An Alternative Test Method for Determining Hardened Air Void Parameters for Concrete Pavement

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Abstract: This research has developed an alternative test method for determining hardened air void parameters that follows ASTM C457 Procedure C, but does not require image analysis software or the required training and skills to develop or use one. Instead, the alternative technique uses a sophisticated microscope that has on-board counting and measuring capabilities, that are easy to use and easy to repeat. The technique was developed and tested on concrete mixtures with four target air void percentages (control at 2%, low at 3 – 5%, medium at 5 – 7%, and high at >8%) and compared to ASTM C457 Procedure A and B. The concrete produced was a conventional pavement mixture, which was a straight cement mixture with a target 3" slump. The results show that the alternative technique is not only just as accurate as the other methods, but it reduces the analysis time, human judgment calls, and the development and maintenance of a sophisticated image analysis program, which are all typical issues with any of the three procedures described in ASTM C457. Additionally, the alternative technique produces all hardened air void parameters that ASTM C457 provides. This study also demonstrates new potential issues that could result in erroneous hardened air void results when following ASTM C457. Lastly, this study provides a detailed description of the equipment necessary to produce the alternative method including a very detailed list of steps required to obtained the hardened air void parameters.
Keywords: Concrete; Durability; Air voids; Characterization; Polish; ASTM procedures.

1. Introduction

The impact of the air void system in hardened concrete 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 [1]. 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 [2 – 10]. 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" [11]. This method, initially proposed by Brown and Pierson [12], 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), and iii) the 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. One major source of variability is the methodology involved in Procedure A and B such that multiple human judgment calls are made and that both procedures could completely miss air voids, simply by the nature of the procedure.

The significance of this study is to develop and present an alternative test method for determining the hardened air void parameters for concrete samples. As previously stated, there are many issues with the current standard (ASTM C457) which could be addressed through the development of an alternative test method.

The specific research objectives are as follows:

1) Develop an alternative hardened air void test method that captures all air voids in the sample and does not rely on human judgment calls.

2) Produce a list of procedures required to produce the hardened air void parameters.

3) Compare and discuss the results of the alternative analysis to the Procedure A and B of ASTM C457.

2. Literature review

The ability of concrete to survive a cold and wet environment is primarily dependent on the air void system in the hardened concrete [13]. This is why it is extremely important to accurately and quickly characterize the air void system as promptly as possible. 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 [6] 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 [14]. Because of the pores small size water cannot freeze inside them at typical temperatures, although the water in them may be supercooled [7]. During hydration, the voids left behind as the mixing water is removed from the system are known as capillary voids [15]. 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 [8, 14 – 17]. 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 [11]. Entrapped air voids are typically isolated from other entrapped air voids and provide no benefit to the concrete [7]. 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.

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+, Ca2+, and Cl- in water also depress the initial freezing point [18]. The damage due to freezing action is mainly dependent on the degree of saturation [19]. Damage is unavoidable if the critical degree of saturation of approximately 86% for all of the voids is reached [20]. The following mechanisms are believed to be primarily responsible for freeze-thaw deterioration [14, 21]:

1) Hydraulic pressure generated by freezing in capillaries.

2) The diffusion of supercooling gel water into capillaries after freezing.

3) Osmotic pressures resulting from the partial freezing in capillaries of solutions that have local concentration differences.

4) 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 [13].

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 [22] 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 [5] 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 ~39°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 [1, 23, 24], and so the phenomenon has the potential to reduce compressive strength [25-26]. However, Riding et al. [27] 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. 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 [1, 5, 24]. Powers [5,6] 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.

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 [1] and x-ray imaging [24], 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 [28]. However, recent studies have found that much smaller air voids and spacing factors also play an important role in freeze thaw durability performance [20, 21, 23]. 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. Methodology

In order to properly develop and compare the alternative characterization method, multiple samples were produced and analyzed. For this investigation one w/c was used (0.45) along with four target air void percentages (control at 2%, low at 3 – 5%, medium at 5 – 7%, and high at >8%). One polishing medium was investigated (diamond) at 6 different polishing pad grit sizes (260μm, 125μm, 70μm, 30μm, 15μm, and 6μm). Six different polishing pad grit sizes were used in order to determine if the degree of polish has any effect on the alternative technique.

Regarding the specific concrete constituents, a Type I/II ordinary portland cement was used for all mixtures. 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 1.
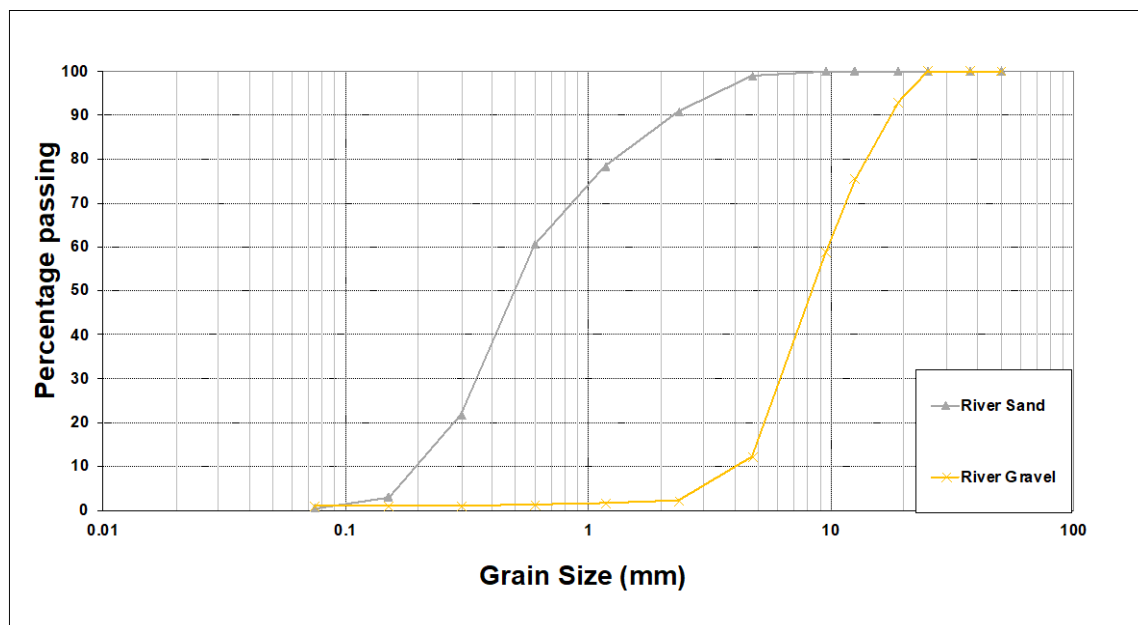

Figure 1. Particle size distribution for river gravel and river sand.

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.

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 (control: 2%). Table 1 shows the mixture proportions of the four mixtures produced.

Table 1. Concrete mixture proportions.

Material	Control	Low Air	Medium Air	High Air
Type I/II Cement (lbs/yd^3)	565	565	565	565
Water (lbs/yd^3)	254	254	254	254
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%

The rationale for only using one w/c of 0.45 is that is the standard w/c for concrete pavements that will be subjected to freeze-thaw conditions [28]. The mixture design proportions and constituents used were selected for similar reasons. The four different air contents were selected as they provided an even distribution of air void ranges for the investigation. Lastly, only one polishing medium was selected as the medium has no effect on the hardened air void parameters, however, the degree of polish could have an effect. There six polishing levels were selected, and the ones selected are the ones that are the most commonly available types and sizes for purchase in the United States.

4. Description of 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 2.

Figure 2. Keyence – VHX 7000 Series Digital Optical Microscope.

As seen in Figure 2, 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 including, focus, magnification, stage orientation, lighting, and more.

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 has to be completed incrementally as the sample is re-positioned below the lens. However, the stitching feature on the Keyence – VHX 7000 will automatically move, 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 and the user to view the entire sample in one singular image. 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 actually 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 desired assessment area. 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 of the assessment area. 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. The size of the smaller circle used for the assessment area had an approximate diameter of 3.9-in (99mm). The user can establish this same circle be used for future analysis to maintain consistency across other analysis. This was done for the analysis completed in this study for consistent comparison. A fully stitched and measured sample can be seen in Figure 3.
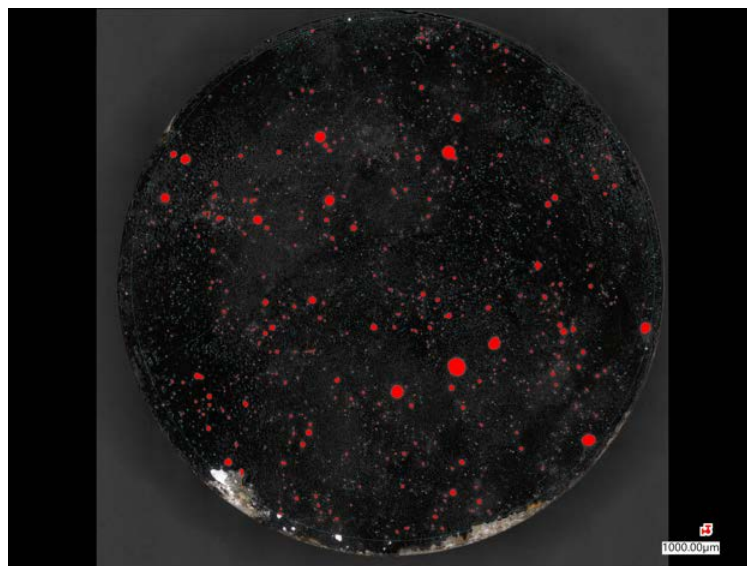

Figure 3. Stitched and Measured Sample.

In Figure 3, a contrast enhanced and polished sample 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 3, 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 (entrapped or entrained) 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 3, 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) [1] 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 [1]. 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 4.
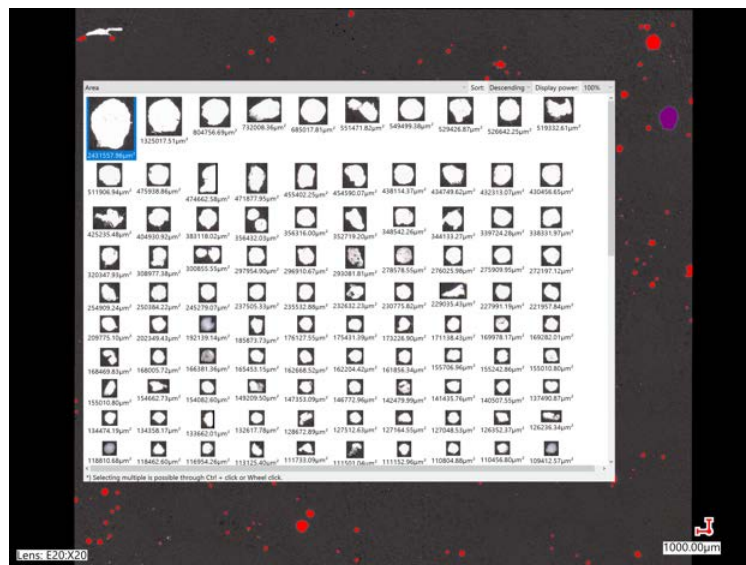

Figure 4. Individual Air Void Analysis Display.

As seen in Figure 4, is a window with all of the air voids that were counted and measured in this analysis; only a portion is shown in Figure 4, but the operator can scroll the window down to see the remaining air voids. In Figure 4, the window is set to display the air voids from the 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 3, 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, an entrapped air void, which tend to be irregular in shape, or not an air void at all, 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 3, 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 3 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 5.
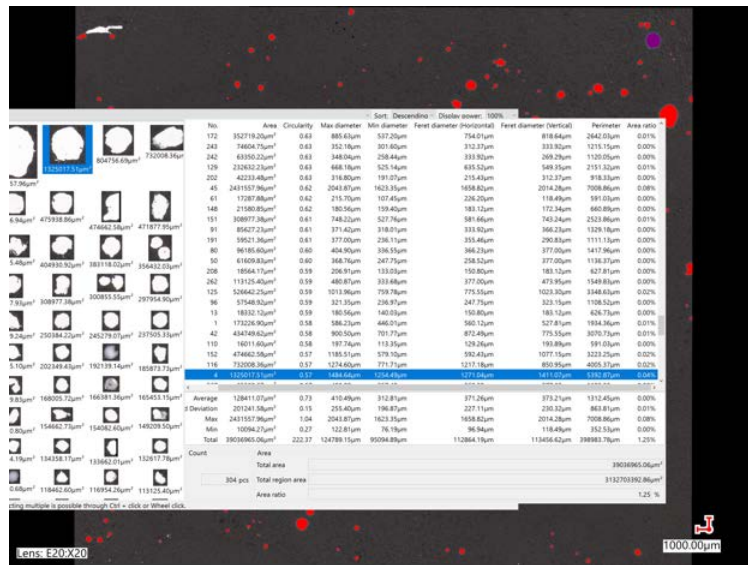

Figure 5. Object Measurement Information.

As seen in Figure 5, 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. A Feret diameter is defined as the maximum horizontal or vertical measurement. Therefore, each measurement is exactly, and only, across the horizontal or 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 5. 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 5, 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 6.

The split window view shown in Figure 6 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 6. 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. 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 who choose to use this method. 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.
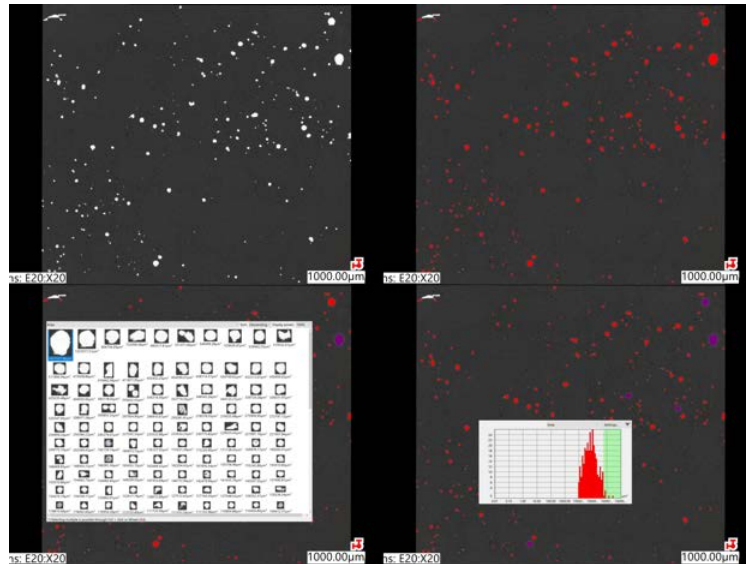

Figure 6. Split Window View on Keyence Microscope.

5. Calculations

As discussed previously, the standard counting and measuring assessment is able to 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 1, 2, 3, and 4 from ASTM C457-16 [11]. The first of the equations, Equation 1, can be seen below.

$$\alpha = \frac{4N}{T_a} \qquad (1)$$

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 2.

$$\frac{p}{A} = \frac{T_p}{T_a} \qquad (2)$$

Where p is the paste content in %, A is the Air Content in %, and T_p is the traverse length through the paste. As seen in Equation 2, 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 2 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 3 can be used to determine the Spacing Factor, if the Paste-Air Ratio is greater than 4.342 then Equation 4 should be used to calculate the Spacing Factor. Equation 3 and 4 can be seen below.

$$\bar{L} = \frac{T_p}{4N} \tag{3}$$

$$\bar{L} = \frac{3}{\alpha}\left[1.4\left(1 + \frac{p}{A}\right)^{1/3} - 1\right] \tag{4}$$

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 7.

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	in^2/in^3
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 7. Screencap of Excel Specific Surface and Spacing Factor Calculator.

As seen in Figure 7, 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 function 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.

6. Results

Following the aforementioned analysis and calculation procedure, multiple samples (6 samples) from each target air void range (Table 2) were polished and contrast enhanced in accordance to guidelines outlined in ASTM C457. These samples were then analyzed via a Keyence – VHX 7000 microscope. these six samples were cut from the same cylinder for sufficient surface area and consistent representation of the entire sample, and sample bias. Additionally, six different polish pad grit sizes were investigated to assess whether the degree of polish has any influence on the alternative technique. For this initial analysis, 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 of 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 or possibly an entrapped air void, which can be irregular in shape (i.e., not circular). 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 2.

As seen in Table 2, 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. Similar to 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 [3-6]. 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 2 are the first measurements produced from the Keyence microscope with no minimum or maximum object size constraints used. 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 judgment 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 1 and 3. In Equation 1, N (number of air voids or measured objects) is in the numerator and in Equation 3, N is in the denominator, therefore as this number gets larger, the values from Equation 1 (Specific Surface) will get larger and the values from Equation 3 (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 Tp increases proportionally to Ta, and Ta increases proportionally to the size of the object. Therefore, with a very small air void, N will increase by a value of 1, and Ta and Tp would only increase by the measured length of that air void, which would likely not be a significant increase to the total Ta and Tp 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 [1] and Taylor et al. [3] state that the size of entrained air voids is in the range of 10 – 1000μm (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 [3]. The size and function of pore types in concrete can be seen in Table 3.

Table 2. Initial Hardened Air Void Analysis Results Completed with the Keyence – VHX 7000.

Control - ASTM C231 Measured Air Content = 1.8%, SAM No. = 0.65							
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							
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							
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							
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**

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 1 and 3, and produce higher than expected values for each. According to Hover [1] and Taylor et al. [13] 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 – 1000μm (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 input into Excel calculator shown in Figure 7. 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. 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 (which includes capillary voids, entrained air voids, and entrapped air voids), based off the comparison between both ASTM C231 and both ASMT C457 procedures and to that of the literature [3-6]. 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 2, 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 – 1000μm (0.0004 – 0.039 in.) in diameter, the analysis is removing capillary voids and 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 and/or osmotic pressure during Freeze - Thaw cycles [1 -3, 5-6]. 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 4.

Table 3. Pore type and function related to freeze-thaw durability [13].

Pore Type	Diameter	Location	Effects on Freeze 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

As seen in Table 4 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 [1-4]. 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.

In order to fully demonstrate the alternative technique comparison, the results have been compared between the other ASTM C457 procedures as well as ASTM C231. The results can be seen in Figures 8 – 11.

As seen in Figures 8 – 11, 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 [1-4]. 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 of the air voids in the specimen. Additionally, ASTM C457 states that the analysis should be distributed evenly over the sample area [11]. 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 [11]. 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. Therefore, with the alternative analysis over 12 in^2 (77.4 cm^2) 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 [11]. 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.

Table 4. Secondary Hardened Air Void Analysis Results Completed with the Keyence – VHX 7000.

| Control - ASTM C231 Measured Air Content = 1.8%, SAM No. = 0.65							
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							
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							
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							
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**
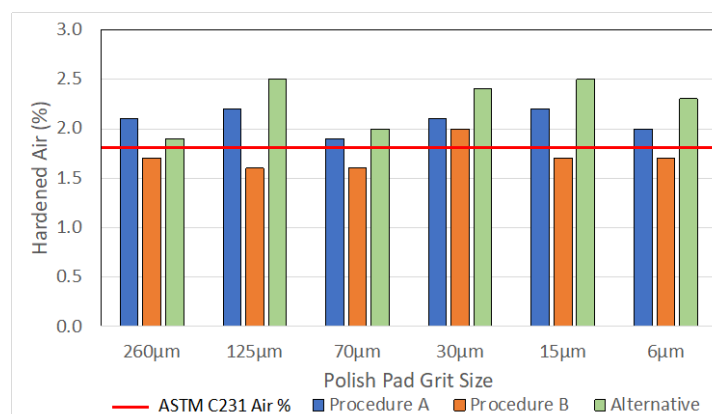
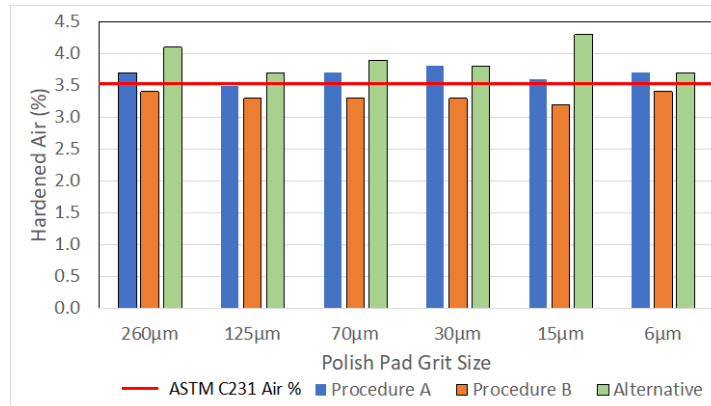**Figure 8. Control (2% Target) Hardened Air Void Comparison.**

Figure 9. Low Air (3-5% Target) Hardened Air Void Comparison.
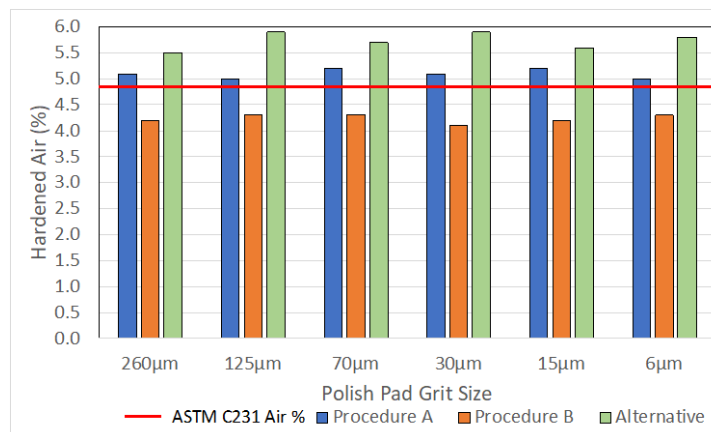

Figure 10. Medium Air (5-7% Target) Hardened Air Void Comparison.
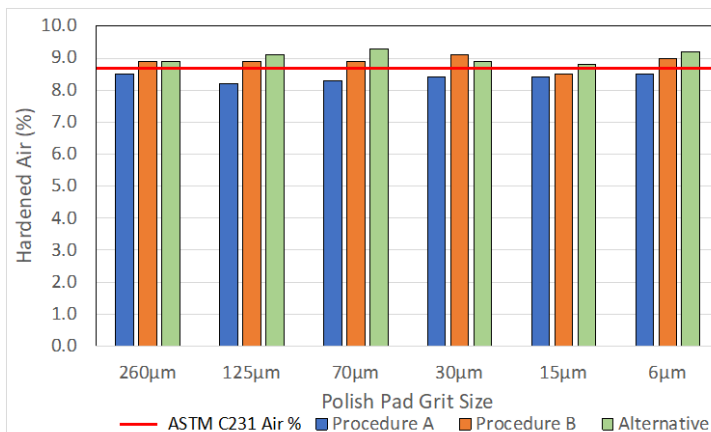

Figure 11. High Air (>8% Target) Hardened Air Void Comparison.

Additional comparison between the alternative analysis and each ASTM C457 procedure can be seen in Figures 12 and 13.

Figures 12 and 13 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 C547 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 [11], where the standard reports a range of air content percent of 0.82 for within lab precision and 1.16 for between lab precision. Therefore, the differences between the results are likely negligeable. 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.
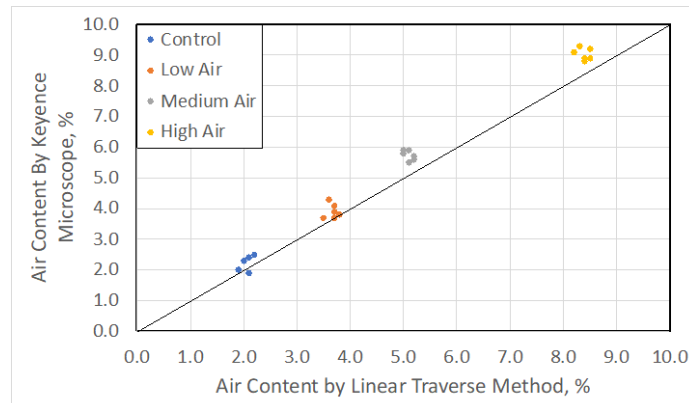

Figure 12. Comparison Between Alternative Method vs. Linear Traverse Method (ASTM C457 Procedure A).
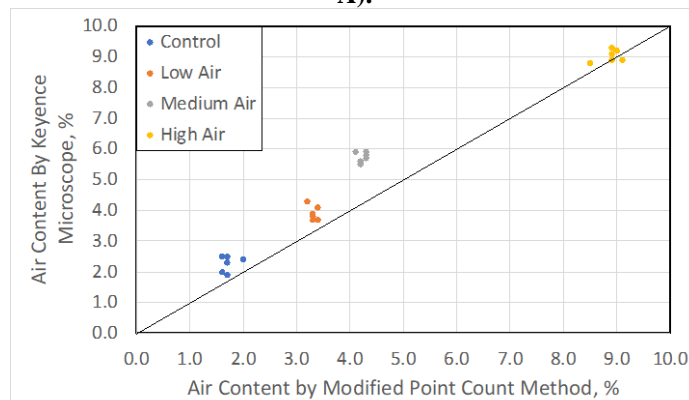

Figure 13. Comparison Between Alternative Method vs. Modified Point Count Method (ASTM C457 Procedure B)

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 14 and 15. The values in these figures represent the average across all of the polishing pad sizes. As discussed, these results were statistically similar, thus representing their average for comparison is acceptable.

As seen in Figures 14 and 15, the respective results are very comparable. Looking at the average spacing factor results (Figure 14), the results for each air percent are not only similar but they are as expected with reference to the literature [1-4]. 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 μm) 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 [1-4]. 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 negligeable. Using the same p-value, the statistical analysis revealed that the difference between each method is negligeable.
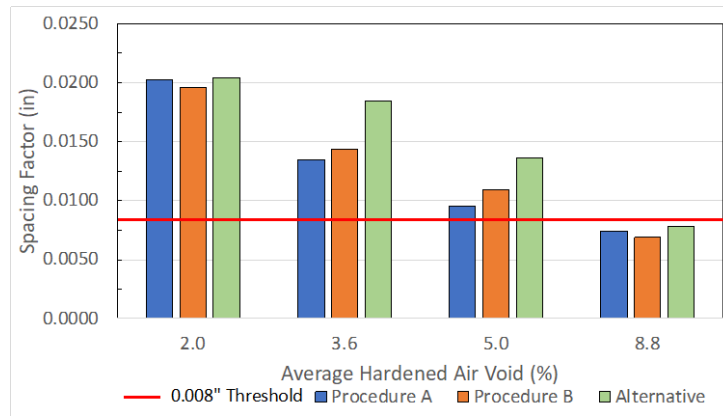
Figure 14. Average Spacing Factor Results for the Alternative Method and ASTM C457.
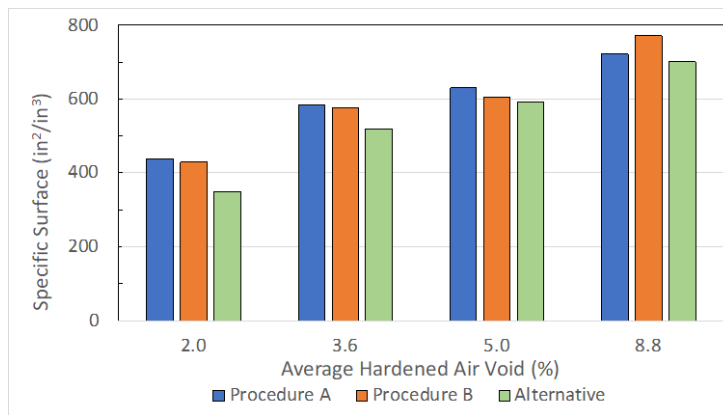

Figure 15. Average Specific Surface Results for the Alternative Method and ASTM C457.

7. Required equipment and recommended procedures

Table 5 lists the required equipment necessary to complete the alternative air void characterization method for hardened concrete samples.

Table 5. Required Equipment to Complete an Alternative Hardened Air Void Characterization.

Item
VHX 7000 Controller
Fully Integrated Camera
Console "Monitor/Computer"
Focus View Motorized XYZ Stage – (**100mm range of motion**)
20-100X FI Lens
100-500x FI Lens
VHX Support Package

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 16.

As seen in Figure 16, 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 16 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.

Figure 16. Screencap of VHX Software User Window with a Fully Stitched Concrete Sample.

7.1 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.

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.

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.

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.

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).

8). 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 17.
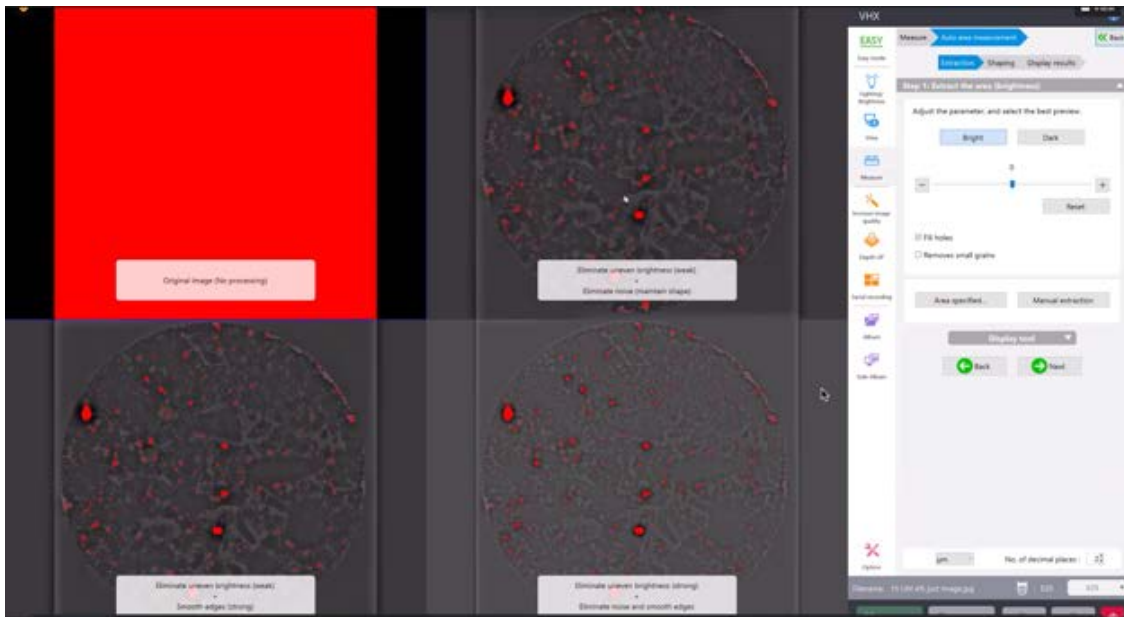
Figure 17. Example of the Measuring Preview Window.

As seen in Figure 17, 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. Also seen in Figure 8, 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 18.

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.
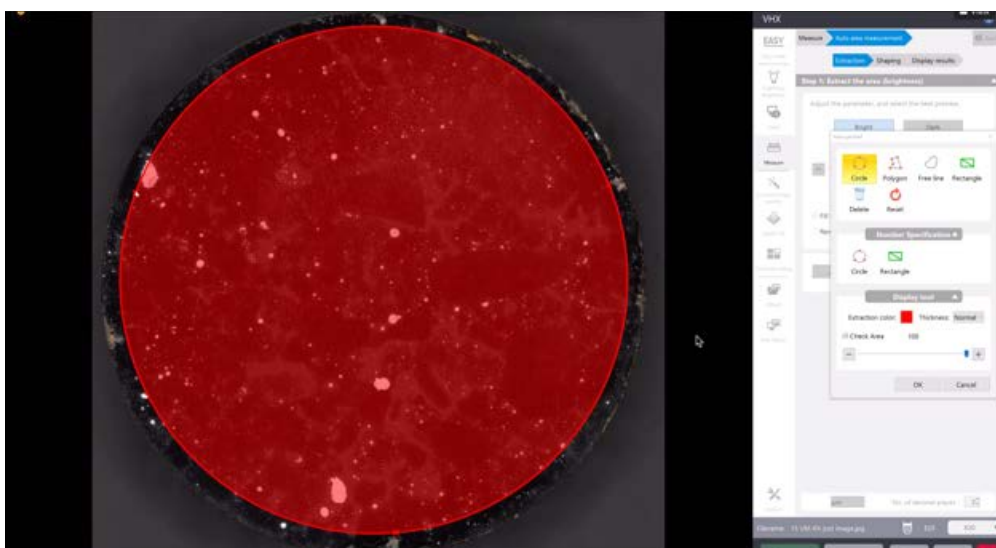

Figure 18. Example of the Area Specified Circle Procedure.

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

A new preview of the measuring analysis will appear like that seen in Figure 8, 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 19.
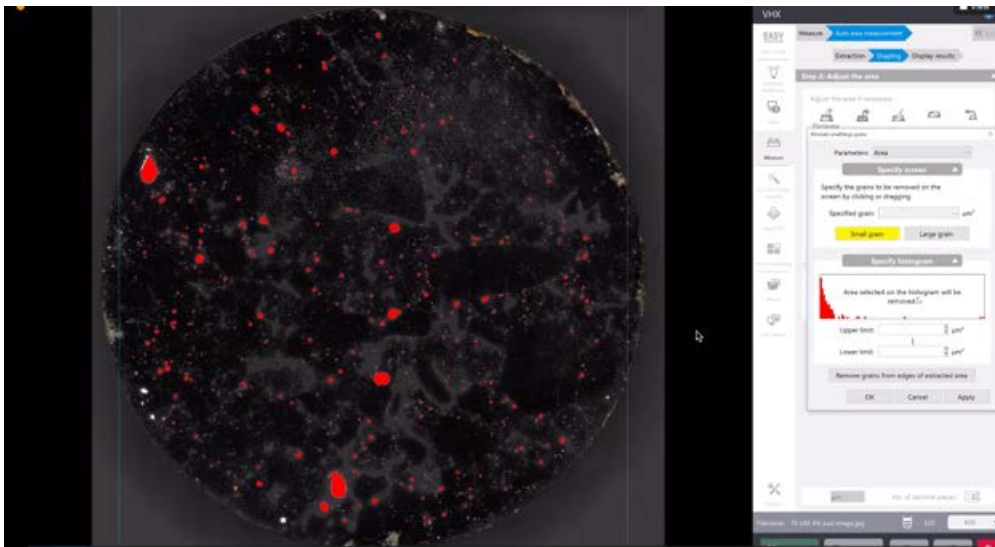

Figure 19. Example of Eliminate Grains Pop-up Window.

13). To apply a circularity parameter, click the parameters drop down menu (which defaults to Area, as seen in Figure 19), 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".

15). At this point the initial analysis is complete, and the auto area measurement results will automatically appear on the screen via a pop-up window and the user options section on the right-hand side will switch to provide result analysis options. An example of this can be seen in Figure 20.

As seen in Figure 20, the results of the initial analysis will be displayed on a new pop-up window. From here the user can see the Area Ratio result, which will correspond to the Hardened Air Void % of the sample analyzed.

16). To save the raw data, click Save CSV and a save the .csv in the user's desired file location on the computer.

17). 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.

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 4 – 6.

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.

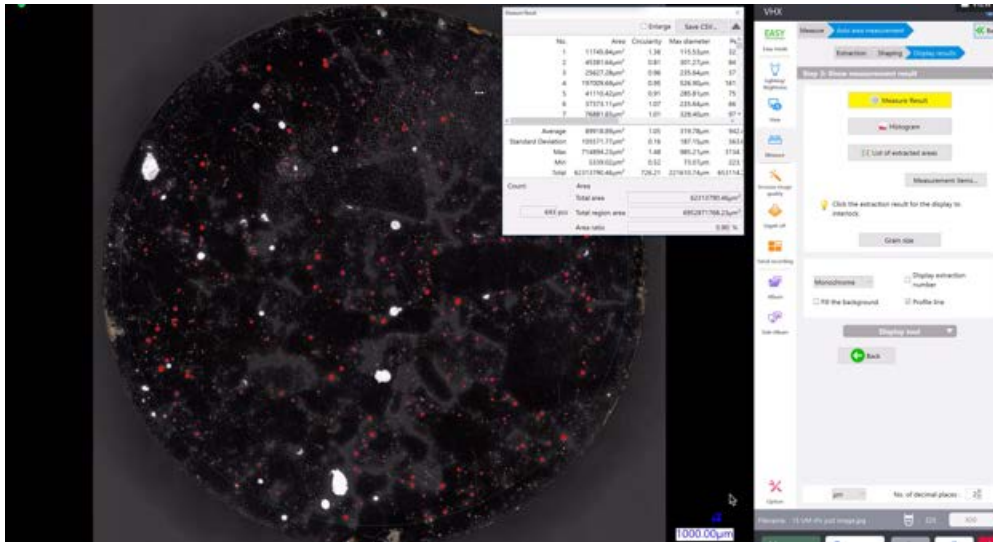20). This completes the initial analysis.

Figure 20. Example of Auto Area Measurement Results.

7.2 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 of 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 19.

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.

8. Conclusions

Based off the results presented and discussed, 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 reproducible from user-to-user and lab-to-lab, but is easier, quicker, more reliable, and less reliant on human decision making. Additionally, the following conclusions can be made that correspond with the study's objectives.

    1) This study has successfully developed an alternative hardened air void test method that captures all air voids in the sample and does not rely on human judgment calls.

    2) The alternative test method developed produced all hardened air void parameters required to analyze the concrete air void system.

    3) All hardened air void parameters were statistically insignificant with the conventional ASTM C457 method; however, the method was quicker, more robust, and reliable.

    4) Through the development of the alternative method additional issues with ASTM C457 were pointed out that may produce inaccurate hardened air void parameter results.

    5) This study has also produced a list of procedures required to produce the hardened air void parameters (for both an initial analysis and a secondary analysis)

    6) This study has also compared and discussed the results of the alternative analysis to the Procedure A and B of ASTM C457, which include all hardened air void parameters. The results also demonstrate a good correlation.

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