; Table 2-1 lists the assumed initial and final set times of the variations based on the calorimetry data. Note that the assumed initial set time is defined as the time where the slope of the rate of heat evolution is greatest and the assumed final set time is defined as the peak of the C_3S depletion peak. Reducing the water-to-cement ratio from 1.5, which is more typical for a grout, to 0.45, which is more typical of a concrete and therefore more widely reported in the literature, significantly affects the hydration of the mix. Though previous studies have demonstrated that reducing the w/c of the mix increases heat flow and decreases hydration time (Lura et. al, 2015), these studies mainly focus on small ranges of w/c that are relevant for concrete applications (0.35-0.50). Thus, the effect of decreasing w/c is visible, but not significant. The significant increase in heat and decrease in hydration time for the w/c = 0.45 mix versus the w/c = 1.5 mix observed in this study reinforces the fact that trends observed in low w/c concrete mixes should not be extrapolated to higher w/c grout mixes. Additionally, decreasing the w/c results in a rate of heat evolution that appears to be continuously decreasing after days of testing. Thus, this lower w/c mix might be expected to have more short term and less long-term strength gain.

The effect altering the base case by adding 2.0% SP by weight of cement is significant. The reaction is extremely delayed, and the heat is significantly reduced. A delayed reaction and reduced heat are expected because SP is a retarder. The extremely low rate of heat evolution during Stage II of hydration indicates little increase in viscosity due to hydration product precipitation. This is expected because SP disperses the particles and reduces buildup of structure. However, the amount of retardation and heat reduction seen in this sample appears quite extreme. One possible explanation is that the TAM AIR isothermal calorimeter is made for lower w/c ratio (concrete) mixes. These mixes tend to have little to no bleed and more uniform cement concentrations throughout the height of the ampule. Higher w/c ratios, on the other hand, tend to have higher bleed and more non-uniformity in cement content with height as SP is added, as will be discussed later in this study. Therefore, the TAM AIR may not be accurately capturing all the changes occurring in the high w/c and high SP sample.

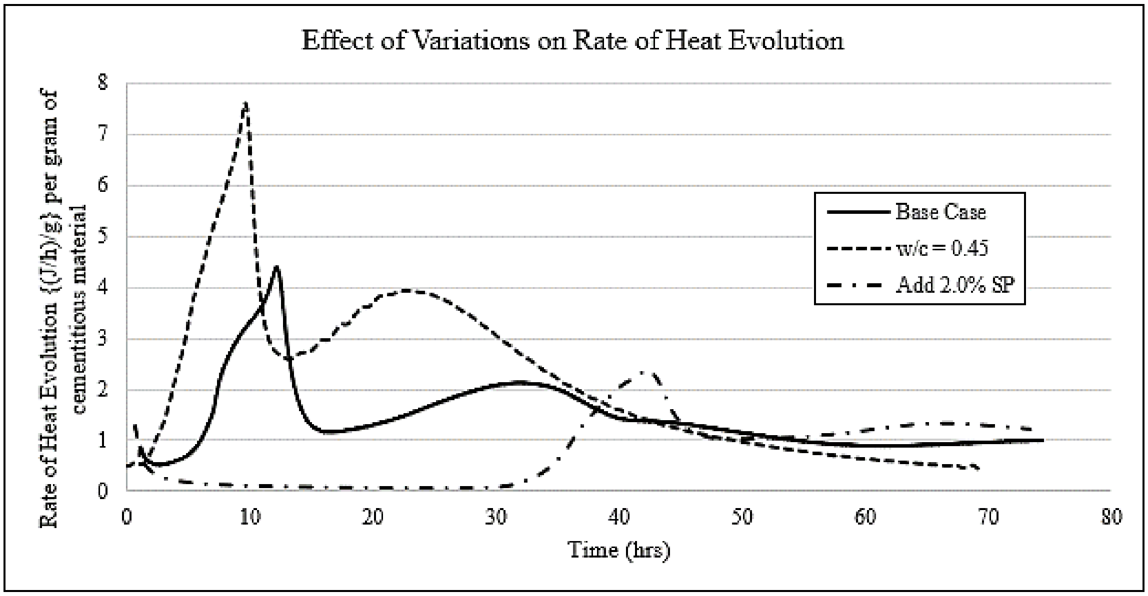

Figure 2-5: Effect of varying water to cement ratio (w/c) and including superplasticizer (SP) on the rate of heat evolution in cement grout

Table 2-1: Effect of varying water to cement ratio (w/c) and including superplasticizer (SP) on the assumed initial and final set times of cement grout based on calorimetry data

	Assumed Initial Set Time (hr)	**Assumed Final Set Time (hr)**
Base Case	7.5	12
Lower w/c	3.0	9.5
with SP	37	42.5

Figure 2-6 shows the cumulative heat evolution curves for the mixes tested. The low w/c mix shows high initial strength gain and decreasing strength gain with time. The tests with the higher w/c show significant slopes even after 75 hours, which indicates that the strength is still significantly increasing. All three curves show a period of low strength gain (lower slope) caused by the dip between the second and third hydration peaks. As shown in Figure 2-3, this does not occur in OPC. This “plateau” may be useful in grouting for tunneling applications where it is desirable for the grout to set, but not necessarily reach a high strength before the TBM progresses through the treated zone (Ivanova et. al, 2016).

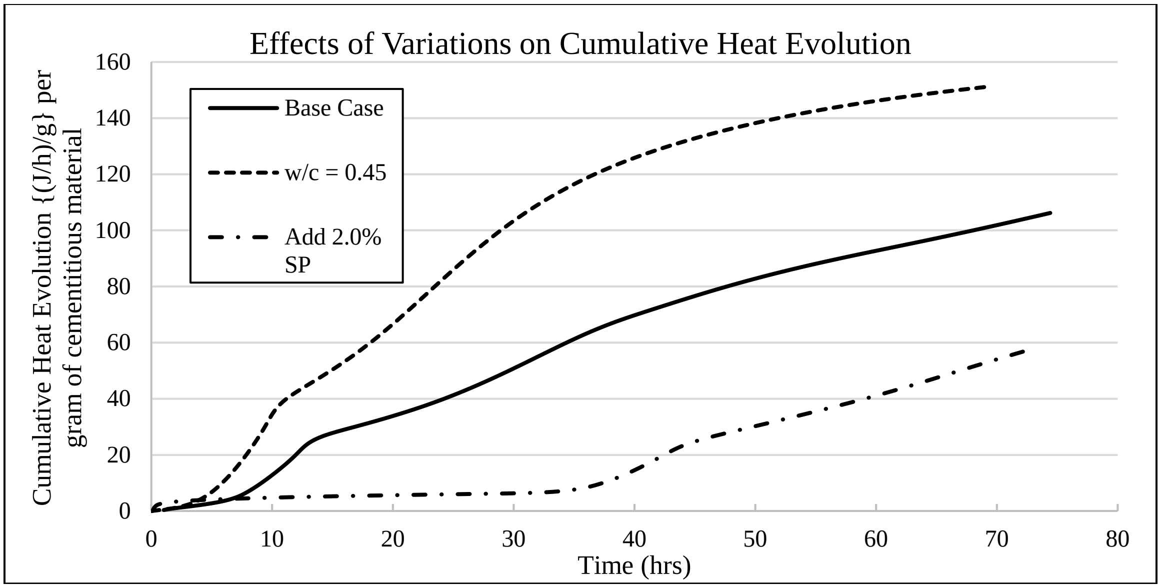

Figure 2-6: Effect of varying water to cement ratio (w/c) and including superplasticizer (SP) on the cumulative heat evolution of cement grout.

Chapter 3: Mechanical Characterization of the Grouts: Rheology and Internal Stability

3.1 INTRODUCTION

Two of the most important properties of a cement grout are its rheology and internal stability. The rheology of the grouts have long been used to determine the groutability and grout penetration depth of grouts into soils and rock fractures (Gustafson and Stille, 1996; Santagata and Santagata, 2003; Axelsson et al., 2009; Yoon and El Mohtar, 2014; El Mohtar et al., 2013). Due to the many problems associated with its use, an unstable grout is undesirable in most cement grouting operations (Rosquoët et al. 2003, Naudts et al. 2004, Tan et al. 2004, Bremen 1997). This chapter discusses rheology and internal stability measurements performed on the ultrafine cement used in this study.

3.2 RHEOLOGY

The rheology of a grout significantly affects how the grout will perform in permeation grouting applications. Rheology describes the plastic flow characteristics of a material. In general, cementitious grouts behave as a non-Newtonian, shear thinning fluids that exhibit a yield stress, or stress at which the material begins to plastically deform. However, this value is small and often difficult to quantify at high w/c ratios. A grout mix can be described by its viscosity, or resistance to flow due to internal friction. However, because grouts are shear-thinning fluids (i.e. its viscosity decreases with increased shearing), there is no single value that can be used to fully describe the behavior of a grout.

In the field, an index test called the Marsh Cone test is used to characterize grout viscosity for quality control purposes. This test is mobile, simple to use and gives an index measurement related to viscosity. The index value is achieved by measuring the time required for a specified volume of grout (usually 1L or 1 quart) to flow through a specified cone. This test is limited to grouts with a yield stress low enough to allow full flow from the cone. Additionally, this test is limited because it only provides an index value and is susceptible to inaccuracies due to human error. For this study, a Physica MCR 301 rheometer (Anton Paar, Graz, Austria) was used to more accurately measure the rheology of the grout mixes (Figure 3-1).

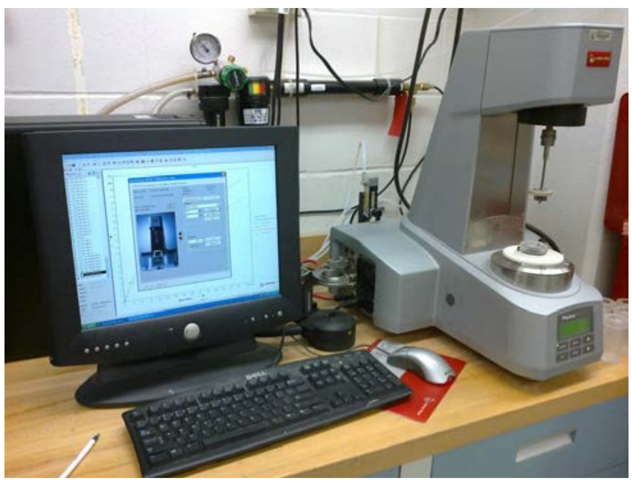

Figure 3-1: Rheometer with data acquisition

3.2.1 Procedure

First, the Physical MCR 301 rheometer is powered on and rheometer software is run on the desktop. The viscosity of a material is affected by temperature; therefore, an internal temperature control unit is used with the rheometer to maintain a constant temperature. For this study, all samples were measured at 22º C. After thermal equilibrium is reached, the rheometer is initialized. During initialization, the rheometer checks the gap sensor and position sensor. This step is less important for vane-and-cup than cone-and-plate geometries; however, in both cases it helps ensure consistency throughout the tests. For the viscosity testing done in this study, the vane and cup geometry is used (Figure 3-2). For this geometry, the rheometer rotates the six-bladed vane at a specified rate and measures the resistance, in the form of torque. The software automatically converts the recorded data into the requested properties, which in this case are viscosity and shear rate. Next, the desired test procedure is input into the rheometer software. As previously mentioned, viscosity is not a unique value for a non-Newtonian material, rather, it varies based on shear strain rate. Therefore, a shear strain rate -ramp type test is run. in which the rate is increased from 1/s to 1000/s over a time of 100 seconds. These parameters were chosen to obtain a reasonable resolution over a wide range of strains while keeping a short test time to avoid hydration effects.

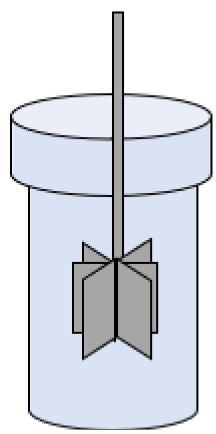

Figure 3-2: Vane and cup geometry for rheometer testing

Once the test schedule is set up, the grout is prepared. The mixes are prepared by measuring out the appropriate amounts of distilled, de-ionized water, and microfine cement to achieve the desired w/c ratio by weight. If superplasticizer (SP) is used, it is added to the water before the cement. As previously mentioned, this order was chosen in order to achieve exact percentages of SP without the use of dosing equipment. The water-SP mixture is then placed in a high shear mixer for 1 minute. Then, the cement is incrementally added over a period of 2 minutes. After all the cement is added, the grout is mixed for 10 minutes. Next, the cement is added to the rheometer cup up to a specified line drawn at an equal height on all cups. A uniform height is kept to remove possible error due to resistance along the stem of the vane. The vane is then inserted into the rheometer. The rheometer is then lowered all the way to its measuring height (in the middle third of the cup to avoid interference from the top or bottom surfaces) and the test is started. Table 3-1 lists the suite of tests chosen to investigate the effects of water-to-cement ratio (w/c) and addition of SP (% by weight of cement):

Table 3-1: Test suite for viscosity testing

w/c	**SP = 0%**	**0.5%**	**1.0%**	**1.5%**	**2.5%**
1.5	√	√	√	√	√
2	√			√	√
3	√	√		√	√
7	√			√	√
10	√			√	√

3.2.2 Results

Each test produced a plot of apparent viscosity versus shear rate. Figure 3-3 shows the plot for w/c = 3 and SP = 2.5%. Note that at a certain point the apparent viscosity stops decreasing and begins to increase. Theoretically, the apparent viscosity should not increase with increasing shear strain rates. This increase is likely due to inertia effects in the rheometer (rather that initiation of hydration); similar trends has been observed in previous testing of bentonite suspensions in which no hydration occurs (Yoon, 2011). Because the "minimum" viscosities occur at reasonable shear rates for what may be expected in grouting applications in the field, the minimum of each plot was selected and designated the “apparent viscosity” of the grout.

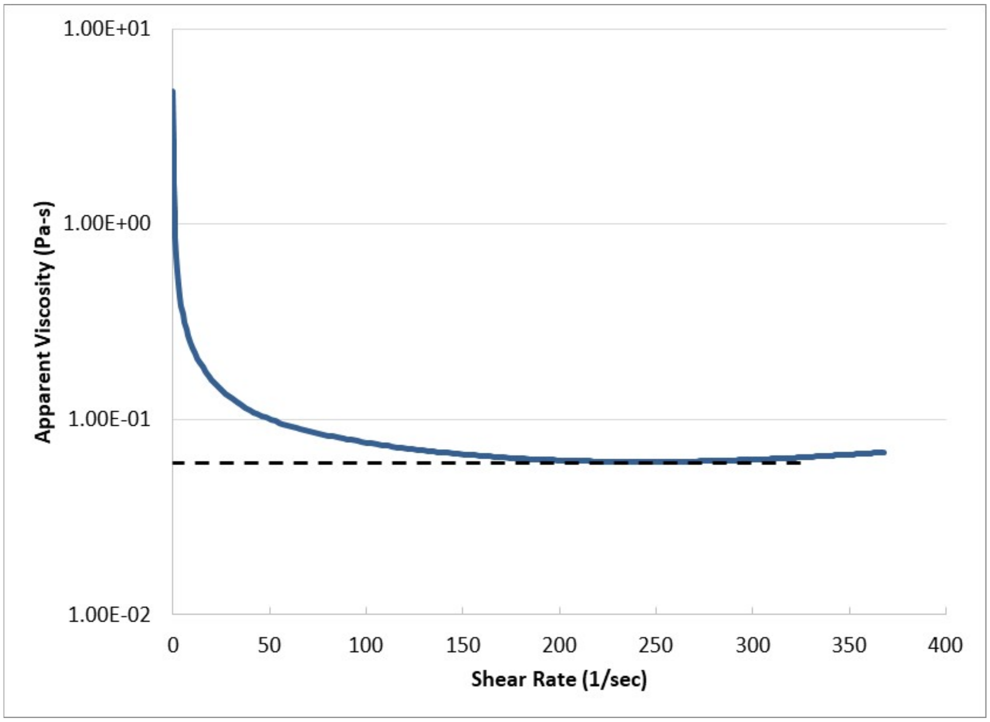

Figure 3-3: Change of apparent viscosity with shear rate

The apparent viscosity values from Figure 3-3 above are plotted in Figure 3-4 and Figure 3-5 below. Figure 3-4 shows the effect of varying the w/c ratio at different levels of added SP. Figure 3-5 shows the effect of varying the percent of SP added at different w/c. Both plots demonstrate that the changes in viscosity for low w/c are much greater than for higher w/c. Additionally, they show that the addition of SP makes a much greater difference at low w/c than high w/c. Figure 3-5 shows that for w/c = 7, adding SP makes almost no difference in the viscosity of the grout. Furthermore, the plots show that the reduction of viscosity associated with adding SP decreases for increasing amounts of SP.

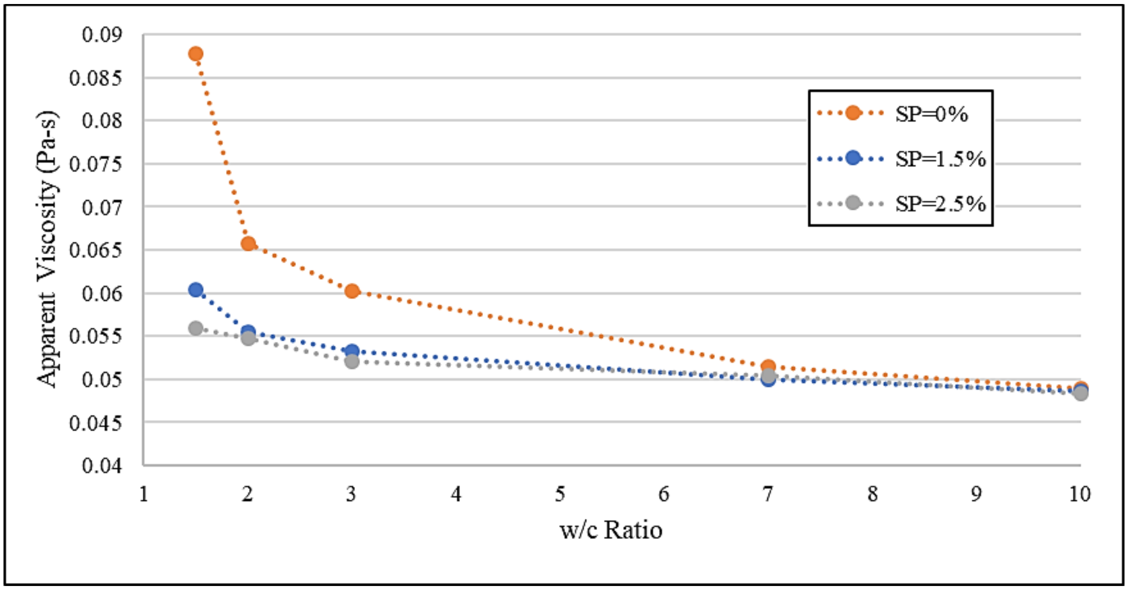

Figure 3-4: Effect of water-to-cement ratio (w/c) on apparent viscosity

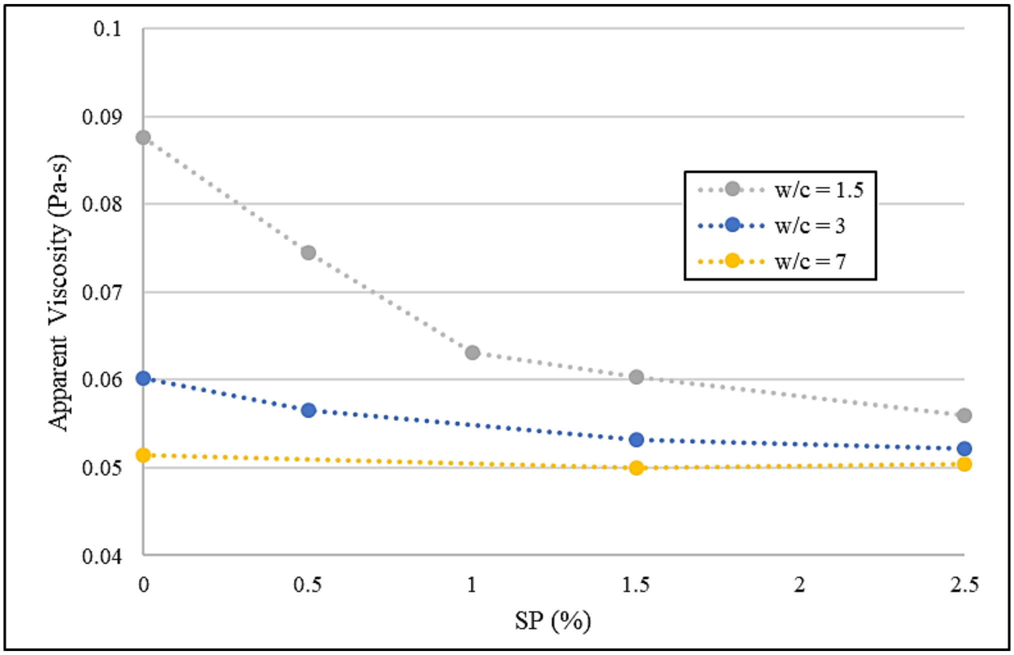

Figure 3-5: Effect of superplasticizer (SP) on apparent viscosity

3.3 STABILITY

In a stable grout, the individual particles are interacting with each other and therefore do not separate. In an unstable grout, cement particles may settle out of suspension or be filtered out of the grout by the formation being permeated. If the cement particles settle out of suspension before setting, the final grouted zone will have non-uniform cement content distribution, with some areas having much lower (away from injection point) or higher (near injection point) cement contents than designed. If the cement particles are filtered out as they pass through the formation, not only will cement contents be uneven, but the buildup of particles near the injection point will cause a significant increase in pressure. This could cause hydrojacking or fractures in the soil/rock mass. Due to the many problems associated with its use, an unstable grout is undesirable in most cement grouting operations (Rosquoët et al. 2003, Naudts et al. 2004, Tan et al. 2004, Bremen 1997). Therefore, it is important to understand how different factors effect stability when designing a grout mix. Current commonly used methods of describing the stability of a grout include the static column bleed test and the API (American Petroleum Institute) filter press test. The API filter press is typically more applicable for lower w/c, lower bleed grouts. Many of the higher w/c grouts used in permeation grouting applications go through the API filter press test too quickly for reliable measurements. Therefore, the stability of the microfine cement grouts used in this study was measured using static column bleed tests.

3.3.1 Standard Column Bleed Tests

Bleeding can be defined as the escape of pore water from the grout suspension. Bleeding is caused by sedimentation and consolidation and is influenced by water-to-cement ratio, cement particle size distribution, hydration and flocculation (Draganovic, 2009). Bleeding, or the escape of pore water from the grout suspension, can be caused by four mechanisms: sedimentation, consolidation, hydration and flocculation (Draganovic, 2009).

Sedimentation is a fairly well-understood process in which particles in a fluid are pulled downwards by gravity. In most applications, Stokes Law is used to calculate sedimentation velocity. However, Stokes Law assumes that only gravity influences the sedimentation of particles, and this is certainly not true of cement. In fact, the behavior of colloidal particles, such as cement, is controlled more by surface effects than gravity (Hakan and Draganovic, 2012). Stokes Law requires knowledge of the size of the settling particles; however, cement particles are often flocculated. Therefore, the size and amount of flocculated particles would need to be known, which is not trivial. Flocculation occurs when attractive forces between particles overcome repulsive forces and cause individual particles to form clumps of particles. Because flocculation is caused by surface forces, cements with higher specific surface area (finer cements) are more flocculated. Flocculation not only complicates the process of identifying particle sizes, but also affects the nature and degree of sedimentation itself. If the particles are flocculated, particles of varying sizes may be clumped together, and therefore will settle together. This will result in a relatively even particle size distribution throughout the height of the settled grout. On the other hand, if the particles are dispersed, larger particles will settle faster than smaller particles and the settled grout will exhibit a particle size gradient throughout its height (Draganovic, 2009). Furthermore, when flocs form, water is trapped inside the voids in the flocs. Therefore, less water is able to escape, which leads to less bleed and a larger volume of sediment than in a dispersed grout. Therefore, due to the strong influence of surface effects, the sedimentation of grout becomes quite complex.

The sedimentation process is complete when all of the particles in the system are in contact. At this point, the particles in contact exert vertical stresses on one another and the grout begins to consolidate. Consolidation is when the water between the previously sedimented particles is forced to the surface of the grout due to increasing overburden stress from newly sedimented particles. The consolidation process continues until the forces acting on the particles are in equilibrium. The amount of bleeding caused by consolidation is dependent on the amount of overburden stress as well as the compressibility of the grout. Flocculated grouts retain water within the flocs and therefore are less compressible than dispersed grouts. Thus, flocculated grouts exhibit less bleeding caused by both sedimentation and consolidation (Hakan and Draganovic, 2012).

Additionally, both the sedimentation and consolidation behavior of a cement grout are affected by the hydration of the cement. As discussed in the previous chapter, the formation of hydration products increases the viscosity and, eventually, the stiffness of the grout. The formation of hydration products also traps water, and thus increases porosity and decreases bleeding.

It is important to note that although a low-bleed, stable grout is desirable, some of the factors that increase stability also increase viscosity. Thus, both must be considered when designing a grout mix.

3.3.2 Results

Water to cement (w/c) ratios of 10:1, 7:1, 5:1 and 3:1 were used in this study. While some of these w/c ratios are too high for most practical applications and are expected to have significant bleed, they were particularly selected because of their distinct bleeding to highlight the difference in the measured stability with minimal impact due to viscosity (the apparent viscosities of the 3:1, 5:1, 7:1 and 10:1 grouts are 64, 53, 53 and 50 mPa.sec, respectively).

The stability of the different grout mixes was first evaluated by measuring the settlement of cement particles in a grout column with time. The method used in this study is a modified version of the method recommended by Widmann (1996). In his bleed test, one liter of grout is poured into a cylinder with a diameter of 60 mm (which gives a sample height of 353 mm), and the development of a clear water layer at the top of the grout column is monitored. Grout bleeding is defined as the height of clear water in percentage of the total grout column height after two hours (Widmann 1996). For this study, a 100 mL cylinder, as well as a test cup with the same dimensions as the rheometer test cup, was used for bleed test readings. The rate of settlement is affected by the different cylinder geometries; however, since this test is mainly used as a qualitative test to achieve a rough comparison between different grouts, this difference from Widmann's recommendations was deemed acceptable. At time zero, a freshly mixed suspension is placed in the 100 ml cylinder (or test cup). The height of the interface between the settling cement and the water is recorded over time until the interface height remains relatively constant. The rate at which the cement separates from the water (bleeding) and the final percentage of clear water at the top of the column give an indication of the grout's stability.

The results from the neat cement grout sedimentation tests are shown in Figure 3-6. The results show that the grouts with higher water-to-cement ratios settled much more and much faster than the grouts with lower water-to-cement ratios. The reduced rate of sedimentation for the lower water-to-cement ratios is likely due to more interactions between the more densely packed cement particles. The high water-to-cement ratio mixes, on the other hand, have less concentrated amounts of cement, which reduces settlement interference due to particle interactions, and less cement overall, which allows for more bleed. Therefore, as is confirmed in the experiment, less bleeding is expected from the 3:1

mix than the 10:1 mix. Additionally, Figure 3-6 indicates that the water-to-cement ratio 10:1, 7:1, and 5:1 grouts behave similarly to each other, while the 3:1 grout behaves quite differently. This observation will be discussed in more depth later in this study.

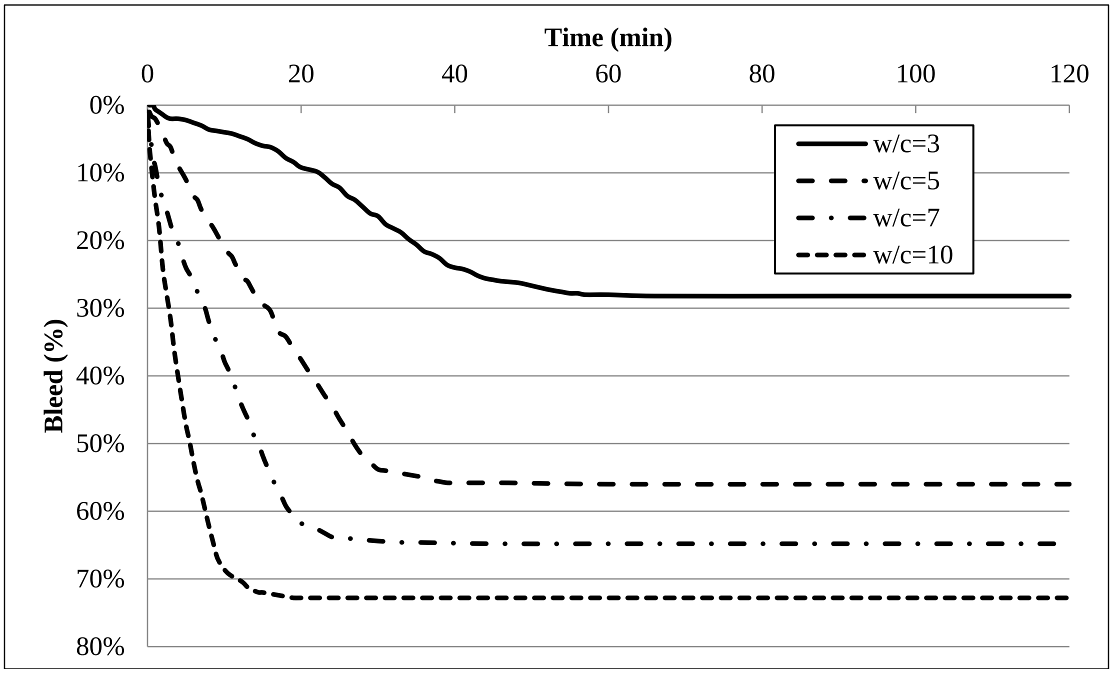

Figure 3-6: Static bleed of neat cement grout mixes

Next, the same w/c grout mixes were prepared with 2.5% superplasticizer (SP) by weight of cement. Superplasticizers are typically added to cement grout mixes to reduce the amount of water needed to achieve the same workability. Because superplasticizers work by dispersing the cement particles, they also effect the stability of a grout. Figure 3-7 shows the results from the 2.5% SP grout static column bleed tests. As before, the amount of bleed is determined by the amount of clear water at the top of the column. When defined in this manner, the 2.5% SP grouts exhibit very little bleed. However, a closer examination of the effect of superplasticizers on grout stability will be presented later in this study.

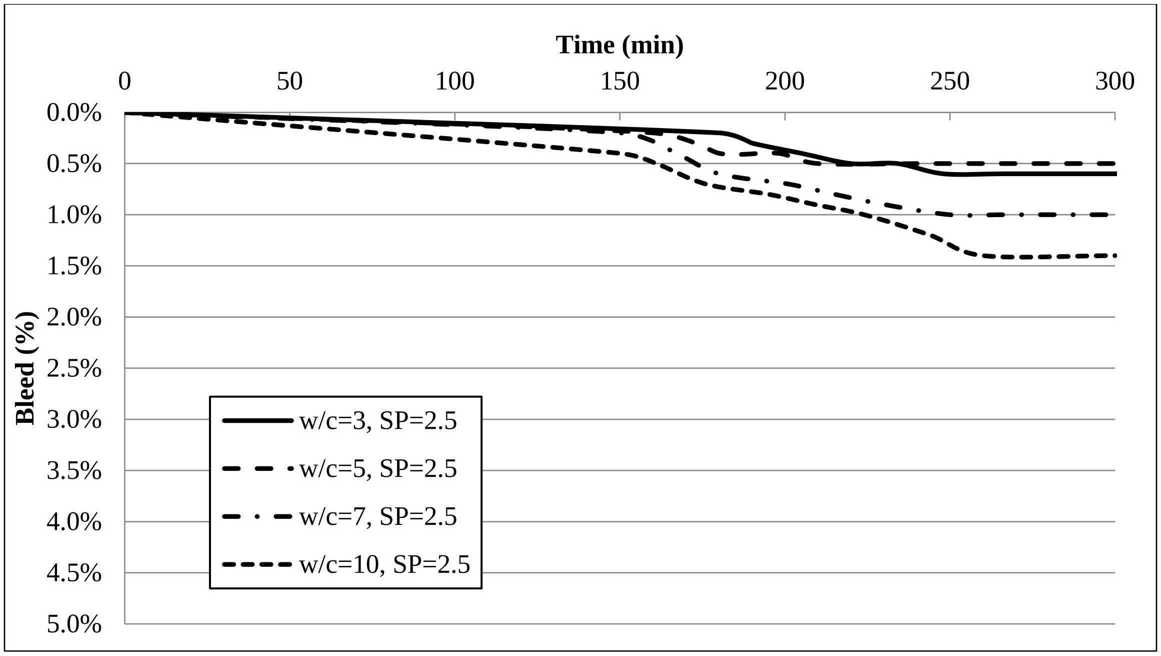

Figure 3-7: Static bleed of cement grout with 2.5% superplasticizer (SP)

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Chapter 4: Using Image Analysis to Improve Understanding of Grout Stability

4.1 INTRODUCTION

In current practice the stability of grout is determined by the amount of bleed, which is a measure of the amount of clear water that appears at the top of a grout column after a waiting time of two hours. This method is used to categorize grouts as either “unstable” or “stable”, based on a limiting bleed percentage. The previous chapter discussed how this is limiting because it does not account for the dynamic condition of grout in the field. An additional weakness of this test method is that it does not describe changes over time. Although these can be recorded, the process is time consuming and plots of change over time are not usually reported. Additionally, the traditional bleed test only reports changes observed at the top of the column and ignores changes in the grout throughout the rest of the column height. This chapter presents a new, more comprehensive method of analyzing changes in grout over the height of a stability column over time using image analysis techniques. The image analysis tests allow for the collection of significantly more data with little to no added effort. Manual pipette tests are used to verify the image analysis results.

4.2 MOTIVATION

Thus far, this study has discussed the importance of stability of a grout as a whole. The phases discussed have consisted of the bleed, which consists of pure water, and the grout, which has been assumed to be a homogeneous suspension. However, this often is a significant over-simplification, especially when additives such as superplasticizer are involved. A good visual example of this can be seen in the two grout columns shown in

Figure 4-1. The column on the right shows a w/c 3:1 grout with no additives. This column exhibits a clear water-grout interface and, as is reported in Chapter 2, this grout is very unstable with approximately 28% bleed. The column on the right is a w/c 10:1 grout with 2.5% SP. In Chapter 3, this is reported as a 1.4% bleed grout. The layer of clear water responsible for this 1.4% bleed designation cannot be easily seen in the picture. Instead, a height range where the grout transitions from cloudy white to grayish-tan can be seen. However, there is no discrete layer transition (more of a gradient) and it is not clear water, so according to the traditional definition of bleed, this cloudy layer is ignored. Nevertheless, any reasonable person who looks at the column could deduce that the grout in this cloudy layer has a much higher w/c than the original mix. Additionally, Figure 4-1 shows a very dark layer at the bottom of the column. Again, though the traditional definition of bleed does not recognize this layer, it would be presumed that this layer has a much lower w/c than the original mix. Furthermore, the presence of a higher w/c zone near the top of the column and a lower w/c ratio at the bottom of the column begs the question, how does the w/c vary throughout the rest of the height of the column?

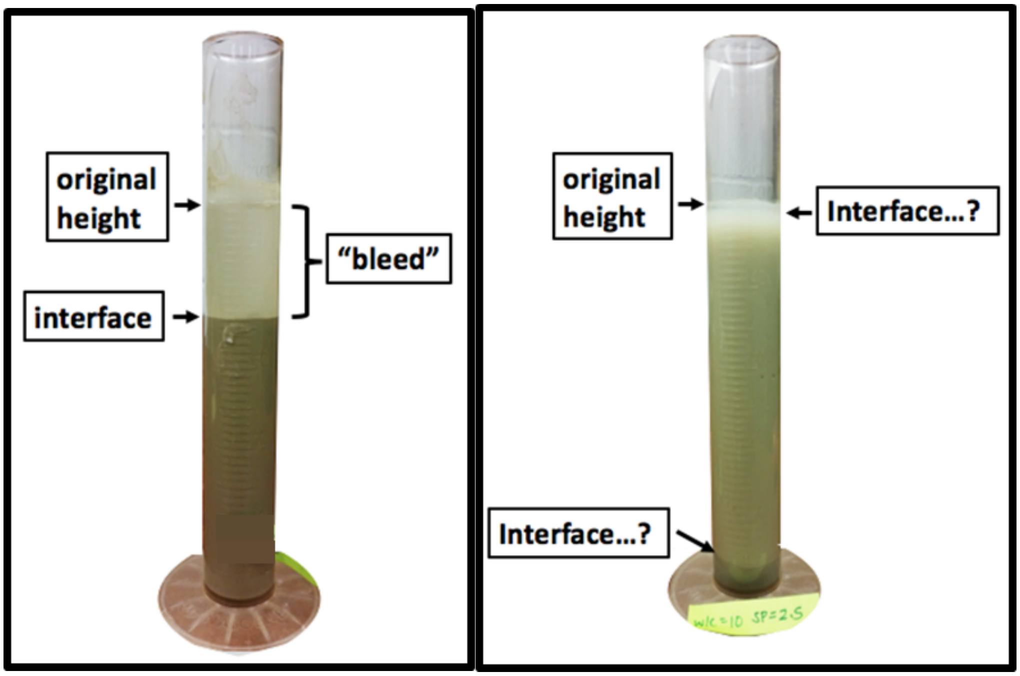

Figure 4-1: "Unstable" w/c 3:1 grout with no additives (left) versus "stable" w/c 10:1 grout with 2.5% SP added (right)

4.3 LITERATURE

The complex nature of changes in a grout column with height over time have been examined previously in the literature. Powers (1939) suggested that bleeding exhibited four unique zones: (1) a clear water zone at the top of the column, (2) a zone of constant water content and constant rate of settling, (3) a transition zone of variable water content and diminishing rate of settling, and (4) a zone of maximum consolidation without settling (Draganovic 2009). However, this study was conducted before the widespread use of microfine and ultrafine cements. Therefore, it neglects the effect of increased flocculation due to finer particles as well as the prolonged suspension of dispersed, ultrafine particles (i.e. the "cloudy" layer). More recent studies have used more advanced techniques to observe the settlement of cement grouts over time. Rosquoet et. al (2003) used gamma ray radiation to determine grout density over time throughout the height of a column. The testing in this study consisted of using a radioactive source (gamma ray) and a scintillation counter that can move vertically along the height of a 1400 mm tall test column. Densities are recorded every 50mm along the height of the column and the test completes one descending half cycle and one ascending half cycle every 8 minutes for 24 hours. Density curves are plotted from the results every 64 minutes up until approximately 7 hours of testing, when no further change in density is observed (Note that this time is consistent with the set time observed for the microfine cement tested in Chapter 2). From these tests, Rosquoet et. al (2003) observed increase in specific density in the lower part of the column and decrease in specific density at the top of the column. The density changes over time for a w/c 1:1 grout are shown in Figure 4-2.

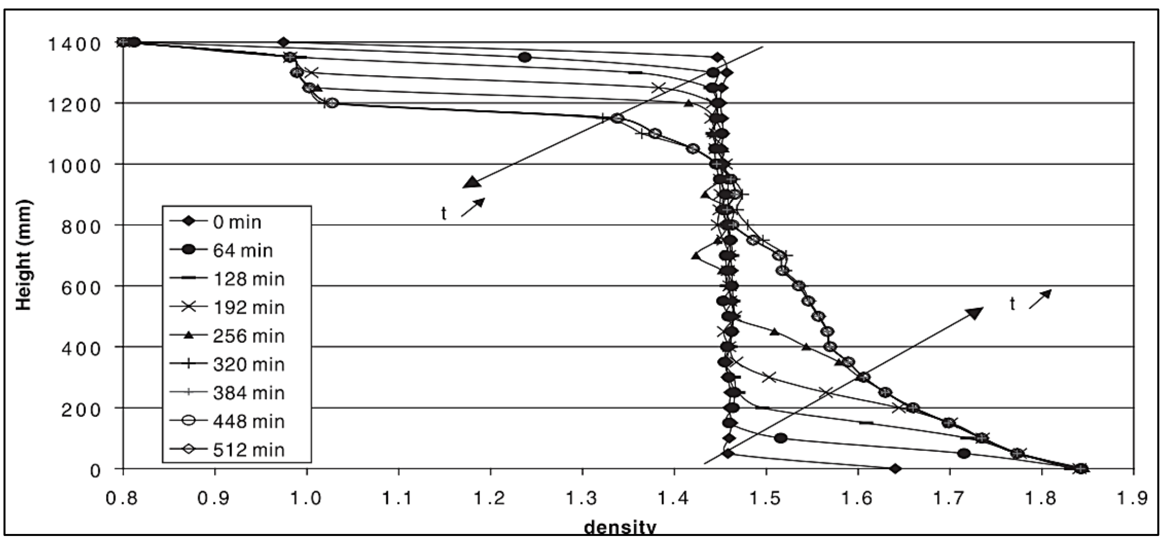

Figure 4-2: Density profiles for a cement paste with w/c 1:1 over a time period of 7 hours and 12 minutes

The tests run by Rosquoet et. al (2003) provide a much-needed advancement in the study of grout stability. However, they are limited in their practicality. First and foremost, these tests require expensive, specialized equipment. The density of the grout is measured from a radioactive source, which requires proper calibration of the gamma-densitometer bench to achieve density measurements for each given type of material. Additionally, the procedure only takes discrete point measurements every 50 mm, and even with this distance between data points, traversing the full height of the column takes 8 minutes. Thus, though this test is useful for research purposes, it has limited practical use. Additionally, though the w/c used by Rosquoet et. al (2003) are much lower than those of interest in this study, the results can serve as an additional verification of the results of this study.

4.4 MANUAL PIPETTE TEST

The manual testing is done using a pipette to take grout samples along the height of a 250 mL graduated cylinder. Each sample consists of 25 mL of grout, for a total of 10 samples along the height of the column. To more easily obtain exact quantities of grout, a pressure panel is used. A tube is connected to the pressure panel and samples are taken by applying a minimal vacuum (1). The samples are then placed in the tins by removing the vacuum and applying a low pressure (2). The amount of vacuum or pressure used was determined based on the viscosity of the grout. Lower viscosity grouts only required slight vacuum and little to no pressure, whereas higher viscosity grouts required higher vacuum and pressure to obtain the samples. Figure 4-3 shows the experimental setup.

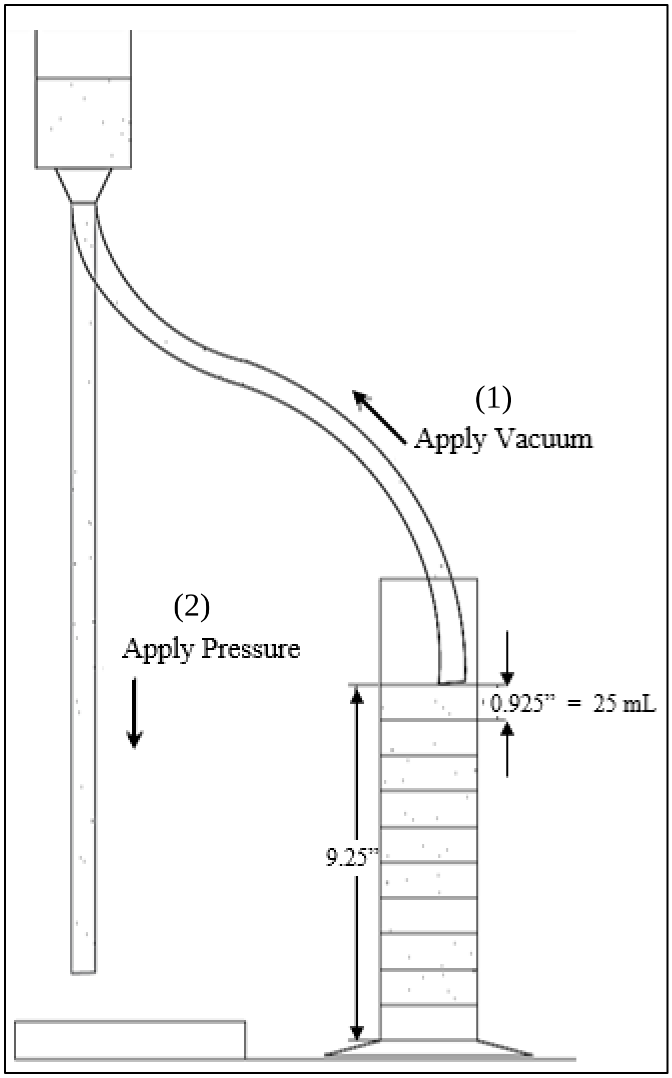

Figure 4-3: Experimental set up for manual test

4.4.1 Experimental Procedure

First, 550 to 800 mL of grout mix is prepared by measuring out the appropriate amounts of distilled, de-ionized water, and microfine cement to achieve the desired w/c ratio by weight. If superplasticizer (SP) is used, it is added to the water before the cement. As previously mentioned, this order was chosen in order to achieve exact percentages of SP without the use of dosing equipment. The water-SP mixture is then placed in a high shear mixer for 1 minute. Then, the cement is incrementally added over a period of 2 minutes. The mix is monitored to ensure the particles are well dispersed and there are no clumps. After the cement is added, the grout is mixed for 10 minutes. After 10 minutes the grout is removed from the shear mixer and immediately placed in two to three 250mL graduated cylinders, depending on how much grout was prepared.

Multiple cylinders are filled with the same mix because at each sample time, the entire 250 mL grout mix is used. By filling multiple cylinders with the same mix, the mix can be tested at multiple times (i.e. 30 min, 1h, 2h, etc.). After the grout is placed, a timer is set for the desired test time for each cylinder. Then 10 water content tins are weighed in preparation for each test. When the desired test time is reached, the grout is sampled from the column. The grout is sampled by placing the sampling tube at the grout-air interface and applying vacuum until 25 mL of grout is collected. During this step much care is taken to only sample from the very top of the grout. The tube is then placed above the water content tin and the vacuum is released. If pressure is needed, light pressure is applied to push the grout out of the sample tube. This is repeated until the last 25 mL of grout is left in the cylinder.

Depending on the sitting time and grout mixture, the grout at the bottom of the cylinder may be quite thick. Therefore, the remaining grout at the bottom is swirled/mixed until the grout can be poured out into a water content container. If the grout at the bottom is too thick to become pourable by mixing alone, 5 mL of water is added to aid mixing. The added weight is noted and subtracted from the total weight. The samples are then weighed and placed in the oven to dry overnight. After the dry samples are weighed, the w/c ratio is calculated at each sample height. The heights are recorded as the center of each sample. Tests were run for the test suite shown in Table 4-1.

$$W_c = W_{t,c} - W_t \quad \text{(Equation 2)}$$

$$W_w = W_{t,g} - W_{t,c} \quad \text{(Equation 3)}$$

$$w/c = W_w/W_c \quad \text{(Equation 4)}$$

where:

W_t = weight of tin

$W_{t,g}$ = weight of tin + grout

$W_{t,c}$ = weight of tin + cement

W_c = weight of cement

W_w = weight of water

w/c = water to cement ratio

Table 4-1: Manual Testing Program

w/c Ratio	Time (min)	SP (%)
1.5:1	0, 30, 60, 120	0, 1.0, 1.5, 2.0, 2.5
3:1	0, 30, 60, 120	0, 1.0, 1.5, 2.0, 2.5

4.5 IMAGE ANALYSIS TEST

In grout samples prepared with superplasticizer, rather observing a clear layer of bleed, a more gradual change in color is observed throughout the height of the column. This indicates that in cases in which SP is added the water-to-cement ratio is also changing more gradually throughout the column. Figure 4-4 shows results from manual testing alongside a photograph of the column for a w/c 3:1, 0% SP grout column and Figure 4-5 shows the results for a w/c 3:1, 2.5% SP grout column after 2 hours of settlement. Figure 4-4 shows an easily identifiable clear layer of bleed, but Figure 4-5 does not. However, in both cases, the changes in cement concentration determined from the manual test appear to track the changes in color along the height of the grout column.

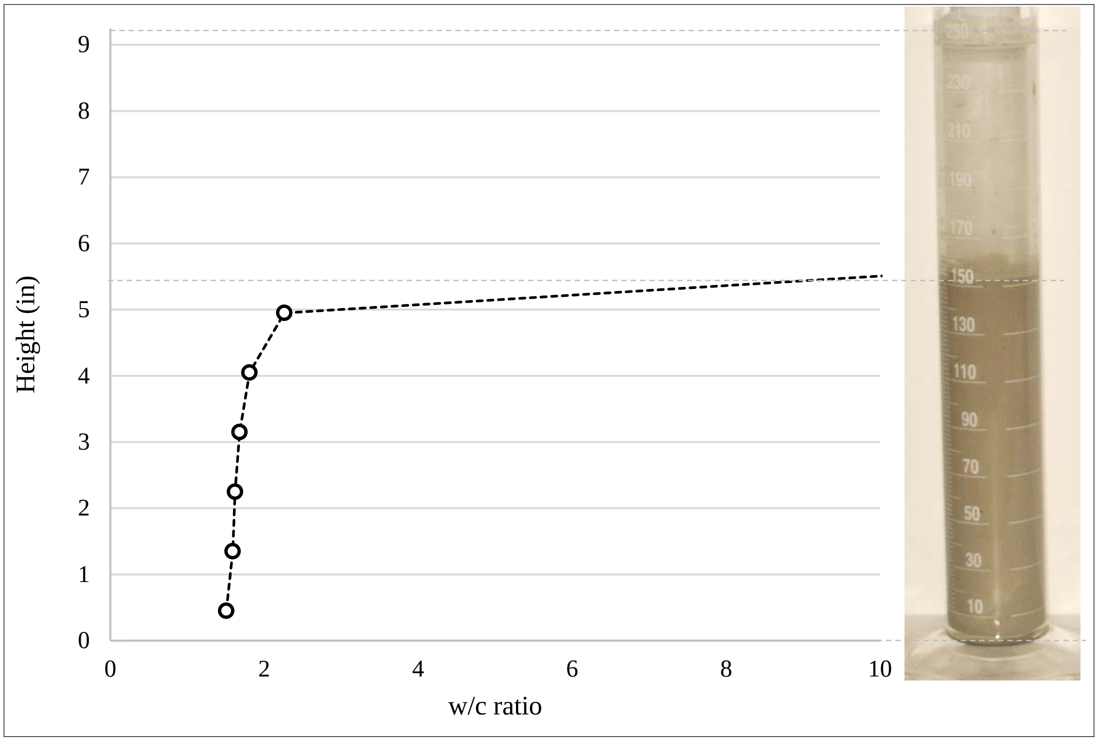

Figure 4-4: Manual test results and photo for a w/c 3:1, 0% SP grout

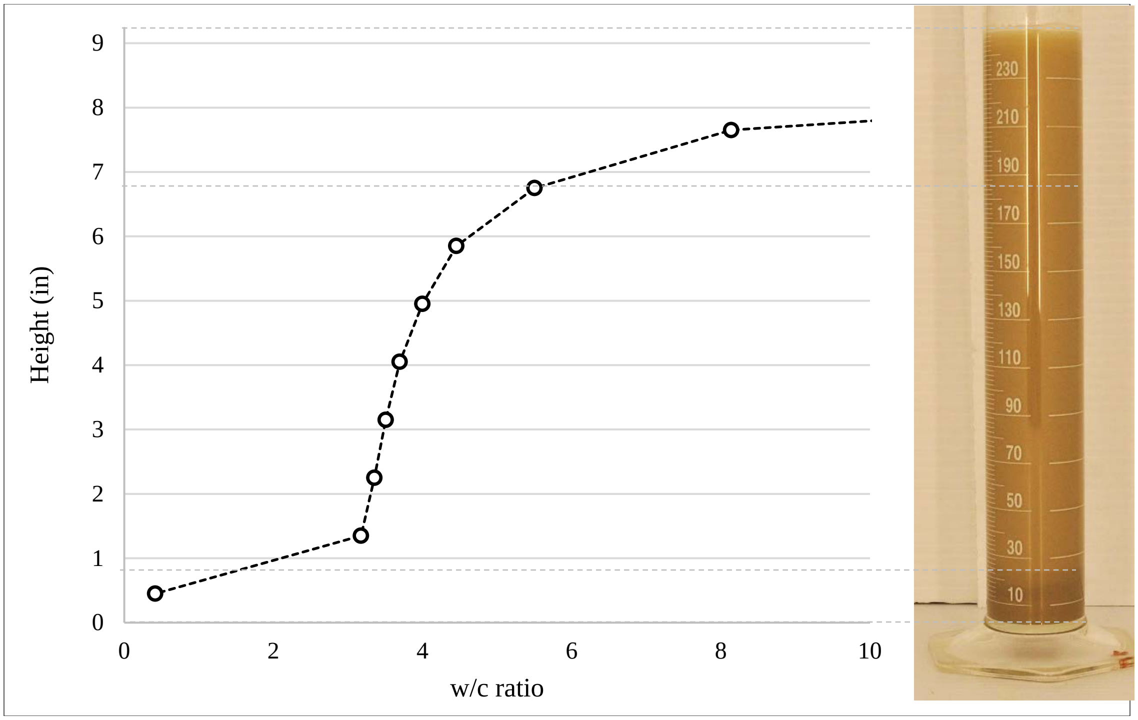

Figure 4-5: Manual test results and photo for a w/c 3:1, 2.5% SP grout

This section of the study first aims to explore the hypothesis that changes in grout color along the height of a sedimentation column over time indicate changes in water-to-cement ratio. Then, use this data to determine a correlation between grout color and w/c ratio and develop a procedure for quantifying this relationship. Ultimately, the goal would be to develop (1) an inexpensive low man-power way to accurately test grout stability (2) a software tool which can generate plots of changes in cement content over time using uploaded image files and (3) a new system for classifying grout stability based on more scientific parameters.

4.5.1 Experimental Setup

Image quality is important for obtaining accurate results using this testing method; therefore, before testing is begun, a controlled photo area must be set up. To limit imperfections/defects in the images due to shadows, poor lighting, etc. a few precautions should be taken. (1) A consistent camera setting should be used for all photographs. Avoid using "automatic" settings. Manually adjust camera settings and keep them constant throughout testing. (2) A constant camera position should be maintained throughout the test. For this experiment, boundaries were mounted on the floor to ensure consistent tripod placement. (3) A photo "area" should be constructed to minimize effects from outside surroundings. This experiment uses a 18" wide by 18" tall by 36" deep white photography chamber with two, 15" bar lights attached at mid-height of the sides at the chamber opening (Figure 4-6 and Figure 4-7).

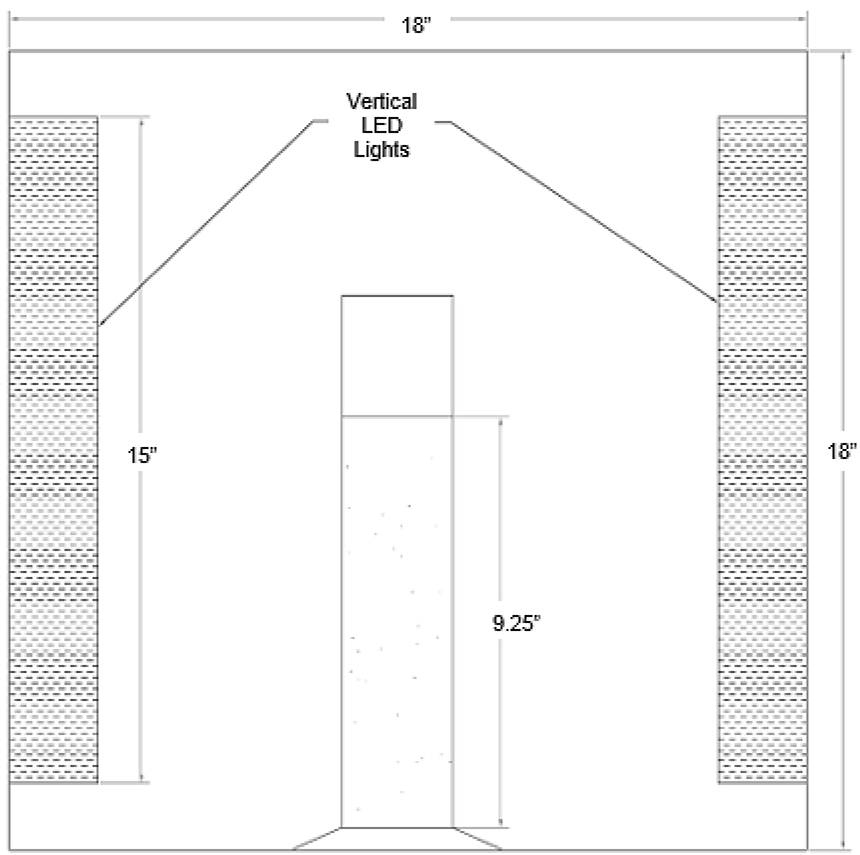

Figure 4-6: Image analysis test set-up, front view

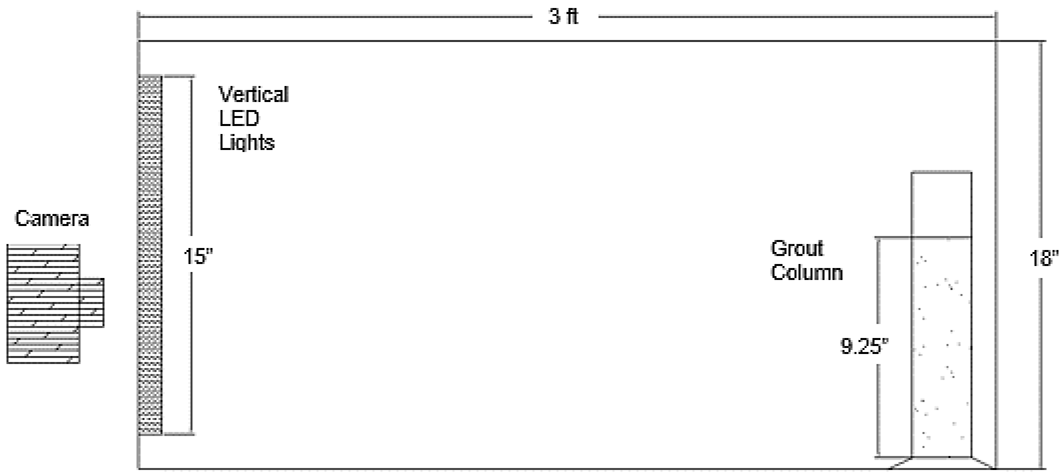

Figure 4-7: Image analysis test set-up, side view

4.5.2 Testing Procedure

To begin the test, a grout is mixed according to the same procedure as the manual test. First, approximately 300 mL of grout mix is prepared by measuring out the appropriate amounts of distilled, de-ionized water, and microfine cement to achieve the desired w/c ratio by weight. If superplasticizer (SP) is used, it is added to the water before the cement. As previously mentioned, this order was chosen to achieve exact percentages of SP without the use of dosing equipment. The water-SP mixture is then placed in a high shear mixer for 1 minute. Then, the cement is incrementally added over a period of 2 minutes. The mix is monitored to ensure the particles are well dispersed and there are no clumps. After the cement is added, the grout is mixed for 10 minutes. After 10 minutes the grout is removed from the shear mixer and immediately placed in a clear glass 250mL graduated cylinder. The cylinder is placed at the back of the photo chamber. Pictures are taken, either manually or using a programmed interval schedule, until desired end time. If desired, the photography chamber may be set up to allow multiple tests to be run at the same time.

4.5.3 Initial Image Processing

After the test period ends, upload the photos into image-processing software. The free software, imagej, was used for this study. Once uploaded into the software, the images should be converted to the image type “Lab stack”. This converts the image from RGB space (8 bits per pixel) to Lab space (32 bits per pixel), which helps in the subsequent analysis. Next, draw a line down the center of the cylinder (avoiding any major glares and graduation lines on the cylinder) using the line tool in imagej (or other comparable software). Then, using the analysis tab, select “plot profile”. This plot shows the color intensity over the height of the column. The color intensity scale goes from 0 (black) to 250 (white). The results from a w/c 3:1, 2.5% SP test are shown in Figure 4-8.

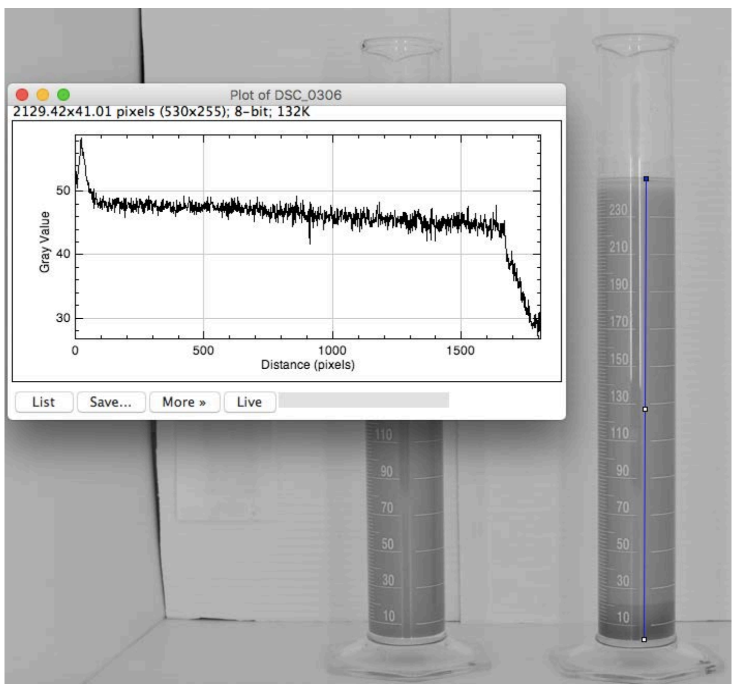

Figure 4-8: Obtaining color intensity from image in imagej

The color intensities recorded in the image are then converted to normalized w/c. Due to the location of the lights, the center height of the column is biased towards slightly higher intensities. This can be seen in Figure 4-9, which shows a w/c 3:1, 2.5% SP grout column at time zero. The intensity should be uniform throughout the height of the column, but the center shows higher intensity than the top and bottom. To remove this, apply the following filter (Equation 14). Note that with future advancements to the setup to eliminate the local impact of light sources, this step can be bypassed.

$$Y_{cor,i} = Y_i + [0.2(|h_i - 0.5h_{tot}|)^2 - 0.4(|h_i - 0.5h_{tot}|) - 0.5] + 0.1\left({}^{h_i}/_{h_{tot}}\right)$$

(Equation 14)

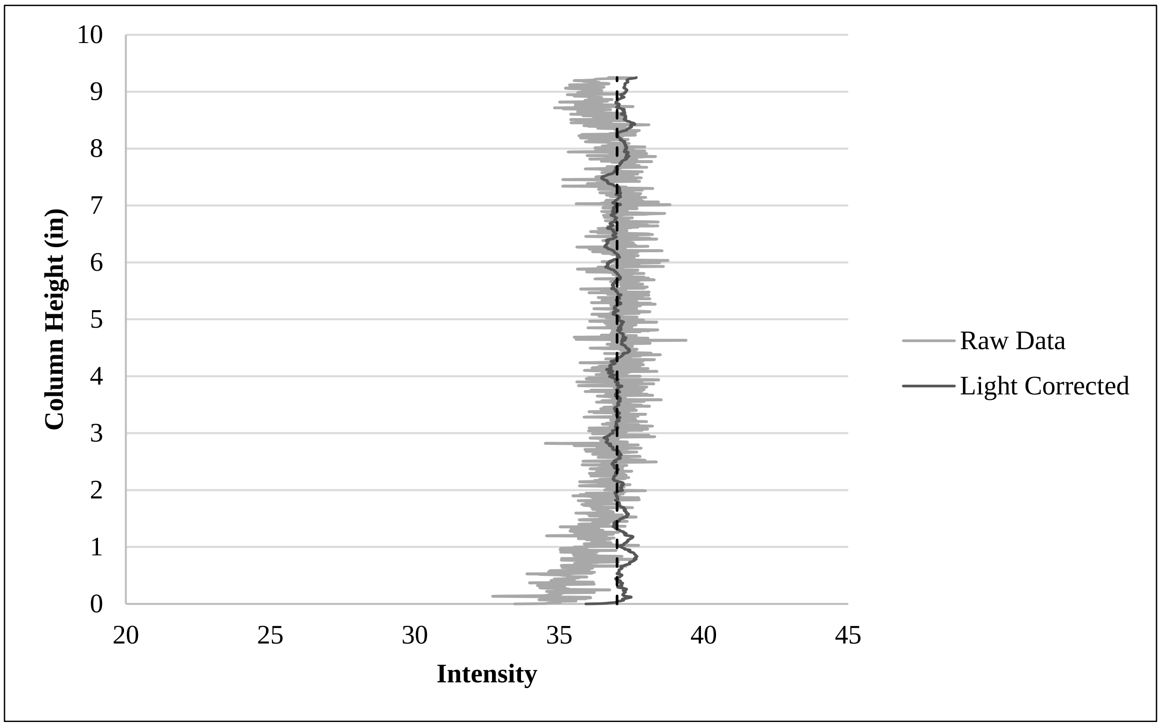

Figure 4-9: Intensity data before and after correcting for lighting

Based on the quality of the image, there may be a distracting amount of noise in the data. To reduce the scatter in the data (smooth the data), average adjacent intensities until scatter is appropriately reduced. For this experiment, which contains approximately 1800 data points, each data point is averaged with the nearest 30 other data points.

4.5.4 Converting Intensity to w/c ratio

Next, the color intensity values need to be converted to equivalent water-to-cement ratios. There likely exists multiple valid ways to accomplish this. In this study, the procedure was determined by plotting image analysis results alongside manual results and identifying the procedure which produced the most accurate results for the samples tested. First, the section of the column that represents the color of the initial w/c of the grout is used to calibrate the results. Using the data between 3.0” and 4.0” (between 80 to 100 mL) works for most grout mixes. The average intensity in this section represents the intensity of the initial water-to-cement ratio, Y_0. Next, take the ratio of the recorded intensity to the initial w/c intensity for each data point. Raise the ratio to the 16th power to get a normalized w/c (i.e. change from initial w/c). Finally, multiply normalized w/c ratio by the original water-to-cement ratio to obtain the w/c ratio at a given location and time.

$$w/c_{norm} = \left({}^{Y_{cor,i}}/_{Y_0}\right)^{12} \quad \text{(Equation 5)}$$

$$w/c_{t=ti} = w/c_{initial} * w/c_{norm} \quad \text{(Equation 6)}$$

Once again, this is an empirical fit based on the observed correlation between the normalized manual pipette data (w/c / w/c$_i$) and the normalized intensity measurements (y/y$_0$) (Figure 4-10). Note, the data shown in Figure 4-10 comes from w/c 3:1 tests because w/c 1.5:1 tests had little variations in w/c ratio. Efforts to bring in elements of sedimentation theory can be used to strengthen this correlation in the future.

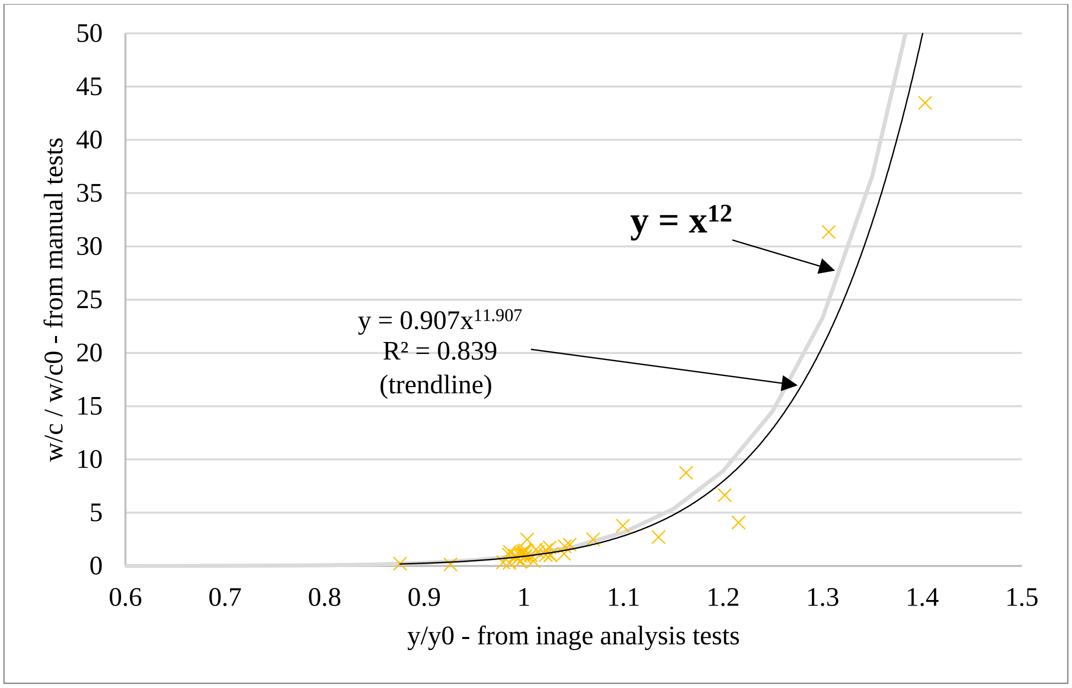

Figure 4-10: Correlation between pipette manual measurements and normalized intensity from image analysis

4.6 Combined Pipette and Image Analysis Results

The results for two different water-to-cement ratio grouts, 1.5:1 and 3:1 are presented in this section. The 1.5:1 grout is considered a fairly stable grout. However, its viscosity is too high for use in most permeation grouting programs without the use of superplasticizers. The 3:1 grout is less stable and less viscous. However, it also often requires the addition of superplasticizer for use in permeation grouting programs. The addition of superplasticizer was observed to have a noticeable effect on the stability of a grout and therefore these w/c ratios were chosen.

Figure 4-11 shows the manual and image analysis test results for a w/c 1.5:1, SP 2.0% grout over a period of two hours.

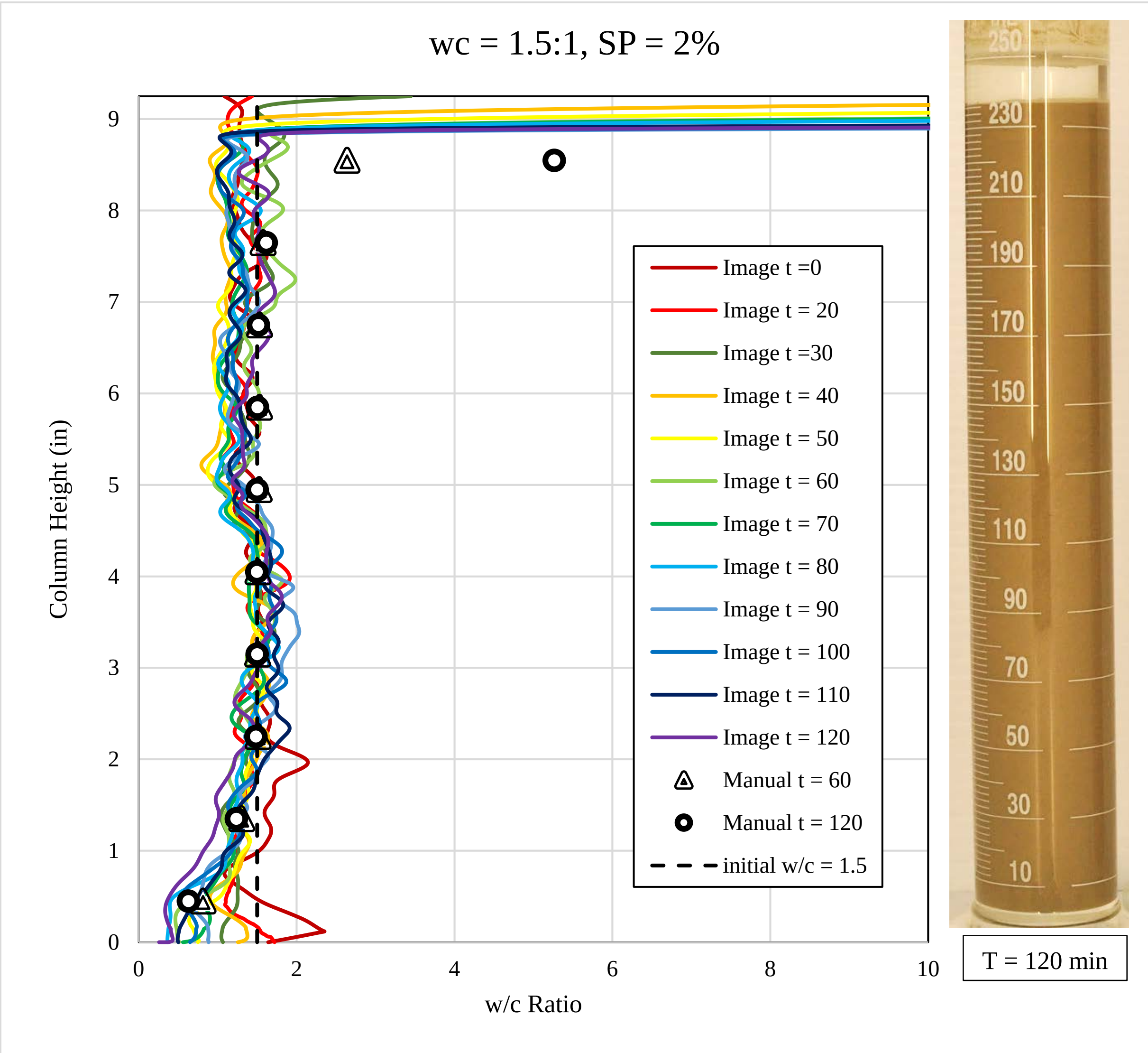

Figure 4-11: Manual and Image Analysis Results for w/c 1.5:1, SP 2.0% grout

As previously mentioned, at a w/c ratio of 1.5:1, the grout is fairly stable and therefore not a lot of change occurs over 2 hours. The most significant change is seen at the top of the column. Figure 4-12 shows a close-up of the top of the column. Note that the top data point from the manual test does not lie on the t = 120 min image analysis curve. This is because the manual test reports an average w/c over the height of sample obtained. Thus, when a large change in water-to-cement ratio occurs over a small change in height, the manual test is not precise enough to determine the actual w/c distribution over that small height. On the other hand, the image analysis test can detect changes in the w/c of the grout continuously throughout the height of the column. Note that fluctuations exist in the image analysis data due to image imperfection; however, the overall trend is easily identifiable.

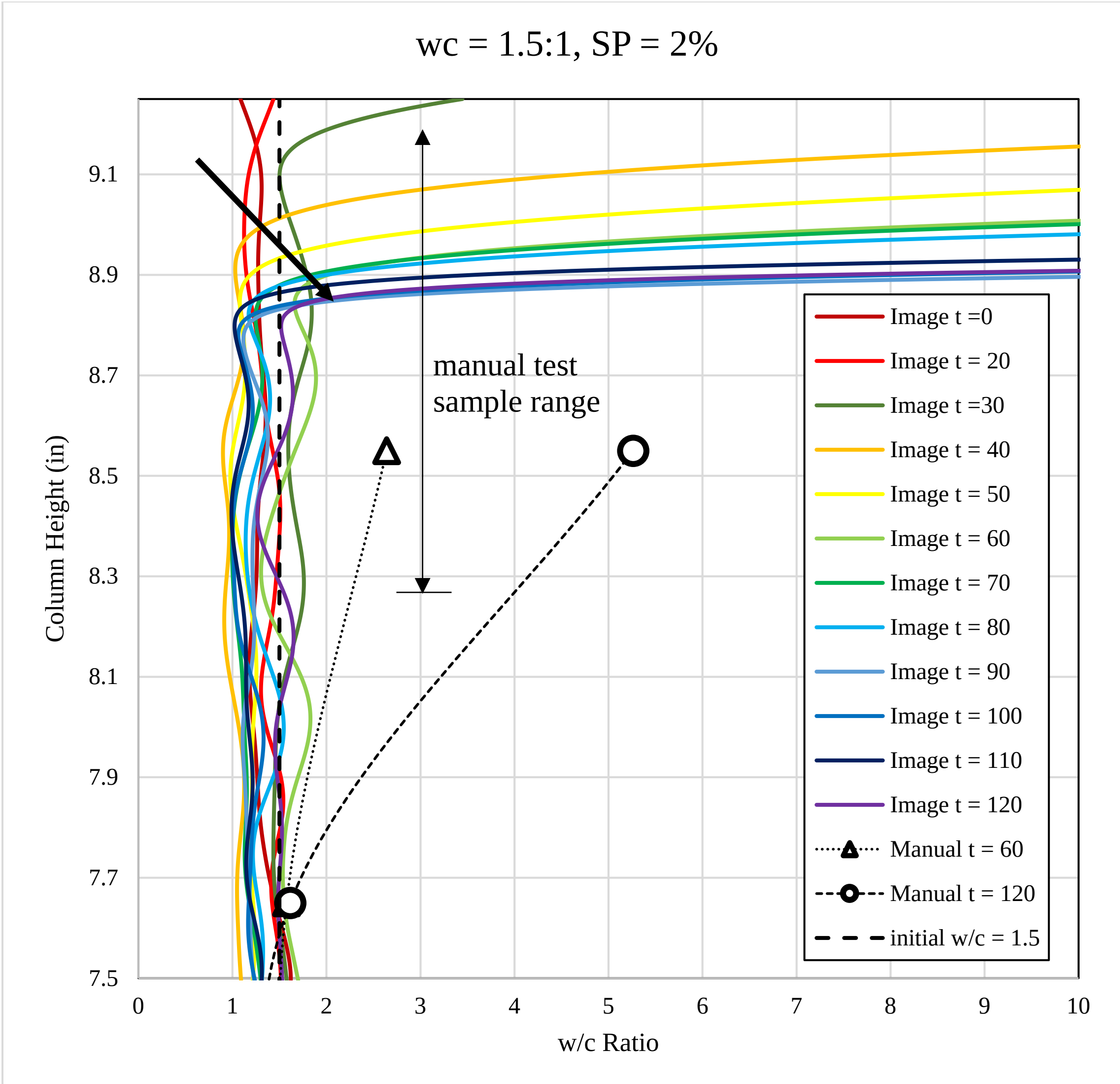

Figure 4-12: Close-up of top of w/c 1.5:1, SP 2.0% grout column

In grout, stability is the result of inter-particle interactions between the cement particles that create a structure within the grout. This structure resists downward settlement of the cement particles and the upward movement of water. In low w/c grout, such as w/c 1.5:1, the high percentage of cement particles in the grout results in significant structure. This is what causes it to behave more stable. Adding superplasticizer neutralizes the forces between particles and promotes dispersion of clumps. Neutralizing the inter-particle forces results in a desirable reduction in the viscosity of the grout but enhances the chances of particle settlement. On the other hand, breakage of clumps will increase viscosity but decrease particle settlement due to smaller individual particles. Because the cement particles are settling as individual particles rather than clumps, they can achieve a higher final concentration at the bottom of the bleed column test, even though it might take longer to reach this point (Figure 4-13). Based on this contradictory impact of SP on rheology and internal stability, it is very critical to determine the optimal SP content for a given w/c ratio grout through experimental testing as that proposed in this study.

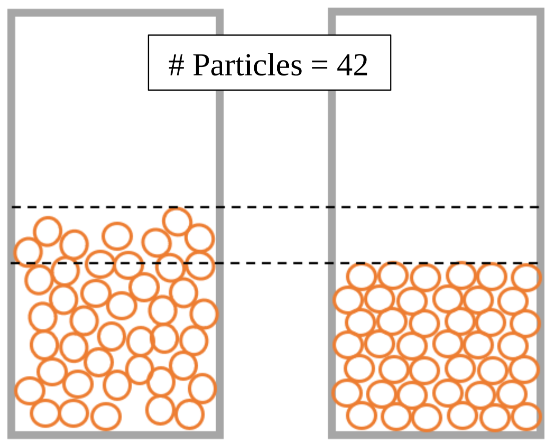

Figure 4-13: Non-dispersed vs. dispersed particle settlement

In a higher w/c grout, such as w/c 3:1, the lower concentration of cement particles causes a looser structure to form. Thus, there are less inter-particle interactions that prevent cement particles from settling and water from escaping to the top of the column and more bleed occurs. Adding superplasticizer to a w/c 3:1 grout will have the same ultimate effect as adding superplasticizer to a w/c 1.5:1 grout; the cement particles can ultimately reach a tighter formation. However, depending on the amount of superplasticizer added, the way in which the cement particles settle into this ultimate formation varies.

Figure 4-14 shows photographs of five grout columns with water-to-cement ratio 3:1 after two hours (120 minutes) with 1.0%, 1.5%, 2.0%, 2.5% and 3.0% superplasticizer added. Figure 4-15 shows the results from the manual and image analysis tests for these columns. Figure 4-14 and Figure 4-15 show that depending on how much superplasticizer is added, the grout behaves quite differently and seems to reach one of three distinct states after two hours (120 minutes).

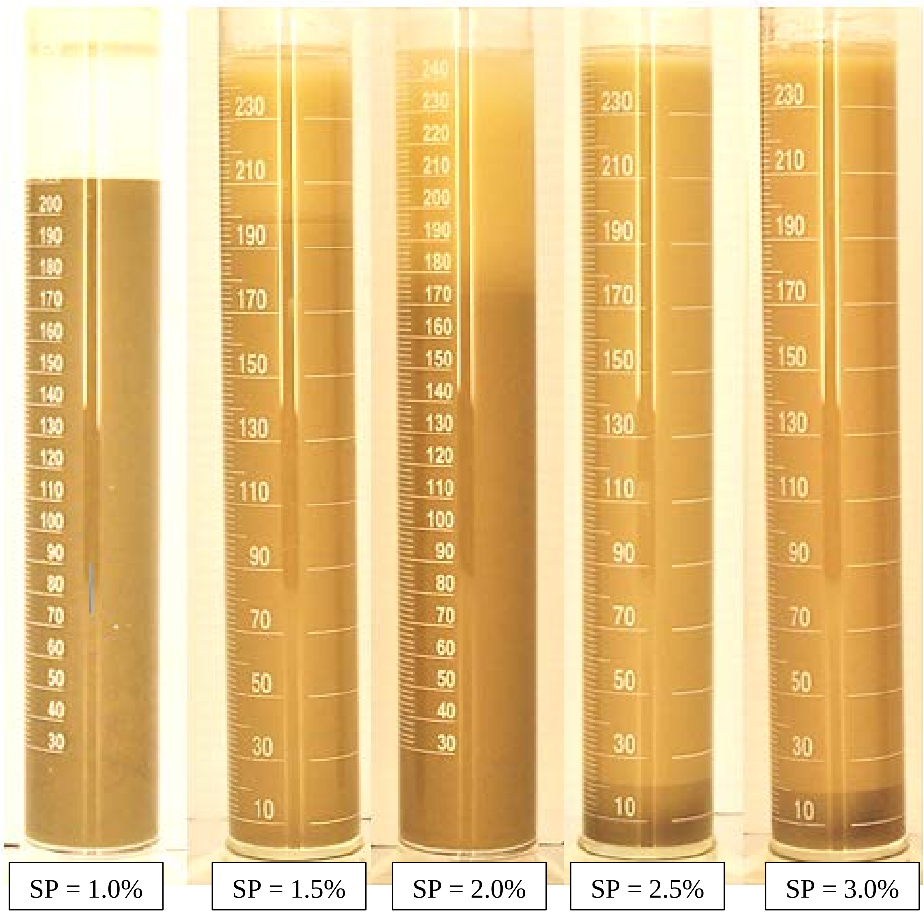

Figure 4-14: Photographs of w/c 3:1 grout after 120 minutes

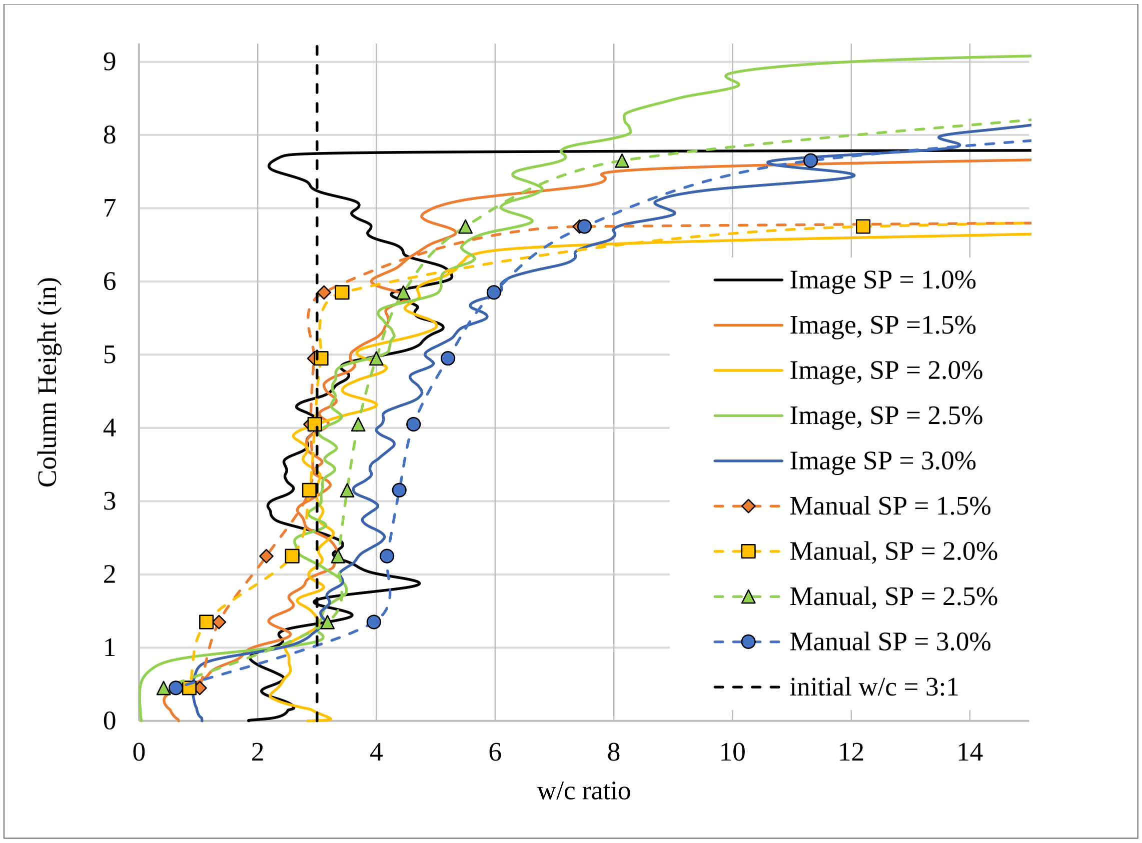

Figure 4-15: Manual and image analysis results for w/c 3:1 grout after 120 minutes

For low percentages of added SP (1.0%), the grout behaves like a mix without SP where a defined interface between the settling grout and clear bleed water can be seen (Figure 4-16). At this percentage of added superplasticizer, the rheology of the grout may be affected, but the stability remains relatively the same.

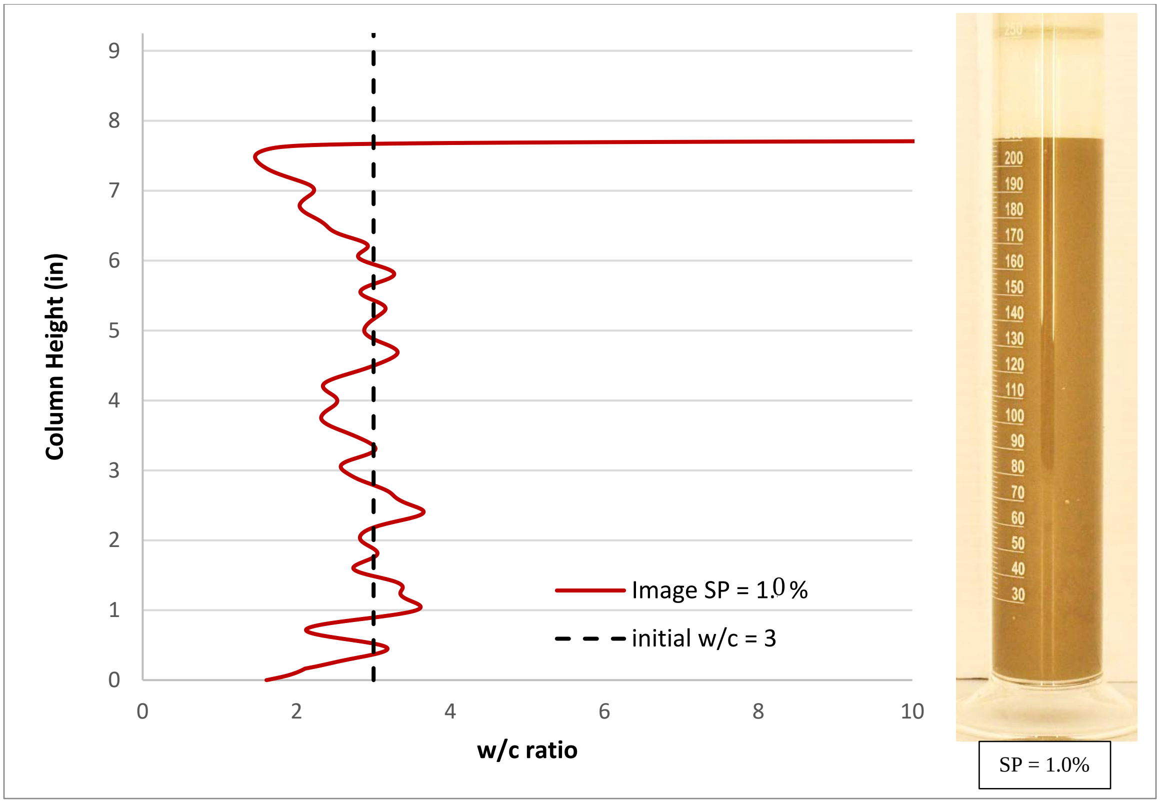

Figure 4-16: Behavior of w/c = 3 grout with low amounts of added SP (1.0%)

As the amount of added superplasticizer increases from 1.0% to 1.5% and 2.0%, the behavior of the grout changes. Instead of a clear layer of bleed on top of the column and a relatively uniform grout mass below the interface, a “cloudy” layer made up of a low concentration of suspended cement particles forms at the top of the column (Figure 4-17). This occurs because for a w/c 3:1 grout, this amount of added SP is enough to reduce interparticle interactions enough that some of the particles become isolated and stay in suspension as the rest of the structure settles. Below the cloudy layer exists a thicker layer of grout. However, unlike the 1.0% SP case, this layer is not uniform. Instead, the layer begins at a w/c higher than the initial w/c and transitions to a w/c lower than the initial w/c (Figure 4-17). Once again this is due to the addition of superplasticizer breaking up the structure that exists within the grout. Instead of the structure compressing as the water travels to the top of the column, enough structure is broken down that isolated particles can travel further down the column as the remaining structure compresses. Additionally, though the 1.5% SP grout and 2.0% SP grout behave similarly, a more defined interface can be seen in the less dispersed, 1.5% SP grout, whereas the interface in the more dispersed, 2.0% SP grout appears more blurred. Also, as previously mentioned, in the more dispersed, 2.0% SP grout, the interface between the cloudy layer and the thicker grout structure occurs lower than in the less dispersed, 1.5% SP grout. From the photographs of the grout columns, the 2.0% SP grout appears less stable; however, using conventional definitions of bleed and stability, this could not be explicitly stated. On the other hand, the image analysis results more clearly indicate that the 1.5% SP grout is the more stable mix.

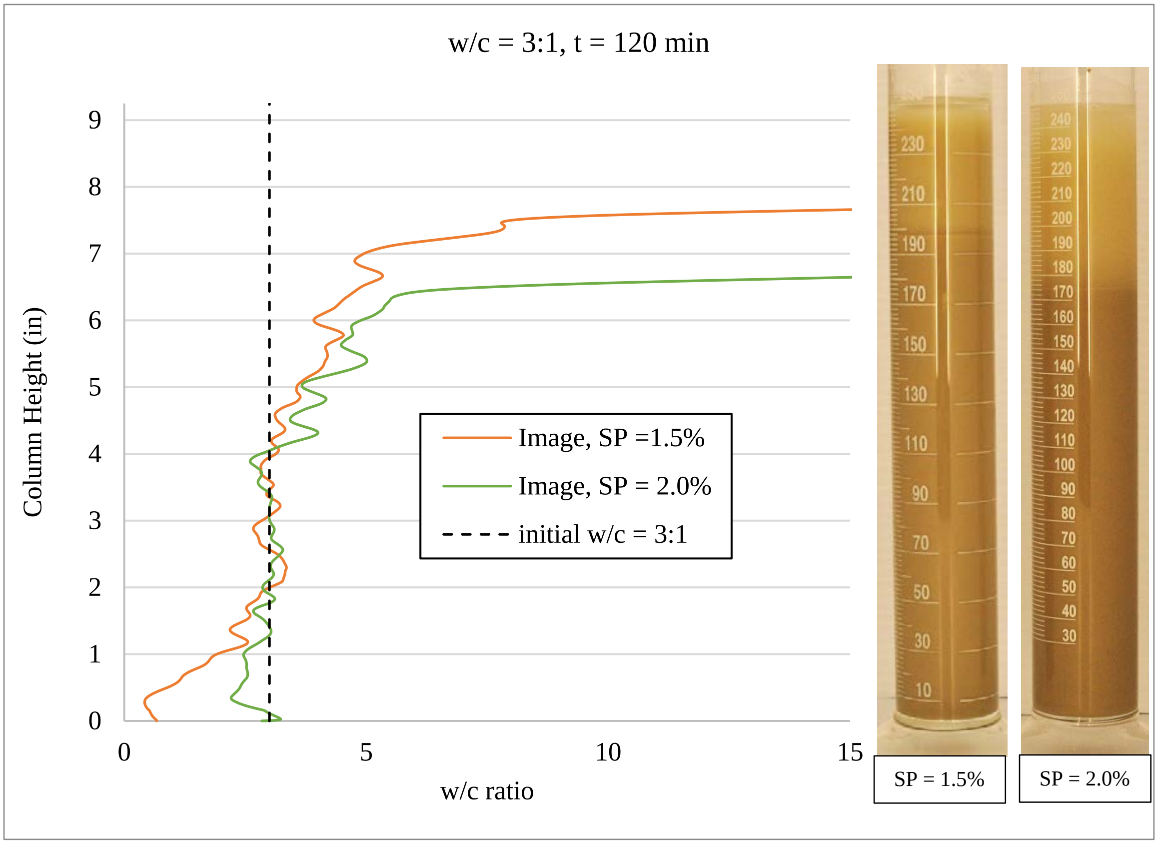

Figure 4-17: Behavior of w/c 3:1 grout with 1.5% to 2.0% added SP after 120 minutes

Above 2.5% SP added, another change occurs in the behavior of the grout. The particles become so dispersed that the structure is significantly reduced, and the particles settle more like individuals than a unified group. Particles at the base of the column reach an even tighter formation. Low concentrations of cement particles remain suspended at the top of the column. The result is a gradual transition from high to low water-to-cement throughout the height of the column (Figure 4-18). The trouble with grouts at this percentage of added SP is that the photographs of the columns make the grout look like it is stable (i.e., there is no obvious bleed). However, the image analysis test results show that for the majority of the column height, the grout is above its design water-to-cement ratio. Furthermore, at the base of the column, the grout is significantly below its design water-to-cement ratio. This non-uniformity could cause issues if the grout were to be used in the field, yet current practices do not include a way of describing and accounting for this.

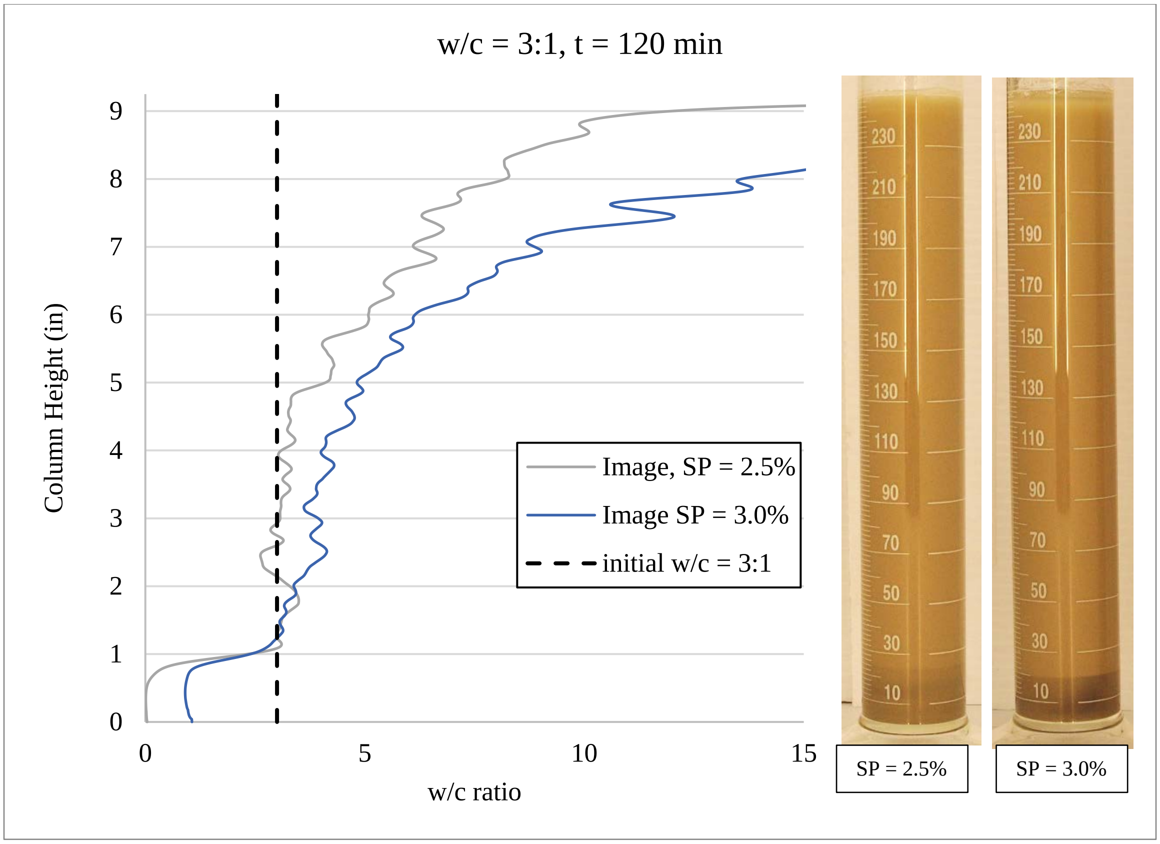

Figure 4-18: Behavior of w/c =3 grout with higher amounts of added SP (2.5%)

Overall, the use of image analysis to study grout stability shows promise for allowing greater inspection of how adding SP affects grout stability as well as how the particles settle over time.

Chapter 5: Dynamic Stability Tests

5.1 Introduction

As evidenced by the previous chapter, one of the important properties of a cement grout is its stability. Due to the many problems associated with its use, an unstable grout is undesirable in most cement grouting operations (Rosquoët et al. 2003, Naudts et al. 2004, Tan et al. 2004, Bremen 1997). Currently, the methods of determining the stability of a grout (bleed test, API filter press, etc.) only look at grout in a static condition; however, these tests may not be representative of grout behavior as it is being injected in the field (this includes pumping through the lines as well as penetration through the target soil). To get a better understanding of how a grout behaves as it is flowing in the field, dynamic stability conditions must be considered. Thus, this chapter aims to introduce a method for characterizing the dynamic stability of grout.

This chapter investigates experimentally the performance of microfine cement at a range of different water-to-cement ratios. It explores two different tests of grout stability, a static column sedimentation test similar to that described by Widmann (1996) and ASTM 940, and the newly proposed dynamic grout stability testing employing the use of a Physica MCR 301 rheometer (Anton Paar, Graz, Austria). In addition to the two sedimentation testing methods, the experimental program included 1-D permeation tests, and unconfined compression tests on the grouted sands. The results from the permeation and unconfined compression were used to determine which of the two stability tests is better at capturing the stability of the grout during its flow through the sand.

[1]El Mohtar, C., Miller, A.K., Jaffal, H. (2017). "Introducing a New Method for Measuring Internal Stability of Microfine Cement Grouts". Grouting 2017.
Contributions: El Mohtar – Assisted in designing research and revising paper; Jaffal – Assisted in performing 2D permeation testing and UCS testing

5.2 STABILITY TESTING

First, the stability of grout mixes is evaluated using traditional static column settlement tests. Next, static and dynamic settlement tests are conducted using a rheometer and compared to the traditional tests' results. Then the actual performance of the different cement grouts is determined by permeating them through a two-foot long sand column. The data from this test are used to analyze the propagation of the different grout mixes. Finally, the strength of the grouted sand is measured at different distances from the injection point. These measurements reflect the uniformity (or lack of uniformity) of cement content within the grouted sand. Water to cement (w:c) ratios of 10:1, 7:1, 5:1 and 3:1 were used in this study. While some of these w:c ratios are too high for most practical applications and are expected to have significant bleed, they were particularly selected because of their distinct bleeding to highlight the difference in the measured stability using the two different methods with minimal impact on the results due to viscosity (the viscosity of the 3:1, 5:1, 7:1 and 10:1 grouts are 64, 53, 53 and 50 mPa.sec, respectively).

5.2.1 Standard Column Stability Test

The stability of the different grout mixes was first evaluated by measuring the settlement of cement particles in a grout column with time. The method used in this study is a modified version of the method recommended by Widmann (1996). In his bleed test, one liter of grout is poured into a cylinder with a diameter of 60 mm (which gives a sample height of 353 mm), and the development of a clear water layer at the top of the grout column is monitored. Grout bleeding is defined as the height of clear water in percentage of the total grout column height after two hours (Widmann 1996). For this study, a 100 mL cylinder, as well as a test cup with the same dimensions as the rheometer test cup, was used for bleed test readings. The rate of settlement is affected by the different cylinder geometries; however, since this test is mainly used as a qualitative test to achieve a rough comparison between different grouts, this difference from Widmann's recommendations was deemed acceptable. At time zero, a freshly mixed suspension is placed in the 100 ml cylinder (or test cup). The height of the interface between the settling cement and the water is recorded over time until the interface height remains relatively constant. The rate at which the cement separates from the water (bleeding) and the final percentage of clear water at the top of the column give an indication of the grout's stability.

5.2.2 Dynamic Stability Test

The standard stability test method described above does not take into account that during its use in the field, a grout will be flowing, not stagnant. Therefore, a new method to evaluate grout stability was developed to take the grout conditions in the field into account. The main concept behind the new proposed method is to measure changes in the properties of a grout while it is being subjected to shearing to simulate field conditions. These tests were performed using a Physica MCR 301 rheometer (Anton Paar, Graz, Austria). A vane was attached to the rheometer motor and inserted into a cup filled with grout. The vane was then used to maintain a constant shearing rate to simulate the dynamic conditions the grout would experience in the field as well as measuring the grout properties over time.

The dynamic sedimentation tests consisted of subjecting grouts to constant mixing (at different speeds) with limited interruption to record the storage (elastic) modulus at predetermined time intervals. Due to the viscoelastic response of the grouts, at each recording, the modulus was measured for 30 seconds and the average of the last four readings is recorded as the representative value for that time interval (Figure 5-1). A similar modulus recording schedule was also performed without mixing to simulate the static condition that traditionally would be tested using sedimentation columns.

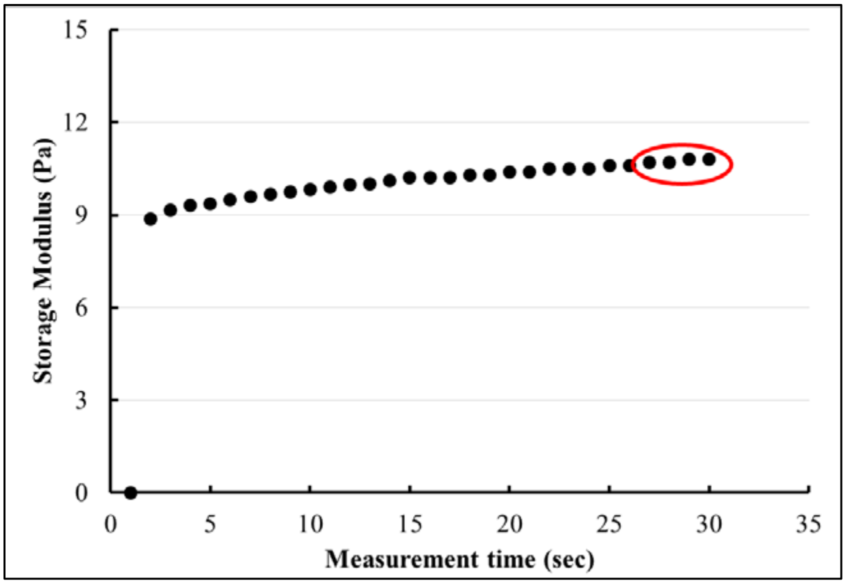

Figure 5-1: Storage modulus recording over 30 seconds per reading. the average of the last 4 readings is used for that time interval.

5.3 PERMEATION TEST

The performance of the different cement grouts is evaluated by permeating each mix through a two-foot long, 2.8-inch diameter sand column (Figure 5-2). The 2-foot column consists of four 6 in. long split tubes that can be fixed independently in place before placing the rest of the top tubes and the top cap. The sand is pluviated into the 6 in. tubes individually to allow for a more uniform sample preparation. Two 1.5-inch long tubes filled with filter material, gravel and coarse sand, are added to the top and bottom of the stack of 6-inch tubes, as shown in Figure 5-2. The filter material helps create a uniform flow of grout into the sand column and reduces risk of sand plugging the outflow tubes.

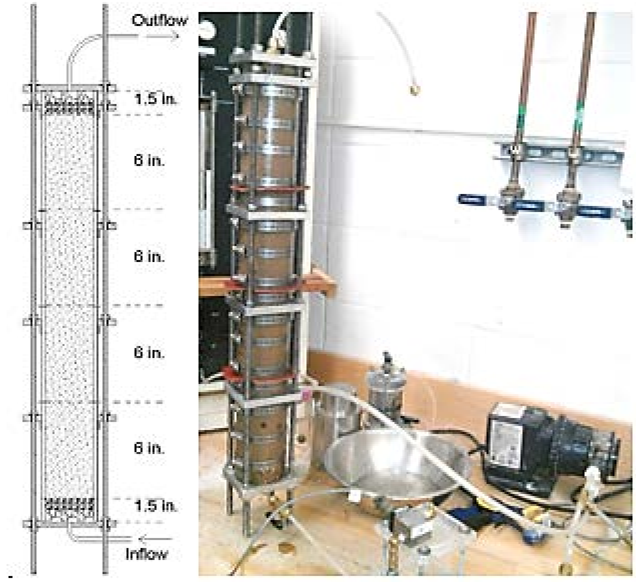

Figure 5-2: Constant flow permeation setup

After the sand column is constructed, it is flushed with CO_2. This step is done before water flushing in order to achieve a higher degree of water saturation later. Then, the sand column is flushed with water for at least two pore volumes. Finally, the sand column is flushed with cement grout while pressure at the injection point and grout front's height are recorded with time. A constant flow pump with a flow rate of 100 ml/min is used for water and cement flushing.

5.4 Unconfined Compression Test

The strength of the grouted sand is measured at different distances from the injection point using the unconfined compression test. Each column produces four 6 in. high, 2.8 in. diameter cemented sand specimens for unconfined compression testing. The unconfined compressive strength of each of the 6-inch samples is measured after a setting time of three days. While the 7 and 28 days are more commonly used for deign values of the compressive strength, the 3 days were deemed suitable for this study since the values were used to a relative comparison rather than a final design strength value. A displacement rate of 1% per minute is applied for all samples. This test gives a direct evaluation of each grout's performance. The variation of strength as function of the distance from the injection point reveals the quality of the grouting operation's outcome. A non-uniform strength profile indicates a low-quality end product, due to filtration of cement particles.

5.5 RESULTS AND DISCUSSION

In this section, results of the previously described tests are presented and discussed. First, the results of the sedimentation column test are presented. Then, the rheometer testing results are shown to demonstrate the newly proposed dynamic stability testing. Next, permeation tests' results, including pressure at the injection point against time are presented. Finally, the unconfined compressive strength profiles of the grouted sand columns are presented. The strength profiles provide a direct measurement of the uniformity of cement propagation and the quality of grouted soil. The results of the permeation and strength tests are compared against the results of the different sedimentation tests to see which one better predicts performance.

5.5.1 Standard Stability Test

The results from the neat cement grout sedimentation tests are shown in Figure 5-3. The results show that the grouts with higher water-to-cement ratios settled much more and much faster than the grouts with lower water-to-cement ratios. The reduced rate of sedimentation for the lower water-to-cement ratios is likely due to more interactions between the more densely packed cement particles. The high water-to-cement ratio mixes, on the other hand, have less concentrated amounts of cement, which reduces settlement interference due to particle interactions, and less cement overall, which allows for more bleed. Therefore, as is confirmed in the experiment, less bleeding is expected from the 3:1 mix than the 10:1 mix. Additionally, Figure 5-3indicates that the water-to-cement ratio 10:1, 7:1, and 5:1 grouts behave similarly to each other, while the 3:1 grout behaves quite differently. This observation will be discussed in more depth later in this paper.

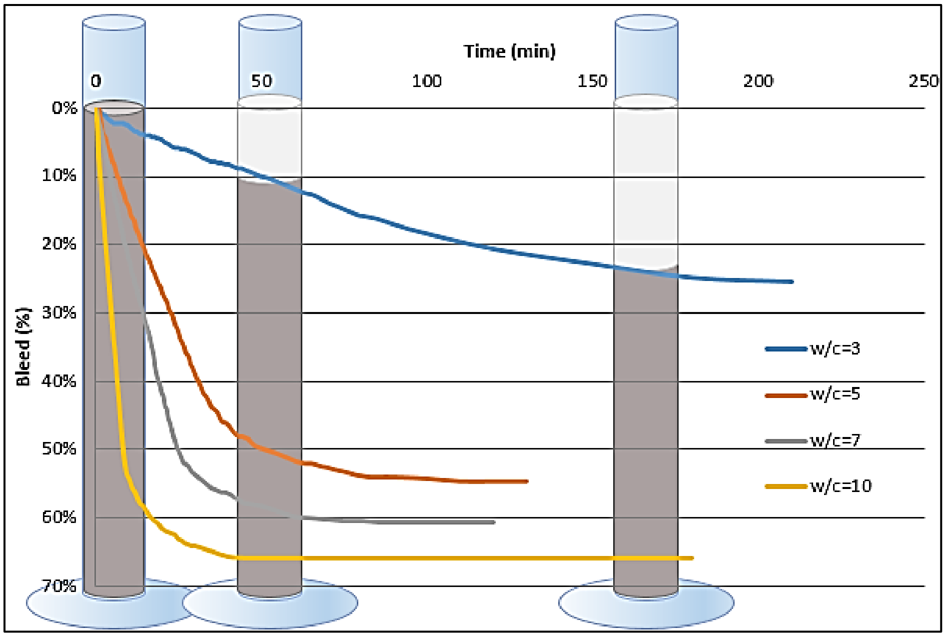

Figure 5-3: Sedimentation rates with varying water to cement ratios (w/c)

5.5.2 Dynamic Stability Test

An additional stability test was conducted to evaluate how the grout may behave in dynamic conditions. First, grouts with w:c ratio of 3:1, 5:1, 7:1 and 10:1 were tested under static conditions using a rheometer (same procedure as described above except that the vane is not spinning between readings). The rheometer is used to measure the storage modulus of the grout over time at the middle of the cup. A sample of the results of the static sedimentation tests obtained using this test method are presented in Figure 5-4 for w:c ratio of 7:1. The storage modulus is a measure of the stored energy in viscoelastic materials; therefore, monitoring this property can give an indication of the interactions between the cement particles in suspension in the grout. This can then be interpreted to determine how the cement particles are settling over time. An increase in the storage modulus indicates an increase in cement concentration at the height of measurement (similar to first 12 minutes of measurements), which indicates that cement has settled. A decrease in storage modulus indicates the cement particles have settled below the measurement height and only part of the vane is submerged in the cement while the top half is measuring resistance of water. .After sufficient time, an increase in storage modulus may indicate cement hydration or consolidation of the settled cement. The pictures on Figure 5-4 are taken at the time intervals they are presented of an identical cup with same mix inside it but put on the side and not in the rheometer. The measured modulus values reflect the sedimentation process relatively well when no mixing is introduced including minimal constant measurements when the top of the sediment is below the base of the vane.

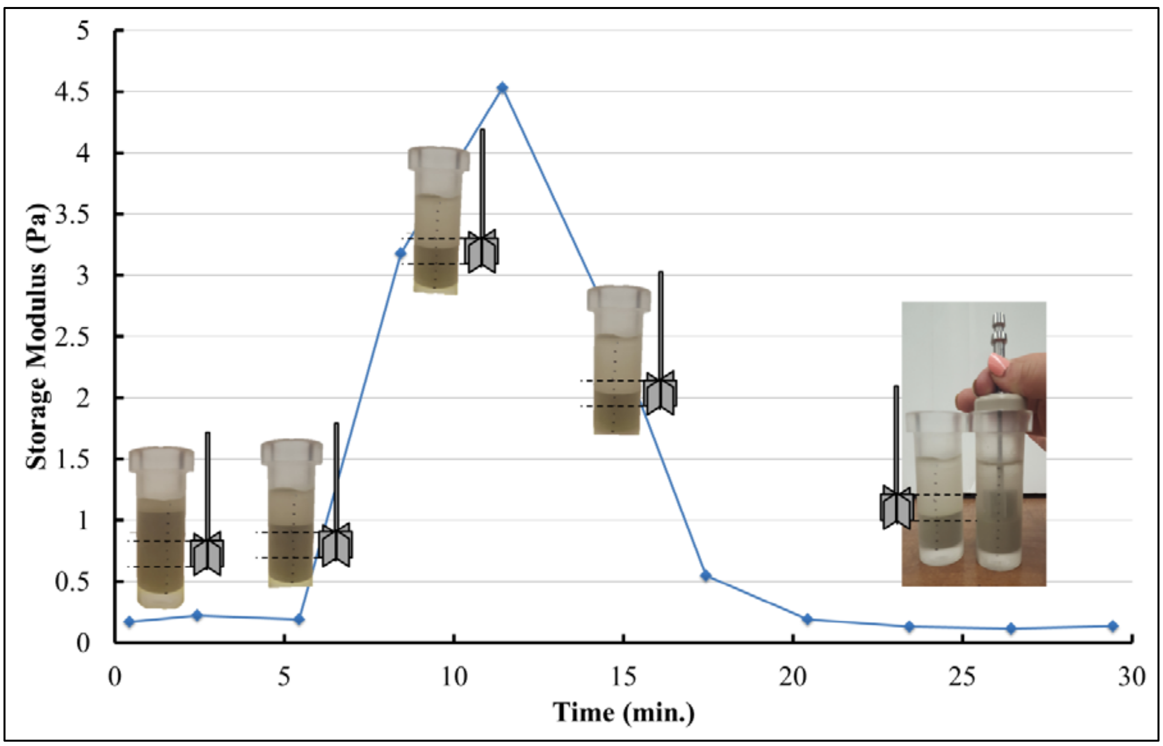

Figure 5-4: water to cement ratio 7:1 grout static sedimentation test using a rheometer

Figure 5-5 (a) through (d) shows the height of the cement-water interface for the 3:1 water-to-cement ratio grout in the rheometer test cups after 0, 30, 60 and 90 minutes. The vanes superimposed on the cups represent the height at which the vane measurements were taken throughout the test. For the water-to-cement ratio shown, 3:1, the vane remained below the level of the settling cement throughout the test. This was not the case with the higher water to cement ratios.

The rest of the static filtration results, using the rheometer, are included in Figure 5-6 along with the results from the dynamic testing. For the dynamic conditions, flow was simulated by using the vane rheometer attachment to mix the grouts at constant rotation speeds of 6 rotations per minute (R=6), 60 rotations per minute (R=60), and 300 rotations per minute (R=300). The curves shown are normalized by the initial recorded storage modulus per w:c ratio. Under static conditions, the 10:1, 7:1 and 5:1 grouts show a similar trend of increasing normalized modulus up to a peak value then decreases afterwards whereas the 3:1 grout didn't experience any peak and decrease. This results are consistent with those from the standard bleed tests where the 3:1 grouts responded differently than the higher w:c grouts.

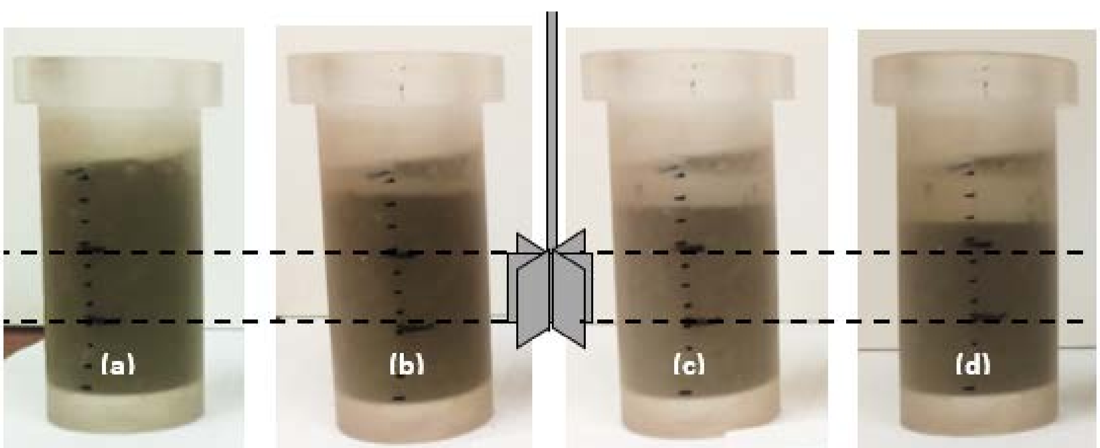

Figure 5-5: Rheometer test cups with w/c 3:1 at (a) t= 0 min, (b) t = 30 min, (c) t = 60 min and (d) t = 90 min

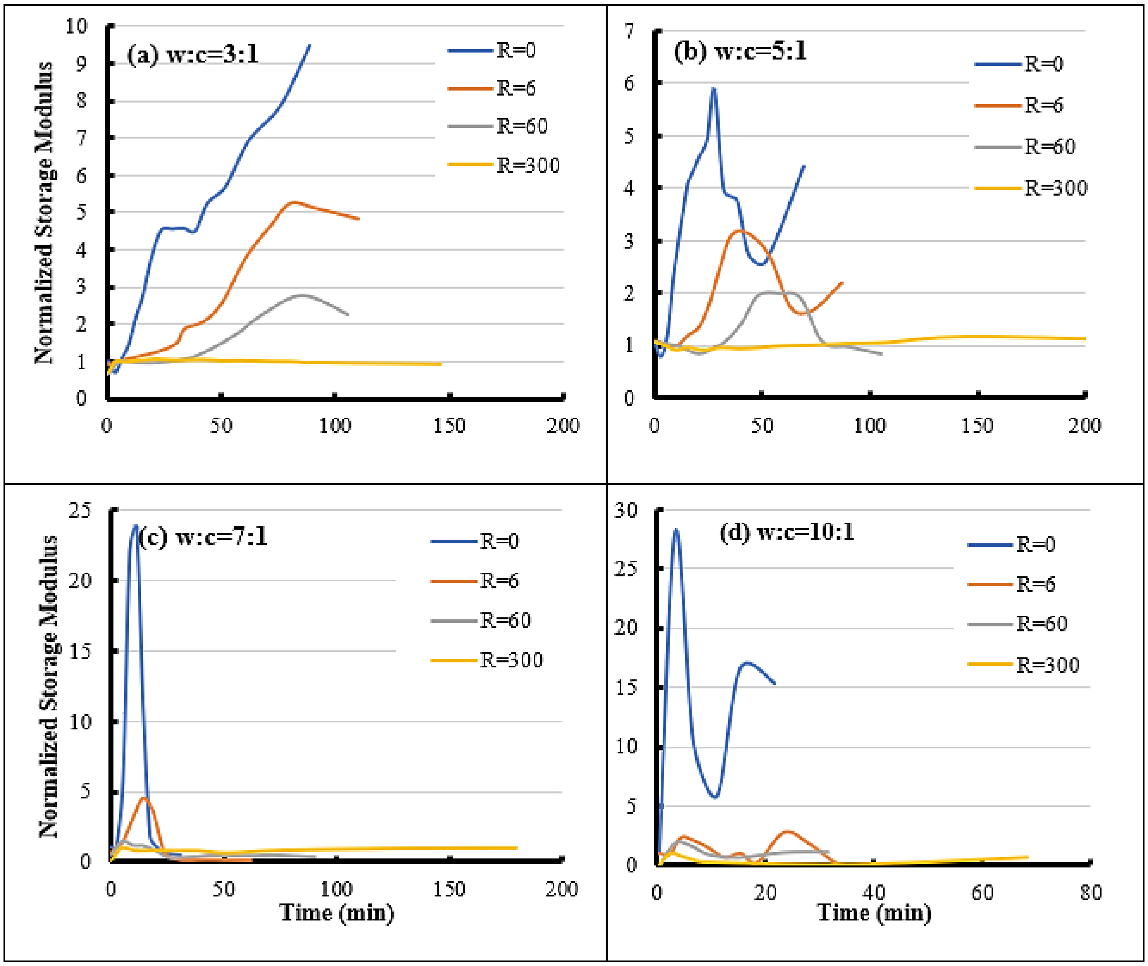

Figure 5-6: Dynamic sedimentation tests (normalized by initial storage modulus readings)

Figure 5-6 shows that even adding a minimal amount of rotation (R=6/min) greatly effects the measured behavior of the grout. Adding rotation (simulating dynamic conditions) increased the stability of the grouts. Dynamic conditions cause the storage modulus to peak at a lower value (if at all) and after a longer time. The 10:1 grout did not exhibit this relationship as clearly as the other water-to-cement ratios; however, this is likely due to noise in the data and the rheometer's lack of capability to accurattly measure changes at such low storage modulus values. For the mixes other than the 10:1, adding rotation directly affected the rate of bleeding. As the mixing rate increased, the overall bleeding as well as the rate at which cement settled out of the mix decreased. Another behavior that can be seen from these tests is that adding high rotation (R = 300/minute) caused the samlple to retain the same storage modulus throughout the duration of the test.

Figure 5-7 shows the 3:1 water-to-cement ratio grout before and after the R=300/min test (after 150 minutes). These pictures are consistent with the data as little to no difference can be observed between the before and after pictures. This indicates that if mixing is high enough, sedimentation barely occurs. Figure 5-8 shows the plots of the different water-to-cement ratio grouts tested with the 60 rotations per minute mixing speed. In contrast to the static rheometer and static sedimentation tests, the results from the dynamic sedimentation tests show the grouts with w:c ratios of 7:1 and 10:1 had very early peaks while the 5:1 and 3:1 grout mixes had delayed peaks.

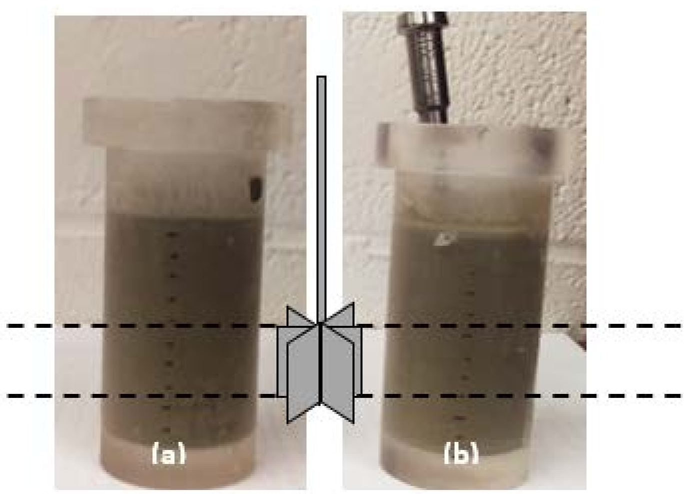

Figure 5-7: w/c 3:1 grout (a) before and (b) after testing with 300 per minute rotation rate

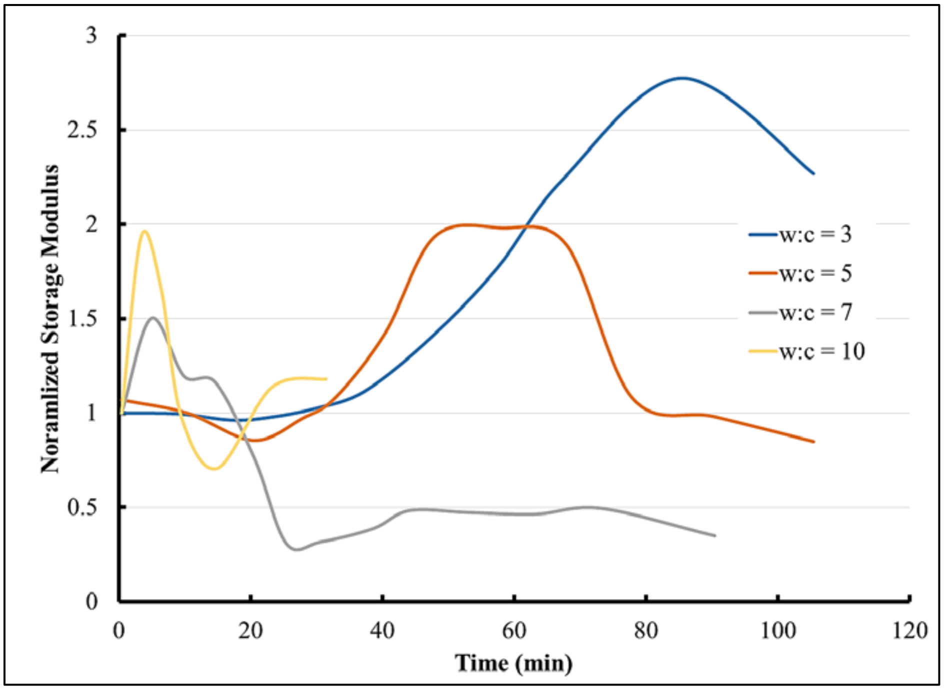

Figure 5-8: Dynamic sedimentation test results for mixing speed =60 rotations per minute

5.5.3 Permeation Test

The pressure at the injection point is recorded using a pressure sensor connected to a data acquisition system. The pressure time curves comparing the performance of grouts at different w:c ratios, are presented in Figure 5-9. For all the different water-to-cement ratio grouts, the pressure-time curves clearly indicate the occurrence of filtration. The tests show that the 3:1 grout results in higher pressure buildup at the injection point due to higher viscosity. Only half a pore volume of 3:1 was permeated before the testing stopped due to excessive pressure buildup; in comparison, 1.3 pore volumes of 5:1, 7:1 and 10:1 were permeated. An important note is that the 7:1 pressure curve is similar to the 10:1 pressure curve, and both the 10:1 and 7:1 pressure curves are different from the 5:1 and 3:1 pressure curves. The traditional column bleed test indicated that the 10:1, 7:1 and 5:1 grouts all would behave similarly, which is not the case in these permeation tests. However, the dynamic sedimentation tests did predict the 10:1 and 7:1 grouts behaving similarly and the 5:1 and 3:1 grouts behaving similarly. This indicates a possible advantage to using this new type of stability test.

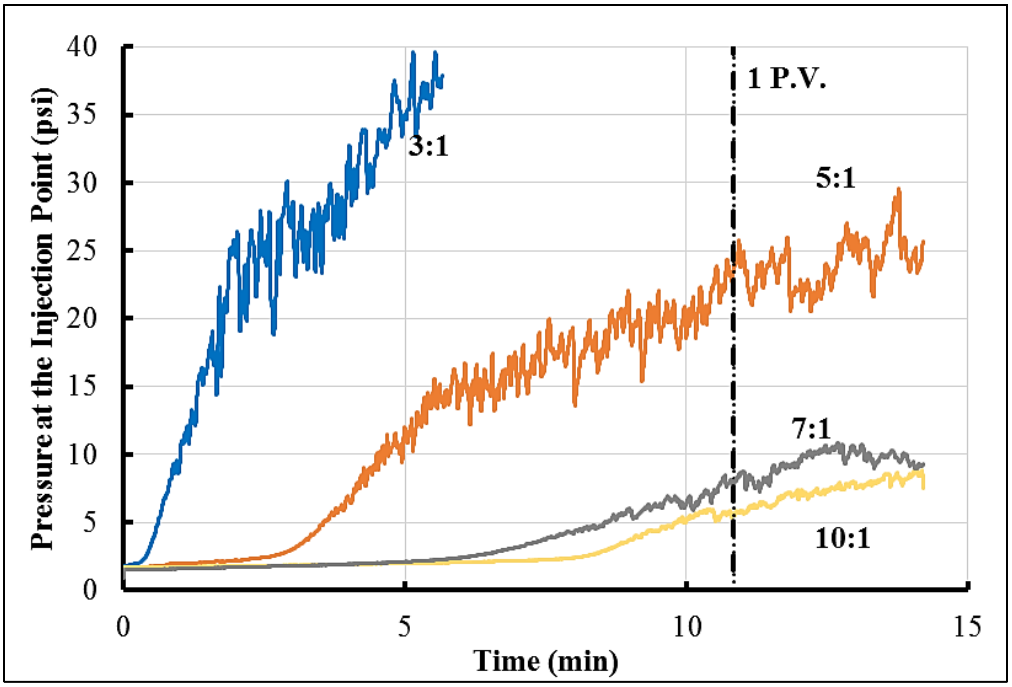

Figure 5-9: Pressure buildup for injecting cement grout at constant flux

5.5.4 Unconfined Compression Test

After three days of setting time, the grouted samples are tested under unconfined compression. The results are plotted in Figure 5-10 with the strength values on the x-axis and the height of the samples' centers relative to the bottom of the sand column on the y-axis. Figure 10 (a) shows that the strength of samples closer to the injection point are higher than those further from it due to filtration and bleeding. The 3:1 water-to-cement ratio sample was not able to permeate the column far enough, and therefore only one sample was available for testing. The strength decreased with increasing w:c ratio except for the 3:1 compared to the 5:1. This can be explained by the lower volume of grout permeated through the bottom quarter of the specimen with the 2:1 compared to 5:1. It should be noted that the 5:1 specimens near the top of the column had comparable strength to those of the 7:1 specimens, implying that the 5:1 grout never delivered a higher cement content to the top portion of the column. Figure 5-10 (b) shows the normalized strength (normalized by the strength of the measured furthest from the injection point) versus depth for all four mixes. The 10:1 and 7:1 curves plot on top of each other indicating similar filtration response while the 5:1 specimens show much higher filtration at the base. This further reinforces the idea that the dynamic stability tests performed using the rheometer might be a more comprehensive predictor of grout performance.

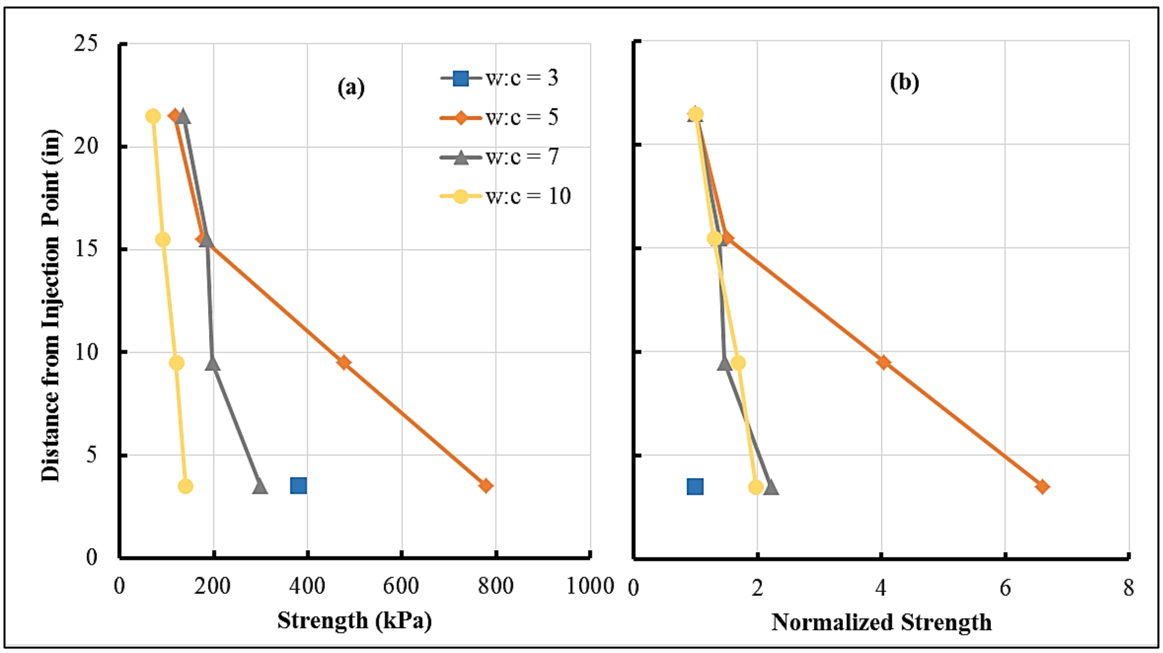

Figure 5-10: Unconfined compression strength (a) absolute values (b) normalized by strength at furthest penetration

5.6 Conclusions

Based on standard static stability tests, all four mixes were highly unstable; however, the 3:1 grout showed a distinct response compared to the rest. Dynamic stability tests were performed using an advanced rheometer by measuring the storage modulus of the grouts over time while being mixed at different speeds between measurements. The dynamic tests provided insight into how grouts may behave while being flushed through soils in the field. These tests showed that sedimentation decreased as mixing increased. Additionally, these tests distinguished between the behavior of the 7:1 and 10:1 grouts compared to the 5:1 and 3:1 grouts.

After the stability testing, permeation tests were conducted at constant flux with monitoring of pressure buildup at the injection point. The pressure curves from the permeation tests showed that the similarities and differences in the behavior of the different grout mixes aligned better with the trends predicted by the rheometer tests than the trends predicted by the column bleed tests. The same conclusion was verified by the unconfined compression tests where the 10:1 and 7:1 specimens should identical change in strength profile along injection path, a profile that is distinct from the one obtained for the 5:1 grout.

Overall, using a rheometer to correlate grout properties with bleeding has the potential to be a very useful tool in understanding and predicting the behavior of grouts. However, this technique needs to be studied further to fully understand its potential. The tests performed were limited and didn't include effect of superplasticizers and other additives. A more in-depth analysis of the change in grout properties while under high mixing is still needed and should be a topic of further research.

Chapter 6: Developing a New Grout Filtration Test

6.1 INTRODUCTION

This chapter documents the development of a new filtration test specific for grouting applications as compared to the API filter press that was developed for Petroleum applications, but later adapted as an index test for grouting. The new proposed testing can be performed on site or in the lab to evaluate the performance of a grout mix under field conditions. This test allows the engineer in the field to evaluate the penetrability of a grout, mixed using field materials and mixing equipment, through soil samples representative of field conditions. In this way, multiple simple tests can be performed on site to determine an optimal grout mix, and the need for an expensive and time-consuming lab testing program is reduced.

6.2 NEW TESTING SETUP

This section presents the design of new, simplified, filtration test setup along with the testing procedures to quickly measure the performance of a grout in the lab or in the field. The suggested setup (filtration cell to the right and compaction rod to the left) is presented in Figure 6-1 below:

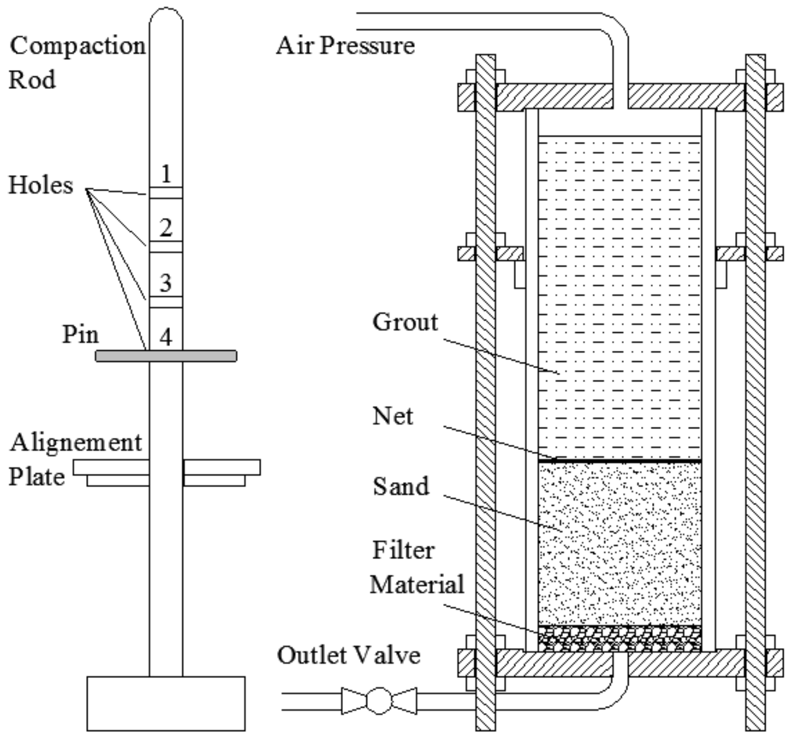

Figure 6-1: Filter press/small scale column test setup

The filtration cell consists of an 8.5 in. long acrylic tube with an internal diameter of 2.86 in. held between two bottom and top plates using four threaded bars. An intermediate plate is used to hold the tube in place to allow the construction of the sample and the addition of grout before placing the top plate. The top plate is connected to an air pressure line to pressurize the grout and push it through the sand. The bottom plate has an outlet line through which the filtrate exits the cell once the outlet valve is opened.

The compaction rod is used to compact the sand sample to a predetermined density, ideally to replicate the density in the field. Since constructing the sand layer to a predetermined density is one of the most time-consuming steps of the test, in addition to often being challenging, the compaction rod is designed to allow an easy and accurate method to do it. The platen connected to the bottom of the compaction rod has a slightly smaller diameter than that of the tube. An alignment plate is placed at the top of the tube to keep the compaction rod centralized during compaction. Four horizontal holes are drilled through the compaction rod (holes 1 to 4), and by placing the pin into any one of them the compaction rod can be used to control the compacted height of the different layers as explained in the sample preparation section.

6.3 SAMPLE PREPARATION

First, the acrylic cell is held in place on top of the bottom plate using the intermediate plate. Then, a course net is placed at the bottom to prevent the filter materials from falling into the outlet line. A 0.5 in. thick layer of filter material, consisting of coarse sand, is placed above the bottom net, and is compacted using the compaction rod with the pin being in hole #1. The filter material layer is placed below the sand to allow drainage below its entire cross-sectional area and ensure uniform fluid flow through the sand layer.

Then the sand sample, through which the grout flow is to be evaluated, is constructed in three lifts. The sand layer has a total thickness of 2.5 in. made of three 0.833 in. thick lifts. Based on the desired sand density the required weight of sand per lift can be calculated (considering that the volume of each lift is 5.37 in^3). In this study, all samples were compacted to a density of 101 pcf, which corresponded to a lift weight of 0.314 lbs. The grain size distribution curve for the sand used is shown in Figure 6-2.

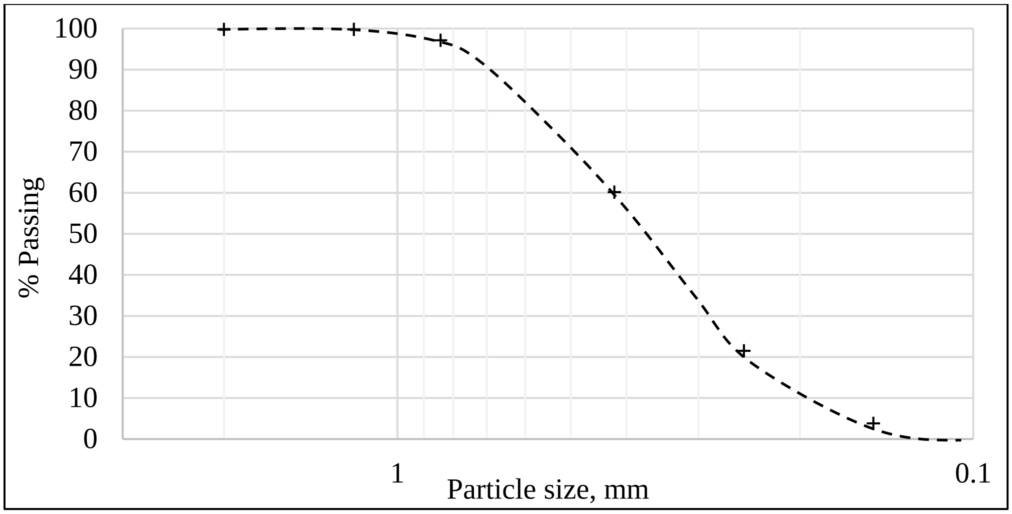

Figure 6-2: Grain size distribution of test sand

The calculated weight of sand is then added, the pin is placed in hole # 2 for the first sand lift, and the added sand is compacted until the pin hits the alignment plate sitting at the top of the cell. This step is repeated for the second and third layers with the pin placed in holes 3 and 4, respectively. Figure 6-3 illustrates the end of compacting the third and final sand lift. After the sand column is compacted, a course net (larger opening size than the average void space of the sand) is placed on top of the sand layer to keep the sand in place while the grout is added on top of it.

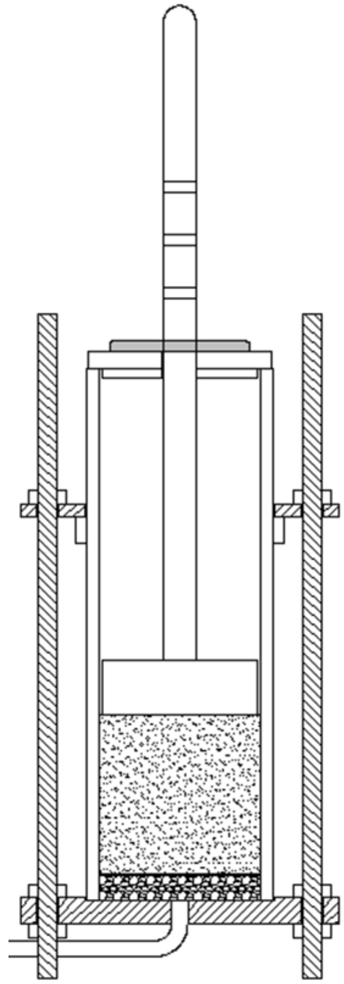

Figure 6-3: Sample preparation for the filter press/small scale column test

6.4 FILTRATION TEST

Before adding the grout, the compacted sand column is flushed with water to achieve saturation (actual degree of saturation is not measured). If, based on the soil conditions in the field, it is desirable that the soil remain unsaturated, then this step may be skipped.

To saturate the sample, attach a water tank with a higher water level than the height of the sand to the bottom outlet of the apparatus (Figure 6-4). Place the compaction rod back into the apparatus and apply a small amount of pressure either by placing a small weight on top of the rod or by hand. This will prevent disturbance of the sand during flushing. Open the valves and let the head difference between the tank and the specimen drive the flushing of the sand column. Once the water reaches a level above the top of the sand column, the water tank may be disconnected and excess water on top of the sand column should be drained through the bottom valve until the water level is even with the top of the sand layer.

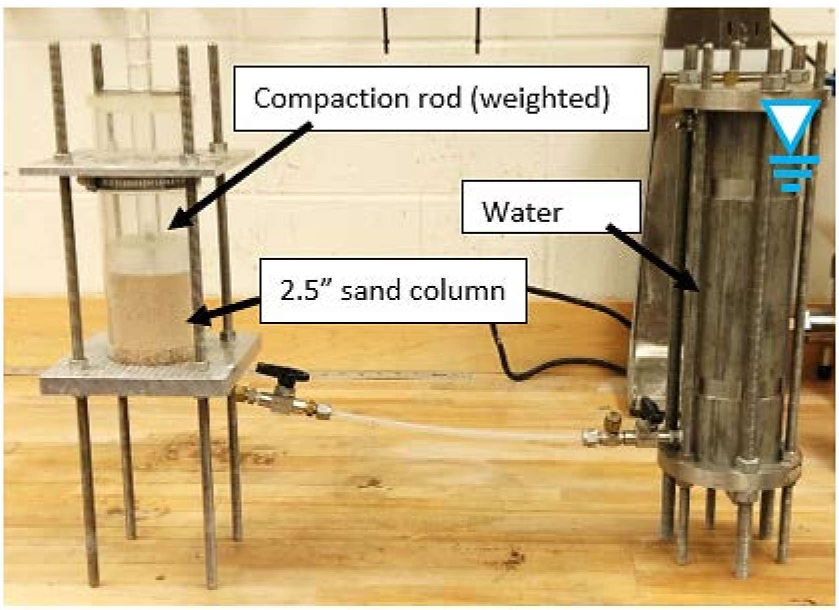

Figure 6-4: Water flushing of the sand column

Optional: Once the water level in the apparatus no longer continues to rise, add a small amount of pressure to the water tank (approximately 2 psi) to achieve a higher saturation in the sample. The added pressure will cause the water level in the apparatus to rise higher than the top of the sand column. Continue flushing until the water level reaches near the top of the apparatus. At this point, close the valves and carefully remove the compaction rod. Open the bottom valve and allow excess water to drain until the water level is even with the top of the sand layer.

Next, making sure the bottom valve is closed, carefully pour 5 inches of grout on top of the sand layer (Figure 4). Any infiltration into the sand layer at this point should be noted. Secure the top plate to the apparatus and attach the air pressure line to the top plate. The amount of pressure applied to the top of the grout should be consistent with conditions expected in the field. In this study, a pressure of 2 psi was applied to all samples. Open the top valve to apply pressure. Again, any amount of grout penetration into the sand should be measured and recorded.

6.4.1 Simplified Field Procedure and Analysis

If this test is to be run in the field rather than the lab, this simplified procedure should be followed. After the soil column is assembled and the grout is added, record the weights of at least five 50 mL volumetric flasks. Additionally, place a bowl below the exit valve to collect the effluent grout. Open the bottom valve to start the test. As water (and then grout) start exiting, insert one of the 50 mL flasks every 20 seconds, collect 50 mL of grout and wait till the end of the 20 seconds to insert the second flask. If it takes longer than 20 seconds to collect 50 mL, wait 5 seconds after filling up the previous sample before collecting the next one and mark down the extra time needed. If after the first 20 seconds only water is exiting the tube (i.e. the grout has not yet reached the exit valve) make note of this and wait until grout begins to exit the tube before taking samples. Continue taking samples until one of these conditions is reached: the grout reaches a level of approximately 0.5 inch above the sand layer; 5 minutes of grout exiting have passed; grout stops flowing;

or the effluent contains only water. Once the test is finished, weigh the 50 mL volumetric flasks and calculate the density of the effluent grout in each flask.

The field results can then be interpreted in a few ways. The performance of the grout during the filter test can be interpreted visually by noting the flow rate of the grout, how much of the grout penetrates the sand column, the amount of bleed on top of the grout column, and if clear water begins to exit the column by the end of the test. For quantitative analysis, Equation 2 can be used to convert the density of the effluent in each flask to a water to cement ratio. This ratio can then be normalized by the initial w/c ratio of the grout as initially prepared and the results can be plotted over time (and volume with some approximation of flow rates based on time required to fill each flask). The results can then be used to determine the filtration of the grout as the decrease of effluent w/c ratio over time.

$$w{:}c\ ratio = \frac{1-\gamma_f/G_{s,c}}{\gamma_f-1} \qquad \text{(Equation 7)}$$

Where γ_f = measured density of the effluent

$G_{s,c}$ = specific gravity of cement

6.4.2 Laboratory Procedure and Analysis

If the test is to be run in the lab, as was the case for this study, the following procedure should be followed. Note that the simplified field procedure can be followed in the lab as well if less detailed information is required. After the soil column is assembled and the grout is placed, record the weights of 15 to 25 moisture content tins. Open the valve to start the test. As soon as grout begins to exit the column, continuously collect samples for 5 seconds each. It is optimal to use two people for this part of the procedure to ensure continuous collection of samples. If the flow of grout slows, it may be desirable to collect samples for a longer time interval (i.e. 10 seconds). If a longer collection time is chosen, make sure to record this on the lab sheet. Continue taking samples until one of these conditions is reached: the grout reaches a level of approximately 0.5 inch above the sand layer; 5 minutes of grout exiting have passed; grout stops flowing; or the effluent contains only water. Weigh the samples and oven dry. Record the oven dry weights.

A continuous w/c curve can then be obtained by the following equation:

$$w/c = \frac{w_{grout} - w_{dry}}{w_{dry} - w_{tin}} \qquad \text{(Equation 8)}$$

Where w_{grout} = weight of wet grout + tin

w_{dry} = weight of oven-dried material + tin

w_{tin} = weight of sample collection tin

For samples where a lot of grout filtration occurs, the w/c ratio can be quite high, and therefore, different methods are proposed to evaluate the data and the performance of the grout. Since the pressure is held constant for the duration of the test, the flow rates will decrease as filtration increases. Therefore, the flow rate of the effluent should be calculated and plotted over time. The flow rate of the effluent is calculated as follows:

$$q_{effluent} = \frac{V_{effluent}}{t_{sample}} \qquad \text{(Equation 9)}$$

Where:

$$V_{effluent} = \frac{W_{effluent}}{\rho_{effluent}} \qquad \text{(Equation 10)}$$

Where:

$$\rho_{effluent}\ \left[{}^{g}/_{cm^3}\right] = \frac{(w/c+1)}{\left(w/c+\frac{1}{\rho_{cement}}\right)} \qquad \text{(Equation 11)}$$

The percent cement content by weight in the effluent can be used to quantify the performance of a particular grout. The cement content in the effluent is compared to the cement content in the original grout before running the test (a sample of the grout should be put in a similar water content tin, weighed, dried and weighed again to be consistent in measuring the cement content). A lower cement content in the effluent indicates more filtration in the soil column. A grout that maintains its original cement content throughout the duration of the test is ideal. The percentage of cement content in the original mix and effluent grout are computed as shown in Equation 9. The theoretical cement content of the original mix can be calculated from the w/c ratio using equation 10 for comparison.

$$\%C_{eff} = 100\% * \frac{Wcement}{W_{effluent}} \qquad \text{(Equation 12)}$$

$$\%C_0 = 100\% * \frac{1}{(w/c+1)} \qquad \text{(Equation 13)}$$

6.5 LAB TEST RESULTS

Neat cement grouts with w/c ranging from 1.5:1 to 7:1 were tested using the new filtration setup. The results are presented in three different ways to highlight different trends in the data. Figure 6-5 shows the effluent cement content versus time. Figure 6-5 is useful in applications where the strength of the grouted soil is important. It can give an estimate of what amount of cement is actually being delivered to the soil. It also indicates how quickly filtration is occurring.

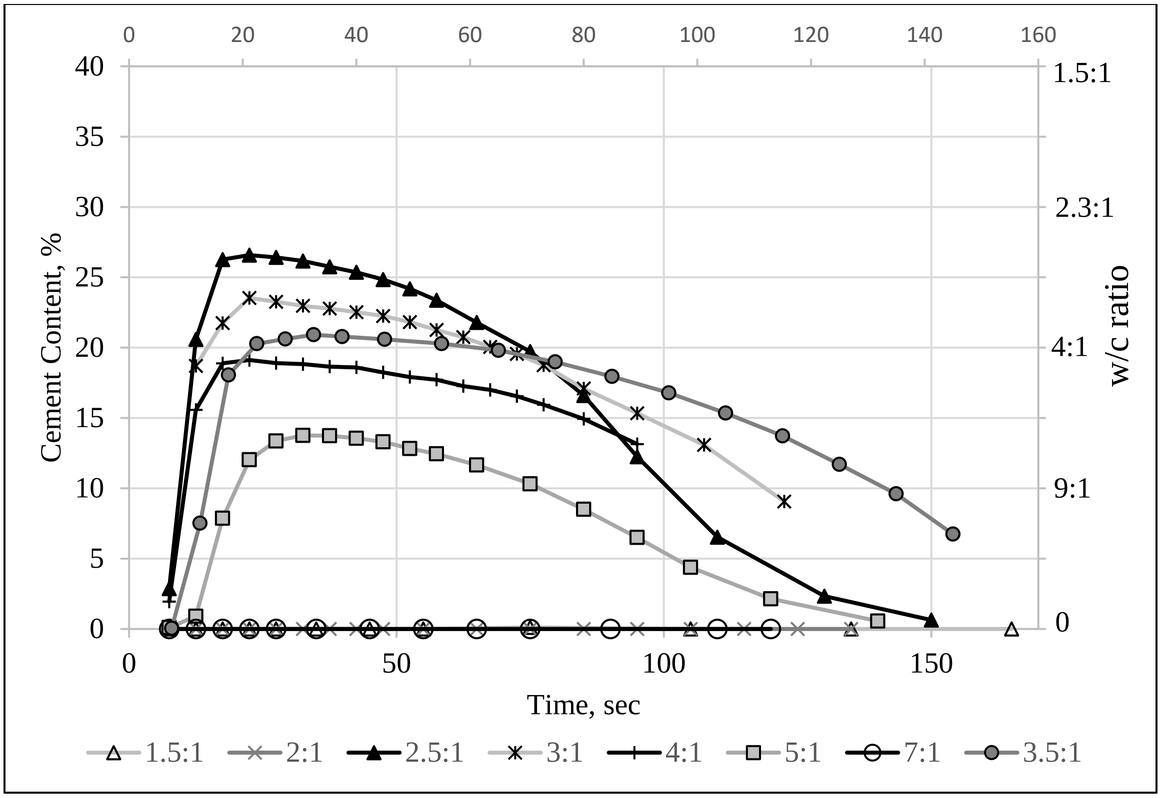

Figure 6-5: Effluent cement content versus time

The rate at which filtration occurs might not be as useful as the distance at which filtration prevents the grout from permeating further. To investigate how far the grout may travel in the soil, the percentage of cement in the effluent can be compared to either the number of pore volumes of effluent or the total volume of soil passed. Figure 6-6 shows the percentage of cement in the effluent versus the number of pore volumes penetrated for the soil used in this laboratory test. This plot shows that although the 2.5:1 mix delivers the highest amount of cement, after flushing about 2 pore volumes the cement content sharply drops off as filtration effects take over. Representing the data this way can be useful for estimating how far the grout might permeate in the field.

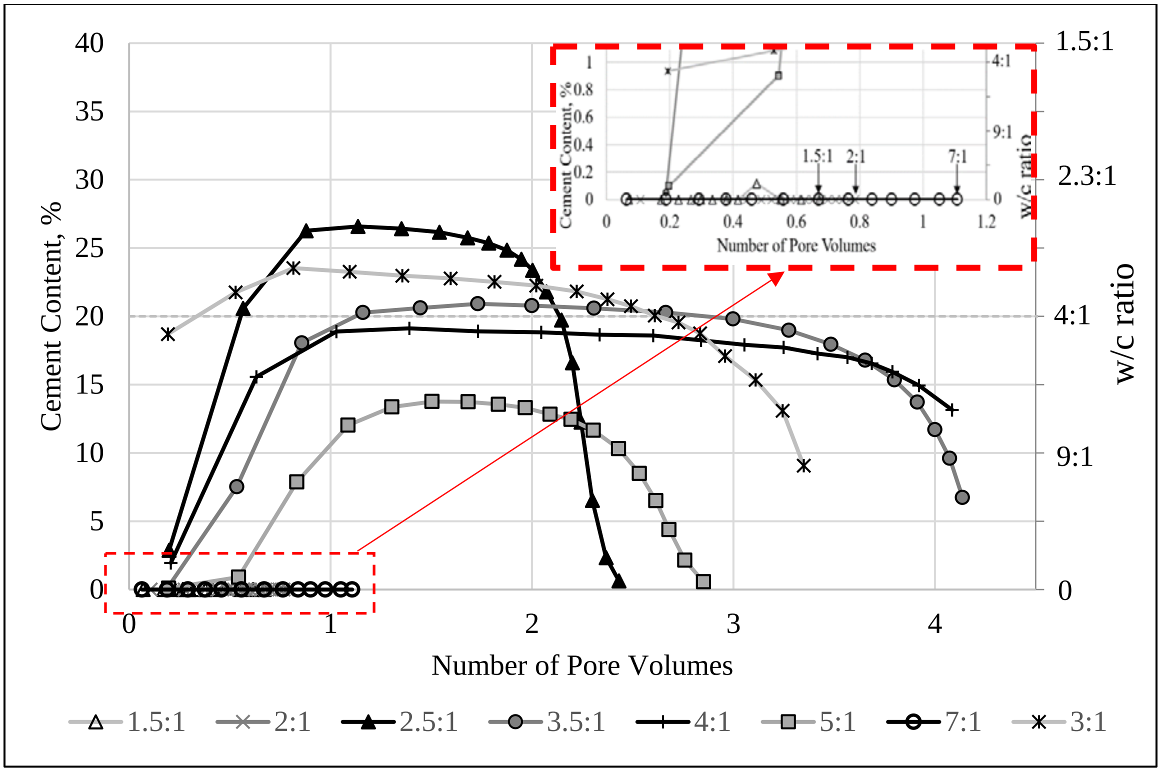

Figure 6-6: Effluent cement content versus number of pore volumes permeated

Because this is a simple, 1-D test, it cannot be used to determine exact volumes of penetration in the field; rather it can be used to compare the performance of different grout mixes. Figure 6-6 indicates that if a larger penetration distance is desired, the 3.5:1 or 4:1 grouts might be the best choice. However, the most desirable grouts for uniform penetration can me more easily identified using the normalized plot shown in Figure 6-7, which shows the effluent cement content normalized to the original cement content over the number of pore volumes of grout that have passed through the sample. This plot clearly shows that the 3.5:1 and 4:1 grout mixes would be expected to penetrate uniformly for a greater volume of soil than the other mixes. It is important to note that these are simply trends, not absolute evaluations of grout performance, i.e. w/c of 3.5:1 and 4:1 won't be the best performers for every application. The type of soil will significantly affect the "peak" performers. Additionally, additives will also significantly affect peak performers. For example, adding 1.5% SP to the 1.5:1 grout mix causes it to flow fairly uniformly through the column, whereas without SP, the mix could not make it through the column at all (less than 0.7 PVs were permeated when the test was stopped as shown in Figure 6-6). Thus, this test is mainly presented as a method of evaluating how one grout may perform compared to another and determine the optimal grout mix for a given formation.

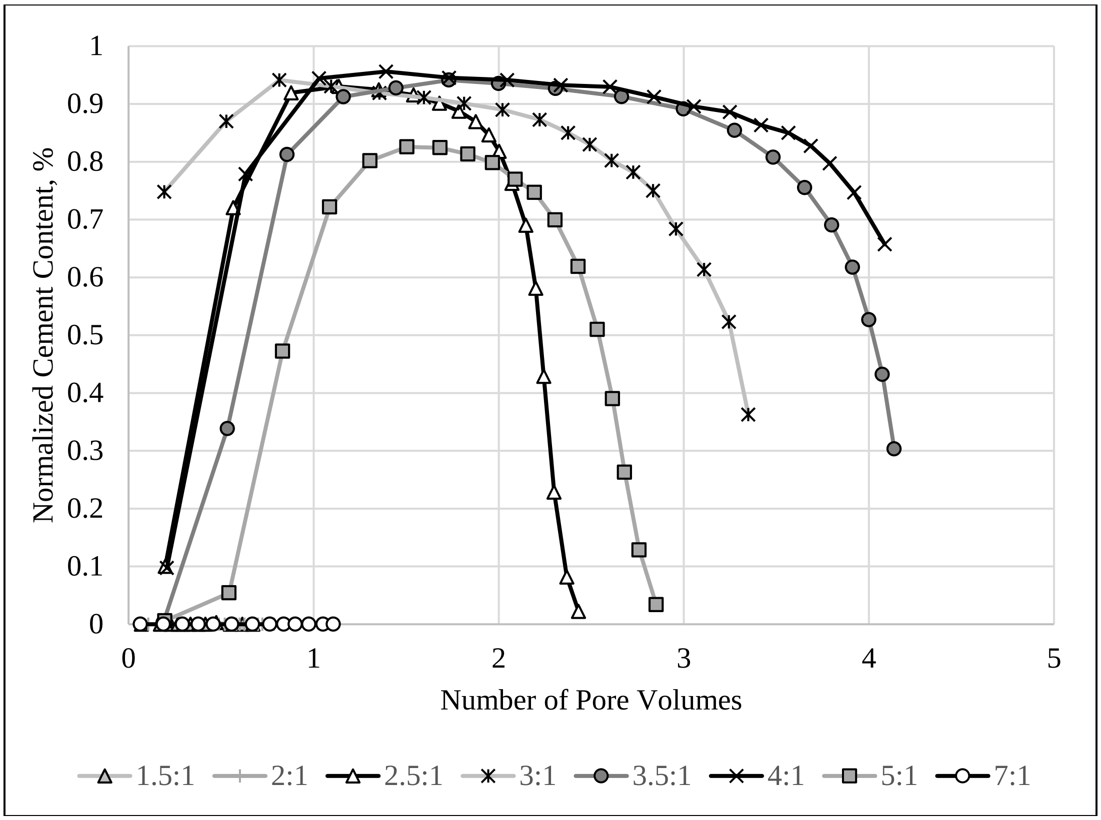

Figure 6-7: Normalized effluent cement content versus number of pore volumes permeated

These tests show that although the viscosity of the grout decreases with increasing w/c, high w/c grouts do not necessarily flow more effectively through the sand column. For this soil gradation, there appears to be a peak w/c (around 3.5:1 to 4:1) where the grout has a low enough viscosity to flow through the pore space, but a high enough stability not to filter out. This is most clearly evidenced by the fact that the three of the neat cement grout mixes that did not show any cement in the effluent include both the lowest and the highest w/c tested. It is intuitive that the low w/c materials would have difficulty permeating through the specimen under low pressure as their high viscosity hinders their ability to flow through the soil matrix. It is less intuitive that the high w/c materials cannot permeate the column, at least not while maintaining its cement content. While at least one pore volume of 7:1 grout permeated through the specimen, the effluent had untraceable cement content. At such high w/c ratios, the cement particles in the grout do not have enough interactions to remain stable. Therefore, individual particles are easily filtered out of suspension by the soil grains as the grout flows through the pores. Thus, these tests demonstrate the limitations of using viscosity as the only grout design parameter; if viscosity alone is used to evaluate the grout, an ineffective mix may be selected.

6.6 FURTHER DISCUSSION

The results of this test confirm that, at least for neat cement grouts, viscosity is not the only rheological property that controls the ability of a grout to flow through a particular soil. Filtration may occur in low viscosity, high w/c grouts because the cement particles in unstable grouts do not have significant enough interactions to prevent them from being “knocked” out of the mixture by soil particles or settled out by gravity. Thus, this simple test exemplified why understanding stability is important.

Unlike viscosity, which is a fairly well understood rheological property, stability is much less well-defined. Currently grouts are typically classified as either “stable” or “unstable”. This is a rather simple definition and a more robust classification of stability is needed. Therefore, the remainder of this study will be used to investigate stability of cement grouts.

Chapter 7: Comparing the Stability Testing, Conclusions and Recommendations

Grout internal stability plays a major role in the successful completion of any permeation grouting job. This study presented the current state of stability testing and proposed additional tests that can be performed to complement and/or substitute the current practice as well as shed light on some of their limitations and the potential impact of such limitations on the results.

7.1 Demonstrating the Importance of Stability: A Quick Experiment

As previously discussed, a stable grout is desirable for permeation grouting applications because a stable grout will permeate uniformly through the ground whereas water will separate out of an unstable grout as it is pushed through the formation. This concept can be clearly demonstrated by a simple experiment with transparent soil. “Transparent soil” is created by saturating soil-sized particles of glass/quartz with a fluid of the same refractive index. This allows light to pass through the sample without differentiating between the “soil” skeleton and the saturating fluid. Thus, the soil appears transparent. An obvious advantage of this is the ability to actually see the behavior of a grout as it permeates a soil.

Two small-scale permeation column tests were run using transparent soil. The columns were built by placing filter material followed by a wire mesh over the grout inflow port at the base of the column. Then the glass “soil” was added by dry pluviation. The “soil” was pre-weighed and carefully placed to obtain similar void ratios in both columns. Next the column was permeated with the same-RI fluid from the bottom up until the specimen was sufficiently saturated. Next, the columns were permeated with two different grouts. Note that this test was used as a visual demonstration of the effect of grout stability, not a quantitative test used for gathering data. Therefore, a highly unstable grout was chosen. A neat cement grout with water-to-cement ratio (w/c) 10:1 was permeated through the first column. Then, a w/c 10:1 grout “stabilized” with superplasticizer (SP) was permeated through the second column. Recall that in the static column bleed tests the w/c 10:1 neat grout had a bleed of approximately 65% while the w/c 10:1 grout with SP added had a bleed of 1.4%. Figure 7-1 shows the transparent soil column at the start of the tests as well as the grout mixes at the same time after the start of permeation. From Figure 7-1, it can be seen that while the stable mix retains a uniform color throughout the height of the column, the unstable grout shows significant loss of color with height. This is due to the cement particles being filtered out of the unstable grout and only the water progressing as it is pushed through the column. Because both mixes started with the same w/c ratio, the unstable grout will result in a much higher cement content at the base of the column than the stable grout and a much lower cement content at the top of the column. Thus, the unstable grout produces a column with higher strength than designed for close to the injection point as well as lower strength and higher permeability than designed for farther from the injection point. The results from this quick test indicate the importance of grout stability and the need for a better understanding of this critical property.

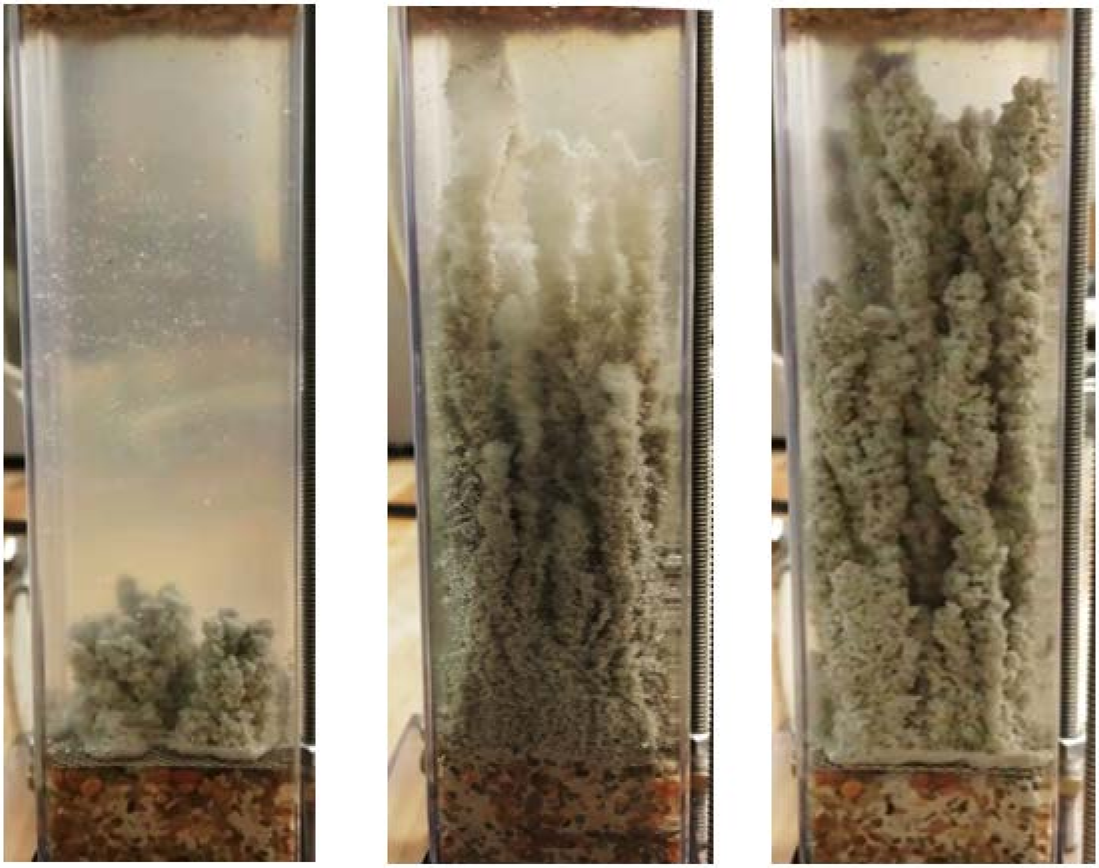

Figure 7-1: Start of test (left) Unstable grout mix (center) More stable grout mix (right)

7.2 FINAL THOUGHTS ON STABILITY TESTING RESULTS

In grout, internal stability is the result of inter-particle interactions between the cement particles that create a structure within the grout. This structure resists downward settlement of the cement particles and the upward movement of water. For low w/c grout, such as w/c 1.5:1, the high percentage of cement particles in the grout results in significant structure. This is what causes it to behave more stable. Adding superplasticizer neutralizes the forces between particles and promotes dispersion of clumps. Neutralizing the inter-particle forces results in a desirable reduction in the viscosity of the grout but enhances the chances of particle settlement. On the other hand, breakage of clumps will increase viscosity but decrease particle settlement due to smaller individual particles. Because the cement particles are settling as individual particles rather than clumps, they can achieve a higher final concentration at the bottom of the bleed column test, even though it might take longer to reach this point (Figure 4-13). Based on this balance between positive and negative impact of SP on rheology and internal stability, it is very critical to determine the optimal SP content for a given w/c ratio grout through an appropriate testing that can allow us to capture the kinetics of the concentration of the grout suspension.

7.2.1 Impact of Initial Setting Time

The addition of superplasticizer affects more than just the rheology and internal stability of a grout. As presented in Chapter 2, the addition of SP significantly affects the hydration of a grout (Figure 2-5, Figure 2-6 and Table 2-1). For a w/c 0.5 grout, adding 2.0% SP was shown to increase the assumed initial set time from 7.5 to 37 hours. This marked delay in set begs the question, should stability of a grout with added SP be monitored longer than the traditional two-hour stopping point? Figure 3-6 shows that for a w/c 3:1 neat cement grout, practically no additional settlement occurs after approximately 60 minutes. However, when superplasticizer is added (Figure 3-7), changes are occurring even past 200 minutes. Figure 7-2, which shows the change in w/c ratio over the height of a 250 mL cylinder over a period of two hours for a w/c 3:1, SP 2.0% grout, also indicates the need for a longer test period. After 120 minutes, significant changes are still occurring in the grout, and therefore the state of the grout after that amount of time should not be considered representative of the final state of the grout. In conclusion, given the delayed set time that results from adding superplasticizer to a grout, an increased stability time is necessary for getting a more complete understanding of the behavior of the grout.

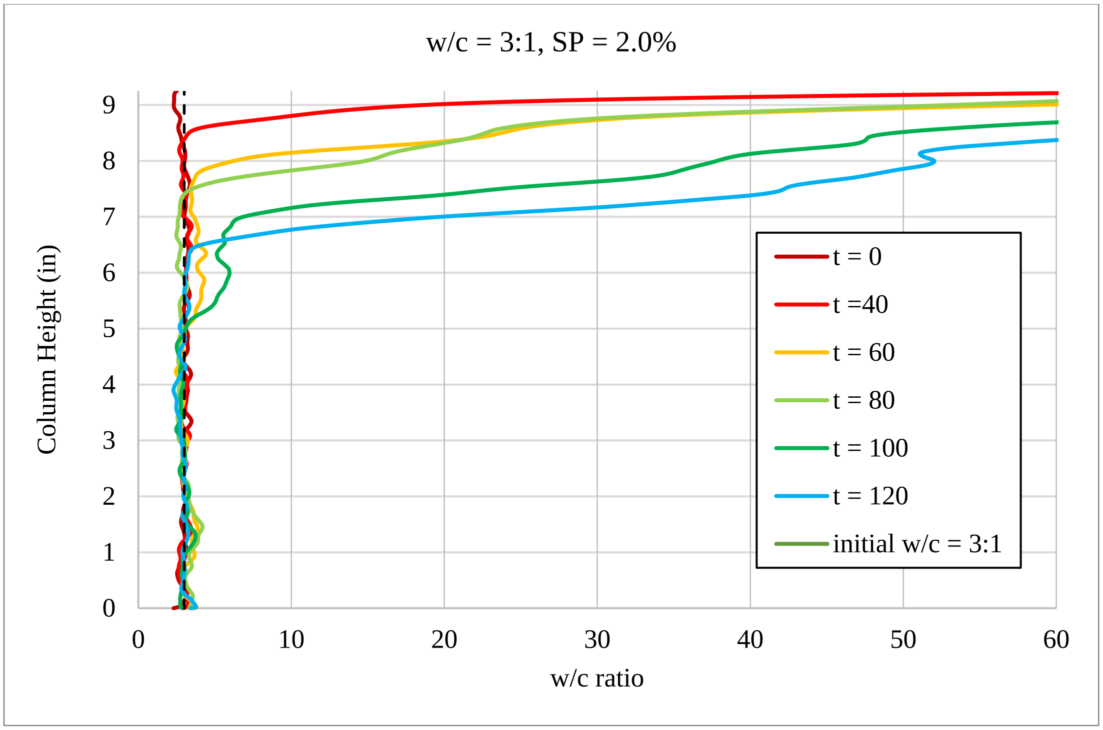

Figure 7-2: Settlement of a w/c 3:1 grout with 2.0% added SP over a period of 120 minutes (image analysis results)

7.2.2 Comparing Static Stability Tests

Image analysis testing allows for more "insight" on the internal stability of the grouts. This allows for better understanding of the changes in w/c content over time across the column and help shed light on the impact of SP beyond what the results of a standard bleed test would provide. One of the first observations is that adding SP has a much greater impact on the stability of higher w/c grouts than lower w/c grouts. This can be seen in Figure 7-3 by comparing grouts with w/c ratios of 1.5 and 3 with a range of SP%. The grout with w/c ratio of 1.5 didn't show a major difference stability with increasing SP% while the w/c ratio of 3 grout showed maximum stability at SP = 2.5% with lower stability at higher and lower SP content. If we did a similar comparison between results presented in Figure 3-6 and Figure 3-7, the impact of 2.5% SP seems significant to all w/c ratios decreasing the bleed to below 0.4% for all mixes. This bias in the results is due to the definition of bleed in standard tests as "clear water" on top of the column. For example, the grout with w/c ratio of 3:1 and SP2.5 would have been reported to have 0.2% bleed based on standard column bleed test (Figure 3-7) while the results in Figure 7-3 clearly indicate that the minimal "clear bleed water" is not a true indication that the grout remains stable and the w/c ratio varies with height.

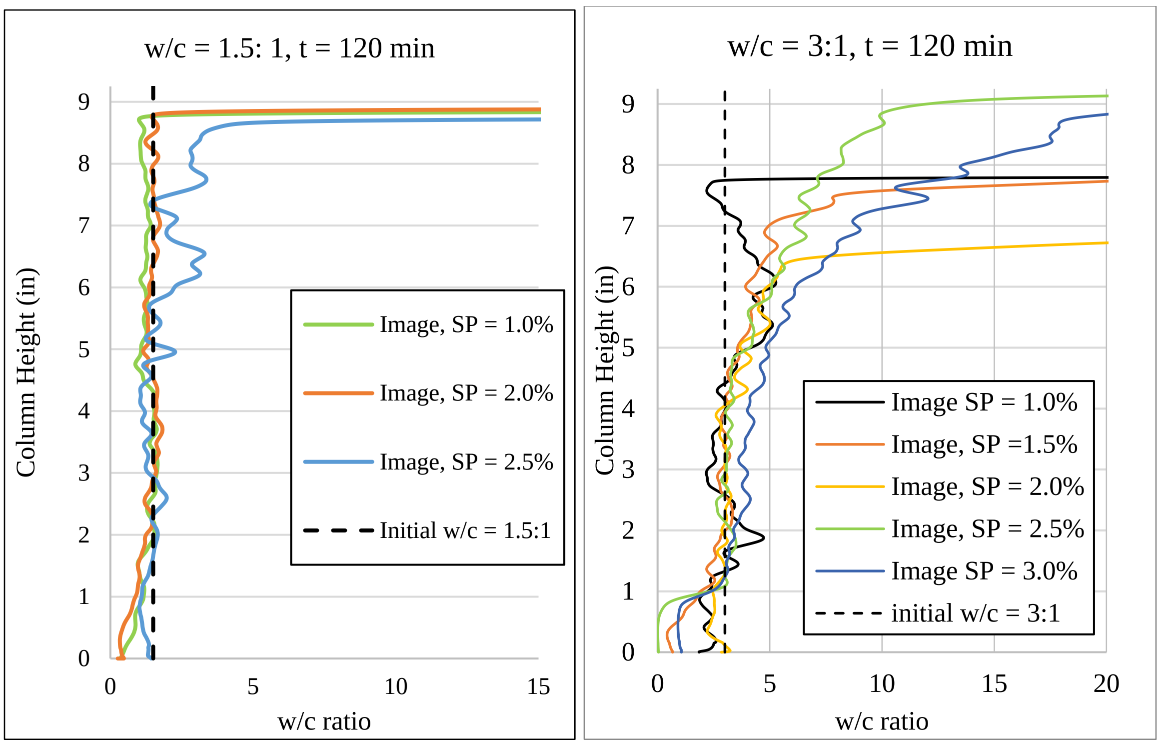

Figure 7-3: Impact of adding SP on w/c 1.5:1 grout versus w/c 3:1 grout (image analysis results)

7.2.3 Static versus dynamic versus New Grout Filtration Tests

Figure 6-6 showed an interesting trend with grouts at w/c ratio of 3.5:1 and 4:1 outperforming other w/c ratios in terms of retaining most of their soils as flowing through a porous medium. However, all static and dynamic stability tests showed a continuous decrease with stability as w/c ratio increased (although all the tested w/c ratios were equal of above 3:1 in these tests). Figure 7-4 shows the results from the new grout filtration test for grouts at w/c ratio of 1.5:1 (with and without SP) and 3:1. The results show that the 1.5:1 grout as instable (the effluent didn't have any cement content) while 3:1 grout having intermediate stability and the 1.5:1 with SP1.5 grout to be the best performing grout. These results are again contradictory with the static stability tests which showed minimal impact of SP on stability of the 1.5:1 grouts (Figure 7-3).

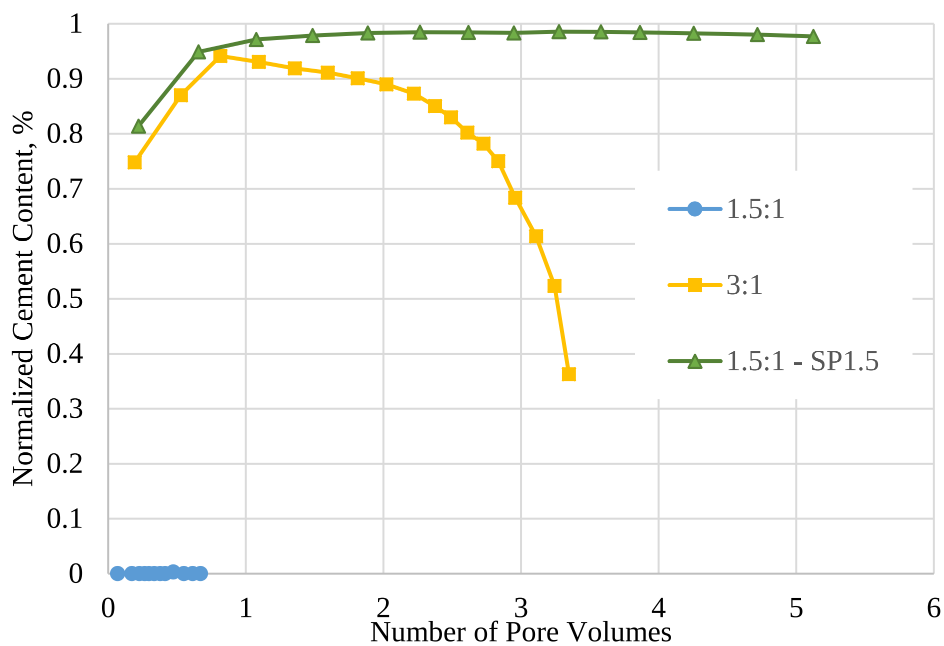

Figure 7-4: Results from New Grout Filtration test for w/c ratio of 3, 1.5 and 1.5 with SP1.5

7.2.4 Final Conclusions

The study presented a comparison of the standard column bleed test to additional proposed testing method for evaluating the internal stability of grouts. The current standard column bleed test is a very easy to run test but can provide misleading information on stability of grouts. Below are some of the major observed limitations, how the new proposed tests addressed them:

1) The bleed test is run for 2 hours regardless of type of cement, w/c ratio or percentage of SP. However, the hydration process is dependent on the chemistry of the cement (including type of cement and additives used) as well as w/c ratio (at least in the w/c ratios of interest for grouting). Therefore, the time of the testing should be adjusted based on the initial setting time.
2) The bleed test only accounts for the percentage of clear water on top of the column. However, this measurement doesn't account for the variation in the grout concentration throughout the column and can lead to over estimation of the stability of the grout. The newly proposed image analysis process allows for determining the distribution of the grout concentration across the height and monitor its progression over time. The imagine analysis provided additional information on the impact of SP on dispersion versus settlement of the cement as well.
3) The bleed test is a static test where the grout is not subjected to any shearing after being placed in the cylinder. However, the grout is rarely in static state shortly after mixing in the field until it reaches its final destination in the formation. The Dynamic Stability Tests were able to validate (albeit mostly qualitatively) that the internal stability of the grouts is higher if subjected to shear as it reduces the settlement of particles under their own weight.
4) The bleed test, along with the image analysis and dynamic filtration tests, only measure internal stability of the grout without addressing the impact of the formation properties on whether filtration would occur or not. A new Grout Filtration Test is proposed that allows for testing the internal stability of grouts as they permeate through a thin layer representing the formation targeted by grouting. This test captures the internal stability of the grout as it flows through

a porous medium and therefore, can be a much better indicator of how grouts would perform in the field. While it is not part of the focus of this study, the new Grout Filtration Test accounts for the grout rheology as well as its internal stability (both factors are critical for a successful grouting).

7.3 RECOMMENDATIONS FOR FUTURE RESEARCH

The current work presents a major advancement towards better understanding and characterizing internal stability of grouts. However, to be able to apply the conclusions on a wider scale, the following future work need to be completed:

1) Additional refinement to the newly proposed testing setups, procedures and analysis can allow us to develop the theory to back up the observations and empirical correlations developed.
2) Complete the set of tests to include the same range of w/c ratios and SP content for all tests to allow for better comparison over the complete range.
3) Perform the tests on multiple cements/microfine cements to expand the results to additional materials and allow for incorporating the variation in the physio-chemical properties of the material into the results. This also applies ot using different formations in the new Grout Filtration setup to capture the impact of the formation properties.
4) Develop industry standards for new testing to improve the characterization of internal stability of grouts using the expanded database.

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