

DEVELOPING AN ALTERNATIVE HARDENED CONCRETE AIR VOID
CHARACTERIZATION METHOD

by

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DEDICATION

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ABSTRACT

This study presents the development and analysis of an alternative characterization method for the hardened air void system in concrete. This study uses ASTM C457, “Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete” as a baseline characterization method and attempts to thoroughly reduce the long sample preparation and characterization time required to complete the test. An investigation on the sample preparation requirements was first completed, in which new optimized cutting and polishing procedures were developed, along with improved contrast enhanced procedures, to accommodate the alternative characterization method. The new characterization method also eliminates the reliance on user-judgement and reduces human error. The new characterization method uses a Keyence digital optical microscope to analyze the processed hardened concrete sample, and automatically counts and measures the air void system. The microscope then provides an excel report on the analyzed system, which can then be processed to produce key hardened air void system information such as the maximum and minimum air void size, dispersion, circularity, air void percentage (amount of air) and further information such as the spacing factor and specific surface can be calculated from this data. This entire process was developed by producing four concrete mixtures with different air entraining dosages. The air entraining dosages were added at various ranges sufficient to provide a low (3-4%), moderate (5-7%), and high (> 8%) fresh air contents in the

produced mixtures. A non-air entrained mixture was also included as a reference in the study. The mixtures were then tested for slump (ASTM C143), air content (ASTM C138), unit weight (ASTM C138), Super Air Meter number, and 7 and 14-day compressive strength (ASTM C39). Additional hardened samples were created from each mixture such that they can be cut, polished, and prepared for hardened air void analysis. A procedural analysis was completed to determine the minimum processing steps and time necessary to produce a surface that can be analyzed by both ASTM C457 and the alternative characterization method. The new alternative characterization method showed that testing laboratories would save time, money, and better consistent results by reducing human decision-making. The use of the Keyence VHX-7000 microscope, along with sample cutting, polishing, and color enhancing procedures discussed in this investigation, was found to give operator flexibility of running multiple analyses thru the use of the software in the Keyence microscope. Single run analysis with data stored within the file allows the operator to adjust boundaries, change the air void parameters such as circularity, max and min diameters, and re-run the test in minutes instead of hours.

1. INTRODUCTION

1.1 Background

The impact of the air void system depends on the total volume, size, and dispersion of the air voids in the concrete system, as well as their compatibility with various material properties. Properties affected by the air void system include workability, cohesion, density (fresh and hardened), strength, finish ability, and freeze-thaw resistance. The most important of these in terms of highway concrete for long-term performance is the resistance to freeze-thaw durability. To effectively provide freeze-thaw resistance, the air void system must have a total volume of empty air voids that equals or exceeds the volume of water or ice not accommodated by space in the capillary pore system (Hover, 2006). Additionally, the air voids must be dispersed throughout the cement paste so that nearly all of the paste is within an air-void system zone of influence (Power, 1945, Powers, 1954, Powers, 1954, Natesaiyer & Powers, 1992). The current method for studying the entrained air void system in hardened concrete is ASTM C457 – “Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete” (ASTM Standard C457/C457M C09). This method, initially proposed by Brown and Pierson (Brown & Pierson, 1950), consists of polishing a hardened concrete sawn sample and placing it under a microscope. The operator systematically makes measurements by counting the air voids that come into view. Statistical estimates are produced from the measurements that define the air content, paste content, air-void size distribution, and spatial dispersion. There are three methods described in ASTM C457: i) the Rosiwal linear traverse technique (Procedure A) and ii) the modified point-count method (Procedure B) resulting in equivalent results, and iii)

contrast-enhanced method (Procedure C). Although ASTM C457 is the standard test method for characterizing the entrained air void system in concrete, some significant sources of variability and uncertainty still exist in the test results. The major sources of variability include precision and bias, inherent statistical uncertainty, level of magnification of the microscope, and operator subjectivity. This procedure is subject to human error, laborious, and time-consuming because it relies on the user to make hundreds of critical decisions per sample. The ASTM C457 Procedure C is also costly and requires significant sample preparation, including lapping, polishing, and dying.

1.2 Statement of Problem

Due to the many drawbacks of human error, time consumption, laborious, most laboratories do not perform ASTM C457 and generally only rely on fresh air content values. Because of these effects, there is a need to develop a method to fully characterize the air void system in hardened concrete that is more reliable, less time consuming, cost effective, and easy to complete.

1.3 Objective of Research

The objective of this research is to develop and analyze an alternative test procedure to characterize the entrained air void system for hardened concrete. The new procedure utilizes a Keyence microscope with onboard counting and measuring software to make sophisticated automated measurements of a prepared sawn or drilled core sample. Additional focus was placed on minimizing the sample preparation time required in ASTM C457 Procedure C.

1.4 Research Significance

The information developed in this thesis can help laboratories improve their ability to characterize the entrained air void system of hardened concrete samples. The possible benefits include the following:

1. A new hardened air void characterization method using a Keyence digital optical microscope.
2. More reliable hardened air void characterization (less reliant on human decision making).
3. The new characterization method can be used as a performance indicator for design and quality assurance purposes.
4. More robust and user-friendly hardware.
5. The ability to do in-house analysis (less reliant on costly service laboratories).
6. A comprehensive guide for performing hardened air void analysis including sample preparation, testing, and interpretation of results.

1.5 Thesis Organization

The first part of this report (Chapter 1) is the introduction. It comprises of a background discussion on the air void system, the statement of problem, the objective of this research, the significance of this research and organization of this research report.

The second part (Chapter 2) of this report presents a literature review on the air void system (fresh and hardened) and the different characterization methods to date. The materials and methodology (Chapter 3) provides a detailed account of the materials, mix proportion, equipment and procedures employed in carrying out the various laboratory research. Chapter 4 presents an in-depth discussion on the development and improvement

of the hardened sample preparations steps. Chapter 5 presents and discusses the results obtained from ASTM C457 Procedure A, Procedure B, and the alternative characterization method using the Keyence microscope. This section includes a statistical comparison between the three methods and a discussion regarding advantages and disadvantages of the new alternative characterization method. Lastly, Chapter 6 is the conclusions and lessons learned.

2. LITERATURE REVIEW

2.1 Freeze Thaw Durability of Concrete

The ability of concrete to survive a cold and wet environment is primarily dependent on the air void system in the hardened concrete (Taylor et al., 2021). This is why it is extremely important to accurately and quickly characterize the air void system as promptly as possible. Many aspects of the concrete mixture can influence the air void system which is discussed in the following sections.

2.2 Mixture Ingredients

The amount of free water that can then freeze in the paste capillaries affects the risk of damage to excessive freeze-thaw cycles. A reduction in the volume and size of the capillary voids enhance the freeze-thaw resistance of concrete because there is less freezable water in the system, and the freezing point is lowered in smaller voids due to surface tension effects (Pigeon & Pleau, 1995). The pore system, in turn, is influenced by both the physical and chemical characteristics of the cement and the degree of hydration. Over time and with continued hydration, the continuity of the capillary pores in the paste decreases due to the formation of hydration products (Powers, 1960; Garboczi & Bentz, 1995). Another effect of cement chemistry is that air content may increase with increasing alkali levels because more of the air-entraining agent remains in solution during mixing. However, not all researchers agree with this hypothesis (Pigeon et al. 1992; Greening, 1967; Pistilli, 1983). With proper curing, concrete containing blended cement, slag, or fly ash is reported to have enhanced long-term strength and durability characteristics, partially due to the concrete having a more refined capillary pore system (Pigeon & Pleau 1995, ACAA 2003). The presence of reactive carbon in fly ash may cause air-void stabilization problems and, therefore a negative impact on the freeze-thaw

resistance. Due to their porosity, the carbon particles absorb the air-entraining agents and reduce their effectiveness (Karakurt & Bayazit, 2015; Klieger & Perenchio, 1976; Ramachandran, 1995). The formation of the entrained air-void system can also be affected by concrete aggregates during mixing, as a function of the aggregate size, gradation, shape, and texture, as well as the amount of contaminants (Verbeck & Landgren, 1960; Waugh, 1961; Gaynor, 1967; Powers, 1975; Kaneuji et al., 1980; Hudec 1989; Page & Page, 2007). Because these factors also significantly influence the form of the interfacial transition zone (ITZ), they, in turn, affect concrete freeze-thaw resistance (Rhoades & Mielenz, 1946; Helmuth 1961; Dunn & Hudec, 1965; Sawan, 1987; Chatterji & Jensen, 1992). Fine aggregates can also influence the air-void system (Kosmatka and Wilson, 2016). Increasing the amount of medium-sized fine aggregate tends to entrain more air (PCA, 1962) because the aggregates provide a screen to hold bubbles in the paste (Deno, 1966). On the other hand, the use of very fine particles ($> 150 \mu\text{m}$) results in a reduction of entrained air (Kosmatka & Wilson, 2016). The benefits of entraining air were accidentally discovered in the late 1930s when researchers observed that concrete blended with portland and natural cement containing “crushed oil” was more resistant to surface scaling (Jackson, 1944). Researchers also observed that concrete pavements with certain cement were more durable in a freezing environment than other pavements. Researchers found that these more durable concretes had cement that was manufactured with grinding aids, including beef fat, calcium stearate, and fish oil, which acted as air-entraining agents. Many different admixtures have been used as an air-entraining agent with varying results in air bubble size and stability, such as vinsol resin, wood resin, tall oil, vegetable oil acids, and synthetic detergents. Other chemical admixtures can also be

used in concrete combined with air-entraining admixtures to modify concrete properties such as workability and setting time. The interactions between these admixtures and air entrainers can be critical because they may impact the air-void system.

2.3 Paste Porosity

The formation and source of pores in a hydrated cement paste system has a strong influence on the freeze-thaw resistance. Calcium silicate hydrate (C-S-H) is a principal product of cement hydration and Powers (1955) recognized that this product determines the properties of cement paste. The porosity of C-S-H is about 25%, with pores, which are known as gel pores, which are a few nanometers in size (Cordon, 1966). Because of the pores small size water cannot freeze inside them at typical temperatures, although the water in them may be supercooled (Powers, 1958). During hydration, the voids left behind as the mixing water is removed from the system are known as capillary voids (Jennings et al., 2008). The sizes of these capillary pores range from approximately 5 nm to about 1 μm . The volume of capillary pores depends on both the degree of hydration and the original water/cementitious material (w/cm) ratio (Verbeck & Klieger, 1956; Powers, 1960; Cordon, 1966; Aligizaki, 2006; Jennings et al. 2008). Air voids in hardened cement paste can be considered as either entrapped or entrained.

Entrapped air voids occur due to insufficient consolidation. These air voids are typically greater than 3/64 in. (1 mm) in size and are irregular in shape (ASTM C125). Entrapped air voids are typically isolated from other entrapped air voids and provide no benefit to the concrete (Powers, 1954). Entrained air voids are typically 10 to 1,000 μm in diameter and are mostly spherical. Entrained air voids are discrete and uniformly distributed, and therefore have little effect on concrete permeability. A comparison of the sizes of pores and concrete components is presented in Figure 1.

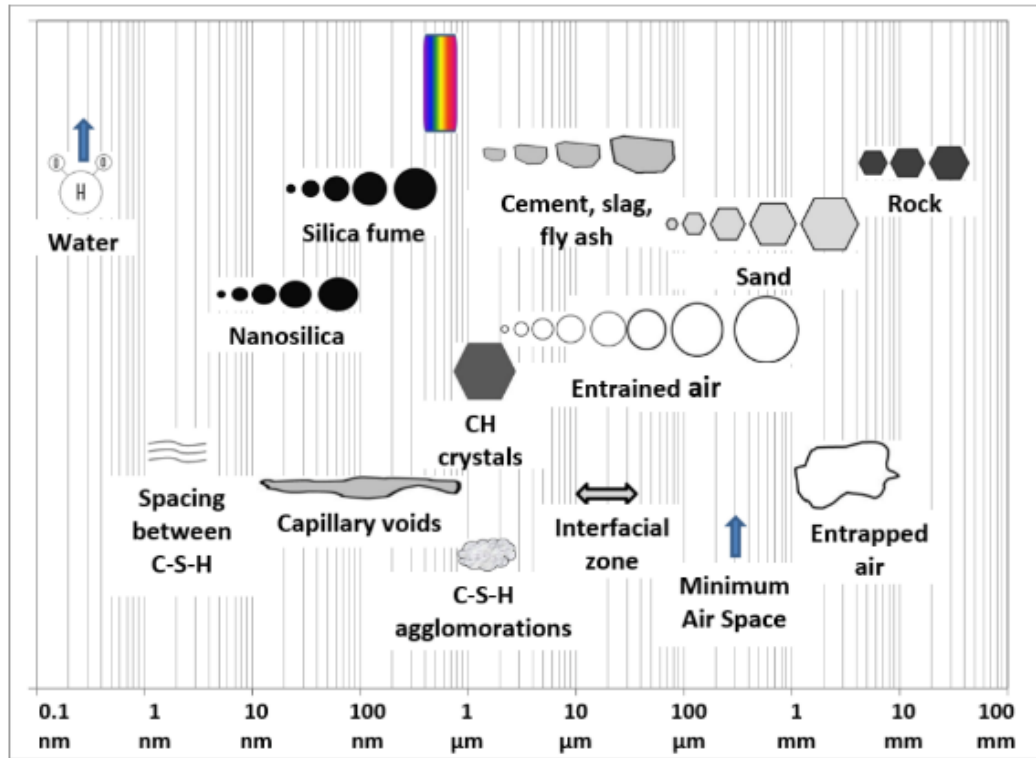


Figure 1. Size of Elements in Hardened Concrete (Taylor et al., 2021)

Table 1 presents a summary of the type of pore and their function related to freeze thaw durability performance of concrete.

Table 1. Pore Type and Function Related to Freez-Thaw Durability (Aligizaki, 2006; Mehta, 198

Pore Type	Diameter	Location	Effects on Freez Thaw
Gel Pore	Intra-gel: <0.6nm	Inside C-S-H	Water in gel pores may travel into capillary pores to reduce solution concentration due to osmotic pressure
	Inter-hydrate: 0.1 - 100 nm	Space between C-S-H and CH	
Capillary Pore	Small: 2-50 nm	Between cement grains and products of hydration	Ice crystals form in capillary pores (> 50 nm) and may generate stresses that damage the paste
	Large: 1-10um		
Air Voids	Entrained: 10-1,000um	Between cement grains and products of hydration	Provide boundaries for water to be forced out in capillary pores due to ice formation; limit hydraulic pressure
	Entrapped: 1 mm		No effect

2.4 Mechanisms of Freeze Thaw Action on Hardened Concrete

Unlike most materials, which become denser during freezing, water expands and becomes less dense when the temperature falls below 32°F. The freezing point of water inside concrete pores depends on the pore sizes, the internal pressure, and the presence of solutes. At higher pressures (> 1 atm), the freezing temperature decreases. The presence of solutes such as K^+ , Na^+ , Ca^{2+} , and Cl^- in water also depress the initial freezing point (Akyurt et al., 2002). The damage due to freezing action is mainly dependent on the degree of saturation (Fagerlund, 1993). Damage is unavoidable if the critical degree of saturation of approximately 86% for all of the voids is reached (Li et al., 2012). The following mechanisms are believed to be primarily responsible for freeze-thaw deterioration (Cordon 1966; Plum & Hammersley 1984):

- Hydraulic pressure generated by freezing in capillaries.
- The diffusion of supercooling gel water into capillaries after freezing.
- Osmotic pressures resulting from the partial freezing in capillaries of solutions that have local concentration differences.
- Differential strains due to localized shrinkage and swelling.

All of the mechanisms are influenced by the ability of fluids to travel through the system, as well as the saturation of the pores. It is difficult for water to penetrate spherical air voids through capillary action due to the nature of surface tension forces; however, external pressures in the pore system may drive fluids into an air void. Damage, then, is significantly influenced by the fluid transport characteristics of the system, as well as the size, shape, and connectivity of the pores (Taylor et al., 2021).

2.5 Effects of Air Voids on Mechanical Properties of Concrete

The mechanical properties of concrete are influenced by each component in the matrix of the mixture. While the major impact of air voids on concrete is on freeze thaw resistance, air voids also have an impact on mechanical properties such as compressive strength and drying shrinkage. It is well understood that increasing the air content of concrete may cause a commensurate loss in strength, which, as a result, may impact the durability of concrete (Sutter, 2007; Kosmatka & Wilson, 2016; Cross et al., 2000). A one percentage point increase in air content reportedly leads to a 2% to 6% reduction in compressive strength (Whiting & Nagi, 1998). The dominant factor behind this reduction in strength is likely that air voids provide a shortcut for crack propagation because they have no strength themselves. Clustering has also been reported as responsible for reduced strength as a function of the air-void system, due to insufficient bonding between the aggregates and the cement paste.

2.6 Air Void System

Total air content is defined as the total volume of air voids expressed as a percentage of the bulk volume of concrete. This parameter has been the basis of acceptance testing since the AASHTO T152/ASTM C231 test method was developed. Despite the fact that the critical parameter is the spacing factor, total air content was a satisfactory measure because the correlation between the two parameters was good. With changes to system chemistry using different materials, this correlation has been diminished, increasing the need to measure other parameters that indicate potential durability, such as the Super Air Meter (SAM) number. Powers (1954) suggested that air voids perform two functions in concrete: limit the hydraulic pressure in the paste during the early age of freezing and limit the formation of ice bodies. Based on the first function,

the space between air voids, also expressed as the spacing factor, becomes the most important factor because, as water expands with decreasing temperatures (below $\sim 39^{\circ}\text{F}$), the water has to flow through saturated pores to find empty air voids before it freezes. Even with the same air content, the spacing factor can be very different between mixtures. Clustering is a phenomenon in which entrained air bubbles preferentially collect around aggregate particles. This clustering reduces the bond between the paste and the aggregate (Lamond & Pielert, 2006; Hover, 1989; Sutter, 2007), and so the phenomenon has the potential to reduce compressive strength (Kozikowski et al., 2005; Cross et al., 2000). However, Riding et al. (2015) reported no correlation between clustering and strength. Regardless, a system of small, stable bubbles close together is desirable to provide a sufficient freeze thaw resistance for concrete exposed to cold, wet weather. Compared to capillary pores, spherical voids are much more difficult to fill with water under capillary action. This has the effect of reducing the degree of saturation of the system at any given time. Unsaturated bubbles provide a space into which expanding water and ice can move, without incurring pressures within the hydrated cement paste, which can ultimately crack and fail the concrete. The bubbles need to be close together to reduce the distance that expanding water has to travel, and they need to be small to reduce the impact on the mechanical properties of the mixture. The principal objective of using air-entraining admixtures (AEAs) in concrete is to provide and stabilize an air-void structure that is able to protect concrete from the deterioration brought by freezing and thawing. AEAs are surfactants that are readily adsorbed at air-water or solid-water interfaces and serve as bubble stabilizers. Figure 2 provides a depiction of this stabilization effect.

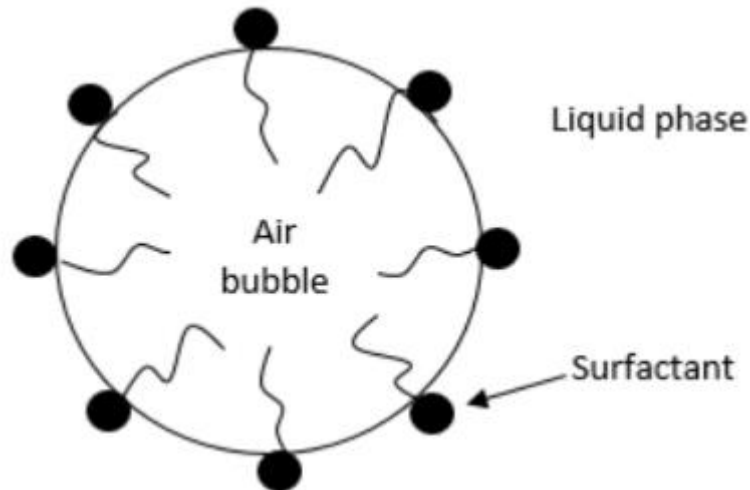


Figure 2. Stable Air Bubble with Air Entraining Admixture (Taylor et al., 2021)

There are currently two explanations as to how the air-void system forms during mixing. One explanation is that, during concrete mixing, air layers are trapped between the folding surfaces of the concrete, and the fine aggregate acts as a “three-dimensional screen” to hold air bubbles within the network of particles (Dolch, 1996). Powers (1968) described another process in which air bubbles are formed in a vortex as the mixture is stirred. Mixing provides the energy to build the interface between air and liquid in large air voids and then splits the large voids into smaller bubbles.

2.7 Measuring and Characterizing the Air Void System

For fresh concrete, the main challenges in measuring the air system are the timeliness of the results, measurable parameters, accuracy, and ease of use. For hardened concrete, the challenges are accuracy, measurable pore sizes, resolution, sample preparation, and comparison between methods. Three conventional methods are used in the field to determine total air content for fresh concrete: pressure meter method, volumetric method, and gravimetric method. The AASHTO T 152/ASTM C231 pressure meter method is the most widely used method for determining total air content. Based on Boyle’s law, applying pressure to a known volume of concrete compresses the voids in it,

which in turn reduces the volume of the concrete itself. The change in pressure of a known volume and the initial pressure of a vessel that is connected to the concrete allows the calculation of the volume of air in the concrete based on the assumption that only the air in the system is compressible. This method is not applicable for concrete that uses high-porosity aggregates. The AASHTO T 196 volumetric method is used to determine the total air content of concrete with lightweight aggregate. For the volumetric method, a container is filled with concrete, water, and isopropyl alcohol and inverted a number of times to release the air from the concrete to allow it to collect in a calibrated glass vessel at the top of the equipment. Air trapped in the aggregate does not affect the test results (Lamond & Pielert, 2006). This method is more operator-sensitive and tedious than the pressure meter method (Pigeon & Pleau, 1995; Lamond & Pielert, 2006).

Underestimation of air content is a potential problem because extracting the smallest air voids from the paste can take more than an hour (Ozyildirim, 1991). The AASHTO T 121 gravimetric method is an indirect method based on comparing the measured unit weight of concrete with the theoretical unit weight (Pigeon & Pleau, 1995; Lamond & Pielert, 2006). The gravimetric method is not appropriate for concrete with lightweight aggregate because the moisture content and specific gravity of the aggregate can be variable. Beyond the three conventional methods, a newer test method uses a different technology. The SAM, has the convenience of the pressure method, while reporting numbers that have been shown to correlate with Freeze-Thaw performance (Ley et al. 2017).

The most commonly tested parameters for expressing the characteristics of an air-void system in hardened concrete are total volume of air, specific surface, and spacing

factor. Conventionally, the average chord length of voids is used to calculate the specific surface and spacing factor. With more recent technologies, such as flatbed scanners used with bubble counter analysis software (Anzalone, 2007) and x-ray imaging (Sutter, 2007), analysis of air-void connectivity, clustering, and spatial distribution is also possible. Currently, most existing test methods only measure air voids a few microns long and longer. Based on these measurements, it is widely accepted that the spacing factor of an air-void system should typically be less than 0.008 in. (0.20 mm) to provide the greatest protection against freeze thaw damage (ACI Committee 221R 1996).

However, recent studies have found that much smaller air voids and spacing factors also play an important role in free-thaw durability performance (Zhang et al. 2012; Ng et al. 2014; Liu et al. 2014). The ASTM C457 microscopical method has long been a reference method in determining the spacing factor and specific surface of air voids in concrete. The linear traverse and point count techniques use a microscope and user interpretation to evaluate an air-void structure. Although this is the most common practice to characterize the hardened air void system of concrete samples, it is the most tedious and susceptible to user error. All three procedures outlined in the standard require extensive cutting and polishing of the sample surface before the sample can be analyzed under a microscope.

3. MATERIALS AND METHODS

Chapter 3 provides descriptions of the materials included in this study as well as the procedures used to accomplish the research objectives. Figure 3 illustrates the flow chart of experimental design followed during this study.

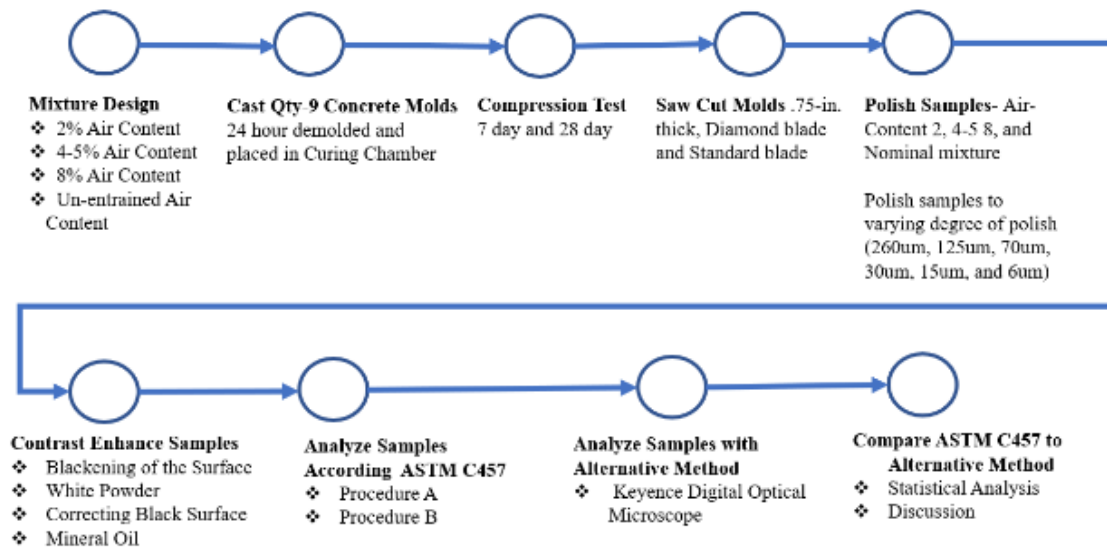


Figure 3. Flowchart of Experimental Design

A Type I/II Portland cement was used for all mixtures. This cement was obtained from a local Central Texas cement producer and was referred in this entire work as PC-1. Table 2 shows the physical and chemical properties of the cement taken from the datasheet.

Table 2. Chemical and Physical Properties of Type I/II Cement

Chemical Analysis %		
Item (%)	Test Results	Spec. Limit
MgO	0.9	6.0 max.
SO3	2.8	3.5 max.
Loss on Ignition	3.2	3.5 max.
Insoluble residue	-	1.5 max.
CO2	2.0	
Limestone	4.7	5.0 max.

CaCO ₃ in limestone	96	70 min.
Physical Test		
Air content of mortar (volume %)	10.1	12 max.
Blaine fineness (m ² /kg)	347	260 min.
Mesh 325 (45 microns) % through	92.5	
Autoclave expansion (%)	-0.006	0.80 max.
Time of setting - Vicat test (minutes)		
Initial- not less than or more than	123	45 - 375
Final	209	
Compressive strength		
1-day, minimum MPa	14.8	
3-day, minimum MPa	26.8	12
7-day, minimum MPa	35.1	19
28-day, minimum MPa	46.5	
False set (OPTIONAL)	80	50 min.

A locally available River Gravel (#67 grading) and River Sand that meets ASTM C33 requirements were used in all mixtures. The particle size distribution of these two aggregates can be seen in Figure 4.

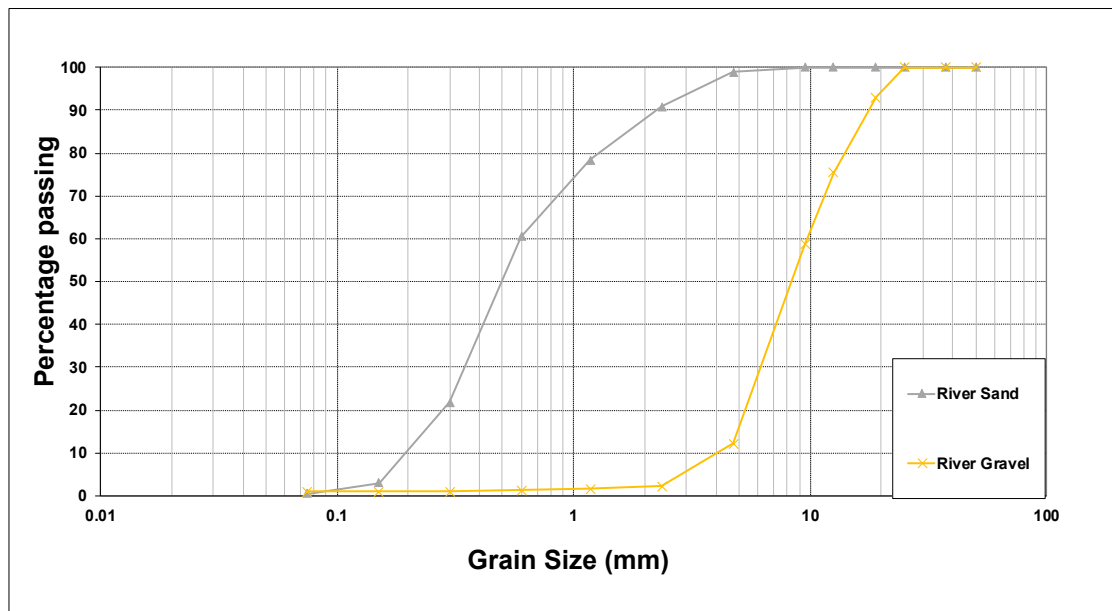


Figure 4. Particle Size Distribution of Aggregates.

The physical properties of the two aggregates can be seen in Table 3.

Table 3:Physical Properties of River Sand and River Gravel.

Aggregates	Bulk specific gravity (OD)	Bulk specific gravity (SSD)	Apparent specific gravity	Absorption (%)	Dry rodded unit weight (lb/ft ³)	Percent void
River Sand	2.54	2.59	2.63	0.96	103.46	36%
River Gravel	2.50	2.55	2.59	2.04	100.49	35%

Only, one AEA was used in this study in order to provide consistent results, which will allow the study to focus on the new characterization method. The air entraining admixture that was used was SikaAIR, which meets ASTM C260.

3.1 Concrete Mixture Design

Four straight cement concrete mixtures, with no supplementary cementitious materials, were produced in this study with the aforementioned materials. The mixtures were simple highway pavement mixtures aimed solely at producing various entrained air contents. A consistent slump of 3-5” was targeting to create similar workability between all mixtures. The air entraining dosages were added at various ranges sufficient enough to provide a low (3-4%), moderate (5-7%), and high (> 8%) fresh air contents in the produced mixtures. A non-air entrained mixture was also included as a reference in the study. Table 4 shows the mixture proportions of the four mixtures produced.

Table 4. Concrete Mixture Proportions.

Material	Control		Low Air		Medium Air		High Air	
	Weight (kg)	Weight (lb)	Weight (kg)	Weight (lb)	Weight (kg)	Weight (lb)	Weight (kg)	Weight (lb)
Type I/II Cement	335	565	335	565	335	565	335	565
Water	168	282	168	282	168	282	168	282
River Gravel (SSD)	1065	1795	1043	1757	1020	1719	975	1643
River Sand (SSD)	710	1197	695	1171	680	1146	650	1096
Target Air (SikaAir)	2.00%	2.00%	3.50%	3.50%	5.00%	5.00%	8.00%	8.00%

3.2 Fresh and Hardened Property Testing

Following mixing of the four mixtures, the slump of the freshly mixed concrete was determined in accordance with ASTM C143 to ascertain if a proper workability was obtained. Secondly, the fresh unit weight and air content was determined in accordance with ASTM C138 and ASTM 231 respectively. Determining the fresh concrete unit weight is necessary to ensure consistency between batches. Obtaining the air content via the pressure method is necessary to determine if the mixtures obtained the targeted air content expected from the design. Following these procedures, the mixtures will then be analyzed using the SAM to determine the air content via the SAM as well as the SAM number. The testing followed the procedures outlined in the operator's manual. While performing the SAM test, the air content is provided, which was used as a confirmation that the air content was within the targeted range. Lastly, nine 4" diameter by 8" length concrete cylinders were molded and cured in accordance with ASTM C192. Nine cylinders were produced to have three specimens to determine the 7-day compressive strength, three to determine the 28-day compressive strength, and three to provide samples to characterize the hardened air void system (ASTM C457). Of the three

additional samples, a portion will be used to develop the new characterization method.

The compressive strength testing was completed in accordance to ASTM C39.

3.2 Developing Alternative Hardened Air Void Characterization Method Hardened

3.2.1 Sample Cutting, Polishing, and Preparation

This step focused on minimizing or removing the sample preparation requirements of ASTM C457, which requires saw cutting, lapping, polishing, and dying of the hardened concrete sawn surface if following Procedure C. These steps are required by ASTM C457, and this task will focus on determining which steps are necessary and which can be eliminated, with an emphasis on minimizing this process. According to ASTM C457 the surface preparation requires the surface to be lapped with successively finer abrasives until it is suitable for microscopical observation. The standard recommends beginning with a nominal 150 μm grit size followed by 75, 35, 17.5 and 12.5 μm grit sizes, and perhaps 5- μm (No. 2500 grit) aluminum oxide. In between each successive lapping the sample is investigated under a microscope to determine if the air voids are clearly distinguishable. In this task, a control sample from each mixture was polished to meet the specifications in ASTM C457, and additional samples from the same mixture were polished to a lesser degree to investigate the possibility of reducing the preparation step. Also required in ASTM C457 Procedure C, is dying the sample surface into a contrast enhanced (black/white) color scheme. This is accomplished by applying black ink to the entire surface followed by a white powder/paste to fill the air voids. This will create a surface that specifically highlights only the air voids. Therefore, for each concrete mixture produced (control, low air, medium air, and high air) multiple hardened air void test samples were produced and polished at various degrees to ascertain the minimum preparation steps for microscope analysis. This task also investigates other

specific preparation variables such as consumable (polishing discs, etc.) type or brand, polishing time, polishing pressure, and others that may impact the processing.

3.3 Alternative Air Void Characterization Method Testing and Development

This step focused on characterizing the entrained air void system using a Keyence digital optical microscope. This step is the critical step in the alternative air void characterization method, in that it uses equipment to do the air void counting and measuring rather than a human making the assessment. Developing this process required demonstrating the full capabilities of the Keyence digital optical microscope that can be used to assess the air voids of the prepared concrete samples. Since many samples will be developed of varying polished degrees, this process demonstrates the optimum preparation extent needed to properly assess the hardened air void system using the Keyence digital optical microscope. In addition to obtaining key entrained air void system data via the Keyence microscope, focus was also placed on quantifying the time required, the ease of use, the repeatability, and the quality of the results. This step also determined the necessary Keyence hardware components and specific procedures necessary to complete this analysis.

4. FRESH PROPERTIES, SUPER AIR METER AND COMPRESSION

STRENGTH RESULTS

Each mixture used a water to cement ratio (w/c) of 0.50 and incorporated low-alkali Type I/II cement, as well as siliceous river gravel and river sand that meets ASTM C33. While a maximum w/c of 0.45 is suggested for concrete that is exposed to freezing/thawing and deicers (ACI 201), a slightly higher w/c of 0.50 was used. This was done to ensure all samples were properly consolidated and to help increase the dispersion of air bubbles within the concrete system, which will consequently help develop the new hardened air void characterization technique. A total of four concrete mixtures were designed according to ACI 211 to produce mixtures with various air contents. A non-air entrained mixture was designed with a target air content of 2%, as well as a low air content (3-4%) mixture, a moderate air content (5-7%) mixture, and a high air content (> 8%) mixture. The air entraining admixture used was SikaAIR, which meets ASTM C260. The concrete was mixed in a drum mixer in accordance to ASTM C192. Initial trial mixtures were first produced to determine the proper dosage of SikaAIR to develop an air content within the desired ranges. Once the appropriate SikaAIR dosages were obtained, three mixtures per concrete proportion were produced to present an average result for each test. Each of these mixtures were individually mixed with sufficient concrete volume to complete all the desired tests. Immediately following mixing, the slump was measured in accordance with ASTM C143. The fresh air content was measured in accordance with ASTM C231 and the Super Air Meter (SAM) number was measured in accordance to AASHTO T118 as well as the Super Air Meter procedures and guidelines provided by the manufacturer. Hardened concrete samples were cast in 4" (diameter) x 8"

(length) plastic molds in accordance with ASTM C192. For compressive strength testing, three-cylinder specimens per mixture, per 7-day strengths and 28-day strengths were produced and tested. Compressive strength testing was completed in accordance with ASTM C39. Additionally, three-cylinder specimens were cast per mixture for future hardened air content measurement and analysis.

4.1 Fresh Property and Compressive Strength Results

A summary of the four mixture proportions along with their results can be found in Table-5.

Table 5. Concrete Mixture Proportions and Results

Material	Control	Low Air	Medium Air	High Air
Type I/II Cement (lbs/yd ³)	565	565	565	565
Water (lbs/yd ³)	282	282	282	282
River Gravel (SSD) (lbs/yd ³)	1795	1757	1719	1643
River Sand (SSD) (lbs/yd ³)	1197	1171	1146	1096
Target Air (SikaAir)	2.00%	3.50%	5.00%	8.00%
Property	Control	Low Air	Medium Air	High Air
Fresh Density (lbs/ft ³)	146	144	143	139
Slump (in)	3.75	4.25	3.5	4.0
Measured Air Content (ASTM C231) (%)	2.1	3.7	4.9	8.3
Air Content (SAM) (%)	2.3	3.4	5.2	8.7
SAM Number	0.61	0.58	0.39	0.12
7-day compressive strength (psi)	5086	4557	4328	3890
28-day compressive strength (psi)	6719	5723	5340	4280

The results shown in Table 5 are all as expected based off the literature, experiences, and the corresponding mixture proportions. The fresh density results decrease with an increase in air content, which is expected since the air has negligible weight and there is less concrete material in the system. The slump values were all within the target 3-5" range. The air values are also within the anticipated target range. Both the

air content obtained from the ASTM C231 pressure method and the SAM meter appear to be in close alignment with each other. The SAM numbers decrease with an increase in air content, which is expected based off the literature (ACI 201, 2016, Ley & Tabb, 2013). A SAM number of 0.20 has been shown to correctly determine, over 90% of the time, whether the spacing factor is above or below the 0.008” limit (ACI 2016, Ley & Tabb, 2013). Therefore, an ideal SAM number is anything less than or equal to 0.20, which only the High Air mixture obtained. All other mixtures were above this value, which suggests the spacing factor is likely above the 0.008” spacing factor limit. The 7-day and 28-day compressive strength values were also as expected. All strengths showed a decrease in compressive strength with an increase in air content. The literature states that for every 1% increase in air content this causes a reduction in the compressive strength of about 500 psi (Ley et al., 2017).

4.2 Hardened Sample Preparation and Development

This chapter is broken up into two major sections, Section 4.1 - the development and discussion of the preparation steps investigated, which includes information regarding why certain equipment, materials, and procedures are recommended. And Section 4.2 - the recommended hardened concrete sample preparation steps necessary for hardened air void analysis using the Keyence digital optical microscope as well as traditional methods outlined in ASTM C457 standard.

4.3 Development And Discussion of Preparation Steps

4.3.1 Sample Cutting

Multiple 4” (dia.) x 8” (length) concrete cylinders were cast and prepared for hardened sample preparation. Therefore, all hardened samples were cut from 4” (dia.) x

8” (length) concrete cylinders. In the cutting process, a wet masonry saw from Humboldt Manufacturing was used as seen in Figure 5.



Figure 5. Wet Masonry Saw by Humboldt.

Two different saw blades were investigated; i) a 20” Husqvarna Masonry Saw Blade (sold by Humboldt) seen in Figure 6a and ii) a 14” Diamond Cut-off Wheel Blade (sold by UKAM Industrial Superhard Tools) seen in Figure 6b.

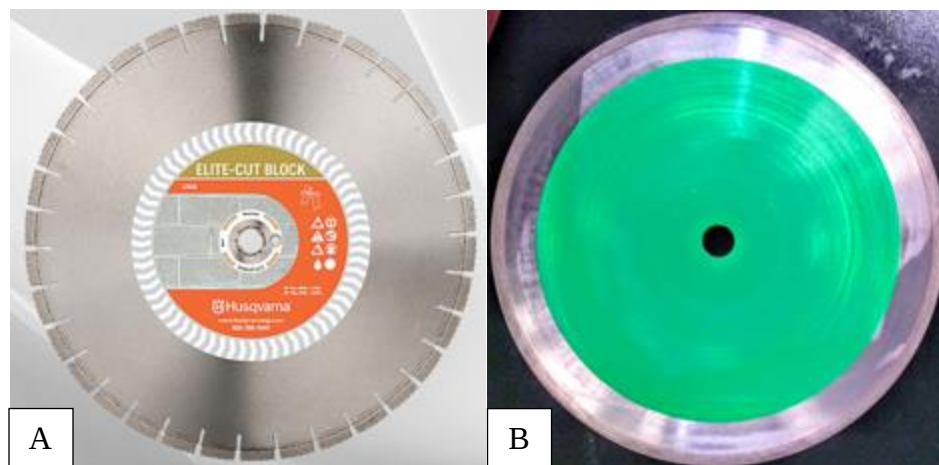


Figure 6. The Two Sawblades Investigated

Two different blades were used in this study to determine the speed and cut quality of the different blades. The Husqvarna is a common saw blade found in concrete materials labs, often purchased together with the wet saw from Humboldt. From

experience, this blade tends to leave small grooving along the cut surfaces. The smoother the cut from the saw blade, will ultimately result in less polishing time in the next steps, therefore an additional diamond saw blade was introduced and determined to level less grooves. After an internet search, and discussions with equipment suppliers, it was discovered that the 14" UKAM diamond cut off blade, could produce superior cuts to the typical Husqvarna blade. Both companies state that their blades can last for "thousands" of cuts, unfortunately the authors were unable to confirm or deny this claim. Currently, the 20" Husqvarna lists for \$499/blade and the 14" UKAM lists for \$254/blade. Due to project time and budget limitations, only two blades were investigated in this study.

When using either blade with the saw, a c-clamp was used to safely hold the sample flush against the edge of the rolling table as seen in Figure 7. This helped to ensure that a straight cut was achieved.



Figure 7. Using a C-Clamp to Mount Concrete Sample to the Saw Table.

The cylinders were cut into nominal 0.75" thick samples. On average one 8" long concrete cylinder would produce 6-7, 0.75" thick samples, as the cylinder became unsafe to mount or hold by hand for further cutting. On average, a single cut from the Husqvarna blade took approximately five minutes. A two-minute cut could be achieved but the

quality of the cut decreased. Even at a five-minute cut speed, the cut surface often exhibited small grooving from the blade along with a raised edge that was protruding from the cut surface. The raised edge is an issue as this edge will first need to be polished through before the majority of the sample will begin to be polished. The grooving was typically very fine and not as exaggerated as the raised edge. It could be seen with the naked eye and felt by hand. It was difficult to capture a photo of the grooving but tended to show up better when the sample was wet in the reflected light. The cut quality of the Husqvarna blade can be seen in Figure 8.



Figure 8. Husqvarna Blade Cut Quality.

On average, a single cut using the UKAM diamond cut off blade would take approximately two minutes, which was the quickest and safest cut speed noted for this blade and saw configuration. It was observed that the cut quality with the UKAM diamond cut off blade, was better than the Husqvarna blade. There was little to no visible

grooving left from the saw and the raised edge issue rarely occurred. An example of the cut quality from the UKAM diamond cut off blade can be seen in Figure 9.



Figure 9. UKAM Cut Off Blade Cut Quality.

Based off these observations, it was concluded that of the two saw blades investigated in this study that the UKAM cut off blade is the best blade to use for cutting hardened concrete samples for hardened air void analysis. The cost of the UKAM diamond cut off blade is lower than the Husqvarna, the cut speed is quicker, and the quality of the cut is superior. The final saw cutting recommended procedures can be found in Section 4.1.5.

4.3.2 Sample Polishing

In this investigation a Struers Abramin auto-polisher was used, which can be seen in Figure 10.



Figure 10: Stuers Abramin Auto-Polisher.

The Stuers Abramin auto-polisher uses a 12" base that can rotate at either 150rpm or 300rpm in a counter-clockwise direction. The polishing arm can apply a pneumatically applied force of 30N – 600N (6.7lbs – 134.8lbs). An aluminum fixture was designed and constructed, in house, to fit in the polishing arm and to mount either a 4" diameter or 4" square concrete sample. The polishing arm rotates at 150rpm clockwise. The auto-polisher can automatically dispense water as a polishing lubricant. Once the desired force, base rotation speed, duration of polish, and desire to dispense water, is selected, the polisher can be engaged, and the polisher will commence for the desired duration.

Based off of the literature and discussions with manufacturers there are two popular mediums used to polish concrete samples, Silicon Carbide paper polishing discs and diamond metal backed polishing discs. Silicon Carbide paper polishing discs are typically sold in packs of 100 and cost up to \$300/100pack of polishing discs depending on the grit size. These types of polishing discs typically have an adhesive backing and

need to be applied to an accompanying metal support disc (\$105/support disc). The base of the Struers Abramin auto-polisher is magnetic (as well as many other auto-polishers) and the support disc will magnetically attach to the base of the auto-polisher. However, once the Silicon Carbide polishing disc is spent, it will need to be removed from the metal support disc and a new one applied. The diamond metal backed polishing discs are sold individually and cost upwards to \$600/disc. However, these diamond polishing discs are pre-adhered to a metal backing, therefore do not require a metal support disc to attach to the polisher.

In this investigation, it was discovered that the first polishing step, using the coarsest polishing medium, is the most crucial in producing a suitable surface for further analysis. In this first polishing step, it is necessary to produce a completely flat polish that removes all grooving from the saw and other uneven surfaces created from the saw cutting. Using the UKAM diamond cut off blade discussed in the previous section drastically reduced the time required to achieve a completely flat polish from the first polishing step. The procedure developed to achieve a completely flat polished surface is discussed in Section 4.5.

Both Silicon Carbide paper polishing discs and diamond metal backed polishing discs were acquired from Allied High Tech Products. It was observed that, when completing the first polishing step, using the Silicon Carbide polishing discs at grade 80 (the coarsest option sold from Allied High Tech Products), would require on average 2-3 polishing discs to achieve a completely flat surface. This was completed at a recommended force range of 150N-300N (33.7lbs – 67.5lbs). This was also a laborious process because a long polishing time could not be used, as the polishing disc had to be

periodically checked to see if it had worn through. It was possible to see that the polishing discs was beginning to wear, as one side of the polishing disc is visible while polishing, but it was difficult to make an accurate interpretation while the polishing disc was spinning and wet. Therefore, the auto-polisher had to be completely stopped, the polishing disc removed, dried with a forced air hose, and then a decision to replace was made. If it was time to replace the Silicon Carbide polishing paper, it had to be removed from the metal support disc and a new one adhered. Removing the polishing paper from the metal support disc was completed with the aid of a handheld paint scrapper tool, a razor blade scraper, and rubbing alcohol for any residue. This process could be sped up with an additional metal support disc, as one additional support disc could be pre-loaded with a new Silicon Carbide polishing disc, and placed on the polisher, and while the sample is polishing with the new polishing paper, the spent polisher paper could be removed, and subsequently pre-loaded with the next Silicon Carbide polishing disc. In addition, to checking the polishing paper, the concrete sample needed to be checked, to determine if a completely flat polish was achieved (specifics regarding this process are detailed in Section 4.5). It was difficult to settle on a specific amount of time to stop the polisher and check the polishing paper and concrete sample, as the degree of polishing required per sample varied. This can also vary on the force applied, the rpm of the polishing pads, and more specifically, the sample hardness, paste amount, and aggregates used in the concrete. On average, with the samples produced in this study, a Silicon Carbide polishing disc would begin to wear out between 20-30 minutes at 300N (67.5lbs) force at a platen speed of 300rpm. As previously stated, 2-3 Silicon Carbide polishing discs were required to achieve a completely flat polish, therefore, at minimum the first

polish could be set for 20 minutes without stopping to save time. Once the sample was observed to be nearly complete, the polishing time was set to 2-5 minutes depending on how close the sample was to complete. Following each short duration, the sample was checked. This procedure was followed in order to preserve the life of the polishing discs, as a limited amount were available for this study due to budget limitations. This process was more involved and required more operator time, however, if one had unlimited Silicon Carbide polishing discs, the polisher could simply be set to 30 minutes at 300N (67.5lbs) for three new polishing discs, and on average the sample would be completely flat following this polishing regimen.

It was observed that the diamond polishing discs were less labor intensive and had a much longer life than the Silicon Carbide counterparts. Since the diamond discs are pre-metal backed, the polishing paper does not have to be removed or adhered to a support disc, so that process is completely removed with these polishing discs. Additionally, these polishing discs last much longer. At the time of this writing approximately 50 surfaces have been polished, and there is no sign of wear on any of the discs. According to the manufacturer 100s of samples can be polished per disc. It was also noticed that, on average, it took on average 20 minutes to polish a sample completely flat with the coarsest diamond polishing disc (260 μ m) at 300N (67.5lbs) force at a platen speed of 300rpm. Due to the robust nature of the diamond polishing discs, higher forces were investigated in attempt to reduce the polishing time. The optimum configuration to reduce the polish time was 500N (112lb) at a platen speed of 300rpm with water as the lubrication. This configuration would produce a completely flat surface at an average polishing time of 10 minutes. Based off of these observations, it was concluded that the

diamond metal backed polishing discs are worth the cost and are recommended for use in this process. The final polishing recommended procedures can be found in Section 4.16

4.3.3 Contrast Enhancement of Sample

This process is necessary for further hardened air void analysis using the Keyence digital optical microscope. It is possible to use the microscope on the non-contrast enhanced samples, however, the on-board counting and measuring software will not be able to distinguish between all of the features on the surface of the sample. Therefore, it is necessary to enhance the contrast between the air voids and the remaining portion of the sample. The final recommended contrast enhancement steps can be found in Section 4.8.

4.3.4 Blackening of Sample

Two different blackening methods were attempted in this investigation i) using a black permanent marker, and ii) using water-soluble acrylic ink and roller.

4.3.5 Black Permanent Marker

Previous studies have developed a hardened concrete air void analysis technique using a flatbed scanner (BS EN, 2005; Fonesca & Scherer, 2015; Carlson et al, 2005; Chatterji & Gumundsson, 1977; Pade & Elsen, 2002; Peterson et al, 2001; Zhang et al 2005). In these studies, the procedures also require the samples to be contrast enhanced, and the samples to first be blackened out. Most of these authors suggest simply using a black permanent marker, such as a chisel tip Sharpie brand marker. These studies also specify that the ink should be applied in a line-by-line, left-to-right, application in which each sequential line is overlapped with the previous line. After the sample is completely blackened from top-to-bottom with this method, the authors state to apply one additional layer perpendicular to the previously layer, to ensure the sample is sufficiently blackened.

It is understood that this method applies the minimum amount of ink necessary to blacken the surface, and not interfere with the following preparation step. The following preparation step requires the addition of a fine white powder (discussed in further detail in section 4.8). Adding too much ink, or adding it in a quick, random, pattern would result in ink bleeding into the white powder (once it is applied), coloring it a purple/gray color, no matter how long the ink was left to dry. Additionally, coloring the surface in a quick, random, pattern could unintentionally fill the small air voids with ink. It was also discovered that when using a Sharpie brand black chisel tip permanent marker, the ink would not apply evenly, leaving a streaky application. This was true even with a brand-new marker. It was possible that the sample surface was not providing enough friction between the tip to draw ink in an even manner. It was found that a Staples brand black chisel tip permanent marker rarely left a streaky finish. However, it was important to note that both marker brands often left striations (different from the streaky uneven finish) from each individual line-by-line application. As in, the individual lines could still be seen with the naked eye in the light reflection, even with a thorough non-streaky application. This was especially the case, if upon initial application, the marker did not apply evenly, or if the operator accidentally did not overlap the lines properly and required correction. The striations could also be described as a non-homogenous blackening of the surface, as the individual lines (although black) could be individually identified in the light. These striations could be an issue when analyzing the sample under a microscope as they may reduce user accuracy due to differences in light reflections.

4.3.6 Water-Soluble Acrylic Ink and Roller

Fonseca *et al.*, (2015) reported that black permanent markers often contain a resin, leaving the surface sticky. This was noticed with both the Sharpie and Staples

brand permanent markers discussed in Section 4.6. The stickiness is potentially an issue, as the white powder application in the next step could stick to the surface. Therefore, Fonseca *et al.* (2015) suggest using a water-soluble acrylic ink and roller. This type of ink does not contain a resin, so it should not leave a sticky surface. The authors of this report found the use of the water-soluble acrylic ink to be the superior blackening method to the permanent marker. Although, it is slightly more expansive than the markers, this method produce a surface that appeared much blacker than the markers, it was not sticky, and it applied evenly (no striations as with the markers). As discussed more thoroughly in Section 4.14, the last step in the contrast enhancement procedure is to use mineral oil to remove white powder stuck to the surface. With the black marker method, the mineral oil simply removes the fine particle from view, by making them less reflective, and when the sample dries, they come back into view. However, with the ink and roller method, the oil physically removes the fine particles from the surface, since the surface is not sticky, therefore no re-application is needed if the samples are to be analyzed later. Therefore, it is recommended that the blackening of the sample surface be completed with a black water-soluble acrylic ink and roller.

4.3.7 White Powder

In order to enhance the contrast between the air voids and the rest of the sample a white powder is required to fill the air voids. ASTM C457 (ASTM C457) Procedure C, currently says to use a white powder, and offers Barium Sulfate, Wollastonite, or Titanium Dioxide with a median particle size of 2-3 μ m as some suggestions. However, the standard does not provide any further detail on these white powders. Other literature (Powers, 1949) has also suggested using a Zinc Oxide paste for this step. Zinc Oxide is typically found to be the primary ingredient in sunscreen and diaper rash/skin cream.

Irrespective of the brand, these pastes typically list 20% Zinc Oxide as their only ingredient. Therefore, an over-the-counter diaper rash cream with 20% Zinc Oxide was initially used for this step, due to it being readily available and cheap. However, the Zinc Oxide paste caused many problems, the primary being that it tended to dissolve the ink from the surface resulting in the paste becoming a purple/gray color. Therefore, an over-the-counter diaper rash cream with Zinc Oxide was deemed unusable for this step. Fonseca *et al.* (2015) investigated multiple white powders for the same purpose of enhancing the contrast of air voids in a blacked out concrete sample.

The authors investigated the following white powders; Barium Sulfate with a mean particle size of 3 μ m, Titanium Dioxide with a mean particle size of 0.5 μ m, Silicon Oxide with a mean particle size of 2 μ m, and Corn Starch with a mean particle size of 9 μ m. The authors found that the Titanium Dioxide particles were too small and filled in the very small imperfections that remained from polishing. In contrast, the Corn Starch showed to be too coarse. An individual Corn Starch particle would fall into an air void and “bridge” itself from edge-to-edge of the air void resulting in parts of the air void unfilled. This will result in an underestimation of the air void content when the sample is analyzed. Both the Silicon Oxide and Barium Sulfate powders were deemed to be an ideal particle mean size; small enough to properly fill all air voids, but not too small to fill in the very small defects. Barium Sulfate powder was ultimately recommended over the Silicon Oxide powder as it was much whiter and will provide a better contrast between the sample. Wollastonite powder with a mean particle size of 2-3 μ m was difficult to source and purchase so it was not tested. However, Barium Sulfate with a 3 μ m mean particle size was found to be readily available. Based off these findings and

recommendations, a Barium Sulfate powder with a mean particle size of $3\mu\text{m}$ was acquired and used on the polished concrete samples.

4.3.8 Correcting the Black Surfaces

Following the compaction of the Barium Sulfate powder, it was observed that small cracks in the aggregates and even air voids in the aggregates will be highlighted by the white powder. These need to be removed, as they will contribute to the air void analysis and invalidate the results. To remove these features, a black permanent marker can be used to black-out these features. Additionally, a handheld magnifying lens can also be used to assist with this step.

4.3.9 Mineral Oil

It was observed that very small amounts of Barium Sulfate powder would remain after removing the compacted Barium Sulfate powder from the surface. The residual amounts of powders are not embedded in an air void or an aggregate feature but are simply stuck to paste areas. It was found that the best way to remove these small amounts of powder is to apply mineral oil to the surface. Regarding the blacked-out surface with the permanent marker, this process does not technically remove the powder from the surface, but they are removed from view in regard to further analysis, such that the particles are small enough to absorb the mineral oil and become non-reflective. This is pertinent to know, as the mineral oil will eventually dry, and the small amounts of Barium Sulfate powder will become visible again. The drying process takes approximately 10-15min in a laboratory setting at room temperature. Therefore, it necessary to consider when further analysis will take place as this step should take place immediately before future analysis. It is noted that, mineral oil can be re-applied multiple times and it does not affect the analysis.

However, as noted in this report, using a black water-soluble acrylic ink to blacken the surface is recommended. It was also observed that the mineral oil does remove the Barium Sulfate from the surface of the ink-processed samples, and in most instances, the mineral oil does not need to be applied again.

4.4 Recommended Hardened Concrete Sample Procedures

4.4.1 Saw Cutting Materials and Equipment

Table 6 lists the materials and equipment used for the saw cutting procedure.

Table 6. Saw Cutting Materials and Equipment.

Item	Make/model	Current Cost	Website/direct link
Wet Saw	Humboldt	\$4,700	https://www.humboldtmg.com/masonry-saw-8-in-cut.html
Saw Blade	UKAM Diamond Cut-off Blade	\$254	https://www.ukam.com/
C-Clamp	6” Pony C (or similar)	\$14	https://www.lowes.com/pd/Pony-C-Clamp-6-in-2600-SERIES-C-Clamp/1000988714

4.4.2 Saw Cutting Procedures

1. With the UKAM diamond cut off blade attached.
2. Place a 4” (diameter) x 8” (length) concrete cylinder on the rolling carriage tray on the wet saw.
 - a. The concrete cylinder should be up against the rear guide bar, along its long axis.
3. Measure the desired sample thickness by either marking the sample or measuring from the outer edge of the saw blade to the edge of the sample.

4. Once the concrete cylinder is in the desired location, use a c-clamp to secure the cylinder in place ensuring the clamp arm is out of the path of the blade, as seen in Figure 11.



Figure 11. Concrete Cylinder Mounted Wet Saw.

5. Before cutting, ensure that the blade is approximately 1/8" below the rolling carriage tray into the cutting groove. This will ensure the blade cuts all the way through the concrete cylinder.
6. Turn the wet saw on, and slowly move the carriage tray towards the blade to begin cutting.
7. Continue pushing the carriage tray towards the saw blade at a slow pace throughout the entire cut, until the cut is complete.
 - a. Following cutting of the cylinder, select samples in accordance to ASTM C457 sampling section (section 7).

For the purposes of this study, all sawn surfaces were polished for further investigation.

4.5 Polishing Materials and Equipment

Table 7: Lists the materials and equipment used for polishing procedure.

Table 7. Polishing Materials and Equipment.

Item	Make/model	Current Cost	Website/direct link
Auto-Polisher	Struers Abramin	N/A	https://www.struers.com/en/Products/Grinding-and-Polishing/Grinding-and-polishing-equipment
Diamond Polishing Pad	Allied High Tech Products	\$600/disc	https://www.alliedhightech.com/
Crayons	Crayola	\$6	https://www.amazon.com/Crayola-crayola-Crayons-24-Pack/dp/B0096XWNNY/ref=sr_1_7?crid=254B0YDJJ2IDE&dchild=1&keywords=crayons&qid=1624392711&srefix=cryons%2Caps%2C199&sr=8-7

The specific auto-polisher model used in this study is no longer produced by Struers, however, they produce newer auto-polishers with additional capabilities. Allied High Tech Products, who sells the polishing discs, also makes and sells an auto-polisher that can be used for this purpose. It is important to note that an appropriate sample holder fixture is needed for this process. The one used in this study was produced in house. To the authors' knowledge, the manufacture of the polisher will work with new clients to develop an appropriate sample holder for the new user's needs. They also may have sample holders available to purchase.

4.6 Polishing Procedures

1. Prior to polishing a sample surface use a crayon to draw a grid on the surface spaced approximately $\frac{1}{4}$ " x $\frac{1}{4}$ " as seen in Figure 12. This is necessary to help determine if the specimen surface is polished completely flat.

- a. The lines do not have to be perfectly straight or perfectly spaced but choose a color that contrasts with all features on the sample surface.
- b. This is best completed on a dried sample surface.

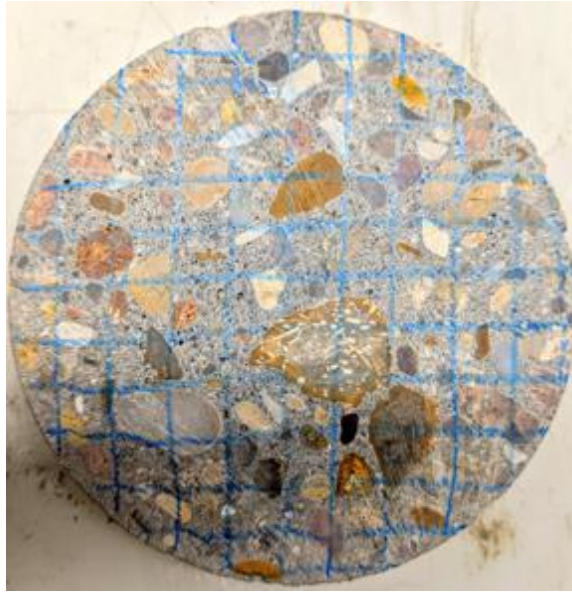


Figure 12. Example of Grid Drawn with crayon

2. Set the polisher force to 500N (112lbs).
3. Place a 260 μ m polishing disc on the polisher.
4. Polish one sample using a 260 μ m disc at a platen speed 300rpm and a force of 500N (112lb) for 10mins using water as lubrication.
5. Following the initial polishing, check the sample surface to see if all of the crayon markings have disappeared.
 - a. Sometimes the crayon markings could be hidden by the water on the surface, therefore, it may be necessary to dry the sample with forced air to check if all markings are in fact gone.

- b. If no visible crayon markers are present, that surface is done with the 260 μ m disc and can be set aside for finer polishing. If any crayon markings are still visible, it is an incomplete polish. An example of an incomplete polish can be seen in Figure 13.

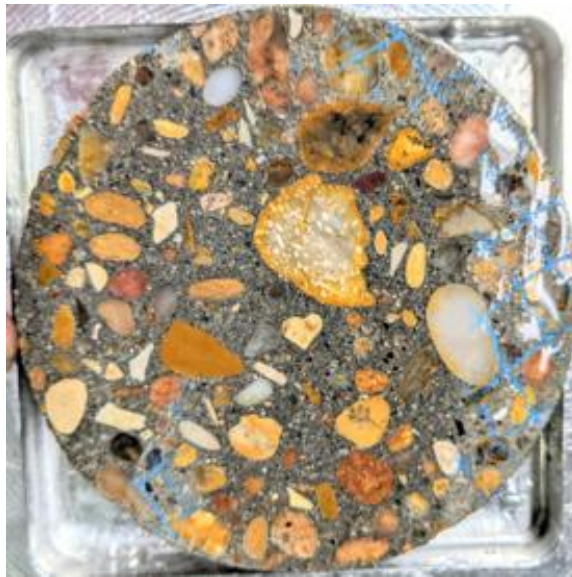


Figure 13. Example of an Incomplete Polish.

- c. If an incomplete polish is noted, it is recommended to polish for an additional 10 minutes at **500N (112lb)** at a platen speed **300rpm** with water as the lubricant.
 - d. On average one 10-minute pass at 500N (112lb) at a platen speed of 300rpm was necessary to achieve a completely flat polish.
6. Before moving to a finer polishing disc, dry the sample using forced air to remove the excess water and any polishing media remaining on the sample surface.
- a. Do not put the air hose nozzle too close (approximately 2”) to the sample surface, as it may damage the air void system.

At this point in the study, it is not known how fine of polish is required for the alternative hardened air void analysis. For non-contrast enhanced samples, ASTM C457 states that the polish needs to be fine enough to show a glossy surface. This can be achieved by polishing with Allied High Tech polishing discs sized 260 μ m, 125 μ m, 70 μ m, 30 μ m, 15 μ m, and 6 μ m. The 6 μ m polishing disc produces the glossy surface. However, a glossy surface may not be necessary for the alternative procedure, thus saving time, money, and resources. This will not be determined until the fall semester. However, it has been determined that once the sample surface is polished completely flat with the 260 μ m diamond polish disc, the force and polishing duration can be significantly reduced. The following procedure can be followed after the sample surface is polished completely flat from steps 1-6 above.

7. Set the polisher force to **300N (67lbs)**.
8. Place a 125 μ m (or 70 μ m, 30 μ m, 15 μ m, 6 μ m) polishing disc on the polisher.
9. Polish the previously sample using the appropriate polishing **disc** at a platen speed **300rpm** and a force of **300N (67lbs)** for **30sec** using water as lubrication.
10. Dry the sample with forced air as previously described.
11. Repeat steps 7-10 with the next smaller sized polishing disc until appropriate polish is achieved.

It should be mentioned that sometimes difficulty will be encountered when preparing the lapped surfaces, such that the air voids could begin to erode or elongate during the polishing procedure. This is usually caused by a weak cement-paste matrix. This effect was not observed with the samples produced in this study, therefore no preparation for this affect was completed. However, if one desires to complete such a

procedure, ASTM C457 describes a procedure in which carnauba is applied to the surface prior to polishing and removed after all polishing procedures are completed. Other researchers such as Fonesca & Scherer (2015) and Carlson et al, (2005) have suggested applying a mixture of equal parts lacquer and acetone to the sample surface prior to polishing, and after polishing is complete, the sample is then soaked in acetone to remove any remaining lacquer/acetone mixture (Fonesca & Scherer, 2015; Carlson,et al, 2005. This could be done with a colored lacquer to help indicate when the sample has been polished completely flat.

4.7 Contrast Enhancement Materials and Equipment

Table 8 lists the materials and equipment used for the contrast enhancement procedure.

Table 8. Contrast Enhancement Materials and Equipment.

Item	Make/model	Current Cost	Website/direct link
Water Soluble Black Acrylic Ink	Speedball (Block Printer Ink)	\$12	https://www.amazon.com/gp/product/B000BYT24U/ref=ppx_yo_dt_b_asin_title_o06_s00?ie=UTF8&psc=1
Rubber Roller (aka Brayer)	Speedball	\$16	https://www.amazon.com/gp/product/B003IFY622/ref=ppx_yo_dt_b_asin_title_o06_s00?ie=UTF8&psc=1 [Used for applying ink]
Barium Sulfate	HighMedia	\$18	https://www.amazon.com/HiMedia-GRM1342-500G-Barium-Sulphate-Extra/dp/B00DYO5K54/ref=sr_1_3?dchild=1&keywords=barium+sulfate&qid=1624412430&sr=8-3
Magnifying Glass	Nazano	\$13	https://www.amazon.com/gp/product/B08PP4RJ5J/ref=ppx_yo_dt_b_asin_title_o08_s02?ie=UTF8&psc=1
Rubber Roller (aka Brayer)	Akiro	\$7	https://www.amazon.com/gp/product/B07YDNKSH6/ref=ppx_yo_dt_b_asin_title_o08_s01?ie=UTF8&psc=1

			[Used for roller compacting Barium Sulfate]
Fiberglass Brush	Lightning Powders	\$17	https://www.amazon.com/gp/product/B001R1TOQM/ref=ppx_yo_dt_b_asin_title_o08_s01?ie=UTF8&psc=1
Razor Blades	Alpine Industries	\$7	https://www.amazon.com/Alpine-Industries-Replacement-Industrial-Grade-Standard/dp/B083V71TZ9/ref=sr_1_6?dchild=1&keywords=4%22+utility+razor+blades&qid=1624412846&s=industrial&sr=1-6
Squeegee	CCyanzi	\$10	https://www.amazon.com/gp/product/B09194154P/ref=ppx_yo_dt_b_asin_title_o08_s01?ie=UTF8&psc=1
Mineral Oil	Scrubbles	\$10	https://www.amazon.com/gp/product/B001B2RG1C/ref=ppx_yo_dt_b_asin_title_o08_s00?ie=UTF8&psc=1
Glass Dropper Bottle	Dropper Stop	\$5	https://www.amazon.com/gp/product/B07W8Y5HYR/ref=ppx_yo_dt_b_asin_title_o09_s00?ie=UTF8&psc=1
Microfiber Towel	Mr. Siga	\$12	https://www.amazon.com/MR-SIGA-Microfiber-Cleaning-Cloth-Pack/dp/B07HRCDDL1/ref=sr_1_13?crd=3Q131XR9MPF4B&dchild=1&keywords=microfiber+towels+for+cars&qid=1625158509&srefix=microfiber+t%2Caps%2C205&sr=8-13

If one intends to use both sides of the concrete sample, it was observed that placing the sample on a 4” diameter neoprene pad (commonly used when compressing concrete cylinders) helped keep the underside of the sample clean and free from excess Barium Sulfate powder. However, this is not completely necessary, but is typically found in a concrete lab. Additional supplies needed, but are not necessary, are an oven capable of reaching and maintaining 230°F (110°C), paper plates, paper hand towels, and a plastic spoon.

4.8 Contrast Enhancement Procedures

4.8.1 Blackening of the Surface Procedure

1. Using a plastic spoon or other utensil, place a small amount of the black water-soluble acrylic ink on a clean paper plate.
 - a. It was observed that a grape sized drop could color 3-4 surfaces.
2. Clean the surface of the polished concrete sample with the fiberglass brush.
 - a. An off-the-shelf fiberglass fingerprint brush was found to work well for this step and future cleaning steps, as the bristles are very soft and will not scratch the polished concrete surface.
3. Using the Speedball Rubber Roller, load the roller with ink by rolling over the top of the ink drop on the paper plate. Do this in a back-and-forth motion until the roller is full of ink as seen in Figure 14.

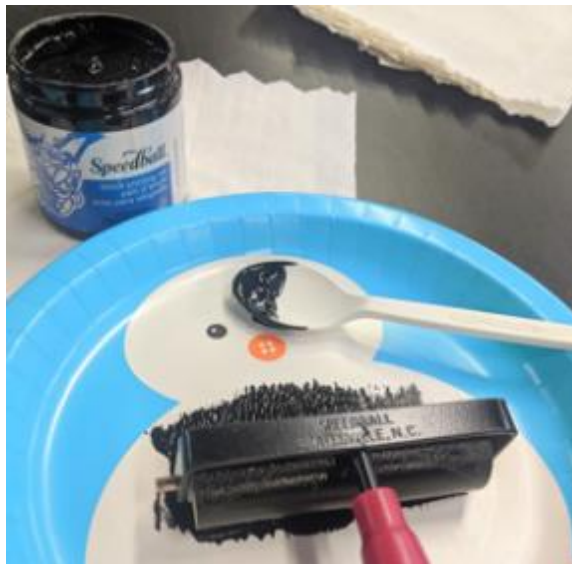


Figure 14. Rubber roller loaded with ink.

4. Apply ink to the surface of the polished concrete sample, by pressing the roller on to the surface and rolling across the sample as seen in Figure 15a-c.

- a. It was observed that 3-4 back-and-forth passes in perpendicular directions was sufficient to blacken the surface.

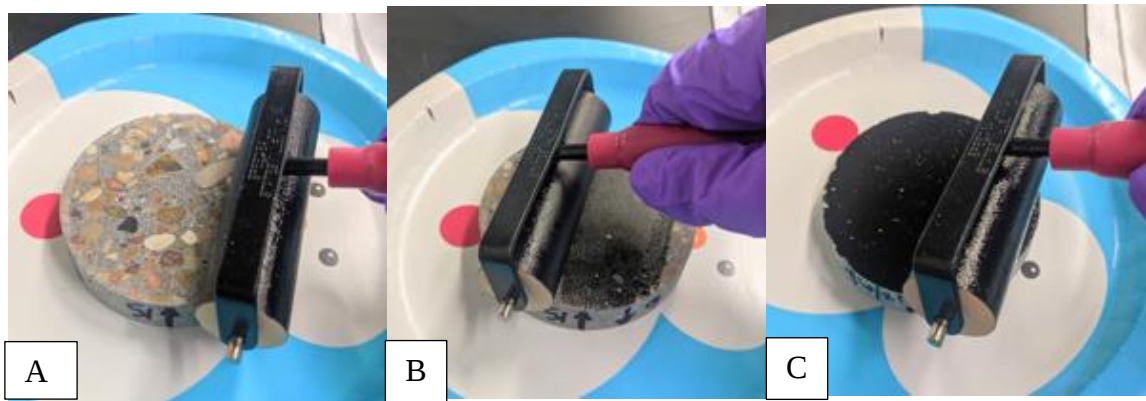


Figure 15.a-c: Applying Ink to Polished Sample with Rubber Roller.

5. Once sufficient ink has been applied to the surface, the sample can be dried in an oven at 230°F (110°C) for 10mins.
 - a. If the underside of the same sample is to be analyzed, it is necessary to ensure that the ink is completely dry, such that the ink does not stick to the table or working surface. Additionally, the sample should be cool to the touch, before applying ink.

4.8.2 White Powder (Barium Sulfate) Procedure

1. Place the concrete sample on a 4" diameter neoprene pad as seen in Figure 16a.
2. Lightly clean the sample surface by brushing it with the fiberglass brush.
3. Lightly sprinkle Barium Sulfate powder on the sample using a spoon or other scooping utensil as seen in Figure 16b. Continue sprinkling Barium Sulfate powder until the entire surface is covered. It was noticed, that sometimes the Barium Sulfate powder will have clumped into small spherical balls. To breakup these clumps the spoon/utensil can be used to lightly cut or crush them as in Figure 16c-d.

4. Use the Akiro 4" rubber roller to roller compact the powder into the air voids. First use a light back-and-forth rolling motion to flatten the powder as seen in Figure 17f. After the powder is flattened by the light pressure pass, complete 2-3 hard pressure back-and-forth rolling passes, followed by 2-3 back-and-forth rolling passes perpendicular to the previous pass.
5. Remove the powder using a 4" single sided razor blade. This was completed by holding the blade at an acute angle to the sample surface, beginning at the edge furthest from the operator, and pulling the blade toward the operator in one, single pass, applying medium, consistent, pressure throughout the pass as seen in Figure 16g-h.
 - a. Take care to not apply too much pressure such that the black ink is removed.
6. Remove the excess powder using the fiberglass brush as seen in Figure 16i. At this point, it can be seen if another pass of the razor blade is needed. If so, complete an additional pass as previously described until all excess powder is removed. After all excess powder is removed, wipe the sample surface using the soft bristle fingerprint brush.
7. To ensure all voids are properly filled with the Barium Sulfate powder, a handheld magnifying lens with 30X magnification, with built in lights, can be used to assess the quality of the air voids. If a lot of air voids are observed to be vacant of the Barium Sulfate powder, it may be necessary to re-do this entire process. If a few voids are vacant of powder, a small amount of powder can be added to that area, compacted with the roller, and removed with the razor blade.

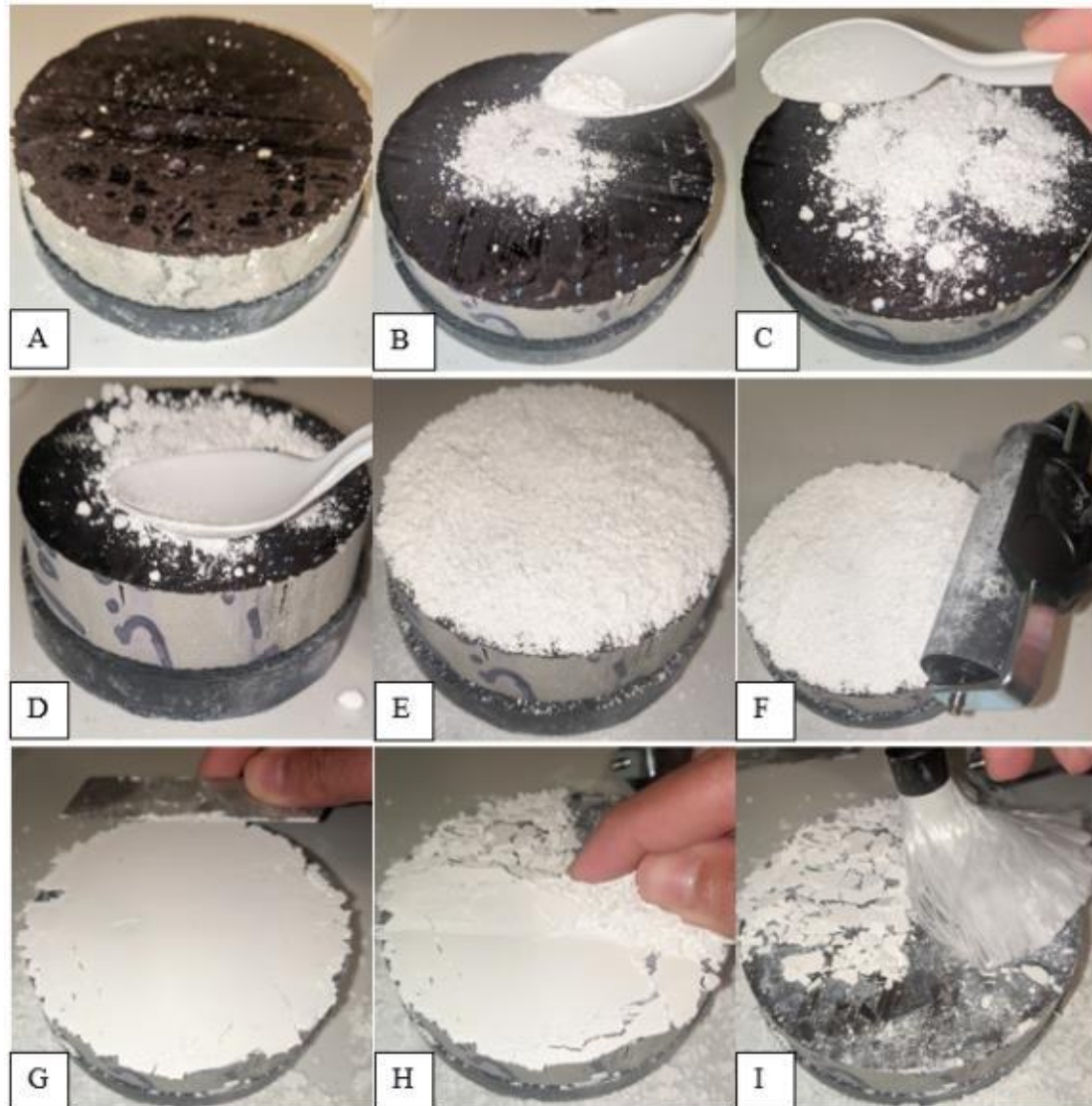


Figure 16. a-i: Enhanced Air Void Contrast Procedure.

4.8.3 Correcting the Black Surface Procedure

Using the handheld magnifying lens, inspect the surface to find any non-air void features that have been filled with the Barium Sulfate powder, such as small cracks or voids in the aggregates. Using a black permanent marker, color in the features found in the previous step.

4.8.4 Mineral Oil Procedure

1. Fill the small glass dropper bottle (30ml) with mineral oil.

- a. Once filled, this step can be ignored in future iterations until empty.
2. Apply 5-6 drops of mineral oil to the sample surface (Figure 17a) and use a nitrile gloved finger to spread the oil as seen in Figure 17b. Add additional drops of mineral oil as needed, followed by spreading with a nitrile gloved finger.
3. Remove excess oil using the 4" rubber squeegee, by placing the squeegee on the most outer edge of the sample from the user, pulling the squeegee towards the user, in one single pass, applying even pressure throughout the pass as seen in Figure 17c. Wipe the edge of the squeegee clean with a clean paper towel, and complete a second pass 90° to the original pass of the squeegee. If excess oil is still visibly present, repeat this process as necessary.
4. Lightly wipe the surface with a clean microfiber towel.

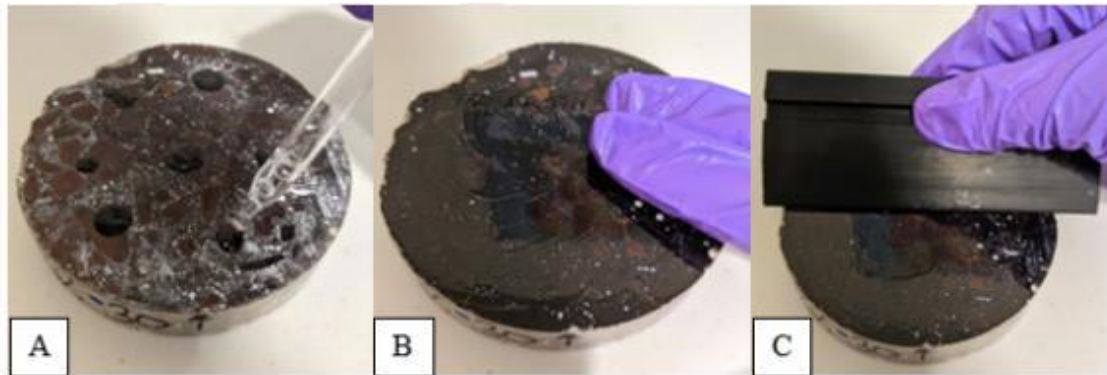


Figure 17. a-c: Mineral Oil Application.

4.8.5 Completed Surface

An example of a completed, contrast enhanced, sample for ASTM C457 assessment can be seen in Figure 18.

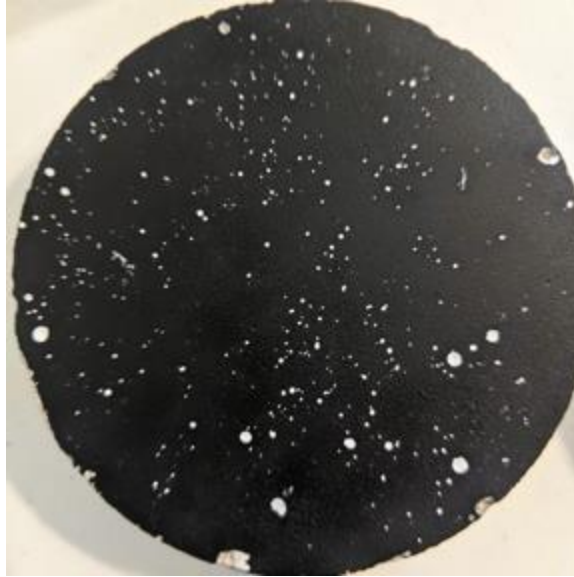


Figure 18. Completed surface for hardened air void analysis.

5. ASTM C457 AND ALTERNATIVE CHARACTERIZATION METHOD

5.1 Methodology

In this step, the concrete samples produced from Chapter 3 and contrast enhanced from Chapter 4 were analyzed in accordance with ASTM C457-16 Procedure A (linear traverse method) and Procedure B (modified point and count method). The procedures outlined in Chapter 4 meet the sample preparation requirements of ASTM C457 Procedure C (contrast enhanced method). ASTM C457 states that both Procedure A and B can be performed on a contrast enhanced sample if additional petrographic data is not required, and additional information can be determined from the mix design (ASTM C457). Additionally, completing Procedure A and Procedure B on the contrast enhanced samples will provide better correlation to the future alternative air void characterization method, which will also be completed on the same contrast enhanced samples. The hardened air void analysis was completed using a Linear Traverse Machine by Humboldt connected to a computer for data processing, which meets the requirements of ASTM C457. Although the system is called a Linear Traverse Machine, the provided software is geared towards completing a modified point and count procedure (Procedure B), in that the stage moves and stops at pre-determined points and information is recorded based on what is observed at each point. Despite this, a linear traverse procedure (Procedure A) can also be completed using the Humboldt Linear Traverse Machine, however, the data must be collected and managed and calculated with excel (or other data management software), in combination with the linear traverse software. The Linear Traverse Machine hardware set-up can be seen in Figure 19.



Figure 19. Linear Traverse Machine by Humboldt.

5.2 Results

In order to ascertain the quality of each polishing level discussed in Chapter 4, Procedure A and B was completed on samples polished at 260 μm , 125 μm , 70 μm , 30 μm , 15 μm , and 6 μm which is all of the diamond polish discs sold and acquired from Allied High Tech Products. Additionally, each sample was analyzed a total of three times each. Since the samples produced in this study are circular, there could be variability in the assessment in regard to where the analysis begins relative to the sample. Both procedures require the starting point of a circular sample to be near the top and at one end of the initial traverse. However, slightly different results would be obtained if that starting point were different. To control this, and to provide average results for each sample, a mark was placed on the side of the sample, and for the first assessment the mark was pointing in the north direction in regard to the traverse stage. ASTM C457-16 Note 8 describes the “East-West (E-W)” direction as the direction from the operator’s left to right, and “North-South (N-S)” refers to the direction perpendicular to E-W, that is, the directions are analogous to those on a map. Therefore, Procedure A and B were completed on one

sample in which the mark was oriented towards the north direction. Then the sample was rotated 45° to the east, such that the mark was oriented towards the north-east direction, then Procedure A and B were completed. Lastly, the mark was rotated an addition 45° to the east, such that the mark was oriented towards the east direction, and Procedure A and B were completed one last time on the same sample. The three data points for each polished surface were then average and reported in Table 9 and graphically presented in Figures 20-23.

Table 9. ASTM C457 Procedure A and B Results.

Control - ASTM C231 Measured Air Content = 1.8%, SAM No. = 0.65							
Procedure A							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	2.1	2.2	1.9	2.1	2.2	2.0	2.1
Spacing Factor, in	0.0191	0.0203	0.0193	0.0221	0.0193	0.0212	0.0202
Specific Surface, in ² /in ³	422	472	459	430	410	430	437
Procedure B							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	1.7	1.6	1.6	2.0	1.7	1.7	1.7
Spacing Factor, in	0.0212	0.0195	0.0218	0.0158	0.0210	0.0183	0.0196
Specific Surface, in ² /in ³	437	420	451	411	431	419	428
Low Air - ASTM C231 Measured Air Content = 3.5%, SAM No. = 0.59							
Procedure A							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	3.7	3.5	3.7	3.8	3.6	3.7	3.7
Spacing Factor, in	0.0128	0.0131	0.0140	0.0155	0.0133	0.0120	0.0135
Specific Surface, in ² /in ³	591	599	585	572	583	573	584
Procedure B							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	3.4	3.3	3.3	3.3	3.2	3.4	3.3
Spacing Factor, in	0.0134	0.0148	0.0140	0.0151	0.0153	0.0132	0.0143
Specific Surface, in ² /in ³	572	582	581	573	582	571	577

Medium Air - ASTM C231 Measured Air Content = 4.8%, SAM No. = 0.39							
Procedure A							
Polish Pad Grit Size	260μm	125μm	70μm	30μm	15μm	6μm	AVG
Air Void Content, %	5.1	5.0	5.2	5.1	5.2	5.0	5.1
Spacing Factor, in	0.0096	0.0092	0.0098	0.0094	0.0098	0.0093	0.0095
Specific Surface, in ² /in ³	627	632	624	639	641	619	630
Procedure B							
Polish Pad Grit Size	260μm	125μm	70μm	30μm	15μm	6μm	AVG
Air Void Content, %	4.2	4.3	4.3	4.1	4.2	4.3	4.2
Spacing Factor, in	0.0110	0.0112	0.0109	0.0115	0.0107	0.0102	0.0109
Specific Surface, in ² /in ³	598	605	613	594	610	611	605
High Air - ASTM C231 Measured Air Content = 8.7%, SAM No. = 0.12							
Procedure A							
Polish Pad Grit Size	260μm	125μm	70μm	30μm	15μm	6μm	AVG
Air Void Content, %	8.5	8.2	8.3	8.4	8.4	8.5	8.4
Spacing Factor, in	0.0072	0.0074	0.0075	0.0071	0.0073	0.0079	0.0074
Specific Surface, in ² /in ³	723	711	722	709	728	735	721
Procedure B							
Polish Pad Grit Size	260μm	125μm	70μm	30μm	15μm	6μm	AVG
Air Void Content, %	8.9	8.9	8.9	9.1	8.5	9.0	8.9
Spacing Factor, in	0.0069	0.0071	0.0073	0.0068	0.0069	0.0064	0.0069
Specific Surface, in ² /in ³	769	772	775	789	742	782	772

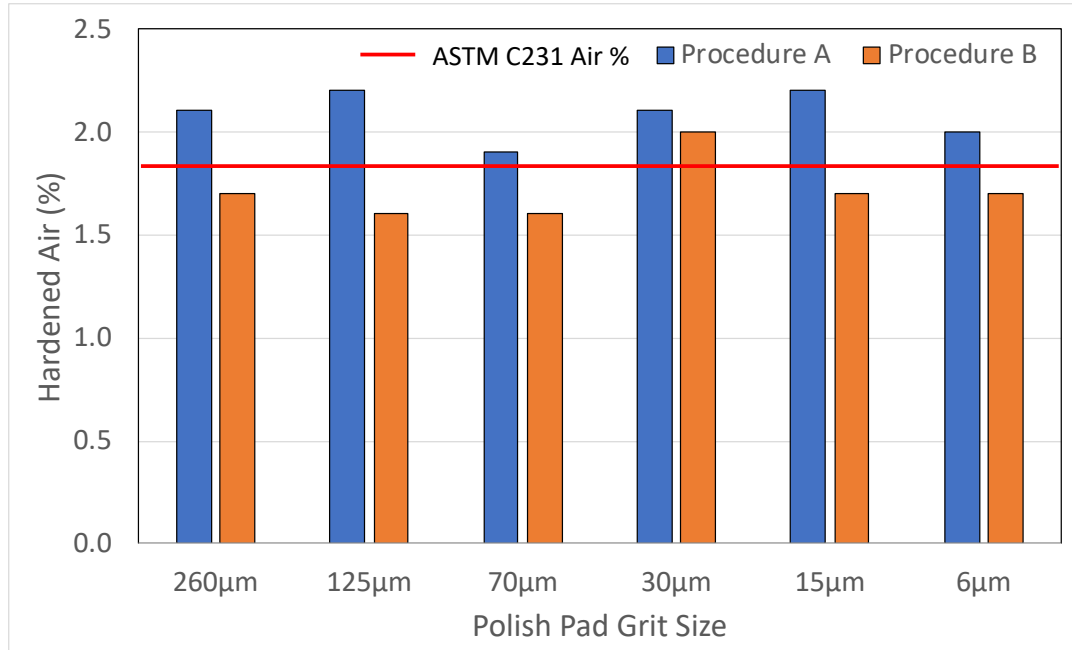


Figure 20. Control (2% Target) Hardened Air Void Comparison.

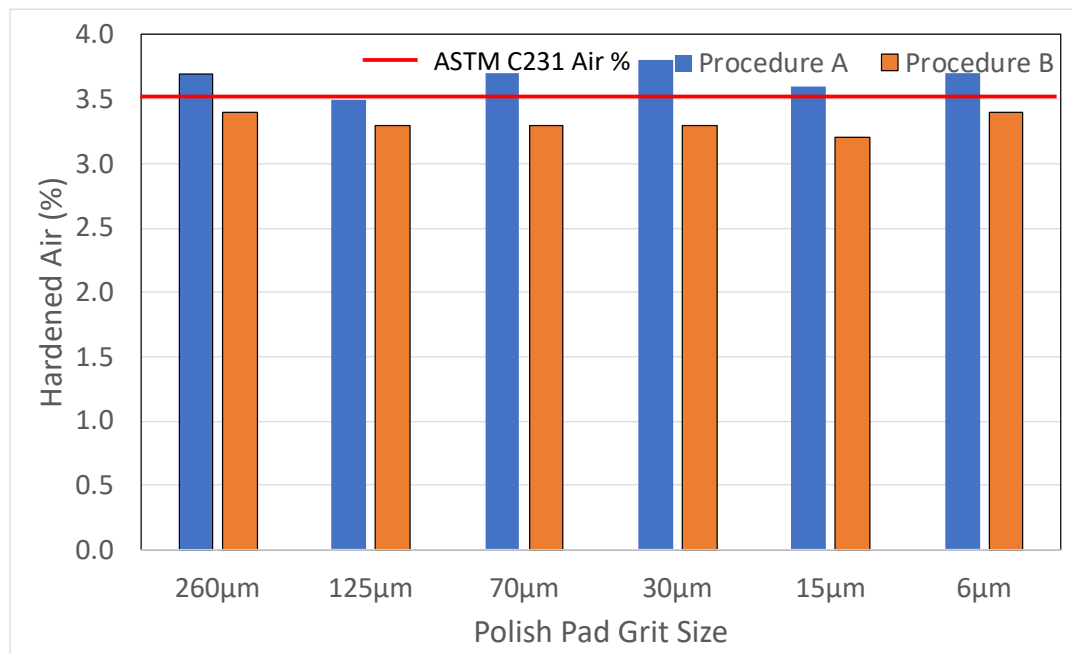


Figure 21. Low Air (3-5% Target) Hardened Air Void Comparison.

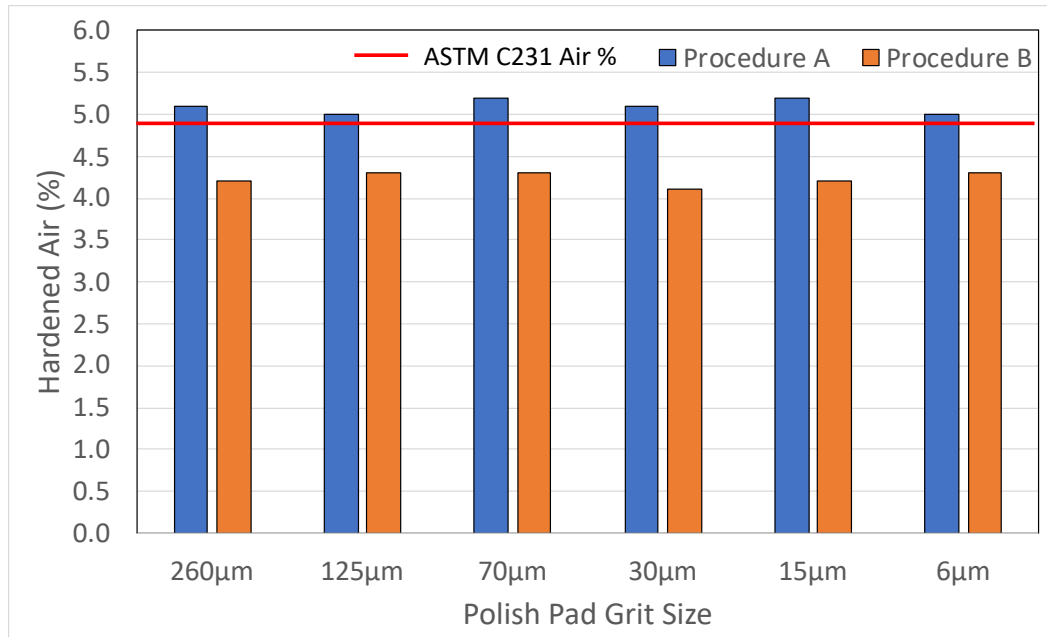


Figure 22. Medium Air (5-7% Target) Hardened Air Void Comparison.

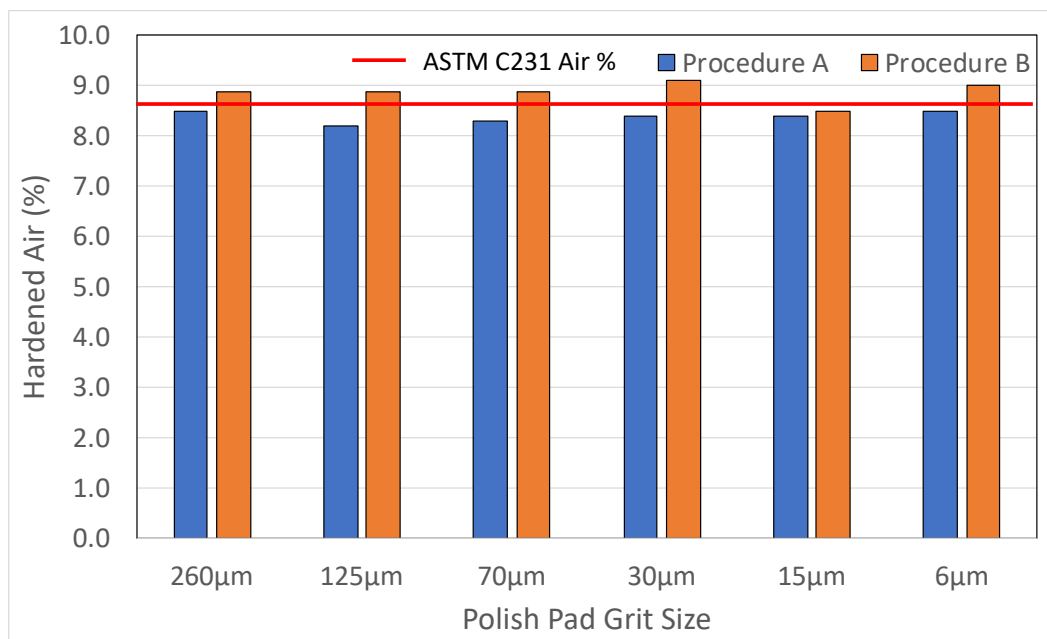


Figure 23. High Air (>8% Target) Hardened Air Void Comparison.

As seen in Table 9 and Figures 20 – 23, both procedures produced hardened air void percentages similar to the fresh air content measured via ASTM C231. Observing

the hardened air void values across each polish pad grit size reveals no significant change between subsequent smaller grit sizes. The average percent difference across each polish pad grit size was 3.8% with a range of 1.4 – 7.9%. This amounted to an average hardened air void difference of only 0.1 across all samples. Comparing the average hardened air void percentage across all polish pad grit sizes to that of the ASTM C231 fresh air percentage, reveals an average percent difference of 6.7% with a range of 2.1 – 14.6%. These fresh versus hardened air void percentage differences are also consistent with the reported values in the literature (ACI 201, 2016; Ley et al., 2012; Pham & Crammer, 2019; Taylor et al., 2021). It is also observed that, on average, Procedure A produced results that were slightly higher than the ASTM C231 results, and Procedure B produced results that were, on average, below that of ASTM C231 results. This result is reversed with the High Air samples. This is believed to be due to the nature of the testing in which, with Procedure B, pre-determined stops are established, and the stops can miss an air void and/or be on the edge of an air void, and it is up to the operator's opinion whether to count the stop in an air void or not. For consistency purposes the same operator completed all of the analysis, such that the decision was subject to the same individual. According to Hover (2006), this decision process can have a higher effect on the measured air void percentage with specimens that contain lower air percentages, as the air voids could be smaller and/or more spaced out. This provides further insight as to why the Procedure B values were consistently lower than Procedure A values, aside from the High Air samples.

In regard to the spacing factor and specific surface values, they also did not show any significant difference between the different polish pad grit sizes. The results for both

are also as expected, and in the appropriate range and agree with the literature (ACI 201, 2016; Ley et al., 2012; Pham & Crammer, 2019; Taylor et al., 2021). When comparing between the two procedures, an average percent difference of 7.5% with a range of 3.1 – 7.0% was observed for the spacing factor, and an average percent difference of 3.5% with a range of 1.2 – 6.7% was observed for the specific surface values. Also, the spacing factor values are consistent with the measured Super Air Meter (SAM) numbers, such that a SAM number of 0.20 or less typically produces a spacing factor of 0.008” or less. As seen in Table 1 the control, low air, and medium air mixtures all obtained a SAM number above 0.20, and all of those mixtures recorded a measured spacing factor of above 0.008”. Whereas the High Air mixture produced a SAM number of 0.12 and recorded an average spacing factor of 0.0069”. As indicated by Ley et al. (2013) this is typically the case approximately 90% of the time and was the case with the measured samples in this study.

Overall, the results of the ASTM C457 characterization were as expected and in an appropriate range. Additionally, the results indicate that, for the samples tested, it is not necessary to polish any lower than 260µm, as long as the 250µm was polished sufficiently as outlined in chapter 4.3 sample polishing. This result indicates that time can be saved on the sample preparation step as well as cost, as the subsequent polish pads do not need to be purchased. It should be noted that, the samples produced and polished in this study did not exhibit any air void degradation or elongation from the polishing process, and thus required no paste strengthening with lacquer or by other means as described in ASTM C457 (ASTM C457-16) as well as other authors (Ley, et al. 2012; Pham &Cramer, 2019). This fact could be the factor that allowed for the subsequent

polishing with finer grits to be insignificant, as the authors did not investigate the impact of paste strengthening. Additionally, all samples polished in this study had a minimum cured age of 28-days, which will lead to a stronger paste. It is likely that paste strengthening is required for younger samples. Additionally, Hover (2006) indicates that faulty or incomplete specimen preparation can be a source of hardened air void characterization error. Therefore, care should be taken to observe the quality of each subsequent polish step with a microscope or handheld magnifying glass as discussed in chapter 4.

5.3 Keyence Digital Optical Microscope

5.3.1 Hardware

For this analysis a Keyence – VHX 7000 series was used, which is the most current available microscope sold by Keyence. The Keyence – VHX 7000 digital optical microscope can be seen in Figure 24.



Figure 24.: Keyence – VHX 7000 Series Digital Optical Microscope.

As seen in Figure 24, the Keyence microscope consists of a monitor/computer unit, the microscope unit (automated stage, optical lenses, and various lighting), and peripherals (controller unit, keyboard, and mouse). The 7000 series is the 5th generation of the Keyence microscopes and delivers measurements with ultra-high accuracy in 4K resolution. The microscope unit can deliver magnifications in the range of 20x – 6000x. This is done automatically, without the need of switching objective lenses or other parts. The stage, in which the sample rests on, is also completely motorized and automatic. The stage has a movement range of 4-inches (100 mm) in both the X and Y horizontal directions, which can easily be manipulated by a mouse click or using the controller unit. The controller unit is also very intuitive, and can control many functions include, focus, magnification, stage orientation, lighting, and more.

5.4 Software Capabilities

The Keyence – VHX 7000 series comes with on board stitching, counting, and measuring software, which is key for the hardened air void analysis. The stitching feature is the critical step that will save time in the characterization process. As with any microscope, the ASTM C457 recommended 50x magnification will not show the entire sample within the magnified image, and any assessment must be completed incrementally as the sample is re-positioned below the lens. However, the stitching feature on the Keyence – VHX 7000 will automatically capture and stitch all areas within the bounds established by the operator. The Keyence stage offers enough actuation, such that an entire 4-in. diameter (or 4-in. x 4-in. square) [100mm. in diameter (or 100mm x 100mm square)] concrete sample can be completely stitched in one stitching operation. This allows the entire sample to be characterized in one complete assessment. It should be noted that the stitching software is sophisticated enough, that it is not simply placing images next to each other, in which there could be an overlap in the analysis, but it is pixel matching hundreds of thousands of pixels across each image, such that it is, in fact, producing one complete image. On a 4-in. (100mm) diameter concrete sample, at a 50x magnification, the entirely automated stitching process can be completed in approximately 10 minutes. Following the stitching, the Keyence microscope has the ability to automatically count and measure any feature desired on the sample. However, the counting and measuring software is more efficient and accurate when the particular area of interest has a different contrast from the background. This is why contrast enhancing the samples is required, as the software will easily pick up all of the white voids from the black background. The counting and measuring process is completed automatically once the user sets the boundary of the assessment. Therefore, the operator

can count and measure specific areas or the entire area if they desire. In this instance, virtually the entire sample was analyzed by drawing a circle around the entire sample to set the bounds. A slightly smaller circle was used as the very edge of certain samples contained edge defects, that would interfere with the analysis, and thus were removed from the assessment. A fully stitched and measured sample can be seen in Figure 25.

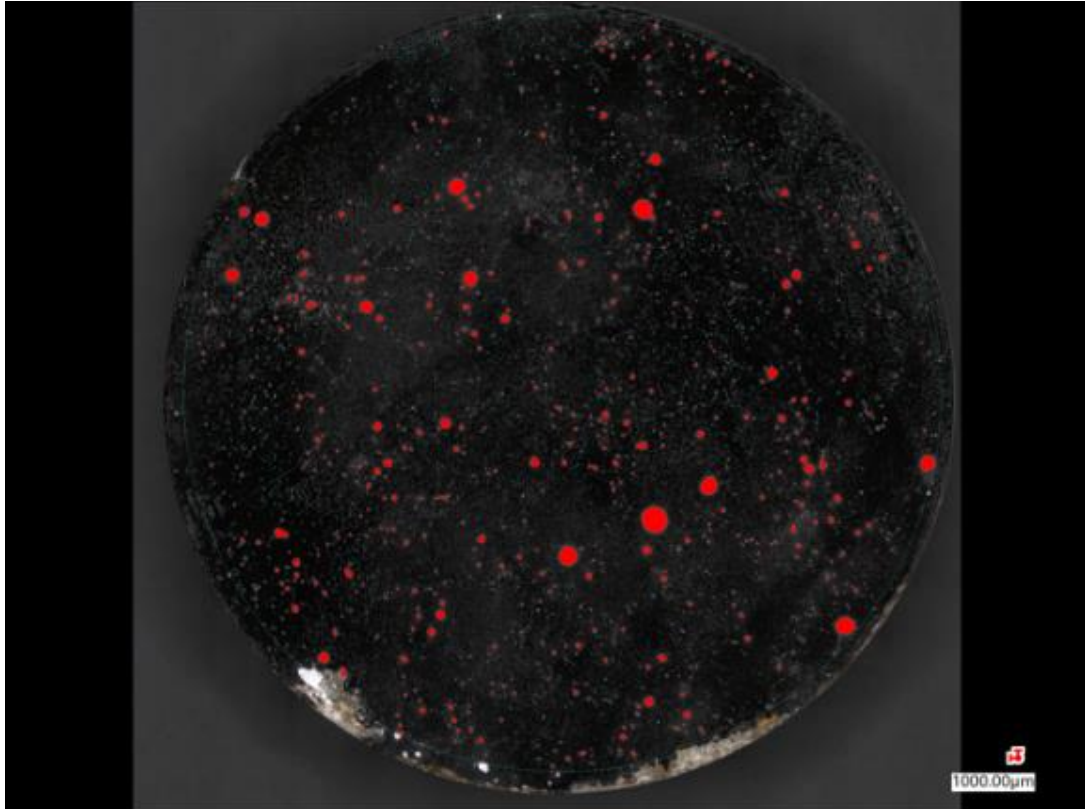


Figure 25. Stitched and Measured Sample.

In Figure 25, a contrast enhanced and polished sample from Chapter 4 can be seen, which was fully stitched, and the voids have been counted and measured. This image simply shows the sample itself, the boundary line, which was input by the operator, and the air voids that have been counted. First, the boundary can be observed as the teal circle that is just inside of the edge of the sample. As previously stated, this can be established by the operator as any desired shape. A circle was chosen as it removes

very little of the sample, while also ignoring the edges of the sample that are chipped from the saw cutting. Excluding these parts ensures that objects in those areas are not counted in the counting and measuring process. The next thing to observe are the highlighted areas in red, which also have a teal outline. These are the objects/air voids that have been counted and measured. Additionally, small, teal-colored dots can be seen throughout the sample. Those are also objects/air voids but are so small that the red color is not visible at this magnification, but only the teal outline is visible, making them appear as only teal-colored dots. These colors can also be changed by the operator, and the current color scheme is the default and was visible amongst the black and white of the sample. Once the sample has been automatically counted in this manner, the user can investigate the sample at any magnification level to either select air voids that were not previously counted or deselect air voids, in which the operator feels were not in fact air voids, such as a defect, crack, or aggregate pop out. For example, in the bottom left-hand corner of the sample shown in Figure 25, there is a small white area that is connected to the chipped off area. This white area is filled with the Barium Sulfate powder, and within the bounds of the counting and measuring area, however, it is clear that it is not an air void due to its irregular shape (non-spherical) and was part of the chipped off area. Therefore, it was removed from the analysis. Additionally, some air voids that are present outside of the counting and measuring bounds can be added in. For example, on the very bottom of the sample in Figure 2, there are a few white, spherical, areas that were not counted due to them being outside of the counting and measuring boundary. For the purposes of demonstration, these were initially left uncounted, but after the automatic counting has taken place, the operator can select these air voids to include them in the

analysis. An additional feature of the counting and measuring software is that before the analysis takes place, the operator can input specific parameters, such as a minimum, maximum, or defined range of objects to count. Hover (2006) describes that the minimum entrained air void size ranges from 0.010–0.020 mm (0.0004–0.0008 in.) up to a maximum size of approximately 1mm (0.039 in.). Entrapped air voids from improper consolidation are typically larger than 1mm (0.039 in.) and have irregular shapes (Hover, 2006). This information can be incorporated into the analysis to distinguish between entrained and entrapped air voids. Another feature of the analysis is regarding the circularity of the counted objects. The analysis measures the maximum horizontal distance and maximum vertical distance of a measured object and compares them to obtain the circularity. A circularity value of 1.00 indicates a perfect circle, as both values are the exact same. Therefore, an additional constraint the operator can input is a circularity range, such that the analysis is not counting objects that would not be considered an air void. Following, the analysis, the software can produce a window in which all of the individual objects (air voids) are shown along with their individual area, as shown in Figure 27.



Figure 26. Individual Air Void Analysis Display.

As seen in Figure 26, is a window with all of the air voids that were counted and measured in this analysis; only a portion is shown in Figure 3, but the operator can scroll the window down to see the remaining air voids. In Figure 3, the window is set to display the air voids from largest to the smallest. From this window, the operator can select an air void from the window (as shown) and the specific air void on the sample will be highlighted (shown in purple on the right-hand side of the figure). Also observed in Figure 26, is an object that was removed from the analysis, which is not highlighted in red, and appears white in color (the original color) in the upper left-hand corner of the figure. Due to the shape of this object, it is likely a defect or not an air void, which is why it was removed. This process can also be completed faster by either entering a circularity constraint as discussed previously, or assessing the window shown in Figure 26, such that

all objects/air voids are highlighted and can be removed quickly and easily. It is important to note that no circularity constraint was used in the analysis shown in Figure 26 for demonstration purposes. However, this method can still be used to quickly assess the measured air voids as a quality assurance procedure. In addition to the individual object images, an additional display window can be shown with measurement information about each object. An example of this can be seen in Figure 27.

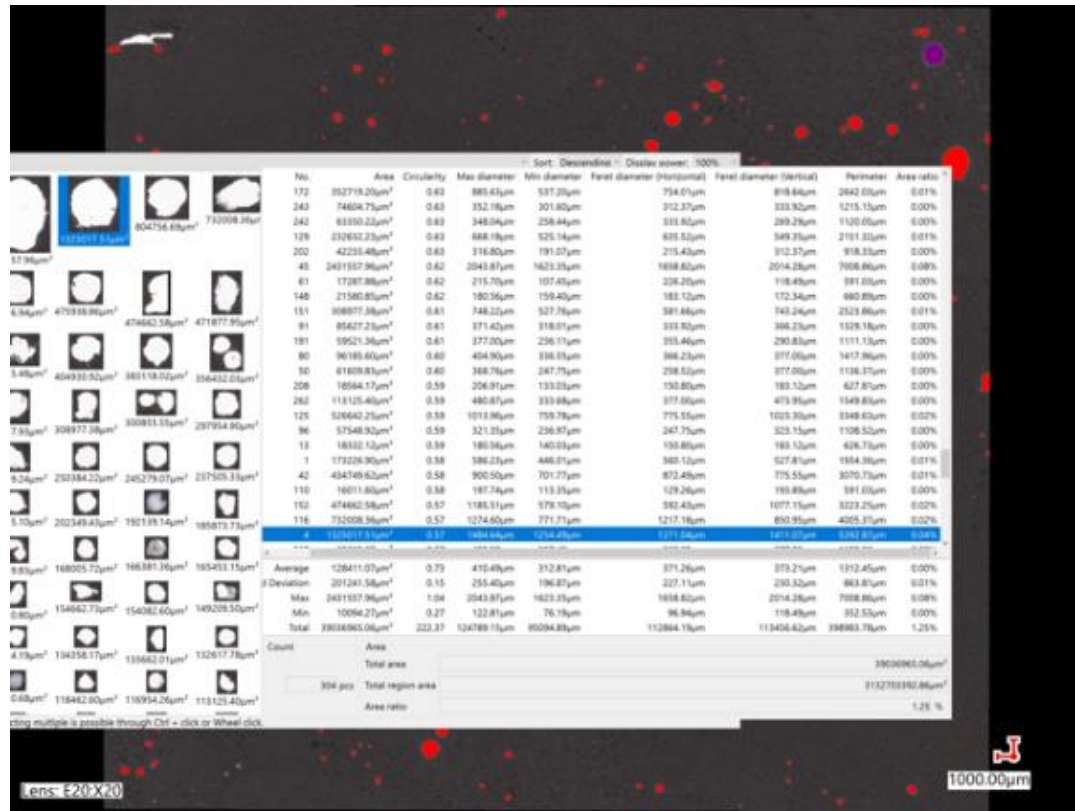


Figure 27. Object Measurement Information.

As seen in Figure 27, the display window shows a lot of information regarding each measured object within the analysis area. The first column shows the individual number of each object, which was assigned by the software beginning in the upper left-hand corner of the counted area. The second column shows the measured area of each individual object. The third column is the measured circularity, as previously discussed.

The fourth and fifth columns show the maximum and minimum diameter respectively. The next two columns refer to the horizontal and vertical Feret diameter, which is the maximum and minimum horizontal and vertical measurement. Therefore, each measurement is exactly, and only, across the horizontal and vertical distance. This is particularly helpful when the object does not have perfect circularity. The next column of information is the perimeter of each object, followed by the last column, which contains the area ratio with reference to the specific area measured. Lastly, the display window shows the total object count, the total area of the objects counted and measured, total area assessed, and the ratio (in percent) between the objects counted and measured and the area assessed. The units can be changed to whatever the operator desires and defaults to the micron scale, which is observed in Figure 27. Once the operator inputs their desired parameter constraints, and reviews the assessment, the final area ratio value can be used as the hardened air void value. The image shown in Figure 4, is an analysis of a small area of a 2% air sample, and not a complete analysis, however it is indicative of a full analysis and shows an area ratio value of 1.25% which is near the ASTM C231 measured air value of 2%. The counting and measuring software can display other information about each measured object, such as first and second moment of inertia, but the ones listed are the only ones relevant to a hardened air void analysis. Beyond specific data, the software can also present a histogram of the measured objects, which could be useful in a hardened air void analysis to assess the size variance of the air voids. Additionally, the operator can do a split screen with up to four images, that can show the original image, the counted and measured objects, the individual object window, and the histogram plot. An example of this can be seen in Figure 28.

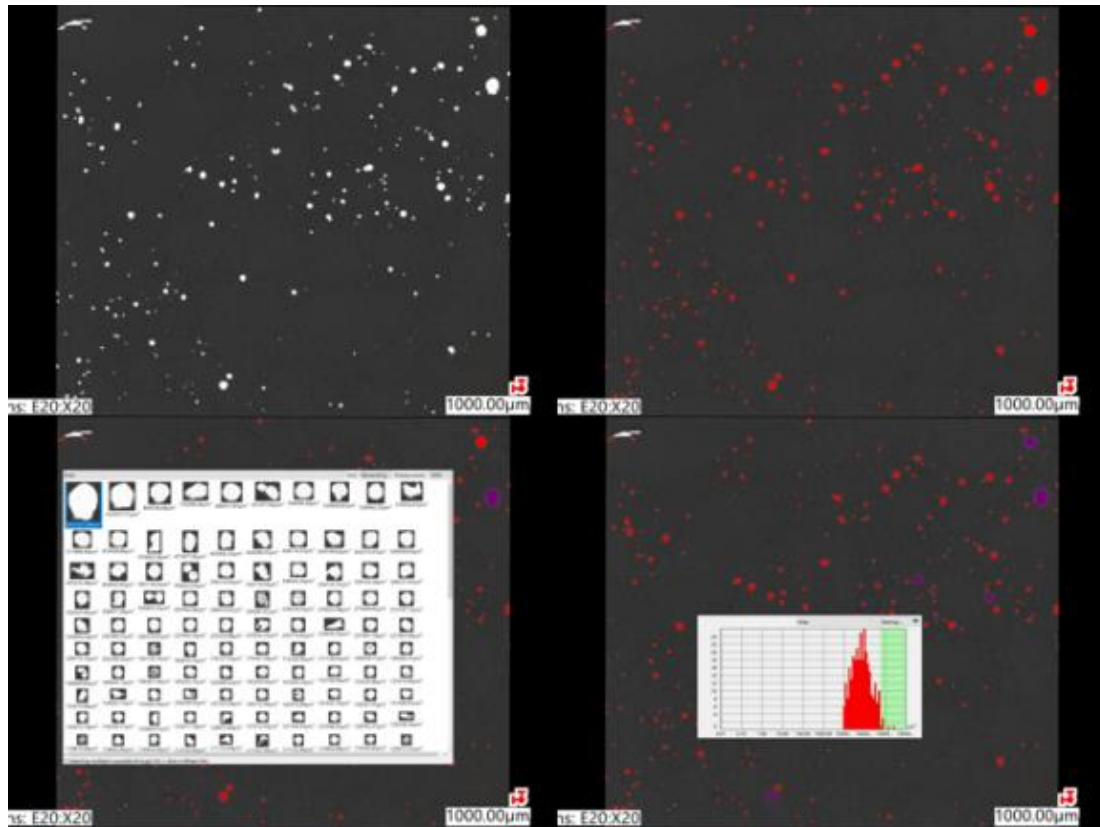


Figure 28. Split Window View on Keyence Microscope.

The split window view shown in Figure 28 can be a very useful tool when assessing the air voids in a concrete sample. Additionally, the user can highlight a specific region within the histogram plot, and those specific objects will be highlighted on the sample image. An example of this can be seen in the bottom right-hand image within Figure 28. The green bar on the histogram indicates the area of interest highlighted on the histogram, and behind the histogram, one can see the objects/air voids that have been highlighted in purple. The objects/air voids that are highlighted in purple correspond to the objects/air voids highlighted in green on the histogram. Following a complete assessment, the operator can capture images at varying magnification, and in any split window configuration, as well as download all of the data in a .csv file, which can be opened and manipulated in excel.

One last feature of the Keyence – VHX 7000 microscope is that it records and remembers all operator input settings. Therefore, upon initial use of the microscope, the operator can configure the ideal lighting, ideal magnification, and all of the counting and measuring constraints, such as minimum and maximum size, circularity, etc. Once all of this information is input, the template can be saved and anytime a new analysis is required, the original image can be opened and all of the settings can be set, based off of that image, automatically. The user does not need to individually look up each setting and manually set those values, the microscope will do all of it automatically. This feature means that any operator at any time can complete consistent analysis at any time. This feature also extends to any other laboratory who has a Keyence VHX 7000 microscope, as that original file can be emailed to that laboratory and opened on a Keyence VHX 7000 microscope (this feature is only compatible with the same series (i.e., 7000 series microscope). At that point, the settings can be extracted in the same manner. This feature can be used to establish interlaboratory consistency amongst all members of the NRRRA. Opening and accessing the emailed file is also simple as the Keyence – VHX 7000 microscope has Windows 10 installed, Wi-Fi, Bluetooth, and many other abilities like any other computer.

5.5 Calculations and Results

5.5.1 Calculations

As discussed in Chapter 2, the standard counting and measuring assessment can produce the hardened air void percentage via the area ratio value. In addition to this value the results from the assessment can be used to calculate the spacing factor and the specific surface values. This was accomplished by utilizing Equations 9, 11, 12, and 13

from ASTM C457-16 (ASTM C457-16, 2017). For consistency, this document will refer to these equations by their ASTM C457-16 equation number as opposed to assigning them new numbers. The first of the equations, Equation 9, can be seen below.

$$\alpha = \frac{4N}{T_a} \quad \text{Eq. 9}$$

Where, α is the specific surface, N is the total number of air voids intersected, and T_a is the traverse length through air. In this instance, the value of N can be the total object count and T_a can be the total horizontal Feret diameter from the Keyence counting and measuring assessment. As opposed to both ASTM C457 Procedure A and B, where air voids can be missed simply due to the inherent nature of the procedure, or they can also be excluded from analysis by the operator's opinion, this analysis would include all air voids that are within the established constraints. Additionally, using the Feret diameter would be as though a Procedure A traverse line is passing directly through the middle of all air voids, as opposed to an area above or below the exact center of an air void, which would only partially include the air void in the analysis. As seen in Equation 9, these values can be used to calculate the specific surface of the concrete sample. This value is calculated first, as it may be required in the spacing factor calculation. The next value that is required in the calculation is the Paste-Air Ratio (p/A), which can be calculated from Equation 11.

$$\frac{p}{A} = \frac{T_p}{T_a} \quad \text{Eq. 11}$$

Where p is the paste content in %, A is the Air Content in %, and T_p is the traverse length through air. As seen in Equation 11, either side of the equation can be used to determine the Paste-Air Ratio. From the Keyence counting and measuring assessment, the air content can be extracted as the area ratio value (as previously discussed) and the paste content can be extracted from the mixture design. With these two values the Paste-Air Ratio can be determined. Once this is determined, Equation 11 can also be used to back calculate T_p by multiplying the Paste-Air Ratio by T_a , which was previously obtained to calculate the specific surface. This step is necessary for future calculations. The next step in the procedure is to compare the Paste-Air Ratio value to that of 4.342. If the Paste-Air Ratio value is less than or equal to 4.342, Equation 12 can be used to determine the Spacing Factor, if the Paste-Air Ratio is greater than 4.342 then Equation 13 should be used to calculate the Spacing Factor. Equation 12 and 13 can be seen below.

$$\bar{L} = \frac{T_p}{4N} \quad \text{Eq. 12}$$

$$\bar{L} = \frac{3}{\alpha} \left[1.4 \left(1 + \frac{p}{A} \right)^{1/3} - 1 \right] \quad \text{Eq. 13}$$

Where $\bar{L}$ is the Spacing Factor (all other variables have been previously defined above). To assist with this analysis an excel file calculator has been developed and is provided with this document. A screencap of the excel calculator can be seen in Figure 29.

Total Air Void (N) [Count from Keyence Analysis]	304
Traverse Length Through Air Void [Total Feret Diameter Horizontal from Keyence Analysis]	4.44 in
Specific Surface (α)	273.9 in²/in³
Paste Percent by Volume (from mix design)	27.4 %
Air Content [Area Ratio from Keyence Analysis]	1.25 %
Paste-Air Ratio (p/A)	21.92
Spacing Factor (L bar)	0.033 in

Figure 29. Screenshot of Excel Specific Surface and Spacing Factor Calculator.

As seen in Figure 29, the calculator requires the following inputs to complete the calculations. The Count value, the Total Feret Diameter (Horizontal), and the Area ratio, from the Keyence counting and measuring analysis, as well as the paste percent, by volume, from the mixture design. The equation located in the Spacing Factor solution cell contains an IF/THEN statement that compares the Paste-Air Ratio to 4.342 and then uses the appropriate equation, therefore the user does not have to worry about which equation to use.

5.6 Results

5.6.1 Results from Initial analysis and Secondary Analysis (max diameter)

Following the aforementioned analysis and calculation procedure, the same samples prepared in Chapter 3 and analyzed in Chapter 4 were also analyzed via a Keyence – VHX 7000 microscope. For this initial study, no minimum or maximum object size constraints were used, however, a circularity constraint of 0.5 – 1.5 was used to remove objects that were not relatively circular. Based off the initial trials with the microscope, objects outside of this range, generally, appear to be defects, cracks, or an irregularity that is not an air void. A range is ideal over a specific value of 1.00, as the resolution of the measurement and minor imperfections in the sample preparation can

affect the absolute perfectness (a value of 1.00) of the circular air void. An initial analysis was completed with no minimum or maximum diameter, to ascertain the range of visible air voids that the microscope can see (and count and measure). Due to the high quality of the Keyence microscope, the resolution, and general state-of-the art nature, it is possible that this piece of equipment could observe more air voids than a traditional microscope. The results from this initial analysis can be seen in Table 10.

Table 10. Initial Hardened Air Void Analysis Results Completed with the Keyence – VHX 7000.

Control - ASTM C231 Measured Air Content = 1.8%, SAM No. = 0.65							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	1.9	2.5	2.0	2.4	2.5	2.3	2.3
Spacing Factor, in	0.0122	0.0093	0.0189	0.0110	0.0089	0.0082	0.0114
Specific Surface, in ² /in ³	642	734	582	715	749	753	696
Low Air - ASTM C231 Measured Air Content = 3.5%, SAM No. = 0.59							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	4.1	3.7	3.9	3.8	4.3	3.7	3.9
Spacing Factor, in	0.0066	0.0073	0.0069	0.0077	0.0061	0.0082	0.0071
Specific Surface, in ² /in ³	831	791	812	799	849	814	816
Medium Air - ASTM C231 Measured Air Content = 4.8%, SAM No. = 0.39							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	5.5	5.9	5.7	5.9	5.6	5.8	5.7
Spacing Factor, in	0.0050	0.0043	0.0044	0.0042	0.0052	0.0055	0.0048
Specific Surface, in ² /in ³	807	815	827	854	812	810	821
High Air - ASTM C231 Measured Air Content = 8.7%, SAM No. = 0.12							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	8.9	9.1	9.3	8.9	8.8	9.2	9.0
Spacing Factor, in	0.0041	0.0049	0.0038	0.0032	0.0044	0.0049	0.0041
Specific Surface, in ² /in ³	1665	1612	1691	1711	1698	1672	1675

As seen in Table 10, the Keyence analysis is able to produce hardened air void results. The hardened air void percentage determined from the Keyence microscope was very similar to that of the fresh air void percentage determined from ASTM C231 as well as both ASTM C457 procedures. Like the ASTM C457 results, it can also be observed that the air void percentage did not significantly change across the different polish pad grit sizes. One thing to note, however, is that the spacing factor values are lower than expected for the respective hardened air void values (ACI 201, 2016, Ley et al., 2012, Pham & Crammer, 2019, Taylor et al., 2021). Similarly, the specific surface values are higher than expected. It is important to note, that the hardened air void values determined from those references were produced with ASTM C457 procedures, and the values shown in Table 1 are the first measurements produced from the Keyence microscope. Therefore, the inherent issues with the ASTM C457 procedures exist with hardened air void values presented from the literature. As previously discussed, the primary issue with this procedure is that both procedures can completely ignore air voids if the linear traverse line does not pass over them, or if the pre-determined stop does not stop in an air void. Additionally, the human error is still present, such that the operator has to make critical judgement calls as to when and where an air void starts or begins, or whether the pre-determined stop stops in an air void or the paste.

In contrast to that, the Keyence analysis counts all objects in the pre-established counting and measuring area within the constraints established by the operator. This results in a high object count that appears in ASTM C457-16 Equation 9 and 12. In Equation 9, N (number of air voids or measured objects) is in the numerator and in Equation 12, N is in the denominator, therefore as this number gets larger, the values

from Equation 9 (Specific Surface) will get larger and the values from Equation 12 (Spacing Factor) will get smaller. This is the case as the value of N can increase at a much faster rate than the other parameters in the equation, as N will increase incrementally irrespective of the size of the object, and T_p increases proportionally to T_a , and T_a increases proportionally to the size of the object.

Therefore, with a very small air void, N will increase by a value of 1, and T_a and T_p would only increase by the measured length of that air void, which would likely not be a significant increase to the total T_a and T_p values. These results are likely due to the fact that no size constraint was applied to the initial analysis in order to allow the microscope to observe all possible objects/air voids it can. Recall that only a circularity constraint of 0.5 – 1.50 was applied with no size (diameter) constraint. Based off of the results of the initial analysis, and the manner to which the spacing factor and specific surface are calculated, a secondary analysis was completed with size constraints. Both Hover, 2006 and Taylor *et al.* 2021, state that the size of entrained air voids is in the range of 10 – 1000 μm (0.0004 – 0.039 in.) in diameter. Objects smaller than this range are typically capillary voids, and objects above this size range are typically entrapped air voids (ACI 201, 2016). The size and function of pore types in concrete can has previously been discussed and shown in Table 1.

Based on the information from the literature, the initial analysis was possibly counting and measuring both capillary pores and entrapped air voids, which as previously discussed will increase N in Equations 9 and 12 and produce higher than expected values for each. According to Hover, 2006 and Taylor *et al.*, 2021 the capillary pores are typically irregular in shape (i.e., not spherical), however if they are within a 0.5 – 1.5

circularity range, they are still being counted by the initial analysis. It is more likely that the entrapped air voids are being counted and measured as they tend to be more spherical in shape than the capillary pores. Therefore, a secondary analysis was completed in which a size (maximum diameter) constraint was applied to only count and measure objects with a diameter within a 10 – 1000 μ m (0.0004 – 0.039 in.) range. The same circularity constraint of 0.5 – 1.50 was applied in the secondary analysis as well. It should be noted that the Keyence software does not require the operator to start this analysis over from the beginning. All the operator needs to do is to open the original assessment of the desired sample and change the desired constraints and run the analysis again. There is no need to re-stitch the image or complete any other set-up. The re-analysis takes less than five minutes per sample to complete, which includes re-opening the file and setting the new maximum diameter constraints. Therefore, the analysis was completed a second time with the mentioned constrained for all samples and the obtained data was exported into Excel calculator shown in Figure 30. However, for this secondary assessment, the total measured Area Ratio from the initial analysis was used. Recall that this value from the analysis is equivalent to the Air Void Percentage as the analysis is done on the entire surface and the assessment is only counting and measuring the white objects, which are air voids filled with Barium Sulfate powder from Chapter 4. The rationale for keeping the same Air Void Percentage from the initial analysis is that the initial analysis is likely measuring an accurate total air void percentage, based off the comparison between both ASTM C231 and both ASMT C457 procedures and to that of the literature (ACI 201, 2016; Ley et al., 2012; Pham & Crammer, 2019; Taylor et al., 2021). Also, both ASTM C231, ASMT C457, and the SAM do not distinguish between entrained and entrapped air

voids, and only report total air void percentage. However, since the Keyence characterization method is counting and measuring all of the air voids in a sample and not a portion intersecting traverse lines or stops, as with procedures A and B of ASTM C457, the analysis is more realistic. Also, the Excel calculator, which uses Equation 11, requires the total measured air content (A). Therefore, the analysis will use the measured total Air Void Percentage from the initial analysis, which did not have a size constraint, but only a circularity constraint. Proceeding in this manner will also yield more realistic and comparable results to ASTM C457. By incorporating a size constraint of 10 – 1000 μ m (0.0004 – 0.039 in.) in diameter, the analysis is removing entrapped air voids, and only measuring the entrained air voids in this secondary analysis. Therefore, the secondary analysis will also provide a more realistic sense of the spacing factor and specific surface as they pertain to just the entrained air voids, as these air voids are the voids primarily responsible for alleviating hydraulic pressure during Freeze - Thaw cycles (Hover, 2006; ASTM C457; ACI 201, 2016; Pham & Crammer, 2019; Taylor et al., 2021). Lastly, proceeding in this manner also better aligns with the Excel calculator developed using the ASTM C457 equations, as the N value will not be very large and distort the results, as with the initial analysis. The results of the secondary analysis (which include the previously measured air void values from the initial analysis) can be seen in Table 11.

Table 11. Secondary Hardened Air Void Analysis Results Completed with the Keyence – VHX 7000.

Control - ASTM C231 Measured Air Content = 1.8%, SAM No. = 0.65							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	1.9	2.5	2.0	2.4	2.5	2.3	2.3
Spacing Factor, in	0.0237	0.0189	0.0215	0.0194	0.0180	0.0209	0.0204
Specific Surface, in ² /in ³	350	329	344	392	315	371	350
Low Air - ASTM C231 Measured Air Content = 3.5%, SAM No. = 0.59							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	4.1	3.7	3.9	3.8	4.3	3.7	3.9
Spacing Factor, in	0.0172	0.0194	0.0199	0.0184	0.0169	0.0189	0.0185
Specific Surface, in ² /in ³	540	504	515	534	532	494	520
Medium Air - ASTM C231 Measured Air Content = 4.8%, SAM No. = 0.39							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	5.5	5.9	5.7	5.9	5.6	5.8	5.7
Spacing Factor, in	0.0154	0.0119	0.0142	0.0123	0.0153	0.0127	0.0136
Specific Surface, in ² /in ³	584	612	550	601	590	621	593
High Air - ASTM C231 Measured Air Content = 8.7%, SAM No. = 0.12							
Keyence Microscope							
Polish Pad Grit Size	260µm	125µm	70µm	30µm	15µm	6µm	AVG
Air Void Content, %	8.9	9.1	9.3	8.9	8.8	9.2	9.0
Spacing Factor, in	0.0089	0.0071	0.0079	0.0084	0.0074	0.0071	0.0078
Specific Surface, in ² /in ³	701	731	715	684	672	709	702

As seen in Table 11 the results from the secondary analysis produced results that are more in line with expected values of similar concretes analyzed and reported in the literature (Ley et al., 2012; Pham & Crammer, 2019; Taylor et al., 2021; Mehta, 1986).

This result is positive and demonstrates that the Keyence VHX – 7000 series microscope

is capable of producing a hardened air void analysis on polished concrete samples. A full in-depth comparison of these results with that of the ASTM C457 results, will be discussed and presented in the section chapter 5.12.

5.7 Alternative Air Void Characterization Method Equipment and Procedures

Table 12 lists the required equipment necessary to complete the alternative air void characterization method for hardened concrete samples.

Table 12. Required Equipment to Complete an Alternative Hardened Air Void Characterization.

Item	Make/model	Current Cost	Website/direct link
VHX 7000 Controller	Keyence	\$39,900	www.keyence.com
Fully Integrated Camera	Keyence	\$12,000	www.keyence.com
Console “Monitor/Computer”	Keyence	\$1,600	www.keyence.com
Focus View Motorized XYZ Stage – (100mm range of motion)	Keyence	\$19,400	www.keyence.com
20-100X FI Lens	Keyence	\$5,800	www.keyence.com
100-500x FI Lens	Keyence	\$6,300	www.keyence.com
VHX Support Package	Keyence	\$2,500	www.keyence.com

5.8 Alternative Air Void Characterization Procedures

In general, the microscope software that is displayed on the monitor/computer unit is very intuitive to use. The software that the user interacts with and controls the microscope always consists of a window showing the microscope view and the user controls on the right side of the display, with large intuitive user options. An example of the VHX software user window can be seen in Figure 30.

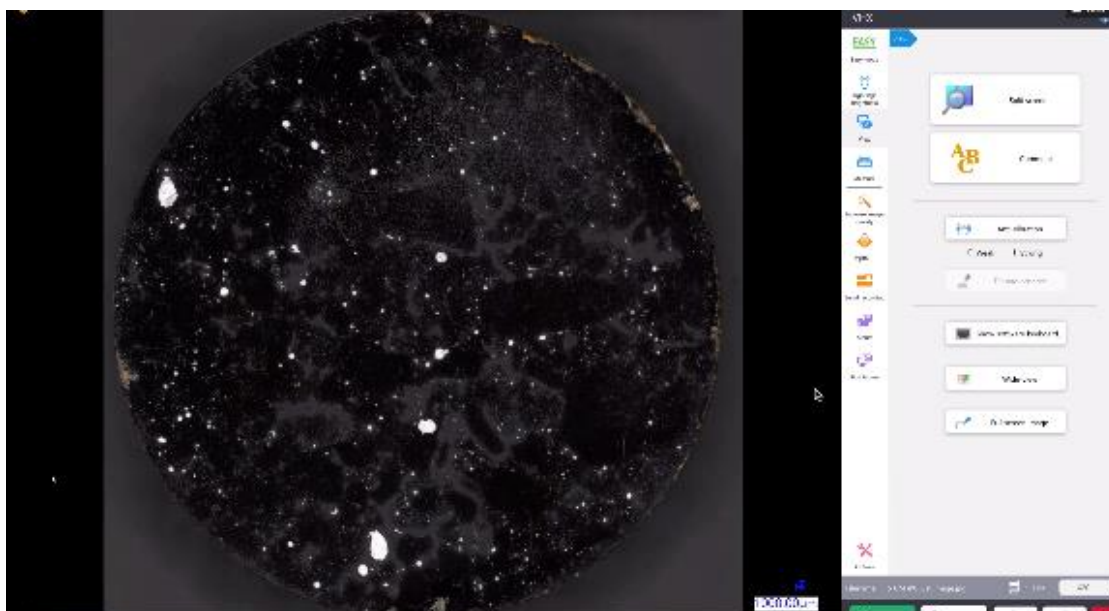


Figure 30. Screenshot of VHX Software User Window with a Fully Stitched Concrete Sample.

As seen in Figure 30, is a screen cap of the VHX software user window, which is displayed on the computer/monitor unit, showing a previously stitched concrete sample. As previously stated, the user has many analysis options on the right-hand side with many intuitive options. As seen in Figure 29 there are eight analysis options to choose from. These are, in order from top to bottom, Lighting/Brightness, View, Measure, Increase Image Quality, Depth Up, Serial Recording, Album, and Side Album. Selecting one of these options leads to more specific analysis techniques.

5.9 Initial Analysis

To complete an initial analysis the following steps are required. Prior to completing these steps, it is assumed that the microscope and all of its required hardware are set up, and calibrated and balanced. For completely new users, set up and calibration is offered by Keyence Personnel.

1. Turn on the Keyence VHX – 7000 series microscope and wait for the system and software to boot up.
 - a. After start-up, the software will ask to initialize the stage.
2. First, ensure the desired lens magnification is selected (for hardened air void characterization 50x should be selected). Initialize the stage by moving the stage vertically (up or down) by rotating the physical stage knob on the right side of the microscope unit. Move the stage up or down until the surface of the stage becomes in focus on the monitor/computer unit. After the surface of the stage is in focus, click initialize and wait for the software to automatically initialize the stage. If any errors are reported, the operator needs to re-initialize the stage.
 - a. This step can be skipped in future analysis if the microscope is put in sleep mode after each use. If it is completely powered off, then the stage needs to be initialized each time.
3. Place a polished and contrast enhanced concrete sample on the stage. Place the sample relatively on the center of the stage.
 - a. Pieces of tape or an erasable marker can be used to ensure each sample is placed in the same spot on the stage each time.
4. Click **Auto-Focus** on the controller unit to focus the lens on the surface of the sample.
 - a. At this point only a portion of the concrete sample is visible at 50x magnification, and in order for the entire sample surface area to be visible, the sample needs to be stitched together.

5. On the user analysis option area, click **Serial Recording**, then click **2-D Image Stitching**.
6. The software will ask the user to set the boundary of the image stitching. Set the stitching boundaries by moving the stage, which is accomplished by moving the dial on the controller unit. Using the dial is intuitive and movement of the stage corresponds to the same movement of the dial. Move the stage, such that the rightmost edge of the sample is in view. Click the right-most edge of the sample to set one boundary. Move the stage such that the top-most edge of the sample is in view. Click the top-most edge to set the next boundary. Continue this procedure with the left-most and bottom-most edge to establish all four boundaries. Click **Begin Auto-Stich**.
 - a. This procedure is the same for circular and square samples.
 - b. Wait for the auto-stitching to finish (this should take approximately 10 minutes or less).
 - c. At this point, the entire sample is now stitched together and visible in the user window, similar to Figure 7.
7. Once the auto-stitching is finished, click on **Measure**, then click on **Auto Area Measurement**, select the **Brightness (normal)** option (which is the default option), then click **Start**.
 - a. This process should take less than 30 seconds.
 - b. After the measuring analysis is complete, the software will display a preview of the measuring analysis based on four different auto area measurement parameters. An example of this can be seen in Figure 32.

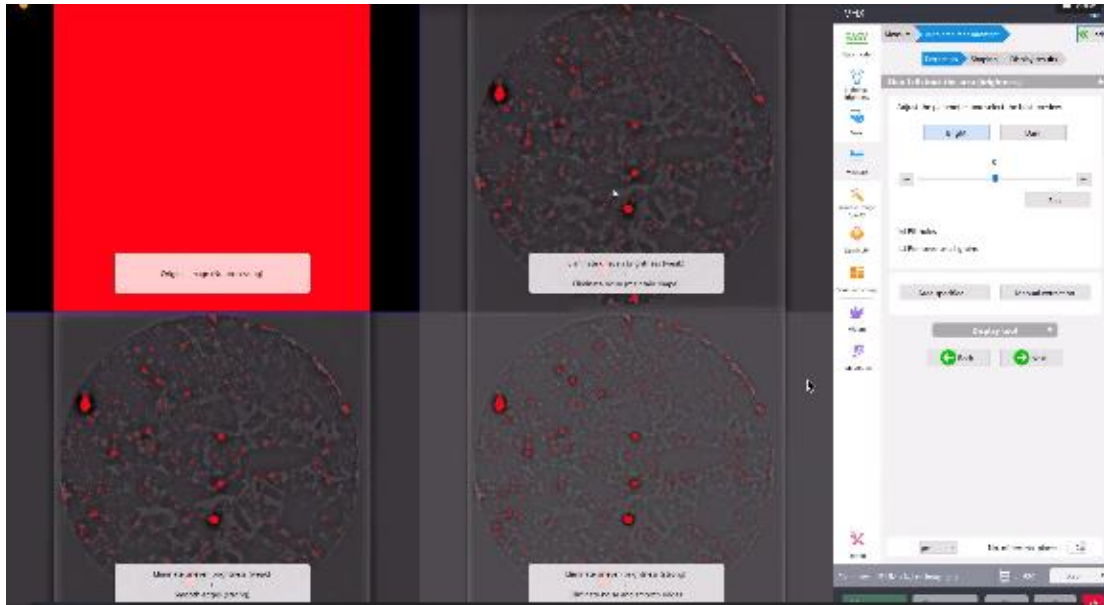


Figure 31. Example of the Measuring Preview Window.

As seen in Figure 31, the auto area measurement software displays a preview of four possible analyses that can be done based on four different parameters. These parameters are, beginning in the top left *Original Image (no processing)*, top right *Eliminate Uneven Brightness (weak) + Eliminate Noise (maintain shape)*, bottom left *Eliminate Uneven Brightness (weak) + Smooth Edges (strong)*, and bottom right *Eliminate Uneven Brightness (strong) + Eliminate Noise and Smooth Edges*. This initial preview demonstrates how the software is auto area measuring the air voids in this study. It is doing so by distinguishing between the black areas and the white areas, which in the case of this analysis, the white spots are the air voids, which were previously enhanced by the processing steps outlined in Chapter 4. Also seen in Figure 32, is that the software has counted some edge defects (non-voids) along the very outer edge of the sample. These should be removed from the analysis. Depending on the degree of the edge from the preparation step, more or less area needs to be removed from the analysis. To do this continue to step 8.

8. Click on **Area Specified...**, which will open a pop-up window with options to specify the area to be assessed. To maximize the area assessed, select **Circle**.

After clicking Circle, there are two options to specify the circle.

- a. Option 1: With **Circle** selected from above, click on the sample near the right most edge. This will establish one point of the circle edge. Establish the second point by clicking near the bottom most edge of the sample, taking care to be inside any edge defects. Lastly, to establish the last point, click near the left most edge of the sample, just inside any of the edge defects. An example of this option can be seen in Figure 32.
- b. Option 2: Click the drop-down menu **Number Specification**, click **Circle**, and type in the desired diameter paying close attention to the units (μm are the default units for length). Then position the established circle.

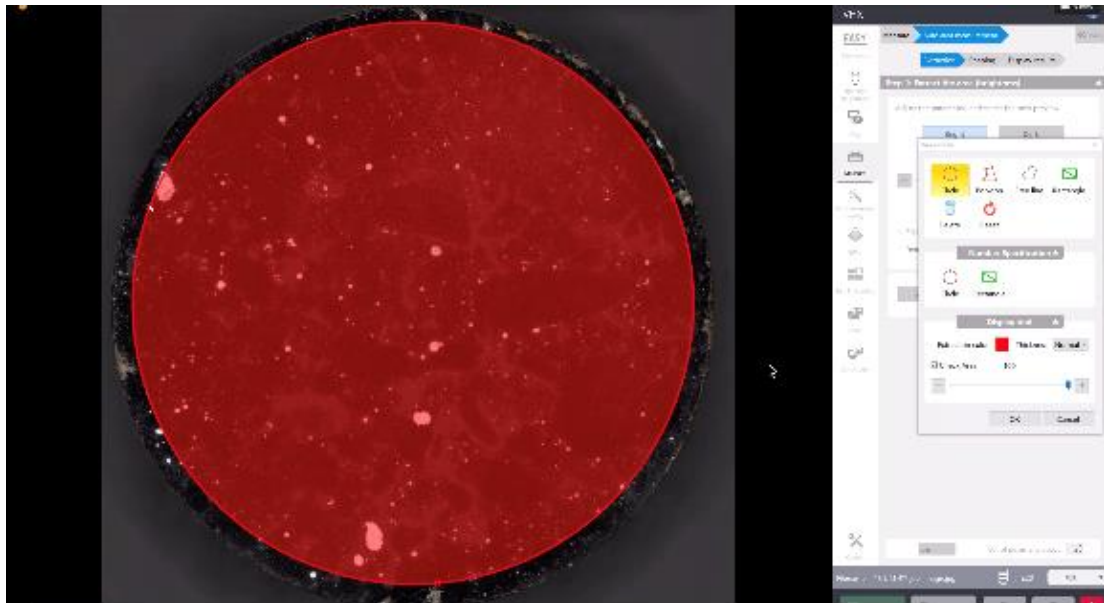


Figure 32. Example of the Area Specified Circle Procedure.

9. After establishing the area to be auto-measured and counted, select **OK**.

- a. A new preview of the measuring analysis will appear like that seen in Figure 32, that will now exclude the rough edges from the sample.
10. On the new preview window, the user needs to select which of the four options they would like to continue the analysis with. For the samples assessed in this study, the bottom left option (*Eliminate Uneven Brightness (weak) + Smooth Edges (strong)*) provided the most consistent and reliable results. Therefore, it is recommended to select that option. Click **the bottom left display window**.
11. At this point, the selected option will be seen in the window with all of the air voids highlighted red that are within the previously established area. This will also move the user into the Shaping portion of the analysis, with new option available on the right-hand side.
12. This is when certain air void size and/or circularity parameters need to be established. For the purposes of the initial analysis, only a circularity parameter needs to be established. To do this, click **Eliminate Grains**. This will create a new pop-up window. An example of this can be seen in Figure 33.

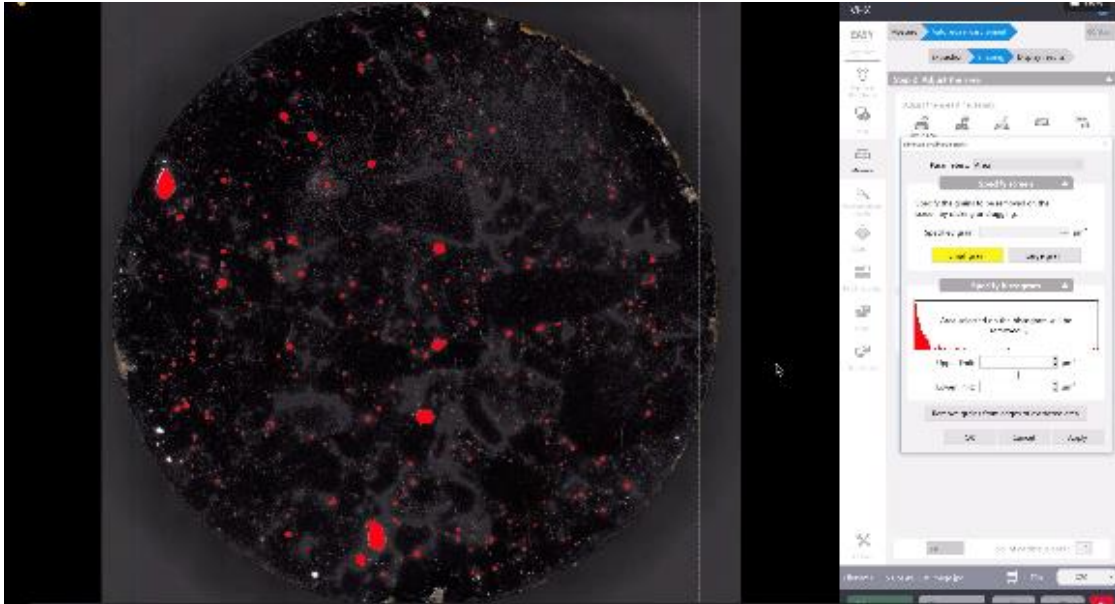


Figure 33. Example of Eliminate Grains Pop-up Window.

13. To apply a circularity parameter, click **the parameters drop down menu** (which defaults to Area, as seen in Figure 33), and select **Circularity**. The pop-up menu will ask for an upper and lower limit in individual input boxes. It is important to note that the software is asking “What grains should it eliminate from the analysis?” Therefore, the following should be done:

- a. Type in 0.5 into the Upper limit input box, click **Apply**. Clicking Apply leaves this window open, clicking OK closes the window, and the user will have to re-select Eliminate Grains to re-access this window.
- b. Type 1.5 into the Lower limit input box, click **Apply**. It is important to remember not to apply a diameter size constraint at this point; that will be done in the secondary analysis.
- c. Click **OK** to close the window.

14. Click “Next ”.

[illegible]

in Figure 34, the results of the initial analysis will be displayed on a new screen. From here the user can see the Area Ratio result, which will correspond to the Air Void % of the sample analyzed.

To save the entire analysis for future reference and manipulation click **Rec** (on the very bottom right-hand side of the screen) and save the file in the user's desired file location on the computer.

- a. This saves the entire analysis, and if opened at another time, the user can still adjust analysis options or extract different data without the sample itself or having to redo the steps up to this point.
18. At this point the user has many additional analysis options such Histogram of the measured air voids, List of Extracted Area, or other Display Tools (and options). This is where the user can complete additional desired analysis or information extraction similar to that shown in Figures 34.
19. For the purposes of calculating the desired hardened air void values, from this initial analysis, the user should write down the Area Ratio value obtained and record it in the provided excel calculator.
20. This completes the initial analysis.

5.10 Secondary Analysis

To complete a secondary analysis the following steps are required.

1. If the analysis is completed immediately following the initial analysis proceed to step 4, if not, continue to step 2.
2. If this is being completed at a later time, ensure the microscope is turned on, booted up, and the stage has been initialized (see steps 1 – 2 in the initial analysis procedure).
3. After the system is ready, click **Album**, then click **Auto Area Measurement (grain count)**. Navigate to where the desired file was saved and double click on the file. After double clicking, a pop-up window will display, asking the user to choose between AAD Playback or Normal Playback. AAD Playback is the option that contains all the previous data that can be manipulated, and Normal Playback is just the saved picture. For the purposes of this analysis, click **AAD Playback**.

4. At this point, the analysis is up to the point where the initial analysis left off. The only change that is required for the hardened air void assessment is to create a maximum diameter size parameter. To do so, click “ **Back**”, so that the analysis is in the Shaping menu.
5. In the Shaping menu, click **Eliminate Grains**. The same pop-up window will display as previously seen in Figure 10.
6. Click on the drop-down menu, and select **Max Diameter**. Double check that the units are in μm , if not either change the units or convert the values below to match the current units on the microscope). The below steps assumes μm are the current units, such that a the analysis is only counting and measuring air voids within a 10 – 1000 μm diameter range.
 - a. Type in 10 into the Upper limit input box, click **Apply**. Clicking Apply leaves this window open, clicking OK closes the window, and the user will have to re-select Eliminate Grains to re-access this window.
 - b. Type 1000 into the Lower limit input box, click **Apply**.
 - c. Click **OK** to close the window.
7. Click “**Next** ”.
8. At this point the secondary analysis is complete, and the auto area measurement results will automatically appear on the screen via a pop-up window as discussed previously.
9. It is recommended that the secondary analysis be saved as stated in steps 16 and 17 in the initial analysis, with the word “secondary” in the file name to distinguish from the initially saved file(s).

10. From the results (or saved .csv file) the user should extract, record, and input the following values into the provided excel calculator.

- a. Count
- b. Total Feret Diameter (Maximum)

11. This completes the secondary analysis procedure.

5.11 Result Analysis and Comparison

As with the ASTM C457 results, the results of the alternative hardened air void analysis were compared to ASTM C231 with respect to the polish pad grit size. For reference, both ASTM C457 procedures were included. The results can be seen in Figures 35-38.

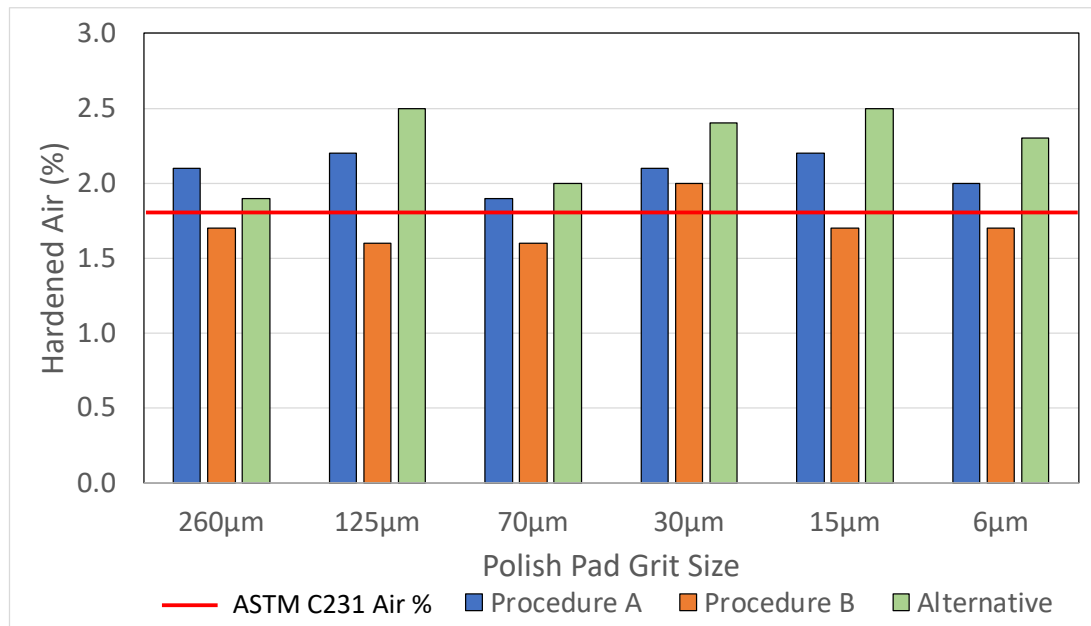


Figure 35. Control (2% Target) Hardened Air Void Comparison.

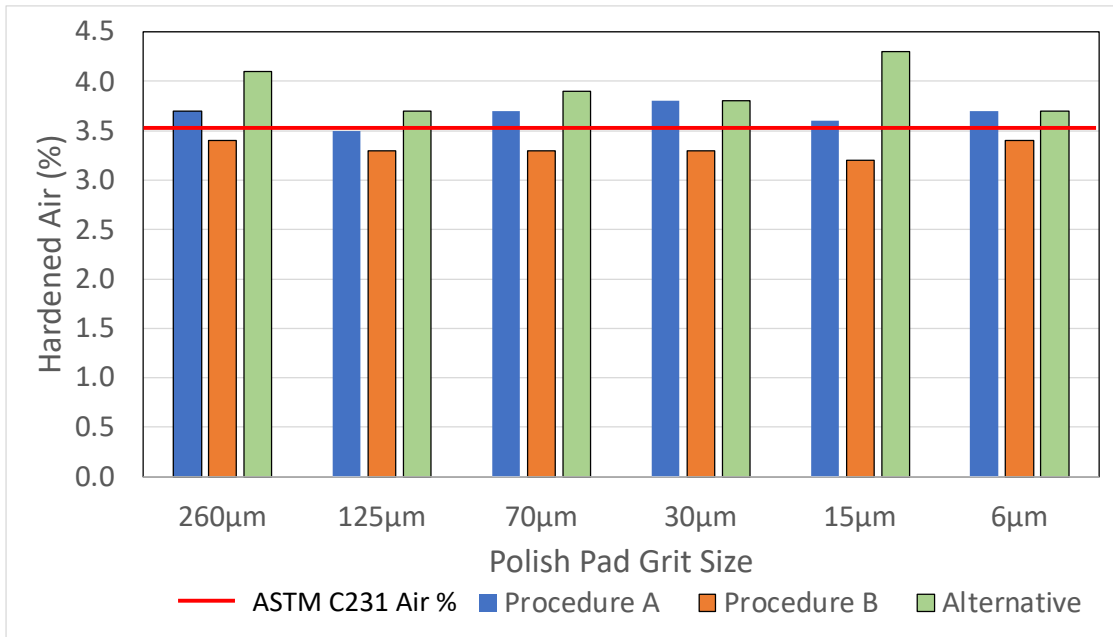


Figure 36. Low Air (3-5% Target) Hardened Air Void Comparison.

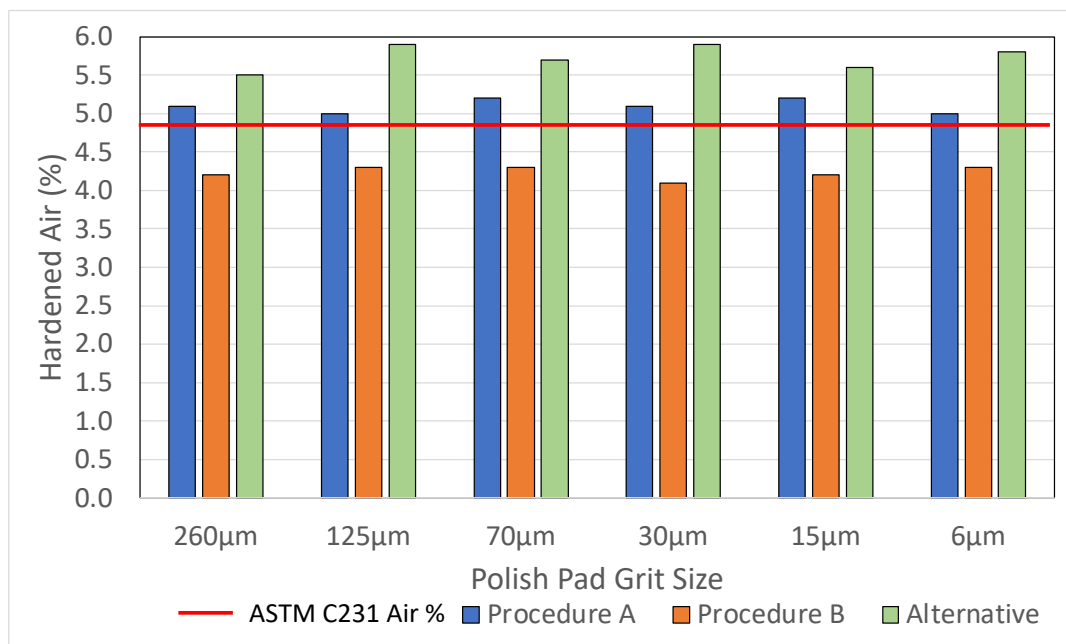


Figure 37. Medium Air (5-7% Target) Hardened Air Void Comparison.

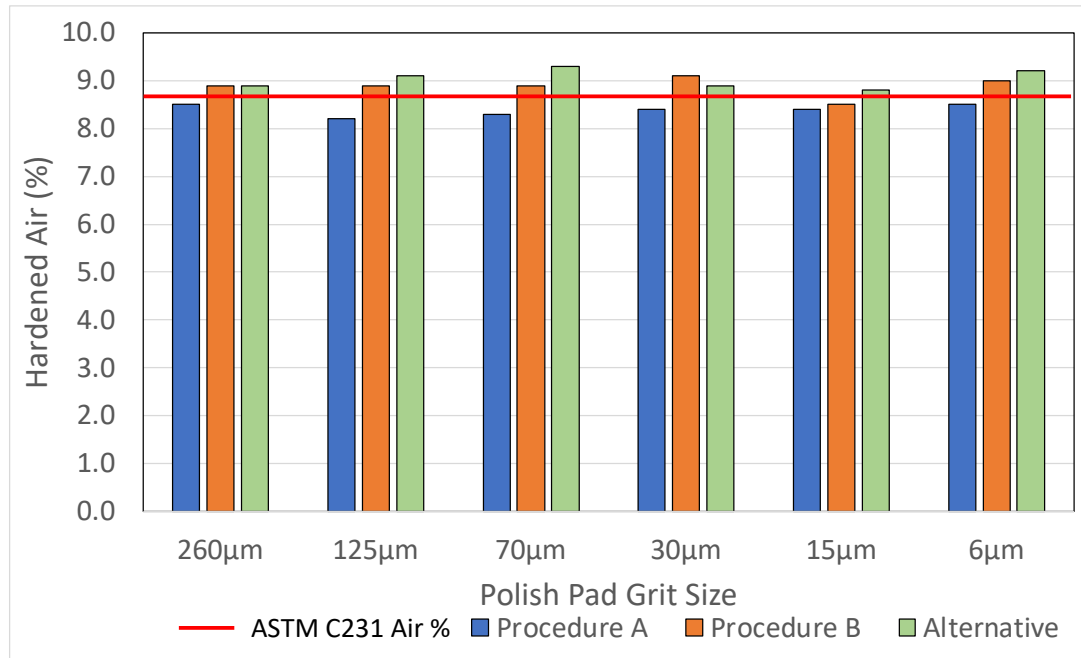


Figure 38. High Air (>8% Target) Hardened Air Void Comparison.

As seen in Figures 35 – 38, the alternative hardened air void analysis produces results that are similar to that of both ASTM C457 and ASTM C231, and are within expected hardened air void values to that of the literature (Ley, et al., 2012; Pham & Cramer, 2019; Taylor, et al., 2021; Mehta, 1986). Within the alternative analysis results, a trend is observed. In general, the alternative analysis produces slightly higher hardened air void results than the traditional methods, especially when the air void percentage is low. This result makes sense as one of the major issues with both ASTM C457 procedures is that both procedures can completely skip over air voids, in that they are not counting all the air voids in the specimen. Additionally, ASTM C457 states that the analysis should be distributed evenly over the sample area (ASTM C457). Therefore, with a circular cross section (like the ones used in this study), an evenly distributed analysis is not doable as the traverse lines would have uneven lengths across the sample (Procedure A) or there will be more or less pre-determined stops (Procedure B).

Therefore, with a circular cross section a perfectly square area is assessed to allow for an evenly distributed analysis – i.e., the traverse lines will all be the same length or there will be an even distribution of pre-determined stops. With a 4 in. (101.6 mm) diameter concrete sample, this typically correlates to approximately 2.75 in. x 2.75 in. (70mm x 70mm) square area. This is determined geometrically, as a 2.75 in. x 2.75 in. (70mm x 70mm) is approximately the largest square that can fit in a 4 in. (101.6 mm) diameter concrete sample. These dimensions are approximate as the concrete sample itself is not perfectly 4” in. (101.6 mm) in diameter. Therefore, beyond the inherent issues with missing air voids or the possibility of user error, entire portions of the specimen are left out of the analysis. The 2.75 in. x 2.75 in. (70mm x 70mm) only represents approximately 50% of the entire sample surface, therefore approximately half of the specimen is not being analyzed. These issues are enhanced when the air percentage is lower, as the air voids are statistically unlikely to be within a confined area, which is one of the reasons for the minimum area requirement in ASTM C457. With the alternative analysis, not only is the microscope counting and measuring all of the air voids in the sample (i.e., not skipping over any), approximately 98% of the sample surface is being assessed. Recall that portions of the sample edges are typically removed from the analysis due to imperfection from the sample cutting and polishing procedures, discussed in chapter 4. Therefore, with the alternative analysis over 12 in² (77.4 cm²) is assessed per sample, which is higher than the minimum assessment area required by ASTM C457 for 0.75 in. (19mm) nominal maximum sized aggregate mixtures (ASTM C457). With the traditional procedures, typically two 4 in. (101.6 mm) diameter concrete samples are

required, to meet the minimum traverse area, which, at minimum, doubles the processing time.

Additional comparison between the alternative analysis and each ASTM C457 procedure can be seen in Figures 39-40.

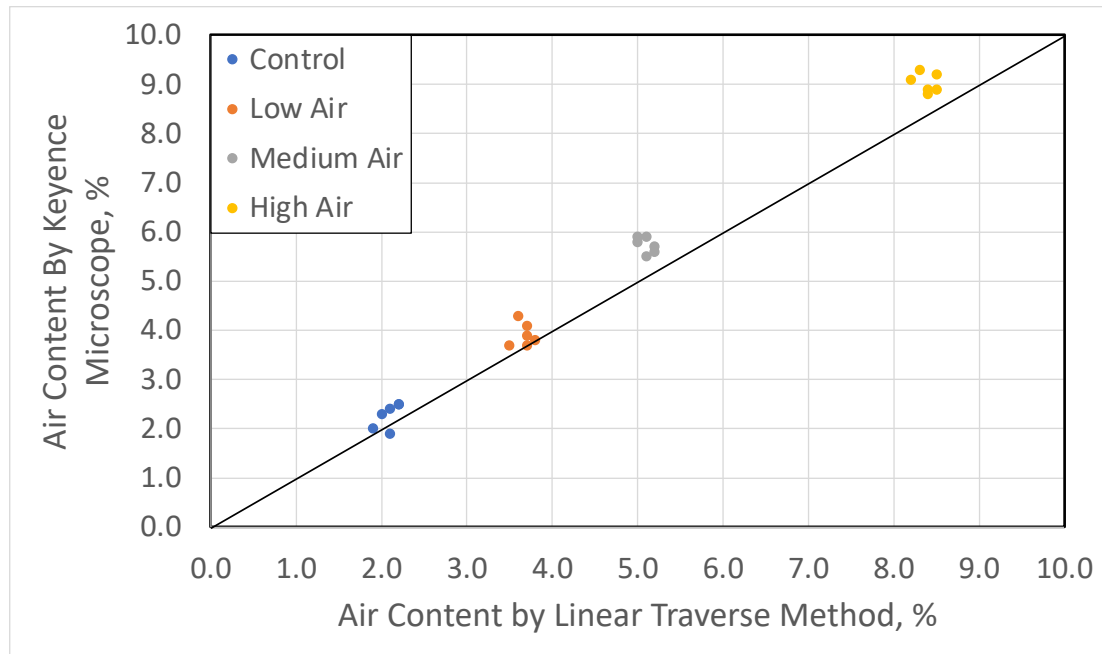


Figure 39. Comparison Between Alternative Method vs. Linear Traverse Method (ASTM C457 Procedure A).

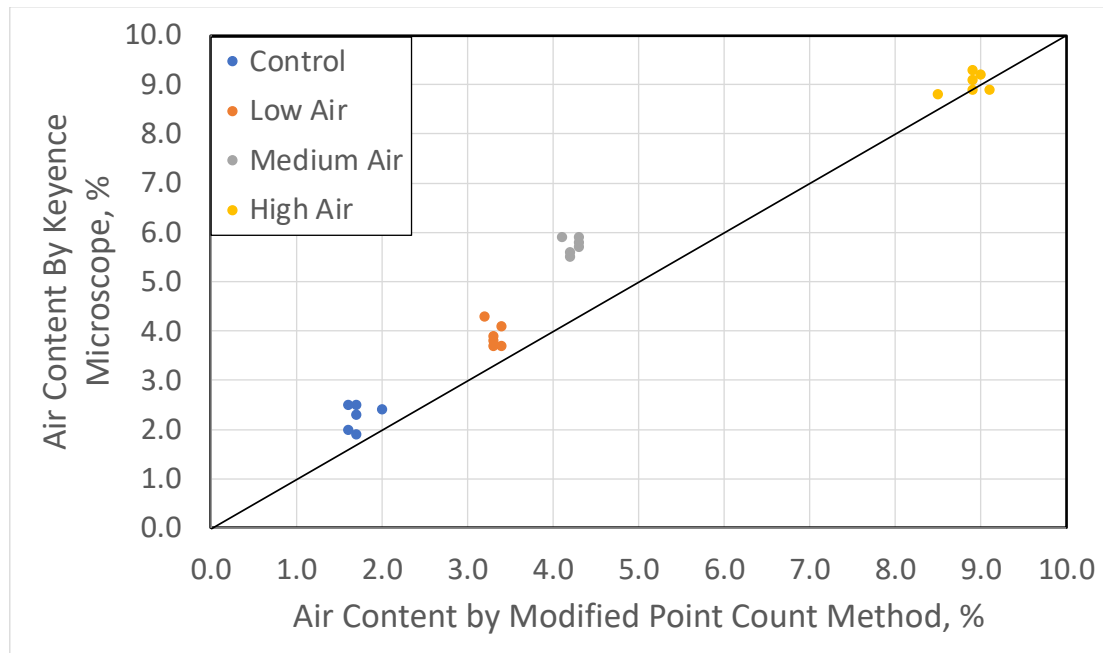


Figure 40. Comparison Between Alternative Method vs. Modified Point Count Method (ASTM C457 Procedure B)

Figures 39 and 40 provided additional visual comparison between the alternative hardened air void analysis using the Keyence VHX – 7000 series microscope to that of the two ASTM C457 procedures. As seen in both Figures, the 45° line represents a “line of equality” which corresponds to a perfect agreement between the two procedures. It is observed in both figures that there is a relatively close agreement between the alternative method and the two ASTM C457 procedures. In general, the hardened air content results favor the alternative method, in that the values are slightly higher than the ASTM C457 procedures. As previously discussed, the slightly higher results from the alternative method are due to the alternative method counting and measuring every possible air void in the sample, and not skipping over any air voids that is very likely in the two ASTM C457 procedures. Additionally, the alternative method has no user-bias or user-error, which produce a more realistic assessment of the hardened air voids in each sample. These slight differences also only correspond to an overall average of a 0.6 difference

between the hardened air void results, with an average specific difference of 0.5 and 0.7 between the alternative method and Procedure A and Procedure B, respectively. These averages are all less than the average precision data reported in Table 4 of ASTM C457-16, where the standard reports a range of air content percent of 0.82 for within lab precision and 1.16 for between lab precision deviations. Therefore, the differences between the results are likely negligible. In order to confirm that these values are in fact negligible, a student t-test was performed on the hardened air void data with a confidence level of 0.05 (p -value). The t-test confirms that the values are not statistically significant between each other, which confirms that the differences are negligible.

When comparing the spacing factor and the specific surface results between the alternative method and the two ASTM C457 procedures, the outcome is similar. A comparison of the spacing factor and the specific surface results can be seen in Figures 41 and 42. The values in these figures represent the average across all of the polishing pad sizes used in chapter 4. As discussed earlier, these results were statistically similar, thus representing their average for comparison is acceptable.

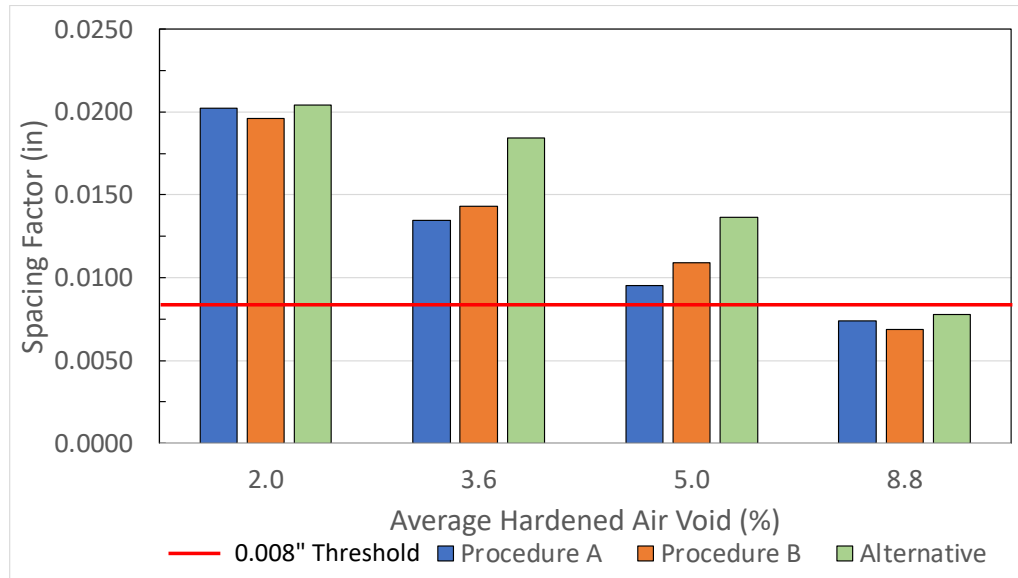


Figure 41. Average Spacing Factor Results for the Alternative Method and ASTM C457.

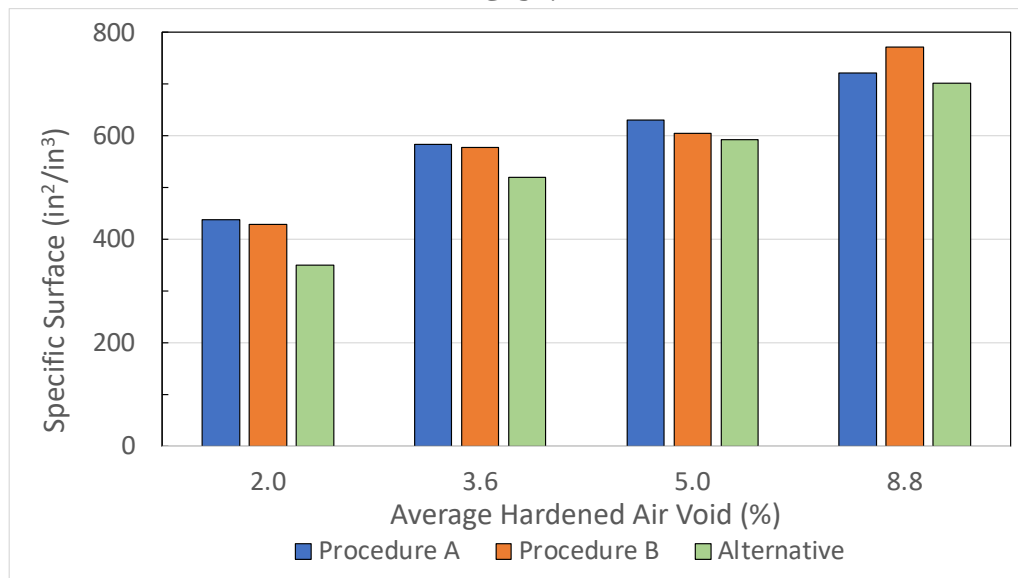


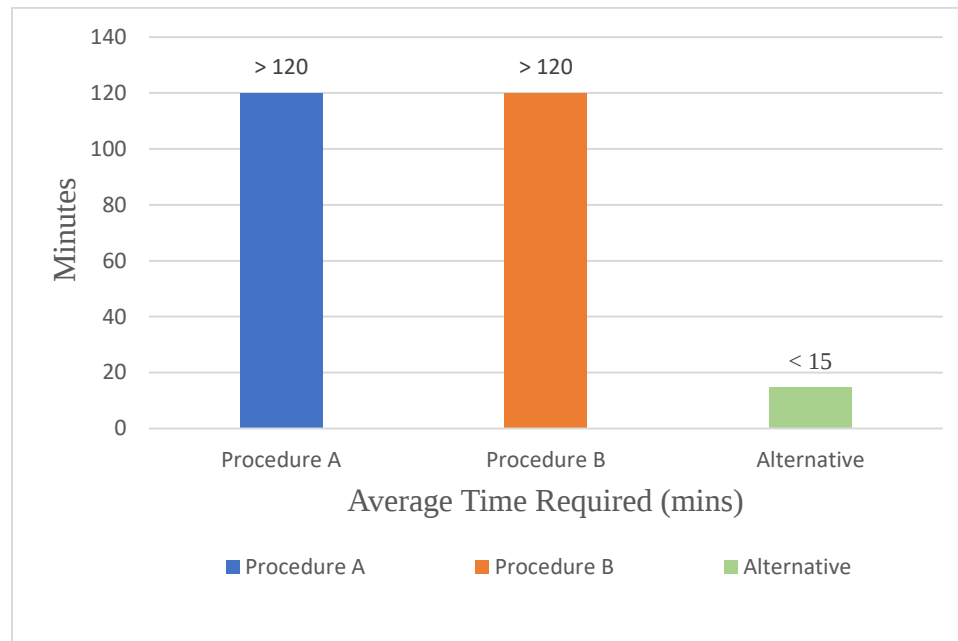
Figure 42. Average Specific Surface Results for the Alternative Method and ASTM C457.

As seen in Figures 41 and 42, the respective results are very comparable. Looking at the average spacing factor results (Figures 42), the results for each air percent are not only similar but they are as expected with reference to the literature (Ley, et al., 2012; Pham & Crammer 2019; Taylor et al.; Mehta 1986). It can also be observed that the high air samples were the only ones in this study to be below the 0.008 in. (200 μ m) threshold,

which typically corresponds to good Freeze-Thaw durability performance. The average difference between the alternative method and ASTM C457 Procedure A and Procedure B is only 0.001 and 0.002, respectively, which is negligible. Comparing the results of the specific surface analysis also shows a similar outcome. The results are not only similar, but they are also as expected with reference to similar samples assessed via ASTM C457 from the literature (Ley, et al., 2012; Pham & Crammer, 2019; Taylor et al., 2021; Mehta 1986). The average difference between the alternative method and ASTM C457 Procedure A and Procedure B is only 23 and 52, respectively, which is also negligible for these values. As was done with the hardened air void data, a statistical analysis was completed on both the spacing factor and the specific surface data to statistically confirm that the differences between the results is in fact negligible. Using the same p -value, the statistical analysis revealed that the difference between each method is negligible.

Based off the results presenting and discussed in chapters 3, 4 and 5 it can be concluded that the alternative hardened air void method using a Keyence VHX – 7000 series microscope is capable of producing quality hardened air void data that is not only reproduceable from user-to-user and lab-to-lab, but is easier, quicker, more reliable, and less reliant on human decision making. It is worth noting the amount of time saved when using the alternative air void method when compared to the two procedures in ASTM C457, which are noted in Table #13.

Table 13. Average Required Time Comparison Alternative Method vs Procedure A and B



6. CONCLUSION AND LESSONS LEARNED

The general conclusion is that when one follows the procedures outlined in this study involving sample preparation, saw cutting techniques, polishing, and contrast enhancement all contribute to an enhanced air void characterization method that improves and delivers consistent results. The ideas in this study as an alternative hardened air void method has been developed which uses a Keyence VHX – 7000 series microscope that can produce quality hardened air void data that is reproducible from user-to-user, and lab-to-lab, is easier, quicker, more reliable, and less reliant on human decision making, thus labs and in-house testing can be more reliable.

6.1 Super Air Meter- Lessons Learned

Calibration and proper training are essential to obtain repeatable results. The operator needs to batch sufficient concrete to practice and to develop a constant technique to become comfortable completing the test. Overall, the SAM is sensitive and insufficient evacuation of air between the concrete and the lid can produce improper results or errors. Additionally, improper cleaning of the rim, or sequence of steps in the procedure can also lead to an error. While the procedure is similar to ASTM C231, the test generally takes a little longer because the procedure requires data readings at three pressures that are done twice, to indicate a SAM number.

6.2 Keyence VHX –7000 Series Microscope

One of the major limitations with the microscope method, is that the sample needs to be contrast enhanced. If not, the microscope will have a much harder time distinguishing between the aggregates and the air voids. The sliced cylinders need to be flat to keep the image in focus at higher magnification.

6.3 Contrast Enhanced Procedure

It was observed that when the samples were stored for more than a day, the applied mineral oil would evaporate and that a reapplication was often necessary. Therefore, it became good practice to reapply mineral oil to each sample prior to any analysis for extra assurance.

6.4 Future Research Recommendations

Further research in this area can be done in the aspects of evaluating other air entraining admixtures from other manufacturers, test other types and size of aggregates, test other types of cements, further SAM testing and improving the SAM testing process, and evaluate other concrete mixture designs.

APPENDIX SECTION

Vita

Ruben Villarreal was born in San Marcos, Texas the son of Raul A. Villarreal and Mary V. Velasquez. He received a Bachelor of Science Technology in Engineering Technology at Southwest Texas State University in 1996. Upon graduation, he worked in industry in the fields of mechanical engineering design and manufacturer of robotic theatrical and architectural lighting in Austin, Texas, and Sunrise, Florida. In August 2020, he entered the Graduate School at Texas State University for a Master of Science in Technology Management (Construction Management) thesis option.

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