

Enhanced Entrained Air Void System Characterization for Durable Highway Concrete

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Abstract

The air void system in concrete provides a strong influence on the behavior of cementitious materials in both the fresh and hardened state, especially as it relates to freeze-thaw resistance and de-icer scaling. The most common test procedure to characterize the air void system is ASTM C457, which involves microscopic determination of the air content, paste content, air-void size distribution, and spatial dispersion of a sawn concrete sample. This procedure is reliant on the user to make hundreds of critical decisions per sample, which requires a lot of time, and is subject to human error. Due to this drawback, most state agencies do not perform this test and generally only rely on fresh air content values. The super air meter (SAM) and the Air Void Analyzer (AVA) are optional alternatives to ASTM C457, however, these devices only provide a “SAM number” or “AVA value”, which only represents a correlated spacing factor value, which are not as accurate as actual measured values [1]. These devices are also lacking in entrained air void system information, which results in a design and analysis deficiency. Therefore, there is a need to develop a method to fully characterize the air void system in concrete that is more accurate, comprehensive, less time consuming, cost effective, and easy to complete. The work outlined in this proposal demonstrates two alternative test procedures to characterize the entrained air void system for durable highway concrete. Proposed Procedure A utilizes a microscope with onboard counting and measuring software to make sophisticated measurements of a prepared sawn sample. Proposed Procedure B uses a flatbed scanner and free software to make the determinations of the same prepared sawn sample. Two different procedures are proposed to offer two options to best suit the current needs and budget of the NRRA. Both procedures will provide an enhanced understanding of the entrained air void system within concrete, specifically the amount of air voids, the air void size, and the spatial dispersion of the air voids.

Introduction

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 that are affected by the air void system include workability, cohesion, density (fresh and hardened), strength, finishability, and freeze-thaw resistance. The most important of these in terms of long-term performance of highway concretes, 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 empty space in the capillary pore system [1]. Additionally, important is that 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 [2-5]. The current state of

the art test method to study the entrained air void system in concrete is ASTM C457 – “Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete” [6] originally proposed by Brown and Pierson [7]. The test method consists of polishing a hardened concrete sawn sample and placing the sample under a microscope. The operator systematically makes measurements, by counting the air voids that come into view and statistical estimates are produced from the measurements that define the air content, paste content, air-void size distribution, and spatial dispersion. There are two alternative methods also 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. Although ASTM C457 is the standard test method for characterizing the entrained air void system in concrete, there still exists some major sources of variability and uncertainty of the test results. The major sources of variability include precision and bias, inherent statistical uncertainty, level of magnification of the microscope, and operator subjectivity. Additionally, the parameters associated with this characterization and the results of these laboratory tests do not always reflect the observed field performance nor do they consider the possible effects on other concrete properties. Therefore, there is a need to identify the characteristics of the air void system that relate to field performance of the concrete system. This information will help highway agencies prepare specifications for concrete procurement that will provide the air void characteristics for freeze-thaw resistance and de-icer scaling needed for enhanced durability of highway structures and pavements.

Objectives

This study will be implemented through the following objectives:

1. Develop two alternative entrained air void system characterization and measuring techniques.
2. Compare alternative techniques to that of the current practice (ASTM C457) and Super Air Meter.
3. Correlate entrained air void system data to the fresh and hardened properties of highway concrete.

Variables

The research team will consult with the NRRRA about testing four various concrete pavement mixtures commonly used by the NRRRA. Mixture details will be provided by the NRRRA personnel and they will be produced and tested in the Texas State University concrete and materials laboratory. The variables tested, with their corresponding test methods, can be seen in Table 1. All necessary variables will be obtained from each test method.

Table 1: List of Variables to be Tested.

Fresh Property Variables	Hardened Property Variables	Durability Variables
Air Content (ASTM C231)	Compressive Strength - 28 day (ASTM C39)	Freeze-Thaw Resistance (ASTM C666)
Air Content (Super Air Meter)	Air Void System (ASTM C457)	De-icing Salt Scaling (ASTM C672)
	Air Void System (Procedure A)	
	Air Void System (Procedure B)	

Questions

The following questions will be answered in the proposed study:

1. Can a superior air void system characterizing procedure be developed to that of ASTM C457?
 - a. How much more accurate is the proposed procedure(s) to that of ASTM C457?
 - b. What is the total time required to complete the proposed procedure(s) compared to ASTM C457?
 - c. How are the proposed procedure(s) more cost effective to that of ASTM C457?
 - d. How easy or difficult is the proposed procedure(s) in comparison to that of ASTM C457?
 - e. What are the specific steps required to complete the proposed procedure(s)?
 - f. How would the proposed procedure(s) be easily implemented across the NRRA?
2. How do the proposed procedure(s) correlate to fresh, hardened, and durability (freeze-thaw resistance and de-icing salt scaling resistance) properties of the NRRA concrete pavement mixtures?

Methodology

The experimental methodology of this proposal focuses on developing two alternative air-void system characterization procedures and measuring techniques to that of ASTM C457. Currently ASTM C457 has many drawbacks, which one of the major issues originates from human error. As described by Hover (2006), “The current test (ASTM C457) requires that the operator make a large number of subtle distinctions and judgment calls based on color, texture, shadow, and the appearance of the feature in question compared to its background (about 1000 such judgments per analysis).” Therefore, an automated, computer-based analysis could eliminate such errors and provide a more reliable air-void system result. Two procedures will be developed, Procedure A, which consists of a sophisticated automated microscope counting software and, Procedure B, which consists of a high-resolution flatbed scanner and imaging analysis software. Both procedures will diminish the errors developed in the more traditional method and significantly reduce the operation time by approximately two hours. This increase in accuracy and decrease in duration will potentially allow the NRRA higher quality assurance over their pavements. The two procedures are outlined below.

Preparation of Sections

In the proposed alternative test method, sampling will remain consistent with ASTM C457 [6], and ASTM C823 [7] for laboratory prepared samples, with minor alterations. Samples will be sawn into sections that are sawn perpendicular to the layers in which the concrete was placed or perpendicular to the finished surface. The individual sections will be sawn so that they are large enough to accommodate the available lapping/polishing equipment available on Texas State University campus. Currently there are two lapping machines by LabX available in the concrete and materials testing laboratory at Texas State University. The sections will first be ground with a nominal 150 μ m (No. 100) silicon carbide abrasive to even the sawn surface and remove any impurities. The surface will be lapped with successively finer abrasives until it is suitable for microscopical observation, as

determined by the operator. The standard recommends an appropriate series of abrasives that would include nominal 75, 35, 17.5 and 12.5 μm grit sizes (No. 220, 320, 600, and 800, respectively), and 5- μm (No. 2500 grit) aluminum oxide. The surface of the sample will be thoroughly cleaned in between sequential grinding periods to remove any grinding compound. This will be done with a soft cosmetic brush under running water, as ultrasonic cleaners may be harmful to the sample [8]. The next step involves coloring the section surface with a colored water-resistant marker, which is followed by filling the air voids with zinc paste. Any excess paste is removed with a sharp blade and the surface will then be sealed and protected using paraffin oil. This entire process is controlled via a microscope to ensure that all air voids have been filled in. If there are any deficiencies in this process, these steps must be repeated. The effect of this process is to remove all possibilities of statistical variance by highlighting only the air voids. Following the preparations of the sections either proposed Procedure A or Procedure B can be used.

Procedure A: Microscopical Measurements

Procedure A for air-entrained system characterization consists of microscopical measurements using a Keyence (VHX 2000) digital microscope, which is currently available in the concrete and materials laboratory at Texas State University. The Keyence VHX 2000 digital microscope is a unique piece of equipment and provides many unique capabilities over a traditional stereo optical microscope. More accurate measurements can be made due to the microscope's high-resolution measurements capability of producing a micrograph with 4800 x 3600 pixels resolution. The onboard software uses a multi-scan method, making it possible to specify a measurement area on a captured image that is nine times larger than a standard image, thus making it possible to perform measurements with greater accuracy. The microscope lens has a magnification capability of 100X – 1000X (transmitted and vertical/lens lighting) and a high range depth of field. A high depth of field allows objects with large variations in surface topography to be focused and accurately observed in a single image, which is impossible with conventional microscope optics. A superior depth of field is essential in preparation of the sawn surface samples. Additionally, significant for accurate air void system characterization is the VHX microscope's function for ultra high-speed image stitching. The automated XY stage moves in a clockwise pattern and captures images at each location. After an image is captured, it will be stitched together with the previous image, in real-time, before the stage moves to the next location. This ultimately provides the operator with a large (20,000 x 20,000 pixel), overall view of the test specimen, while preventing any misalignment typically associated with ASTM C457 standard procedure methods. A schematic of this process is demonstrated in Figure 1.

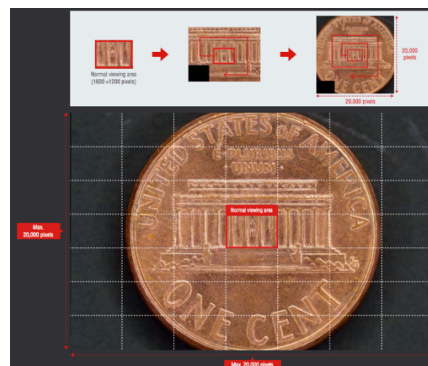


Figure 1: Demonstration of image stitching ability of Keyence microscope.

This unique ability will drastically reduce the processing time typically involved with the traditional methods. Lastly and most importantly is the microscopes ability for real-time accurate measurement and quantification. The system allows for measurements and quantification to be made directly on board the microscope's digital display, instead of having to save the image and use external software to complete any measurements. The primary measuring and quantification function tool that will be used is the automatic area measurement feature. The automatic area measurement feature will automatically select and count or extract key features designated by brightness, color, or by operator selection. A representation of this feature is shown in Figure 2.



Figure 2: Automatic area measurement feature of Keyence microscope.

The major benefit of using this feature is that it removes the human error and operator time involved with both procedures in ASTM C457. In addition to providing the amount and distribution (area ratio) of the air-void system, the automatic area measurement feature can provide information that ASTM C457 cannot provide such as size, shape, specific amount of each shape (histogram), and degree of circularity. Once these parameters have been set and saved, it is possible to recreate the same settings and perform automatic area measurements on as many new samples as necessary. The entire process will take approximately 10-15 minutes per sample, which is a 184% reduction in sample processing time.

Procedure B: Scanner and Image Analysis

In the instance that obtaining a Keyence VHX-2000 digital microscope is not an option, an additional procedure is also proposed. Procedure B consists of the same sample surface processing as described in the sampling and preparation of surface section. After the surface finishing is completed the sample is scanned using a high-resolution flatbed scanner capable of a resolution of 6400 x 9600 dots per inch (dpi). A high-resolutions flatbed scanner can be purchased off the shelf for under \$200. A high-resolution scanner is necessary in order to capture the very fine air voids (~10 μ m or less). An Epson Perfection V600 with a resolution of 6400 x 9600 is available at the Texas State University concrete and materials testing laboratory. Once an image is produced it can then be imported into an image analysis software application. The chosen and recommended software to be used for processing and analysis is ImageJ. ImageJ is a free software application available at <http://imagej.nih.gov/ij/> and is recommended for its capability and accessibility (PC/Mac/Linux). Once the image is opened in ImageJ, the image needs to be converted to gray scale and converted into a binary image by a threshold operation to separate the air voids and solid phases and eliminate any noise. This step is necessary in order for the following step to distinguish between air voids and the solid material. The final step in the proposed Procedure B is to run a histogram analysis across the sample in sequential linear target areas (similar to ASTM C457, but digitally).

What this means is that a ‘target’ area will be highlighted on the sample and a histogram will be produced that provides information regarding the target area. The target area will be a horizontal line across the sample that is approximately 10 pixels wide. The histogram will produce data points on the pixel color in the image, termed ‘gray value’ plot in ImageJ. Due to the previous gray scale conversion of the image, the solid phases in the material will be black and the air voids will be white. Therefore, the histogram will produce data corresponding to the quantity of black pixels versus white pixels, which ultimately relates back to the amount of air voids in the system. Further analysis of this data could yield such information as the distribution, size and location of the air voids. The target area would have to be adjusted and an individual histogram be produced for each transition of the linear target area, which is similar to the linear traverse method of ASTM C457, however the user error and processing times are reduced.

Resistance to Rapid Freezing and Thawing and De-icing Salt Scaling Resistance

Also outlined in this proposal is that of correlating the air-entrained system of hardened concrete to that of its ability to withstand rapid freezing and thawing and de-icing salt scaling. As previously described, the ability of a concrete system to withstand rapid environmental changes in temperature is due to the ability of the freezing water to find space to expand. The available space for expanding ice greatly depends on the distribution and size of the air void system of the concrete. The testing procedure that will be used is ASTM C666 – Method B, “Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing” and the dynamic testing apparatus that will be used will be in accordance to ASTM C215 “Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens”. Additionally, the effect of the entrained air void system on de-icing salt scaling resistance will also be investigated. Salt scaling is a major damage problem for concrete pavements in cold environments. Salt scaling is a type of superficial damage caused by freezing and thawing a saline solution on the surface of concrete, in which the air void system can potentially help resist. The testing procedure that will be used for assessing the resistance to de-icing salts is ASTM C672 “Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals”

The specific testing equipment that is available at Texas State University’s concrete and materials testing laboratory is Humboldt’s H-3185B freeze-thaw chamber that supports the requirements of ASTM C666 and has the ability to cycle continuously between two temperatures for finite number of cycles. The operator can define the ramp rate, the number of cycles, the ramp rate between temperature extremes, and the desired soak duration. Additionally, a Humboldt H-3175 sonometer is available to determine the changes in resonant frequency of the concrete specimens that have been subjected to rapid freezing-thawing cycles. The sonometer will determine the fundamental transverse, longitudinal, and torsional resonant frequencies of the concrete sample (prism and cylinder), which is used to calculate the dynamic Young’s modulus of elasticity, the dynamic modulus of rigidity, dynamic Poisson’s ratio, and the freeze-thaw durability factor. All additional equipment necessary to complete the above tests are available in the Texas State University concrete and materials laboratory.

Documentation of Findings

Key findings that lead to enhanced entrained air void system characterization for durable highway concrete will be communicated to the NRRA for implementation consideration in the NRRA Standard Specifications. The research team shall facilitate implementation of the project findings by documenting the results of the enhanced characterization techniques in the research report and project summary report at the conclusion of the project. The research report will include recommendations and guidelines to the NRRA on the following:

- Sample preparation techniques for enhanced air void system characterization.
- Recommendations and guidelines to perform Procedure A for enhanced air void characterization.
- Recommendations and guidelines to perform Procedure B for enhanced air void characterization.
- Correlation of standard air void system characterization (ASTM C457), Procedure A, and Procedure B characterization to that of fresh and hardened properties of concrete mixtures, including fresh air content (air meter and super air meter [SAM]) and the resistance to rapid freezing and thawing.

Proof of Concept

A similar process as the proposed method (Procedure A) has been previously completed by the author (Torres *et al.*), which demonstrates the capability of this process [8-10]. In the similar study, the crystallization of a small piece of glass was investigated. The Keyence digital microscope was used as the primary investigation technique that first imaged the sample, and then the crystallization quantification was completed using the on-board automatic area measurement feature. Figure 3a shows the imaged glass sample and the corresponding crystallites that were automatically measured using the image processing software. Figure 3b shows the histogram output of the collected data in maximum diameter (μm).

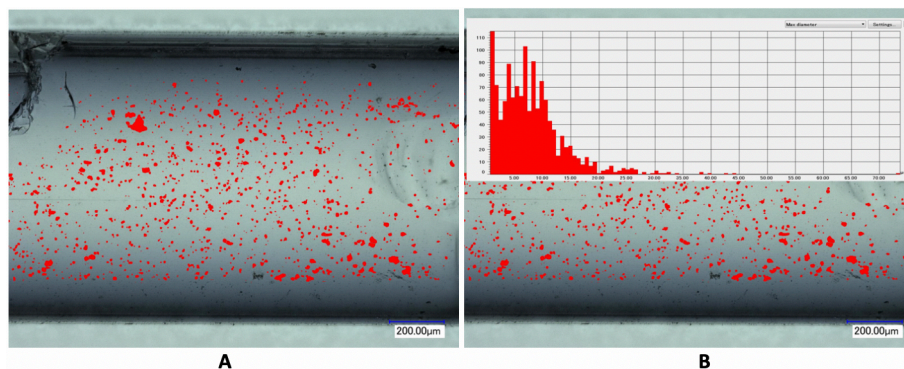


Figure 3a-b: a) Automatic area measurement of glass sample completed by Torres et al. b) histogram of measured areas in max diameter (μm).

Figure 3a-b demonstrates the automatic area measurement capability of the Keyence digital microscope available at Texas State University's concrete and materials laboratory. In addition to the histogram data the software produces a .csv (comma-separated value), which is a tabular data

plain-text file that can be opened and manipulated in Microsoft Excel. Overall, this method can easily be translated into measuring and analyzing the entrained air void system for durable highway concrete. The authors also have experience implementing a similar process to Procedure B [9-11], which provides a proof of concept for use in assessing durable highway concrete. In previous studies, it was required to determine the degree of crystallinity in small glass samples, which is similar to dispersed air-voids in a concrete matrix. The main analysis technique used was ImageJ and its target area histogram technique. Figure 4a-b demonstrates the use of ImageJ on these samples to determine the degree of crystallinity.

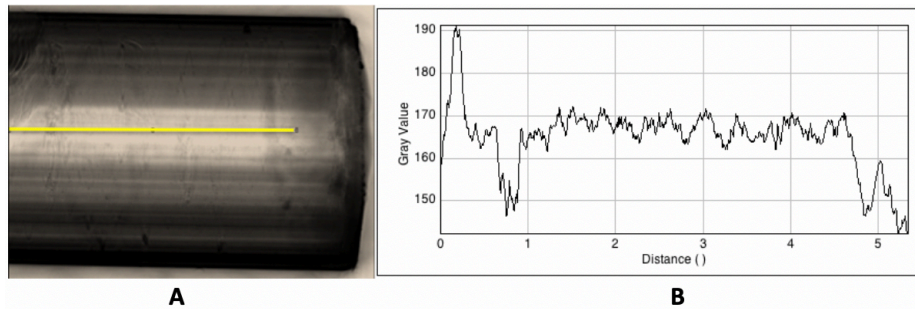


Figure 4a-b: a) ImageJ processing on previous material by Torres et al. Thin yellow line represents the target area b) The histogram output across the length of the target area.

Figure 4a-b demonstrates the research capability and author experience with this type of characterization method. Figure 4b demonstrates the possible histogram output from the target area of the analyzed image. The target area a thin line, which is consistent with the ASTM C457’s linear traverse method. As previously described the histogram produces a ‘gray value’ (200 = black and 0 = white) therefore the corresponding gray value output for a hardened concrete sample in black and white will correspond to an air void or hardened material. The data can then be further analyzed to represent the size, shape and distribution of the sample as the target area is moved. Overall, this procedure (Procedure B) can also be used to analyze the air void system for durable highway concrete and it also has the potential to replace or alter the procedures outlined in ASTM C457.

Schedule

The overall schedule is from June 15, 2020 to December 31, 2021 and a full breakdown is given in Table 2.

Table 2: Proposed Timeline.

Task	2020				2021			
	Summ 2020	Fall 2020	Spring 2021	Summ 2021	Fall 2021	Spring 2022	Summ 2022	Fall 2022
Hire Student(s)	█							
Prepare Materials	█	█						
Concrete Mixing and Casting	█	█	█	█				
Fresh Property Testing (air and super air meter)	█	█	█	█				
Compressive Strength Testing (ASTM C39)		█	█	█	█	█		
Freeze Thaw Testing (ASTM C666)			█	█	█	█		
Salt Scaling Testing (ASTM C672)			█	█	█	█		
Prepare Sample Surfaces				█	█	█		
ASTM C457 Air Voids Characterization					█	█	█	█
Proposed Air Void Characterization Procedures						█	█	█
Analysis							█	█
Write Report to be submitted to the NRRRA								█

Budget

The overall budget of this project is \$120,000, of which \$100,000 is requested from the NRRA and \$20,000 cost share is supported from Texas State University. The budget includes cost for senior personnel (PI and Co-PI) who will dedicate time during the summer months working on this project. Also included is cost for graduate student support who will help complete laboratory mixing and testing. The budget also includes cost to purchase a linear traverse device, which is needed for comparison. The cost for this device is supported by the Texas State University cost share contribution. A breakdown of the budget is proved in Table 3.

Table 3: Proposed Budget.

ITEM	COST
Senior Personnel	\$6,700
Graduate Student Support	\$49,840
Fringe Benefits	\$10,349
Indirect Cost (49.5%)	\$33,111
Total Cost to the NRRA	\$100,000
Cost Share from TxSTATE (20%)	
Humboldt Linear Traverse Method Device (ASTM C457)	\$20,000
Total Cost Share from TxSTATE	\$20,000

Funding this proposal benefits the NRRA by gaining a new entrained air void system characterization procedure that is more accurate, quicker, comprehensive, and more reliable than other methods.

Partnerships

Dr. Anthony Torres (PI)

Dr. Torres received his PhD in Civil Engineering from the University of New Mexico. Dr. Torres's primary research has been in the area of testing and modeling of materials, and the implementation of novel characterization techniques. His technical expertise is based on mechanistic characterization of materials and microstructural characterization of materials through microscopic, SEM, computational analysis and alternative test methods. He has conducted various research projects on evaluation of computational models based on various mechanical theories such as elasticity, micromechanics, and mass transport analysis. Dr. Torres has published numerous research papers on mechanistic material characterization and analytical and/or computational constitutive modeling of materials. He is an active member of the American Concrete Institute (ACI) and participates on multiple committees, such as concrete durability (ACI 201), proportioning concrete mixtures (ACI 211), and concrete with recycled materials (ACI 555).

Dr. Federico Aguayo (Co-PI)

Dr. Aguayo received his Ph.D. from the University of Texas at Austin in Infrastructure Materials Engineering, where he worked under the supervision of Dr. Kevin J. Folliard. Dr. Aguayo's primary research interest have been in enhancing concrete durability through the use of emerging

technologies and alternative cementitious materials. His research group has been active in evaluating the mechanical performance and chemical deterioration processes of various advanced concrete systems, while also working on developing and improving accelerated laboratory methods for predicting their long-term performance in the field. He is a well-established researcher on concrete durability with over 10 years of experience working with private industry and public agencies, especially with the Texas Department of Transportation (TxDOT). He is an active member of the ACI, and participates in several committees related to concrete durability (ACI 201) and material science of cementitious systems (ACI 236).

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