IMPLEMENTATION OF THE SEQUENTIAL AIR METHOD IN LABORATORY AND FIELD STUDIES

By

HOPE HALL

Bachelor of Science in Architectural Engineering

Oklahoma State University

Stillwater, OK

2015

Submitted to the Faculty of the Graduate College of the Oklahoma State University in partial fulfillment of the requirements for the Degree of MASTER OF SCIENCE December, 2017

IMPLEMENTATION OF THE SEQUENTIAL AIR METHOD IN LABORATORY AND FIELD STUDIES

Thesis Approved:

Dr. M. Tyler Ley

Thesis Adviser

Dr. Bruce Russell

Dr. Julie Hartell

ACKNOWLEDGEMENTS

I would like to acknowledge funding from the Oklahoma Transportation Center and Pooled Fund TPF-5(297) and the supporting states. A special thanks to Jason Weiss, Larry Sutter, and Peter Taylor for the discussion over previous research leading into this study. I would also like to thank Dr. Tyler Ley for giving me a chance to work with him during my Masters. Thank you to Dr. Bruce Russell and Dr. Julie Hartell for agreeing to serve on my committee. Justin Becker, Brad Woodard, Zane Lloyd, and Muwanika Jdiobe: it was a pleasure to work with all of you on my research. Thank you to all of the undergraduate students at Bert Cooper Engineering Laboratory for all of your hard work and dedication in providing high quality research.

Acknowledgements reflect the views of the author and are not endorsed by committee members or Oklahoma State University.

Name: HOPE HALL

Date of Degree: DECEMBER, 2017

Title of Study: IMPLEMENTATION OF THE SEQUENTIAL AIR METHOD IN

LABORATORY AND FIELD STUDIES

Major Field: CIVIL ENGINEERING

Abstract: This work validates the Sequential Air Method (SAM) Number by analyzing data from laboratory, industry fieldwork, and a series of concrete mixtures with different admixture combinations. Comparisons are made to the total air content and hardened air-void analysis (ASTM C457) for the 458 concrete mixtures studied. Comparisons are also made to the freeze thaw durability testing (ASTM C666) for the two individual lab data sets with admixtures. Guidelines are established in this work to help users understand the reliability of the air content and SAM Number relationship within a fresh concrete mixture.

TABLE OF CONTENTS

Ch	Page Page
TA	ABLE OF CONTENTS v
LI	ST OF TABLES vii
LI	ST OF FIGURES
I.	INTRODUCTION TO THE QUALITY OF AIR-VOID SYSTEM IN FRESH
	CONCRETE
	1.0 INTRODUCTION
	1.1 AIR-VOID DISTRIBUTION IN FRESH CONCRETE
II.	LABORATORY AND FIELD VALIDATION OF SEQUENTIAL AIR METHOD . 4
	2.0 INTRODUCTION
	2.1 EXPERIMENTAL METHODS
	2.1.1 Laboratory Materials
	2.1.2 Field Materials
	2.1.3 Laboratory Concrete Mixture Procedure and Testing
	2.1.4 Sequential Air Method9
	2.1.4.1 SAM Number calculations 11
	2.1.4.2 Air Content and Aggregate Correction
	2.1.4.3 Estimating Air-void Size by Comparing the Air Volume and SAM
	Number
	2.1.4.4 Variability in Measurement
	2.1.5 Hardened Air Sample Preparation14
	2.2 RESULTS
	2.2.1 Evaluation of the SAM Number compared to the Spacing Factor
	2.2.2 Evaluation of the SAM Number Compared to the Air Content
	2.2.3 Variability in SAM Measurements
	2.3 DISCUSSION

Chapter	Page
2.4 SUMMARY	
III. SEQUENTIAL AIR METHOD TESTING WITH ADMIXTURES	
3.0 INTRODUCTION	
3.1 EXPERIMENTAL METHODS	
3.1.1 Laboratory Materials	
3.1.2 Concrete Mixture Procedure and Testing	
3.1.2.1 Mixtures with Superplasticizer	
3.1.2.2 Shrinkage Reducing Admixture	
3.1.3 Sequential Air Method	
3.1.3.1 SAM Number Calculations	
3.1.3.2 Air Content and Aggregate Correction	
3.1.3.3 Void Size Estimation	
3.1.4 Hardened Air Sample Preparation	
3.2 RESULTS	
3.2.1 Mixtures With and Without a Superplasticizer	
3.2.2 Mixture with Shrinkage Reducing Admixture	
3.3 DISCUSSION	
3.4 SUMMARY	
IV. CONCLUSION	
4.0 SUMMARY	
REFERENCES	
APPENDICES	59
VITA	69

LIST OF TABLES

Table	Page
Table 2-1 – Type I cement oxide analysis	5
Table 2-2 – Admixture references	6
Table 2-3 – SSD Mixture proportions	7
Table 2-4 – A comparison of the coefficient of variation, agreement with Durability	
Factor, and the time required to complete the test	26
Table 3-1 – Type I cement oxide analysis	30
Table 3-2 – Admixture references	31
Table 3-3 – SSD Mixture proportions	31
Table 3-4 – SRA Air Loss	48
Table A-1 – SAM Quantile Curve Values	59
Table A-2 – Oklahoma State University Concrete Mixture Design and Testing Data	60

LIST OF FIGURES

Figure Page
Figure 1-1 – Similar air volumes within two concrete samples
Figure 2-1 – The device used to complete the SAM
Figure $2-2 - A$ graphical representation of the pressures in the top and bottom chamber in
the SAM
Figure 2-3 – Air Content versus Spacing Factor for two laboratory mixtures with similar
air volume and different air-void qualities16
Figure 2-4 – SAM Number versus Spacing Factor for the two laboratory mixtures
previously shown in Figure 2-3 17
Figure 2-5 – SAM Number versus Spacing Factor for 227 laboratory concrete mixtures
completed by two different research groups
Figure 2-6 – SAM Number versus Spacing Factor for 231 field concrete mixtures
completed by 21 different state DOTs with various aggregates and admixtures
Figure 2-7 – Percent agreement between SAM Number and different Spacing Factors for
laboratory concrete mixtures
Figure 2-8 – Percent agreement between SAM Number and different Spacing Factors for
field concrete mixtures
Figure 2-9 – Air Content versus SAM Number for 227 laboratory concrete mixtures
completed by two different research groups
Figure 2-10 – Air Content versus SAM Number for 231 field concrete mixtures
completed by 21 different state DOTs with various aggregates and admixtures
Figure 3-1 – The device used to complete the SAM
Figure $3-2 - A$ graphical representation of the pressures in the top and bottom chamber in
the SAM

Figure

Figure 3-3 – Air Content versus Spacing Factor for 0.40, 0.45, and 0.50 w/cm concrete
mixtures with and without PC1
Figure 3-4 – SAM Number versus Spacing Factor for 0.40, 0.45, and 0.50 w/cm concrete
mixtures with and without PC1
Figure 3-5 – Air content versus SAM Number for 0.40, 0.45, and 0.50 w/cm concrete
mixtures with and without PC1
Figure 3-6 – Air Content versus Durability Factor for 0.40, 0.45, and 0.50 w/cm concrete
mixtures with and without PC1
Figure $3-7 - SAM$ Number versus Durability Factor for 0.40, 0.45, and 0.50 w/cm
concrete mixtures with and without PC1
Figure 3-8 – Air content versus Spacing Factor for six laboratory mixtures with SRA and
one control mixture without SRA
Figure 3-9 – SAM Number versus Spacing Factor for six laboratory mixtures with SRA
and one control mixture without SRA 47
Figure 3-10 – Air content versus SAM Number for seven laboratory mixtures with SRA
and one control mixture without SRA
Figure 3-11 – Air content versus Durability Factor for seven laboratory mixtures with
SRA and one control mixture without SRA
Figure 3-12 – Spacing Factor versus Durability Factor for six laboratory mixtures with
SRA and one control mixture without SRA 50
Figure 3-13 – SAM Number versus Durability Factor for seven laboratory mixtures with
SRA and one control mixture without SRA

CHAPTER I

INTRODUCTION TO THE QUALITY OF AIR-VOID SYSTEM IN FRESH CONCRETE

1.0 INTRODUCTION

Throughout the world, concrete is a widely used material from large structural elements and bridge decks to driveways and sidewalks. This composite material is easy to make and has the ability to be molded into any shape desired. Concrete is made by mixing rock, sand, cement, and water. Today, the science behind concrete mixtures is more complex due to increased emphasis on long-term durability and performance, which has led to the widespread use of various admixtures. However, the overall concept of designing, producing, and constructing long-lasting concrete infrastructure remains.

Understanding the material science behind freeze thaw durability while maintaining other concrete properties has been a research topic for years. Research at Oklahoma State University has shown that the quality of the air-void distribution within a concrete mixture affects the freeze thaw durability of the hardened concrete.

The Sequential Air Meter (SAM) is an AASHTO TP 118 test method that measures air volume and the SAM Number. The SAM is similar to the ASTM C231 Type B air meter in looks; however, the SAM has six clamps instead of four to withstand higher pressures. It also has a digital gauge to allow users to follow systematic instructions, display errors, and calculate the air volume and SAM Number. The SAM consists of six pressure steps. The first step gives air volume and the SAM Number is the difference between the last step and the third step. This will be further explained in the following chapters.

1.1 AIR-VOID DISTRIBUTION IN FRESH CONCRETE

The quality of the air-void system depends on the size and spacing of air bubbles within a concrete mixture [1]. The size and spacing of the bubbles is known as the Spacing Factor specified in ASTM C457. Smaller, well-dispersed bubbles provide finer air-void systems that perform better in freezing and thawing environments than larger bubbles [2-4]. In previous work, the air-void size and spacing within fresh concrete has been studied by measuring the change in response to a series of sequential pressures. The SAM Number was used to measure this air-void distribution. The quality of the air-void system or the Spacing Factor has shown to affect the freeze thaw durability of the concrete. Through previous studies, the SAM Number has shown a greater correlation to freeze thaw testing compared to the Spacing Factor [4].

Figure 1-1 shows two concrete samples containing the same air volume. The left image shows one large bubble and the right image shows small, well-dispersed bubbles. In this study, the left image is referred as a coarse air-void distribution and the right image is referred as a fine air-void distribution. These bubbles act as areas of pressure relief voids for water to move to during freezing and thawing cycles. As water starts to freeze inside

paste, the distance for water to move into the air voids is much shorter in the right sample than the left sample. This smaller, well-distributed bubble distribution provides a more effective air-void system for freeze thaw durability [2, 4-6].



Figure 1-1 – Similar air volumes within two concrete samples.

This work focuses on providing insight into the quality of the air-void distribution within fresh concrete mixtures. Research and test results from laboratories, industry fieldwork, and two individual mixtures with different admixtures will provide information to validate the ability of the SAM to provide immediate insight into the air-void distribution of fresh concrete.

CHAPTER II

LABORATORY AND FIELD VALIDATION OF SEQUENTIAL AIR METHOD

2.0 INTRODUCTION

When concrete experiences a series of freezing and thawing cycles, damage can occur earlier than expected. However, if the concrete mixture contains an air-entrained admixture (AEA) that creates tiny bubbles, then the freeze thaw durability can be improved. Research has shown that small, well-spaced air bubbles within concrete creates pressure-relief regions for water to move to during freezing [2, 4-6]. The quality of the air-void system, the size and spacing of the bubbles, is a mechanism to improve the freeze thaw durability and helps prolong the lifespan of concrete structures.

The current established tests for freeze thaw durability are not able, within fresh concrete, to measure the air-void distribution. While other methods can measure the volume of air in fresh concrete, studies have shown that the air volume is not the only indicator of freeze thaw durability. The small, well-dispersed bubbles improves the quality of the air-void system. The Spacing Factor has represented the quality of the air-void distribution for a concrete mixture; however, measurement of the Spacing Factor requires hardened air-void analysis, which is time consuming and can only be conducted on hardened concrete [2, 3].

In order to measure the volume and spacing of air-voids in fresh concrete, it is most common to use the ASTM C457 method. This measures the Spacing Factor of hardened concrete and takes between 7 and 14 days to complete [2, 3]. The Spacing Factor gives a good understanding of the freeze thaw durability, but does not allow for adjustments to be made to the concrete mixture before placement [3, 4]. The concrete industry needs a test method that provide rapid results for immediate adjustments to ensure that the concrete mixtures placed are durable. This work shows laboratory test results and field test results to support the usefulness of the Sequential Air Meter or SAM test method.

2.1 EXPERIMENTAL METHODS

2.1.1 Laboratory Materials

All of the laboratory concrete mixtures in this research used a Type I cement that met the requirements of ASTM C150. Both the oxide analysis and Bogue calculations for this cement used is shown in Table 2-1. The aggregates used were locally available crushed limestone and natural sand used in commercial concrete. The crushed limestone had a maximum nominal aggregate size of 19 mm (3/4"). One mixture contained a blend of the coarse and intermediate aggregate as well. Both the crushed limestone and the sand met ASTM C33 specifications. All the admixtures used are described in Table 2-2, which met the requirements of ASTM C260 and ASTM C494.

Table 2-1 – Type I cement oxide analysis

Oxide (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	P_2O_5	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Cement	21.1	4.7	2.6	62.1	2.4	3.2	0.2	0.3	-	-	56.7	17.8	8.2	7.8
Fly Ash	38.7	18.8	5.8	23.1	5.6	1.2	1.8	0.6	1.5	0.4	-	-	-	-

Table 2-2 – Admixture references

Short Hand	Description	Application		
WROS	Wood Rosin	Air-entraining agent		
SYNTH	Synthetic chemical combination	Air-entraining agent		
PC	Polycarboxylate	Superplasticizer		
WR	Triethanolamine	Water reducer		
SRA	Glycol Ethers	Shrinkage reducer		

The wood rosin (WROS) and synthetic (SYNTH) AEA are two popular commercial AEAs. Twenty-five different mixture designs were investigated and are shown in Table 2-3. A subset of mixtures were investigated with either a polycarboxylate (PC) superplaticizer meeting ASTM C1017, a midrange water reducer (WR) meeting ASTM C494, or a shrinkage reducer (SRA) meeting ASTM C494. A dose of between 60 and 200 mL/100 kg was used for the superplasticizer to increase the slump of the mixture between 50 mm to 200 mm. Between four and fourteen dosages of AEA were investigated for each mixture to achieve a range of air contents from 2% to 10%. An ASTM C618 Class C fly ash was used in several of the mixtures with a 20% cement replacement by weight.

w/cm	Cement kg/m ³	Fly-Ash kg/m ³	Paste Volume (%)	Coarse kg/m ³	Fine kg/m ³	Water kg/m ³	Admixture Used
0.45	362	0	29	1098	714	163	WROS
0.45	362	0	29	1098	714	163	SYNTH
0.53	362	0	32	1053	682	192	WROS
0.41	362	0	28	1127	722	148	WROS
0.39	362	0	27	1140	730	141	WROS
0.45	362	0	29	1098	714	163	WROS + PC1
0.45	362	0	29	1098	714	163	SYNTH + PC1
0.45	290	72	30	1089	709	163	WROS
0.45	223	56	23	785/573*	634	126	WROS
0.40	290	72	28	1115	724	145	WROS
0.40	290	72	28	1115	724	145	WROS + PC1
0.35	290	72	28	1127	768	127	WROS + PC1
0.40	290	72	28	1115	724	145	WROS + PC2
0.40	290	72	28	1115	724	145	WROS + PC3
0.40	290	72	28	1115	724	145	WROS + PC4
0.40	290	72	28	1115	724	145	WROS + PC5
0.40	290	72	28	1115	724	145	WROS + WR
0.40	362	0	28	1098	742	145	WROS
0.40	362	0	28	1098	742	145	WROS+PC1
0.45	335	0	27	1142	742	151	WROS
0.45	335	0	27	1142	742	151	WROS+PC1
0.50	335	0	29	1115	724	167	WROS
0.50	335	0	29	1115	724	167	WROS+PC1
0.45	268	67	27	1106	792	151	WROS+WR
0.45	268	67	27	1106	792	151	WROS+WR+SRA

Table 2-3 – SSD Mixture proportions

* Mixture used a coarse and intermediate aggregate blend.

Data is also included in this paper from a study completed by the US Federal Highway Administration (FHWA) Turner Fairbanks Research Lab in McLean, Virginia, USA. This allowed an independent evaluation of the method with other materials but similar methods. This work is summarized in other publications [7].

2.1.2 Field Materials

To investigate the use of the SAM in the field, testing was done by either a Department of Transportation or private testing labs from 21 different States and one Canadian Province. Throughout the entire data set, over 15 users recorded SAM test results. This data is from 110 projects with different combinations of air entrainment, water reducer, and superplasticizer admixtures used. Each concrete mixture will use a different combination of aggregates, admixtures, and mixture designs. The types of aggregates were used by states from Alaska to Florida. The mixtures investigated consist of approximately 61% pavement mixtures, 19% bridge deck mixtures, and 20% other air entrained mixtures. Investigating the performance of the SAM on this wide range of materials allows for a large number of variables to be investigated practical to be done in a controlled laboratory setting.

2.1.3 Laboratory Concrete Mixture Procedure and Testing

Aggregates were collected from outside storage piles, and brought into a temperaturecontrolled room at 23°C for at least 24 hours before mixing. Aggregates were placed in the mixer and spun and a representative sample was taken for a moisture correction. At the time of mixing all aggregate was loaded into the mixer along with approximately two thirds of the mixing water. This combination was mixed for three min to allow the aggregates to approach the saturated surface dry (SSD) condition and ensure that the aggregates were evenly distributed.

Next, the cement, fly ash (if used), and the remaining water was added and mixed for three min. The resulting mixture rested for two min while the sides of the mixing drum were scraped. After the rest period, the mixer was started and the admixtures were added. If the

PC, WR, or SRA was used then it was added first and allowed to mix for 15 seconds to 30 seconds then the AEA was added. After the admixtures were added, the concrete was mixed for three minutes.

Samples were made for hardened air-void analysis (ASTM C457). Two 7 L samples were tested with the SAM. These two samples were investigated simultaneously by different operators to determine the average SAM value of a concrete mixture.

2.1.4 Sequential Air Method

The device used to complete the SAM resembles an ASTM C231 Type B pressure meter with some modifications. The meter uses a digital pressure gauge and six restraining clamps instead of the typical four. These additional clamps are required because of the increased pressures during the SAM test. A picture of an initial version of the device is shown in Figure 2-2.



Figure 2-2 – The device used to complete the SAM.

The different components of the meter are shown in Figure 2-2 and are referenced throughout the procedure. The first step in the method is to fill, consolidate, and level fresh concrete in the bottom chamber according to ASTM C231. A plate is used to level the concrete. Next, the rim and seal between the lid and bottom chamber is cleaned. The lid is then secured to the bottom chamber by the clamps. Water is then added through the petcocks to fill the area between the concrete and the lid. Next, the top chamber is pressurized to $100 \text{ kPa} \pm .7 \text{ kPa}$ (14.5 psi $\pm 0.05 \text{ psi}$) and allowed to stabilize. The petcocks are then closed, and the lever is pressed to bring the two chambers to equilibrium while the bottom chamber is hit on all sides with a rubber mallet. This lever is held for at least 10 s to allow the two chambers to reach equilibrium. The value is recorded and used to calculate the volume of the air in the concrete [8, 9]. Without opening the petcocks, the top chamber is pressurized to 207 kPa \pm .7 kPa (30 psi \pm 0.05 psi). The lever is then pressed for 10 s to bring the two chambers to equilibrium while the bottom chamber is hit on all sides. The top chamber is then pressurized to $310 \text{ kPa} \pm .7 \text{ kPa}$ (45 psi ± 0.05 psi) without opening the petcocks. The lever is then pressed for 10 s and the sides of the bottom chamber are again hit with a rubber mallet. This value should be recorded and will be known as Pc₁. The petcocks are then opened to release the pressure on the bottom chamber. Without removing the lid, water is then added to the bottom chamber to fill the area between the lid and the consolidated concrete and the procedure is repeated. The equilibrium pressure after completing the 310 kPa pressure is recorded as Pc₂. The test takes between eight to ten min by an experienced user to complete. Figure 2-3 shows a typical data set and a video of the test is available [10].



Figure 2-3 - A graphical representation of the pressures in the top and bottom chamber in the SAM.

The device in this paper is an improved version over previous publications [11]. The previous version used five pressure steps with a maximum of 517 kPa (75 psi). This test uses three pressure steps with a maximum pressure of 310 kPa (45 psi) and a more sensitive gauge. These changes increase the speed, accuracy, and create new correlations to air-void quality in the test results.

2.1.4.1 SAM Number calculations

From the results in Figure 2-3 it can be seen that the two pressure curves are not exactly the same. To quantify these differences a term called the SAM Number is used. This can be expressed mathematically as:

SAM Number = $(Pc_2 - Pc_1)/c$

Where Pc₂ is the second equilibrium pressure at 310 kPa (45 psi) and Pc1 is the first equilibrium pressure at 310 kPa (45 psi). The value c is a constant that is 1.45 if the units are in kPa and 1.0 if the units are in psi. SAM Numbers in the 303 mixtures investigated ranged from 0.03 to 0.83. The SAM Number is an empirical number that will be correlated to other parameters such as Spacing Factor and Durability Factor. The SAM Number is reported as a unitless value because it does not have a physical meaning and is only used as a correlative number.

2.1.4.2 Air Content and Aggregate Correction

The volume of air in the concrete can be determined by using Boyle's Law from the first equilibrium pressure at 100 kPa (14.5 psi). This procedure is discussed in other publications [8, 9, 12] and matches the same method and procedure used in the conventional pressure meter (ASTM C231). Past experiments with similar equipment have shown that the air content determined by the SAM closely matched results from the ASTM C231 pressure method [7, 11, 12]. Because the procedures are the same and shown to be equivalent, this is not investigated further in this work.

The calculated air volume with the procedure does not include the aggregate correction factor caused by air contained within the aggregate. The procedure to find the aggregate correction factor is outlined in ASTM C231. Since the SAM Number compares the difference between two sequential pressures, any impact caused by the aggregate on the response to pressure should be removed by subtracting the two pressure responses from each other. The application of this procedure on lightweight aggregates is an area of future research.

2.1.4.3 Estimating Air-void Size by Comparing the Air Volume and SAM Number

Concrete mixtures that contain large air bubbles have been shown to not provide a stable air-void system and not be as effective at providing freeze thaw durability as mixtures with smaller bubbles [13, 14]. The industry would benefit from a method that provides immediate feedback so that mixtures could be quickly evaluated to determine the current size of their bubbles and how different variables affect the size of the bubbles.

One way to determine the average size or quality of the air-void system in concrete is to look at the combination of the volume of air and the SAM Number in the concrete. Since the SAM provides both of these numbers after completing a test, this information could be used to rapidly determine the air-void size distribution in concrete mixtures. For a given air volume, the mixtures with a higher SAM Number have bubbles that are on average larger than mixtures with a smaller SAM Number. However, a user does not always realize if the SAM Number that they are investigating is a large or small value for the air content found. Historic data could be used to provide this guidance.

To do this a quantile regression method was used. A quantile regression takes a set of data and estimates the upper or lower bound of the data. For example, the 50th quantile separates 50% of the data for two different variables. The 85th quantile gives a line where 15% of the data is above and 85% of the data is below. For this work, quantile lines of 85% and 15% provide useful guidance for users to understand where the SAM Number falls in relation to the air content found.

This analysis is useful, as it uses the air content and SAM Number to produce a graph that shows where a typical mixture falls along with mixtures that have on average larger and smaller air-voids. This can be helpful for a user make an immediate evaluation of the average void size of a mixture as both the air content and SAM Number can be measured in the fresh concrete. This immediate feedback can allow users to learn how different ingredients or construction procedures impact the quality of the bubble size and spacing in the concrete.

2.1.4.4 Variability in Measurement

The variability of the SAM Number was evaluated by using two or three operators to investigate the same concrete mixture simultaneously. To get more insight into the variability of the method, previous testing was also done by two operators by using water and a calibration vessel that provided a reading of 5% air. By only using water and a calibration vessel, this allowed the variability of the test to be examined without including the variability of the concrete [4].

2.1.5 Hardened Air Sample Preparation

Samples were cut into 19 mm thick slabs, the surface was treated with an acetone and lacquer mixture to harden the surface, and then the samples were lapped with sequentially finer grits. The prepared surface was then inspected under a stereo microscope. After a satisfactory surface was obtained, the hardener was removed with acetone. The sample was then blackened with black permanent marker, the voids were filled with less than 1 µm white barium sulfate powder, and the voids within the aggregates were blackened under a stereo microscope. This process left the surface of the concrete sample black and the voids within the paste white. Sample preparation details can be found in other publications [12, 15]. The surface was then investigated with ASTM C457 method C by using the Rapid Air 457 from Concrete Experts, Inc. A single threshold value of 185 was used for all samples in this research and the results do not include chords smaller than 30 µm. These settings

have been shown to provide satisfactory results with the materials and instrument used and match the practices by others [15-17].

The hardened air-void analysis from Kansas, Iowa, Pennsylvania, and FHWA were completed by their staff with methods that may be different from that described above. This accounted for roughly 47% of the data shown. The hardened air samples that had differences of more than 2% between the fresh and hardened air content were not included in the analysis. This discrepancy could be caused by a fresh air measurement that was not completed correctly, a hardened sample that was not adequately consolidated, or a combination of admixtures that formed an unstable air-void system. An unstable air-void system would cause the fresh concrete to lose air over time. This can cause the fresh air measurements to be much higher than the hardened concrete. Regardless of the reason, any sample that had drastically different fresh and hardened air contents were not compared.

2.2 RESULTS

2.2.1 Evaluation of the SAM Number compared to the Spacing Factor

In the following figures, two concrete mixtures have been compared to show how the SAM relates to air content and Spacing Factor. The only difference between the two mixtures is that one mixture uses a blend of admixtures and the other uses only an Air Entrainment Admixture (AEA). In Figure 2-4, the comparison between air content and Spacing Factor is presented. The linear trend lines are shown for each mixture. At similar air contents, the Spacing Factor is shown to be different from one mixture to another. To compare, the mixture with just an AEA needs approximately 4.5% air to reach a Spacing Factor of 200 μ m, while the mixture with a blend of admixtures needs approximately 7.5% air to reach 200 μ m. This gap between the two Spacing Factors at a similar air content displays the

challenge of strictly using the air content in fresh concrete to determine the quality of airvoid distribution within the mixture. This supports previous research stating that air volume and air-void quality do not relate the same in all mixtures [4].



Figure 2-4 – Air Content versus Spacing Factor for two laboratory mixtures with similar air volume and different air-void qualities.

In Figure 2-5, the comparison between SAM Number and Spacing Factor is presented. The linear trend lines for each mixture are nearly overlapping each other. The similarity between trends displays a correlation between the SAM Number and Spacing Factor for these two mixtures. The SAM Number shows a more accurate representation of air-void quality in fresh concrete than the air volume comparison. This data set shows that the SAM Number better correlates to the Spacing Factor for these two mixtures than the air volume [4].



Figure 2-5 – SAM Number versus Spacing Factor for the two laboratory mixtures previously shown in Figure 2-4.

In Figure 2-6, the relationship between SAM Number and Spacing Factor is represented for 227 laboratory concrete mixtures completed by two different labs. Within this set of data, 71% of the laboratory mixtures were completed at Oklahoma State University and 29% of the laboratory mixtures were completed at FHWA Turner Fairbanks [7]. Refer to the appendix for all of the lab mixtures completed by Oklahoma State University.

There seems to be a relationship between the SAM Number and Spacing Factor as shown in Figure 2-6. As the SAM Number increases then so does the Spacing Factor for the majority of the data. The distributed data could possibly be from variation in test measurements or aggregates and admixture combinations. Past recommendations in freeze thaw analysis have used a single value to determine if a material is recommended for freeze thaw durability. This has also been beneficial in aiding industry implementation. One of the most common values to use is 200 µm. Past work has suggested that a SAM Number

of 0.20 correctly determines if a Spacing Factor is above or below 200 μ m for 88% of the data [4].



Figure 2-6 – SAM Number versus Spacing Factor for 227 laboratory concrete mixtures completed by two different research groups.

While laboratory testing is helpful, it is still unclear if the SAM is a useful tool for field usage. To investigate this, the SAM was used to measure field mixtures completed by either a Department of Transportation or private testing labs from 21 different States and one Canadian Province for 110 projects with different concrete mixtures.

The SAM Number and Spacing Factor are plotted together for the field data in Figure 2-7. A similar trend is shown in both the laboratory and field data. The Spacing Factor limit of 200 µm from ACI 201.2R-16 [18] is displayed in Figure 2-7 as well as a SAM Number limit of 0.20. Four quadrants are created by the two limit values. The data points in the upper right hand quadrant represent 22% of the data set in Figure 2-7. These concrete mixtures would not be recommended for use in freezing climates. These projects may show a reduced lifespan if they are exposed to moisture and freezing temperatures. If these mixtures were found in the field with a tool like the SAM, then they could have been adjusted and would have produced longer lasting concrete. If only a single project could have been helped by this measurement, then it would make a significant savings to the public.



Figure 2-7 – SAM Number versus Spacing Factor for 231 field concrete mixtures completed by 21 different state DOTs with various aggregates and admixtures.

The SAM Number and Spacing Factor can then be separated into four quadrants using these two limit values. Two of the quadrants agree and the other two disagree. For example,

the SAM Number and the Spacing Factor agree above or below a critical value or one measurement is satisfied while the other is not. A quantitative method was used to choose the best SAM Number for a Spacing Factor needed. This method displayed the SAM Number limit to be where the most data fell within the quadrants in agreement. The best correlating SAM Numbers were found for spacing factors of 200 μ m, 250 μ m, and 300 μ m. The results from this analysis are shown in Figure 2-8. The correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μ m agrees with 85% of the data comparisons.



Figure 2-8 – Percent agreement between SAM Number and different Spacing Factors for laboratory concrete mixtures.

For this field data set, the best correlating SAM Number was found for a spacing factor of 200 μ m to compare to the laboratory data. The results from this analysis are shown in Figure 2-9. The correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μ m agrees with 70% of the data comparisons. Figure 2-9 shows that the agreement improves to roughly 78% with a SAM Number of 0.25. Previous research has shown that a SAM Number range from 0.20 to 0.25 agrees with 88% to 83% of the laboratory data

[4]. These numbers are very close and show that the 0.20 SAM Number found in the laboratory data continues to be a conservative measurement to use in the field.



Figure 2-9 – Percent agreement between SAM Number and different Spacing Factors for field concrete mixtures.

It is encouraging that similar relationships can be found for both the laboratory and field data. The use of the SAM Number can be beneficial to the concrete industry, as the SAM Number can be found in the fresh concrete within 10 minutes. This allows the quality of the air-void system to be obtained and for immediate adjustments to be made if necessary. In comparison, the Spacing Factor takes seven to ten days to cut, polish, and analyze a hardened concrete sample to measure the quality of air-void system. The correlation of Spacing Factor and a single SAM Number is significant and displays a general agreement between the two different methods. Since the field data uses a wide range of materials in actual construction conditions, this correlation proves to be a strong validation of the SAM method. Being able to adjust a concrete mixture with the SAM Number before placing it

would be a huge improvement in saving time, money, concrete materials, and expanding the life of in-place infrastructure.

2.2.2 Evaluation of the SAM Number Compared to the Air Content

While comparing the SAM Number to the Spacing Factor shows the validity of the SAM test, it would be helpful to give immediate feedback to the user about the quality of the air-void system in the concrete. The two parameters that are measured in the SAM test are the air content and the SAM Number. It may be possible to compare these numbers and give users much better insight on the average size distribution of their air bubbles based on historic data.

The relationship between the air content and SAM Number is shown in Figure 2-10 for laboratory mixtures. Within this set of data, 71% of the laboratory mixtures were completed at Oklahoma State University and 29% of the laboratory mixtures were completed at FHWA Turner Fairbanks [7].

Two cubic polynomial lines are included to show the 85th and 15th quantile. These lines represent the lower and upper bounds of the SAM Number at a given air content. The lower line represents 15% of the data and the top line represents 85% of the data. These lines are not limitations to the data set, but rather guidelines for the user to understand whether the SAM Number is low or high compared to the volume of air in the mixture. These two cubic lines were found to be the best representation of how the data varies. Other trend lines were investigated but they did not provide a useful representation of the investigated data set.

Equation $1 - 15^{\text{th}}$ Quantile:

 $y = -0.0006x^3 + 0.0186x^2 - 0.1888x + 0.6804$

Equation 2 – 85th Quantile:

 $y = 0.0014x^3 - 0.0102x^2 - 0.1061x + 0.9213$

These lines can help SAM users to understand where their concrete mixture falls compared to other SAM Numbers from a wide variety of tests. The closer the SAM Number is to the 15% line, the finer the air-void distribution. If the number is closer the 85% line, then the air-void distribution is coarser for a specific air volume. These guidelines are based on 227 different concrete mixtures consisting of five different admixtures, eight different water cement ratios (w/cm), and a range of 2% to 10% air contents. It should be noted that these lines are dependent on the mixtures that were investigated. However, the results are helpful as it gives insight into the average size of the bubble system before the concrete has hardened. Due to the wide variety of admixtures, aggregates, and user experience, the 15th and 85th quantiles help to simplify a range that best represents the SAM Number versus air content instead of a single trend line for all test runs.



Figure 2-10 – Air Content versus SAM Number for 227 laboratory concrete mixtures completed by two different research groups.

The relationship between air content and SAM Number is displayed in Figure 2-11 for the field data. The guidelines established from the laboratory data were added to Figure 2-11 to show the relationship between laboratory concrete mixture results and field test results. The scattered data points above the 85th quantile line show that many of the mixtures seem to have a coarse air-void distribution. This means that these mixtures are not as effective in providing freeze thaw durability for a given volume of air. The air-void systems created in these mixtures also may not be as stable.



Figure 2-11 – Air Content versus SAM Number for 231 field concrete mixtures completed by 21 different state DOTs with various aggregates and admixtures.

2.2.3 Variability in SAM Measurements

To consider the variability of the test method, previous research studied the average difference between two SAM Number measurements using water and a calibration vessel. The average difference was found to be 0.008 with a standard deviation of 0.049. This means that on average the two measurements between two meters will be off by 0.008 but that the expected difference between two measurements can vary by 0.10 for a 95% confidence interval (two standard deviations). These numbers are important for users to understand when specifying and using the SAM Numbers [4].

In Table 2-4 the variability of hardened air void test (ASTM C457) and the rapid freeze thaw test (ASTM C666) test were compared to the variance measurements of the SAM

Number. These three measurements intend to measure the air-void system quality in fresh or hardened concrete. The coefficient of variation (COV) was used to compare the three tests. The table shows that all three tests have a COV of below 12%. The SAM Number shows a lower COV than the Spacing Factor. The comparison between the two and the Durability Factor is also shown. The SAM Number shows a higher agreement with the ASTM C666 test than the Spacing Factor.

Table 2-4 - A comparison of the coefficient of variation, agreement with Durability Factor, and the time required to complete the test.

Test Method	Parameter	COV	Agreement with Durability Factor in ASTM C666	Time to complete the test
AASHTO TP 118	SAM Number ¹	6.5%	72%	10 min
ASTM C457	Spacing Factor ²	11.5%	63%	7 days
ASTM C666	Durability Factor ³	4.6%	-	3.5 months

¹Assumes a SAM Number of 0.32 and a standard deviation of 0.019 from this paper

²Assumes a Spacing Factor of 300µm

³From ASTM C666 with a durability factor of 70 and Method B

2.3 DISCUSSION

This work studies the implementation of a new method comparing laboratory data and field data. Using a wide variety of concrete mixtures shows the strength in diversity of the SAM test. The SAM Number provides feedback to the user before the mixture is placed to determine if it needs to be adjusted to meet specification requirements. If the industry were able to adjust concrete mixtures before placement, there would be less rejected concrete mixtures and longer lasting concrete after placement.

The curves on the air content versus SAM Number figures have been established to provide guidelines for users to understand where their concrete mixture stands in relation to a variety of other concrete mixtures in terms of freeze thaw durability. Using these guidelines, new admixtures and aggregates can be studied and adjusted with the SAM Number to figure out how various materials affect the quality of the air-void distribution. This shows great promise to be a tool that can help producers design their concrete, troubleshoot field practices, and provide concrete that has a high confidence of freeze thaw durability.

2.4 SUMMARY

This work compares laboratory concrete mixtures to a large-scale field study analysis to determine the reliability of the SAM test method and give guidance to field users. Two testing laboratories, 21 states, and one Canadian Province investigated results from 458 concrete mixtures.

These specific findings have been made:

- For 227 laboratory mixtures, the correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μm agrees with 84% of the laboratory data comparisons.
- For 231 field mixtures, the correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μm agrees with 71% of the field data comparisons.
- SAM Number versus Spacing Factor data for both laboratory and field concrete mixtures show that the SAM Number and Spacing Factor are correlated and there is agreement for a 0.20 SAM Number and a 200µm Spacing Factor.
• Cubic 85% and 15% quantile lines based on the laboratory data provides a useful tool to evaluate the average void size in fresh concrete mixtures. This can be a useful tool for a user to gain immediate feedback on how their concrete mixtures, material changes, and construction practices impact the average void size in their concrete

Because the SAM provides rapid feedback that is useful, it has the ability to impact each phase within the concrete industry for the better, from materials to producers to construction implementation.

CHAPTER III

SEQUENTIAL AIR METHOD TESTING WITH ADMIXTURES

3.0 INTRODUCTION

Freeze thaw durability of a concrete mixture can be enhanced by the addition of an airentrained admixture (AEA). The tiny, well-spaced bubbles help water escape during the freezing and thawing process [2, 4-6]. The size and spacing of the bubbles represents the quality of the air-void system. This quality works to improve the freeze thaw durability and helps prolong the lifespan of concrete structures.

The air-void distribution cannot be found within fresh concrete with established tests for freeze thaw durability. While other methods can measure the total air volume in fresh concrete, studies have shown that the air volume is not the only indicator of freeze thaw durability. The small, well-dispersed bubbles improves the quality of the air-void system.

The ASTM C457 method measures the volume and spacing of air-voids to find the Spacing Factor of hardened concrete. The process takes between 7 and 14 days to complete [2, 3]. The Spacing Factor gives a good understanding of the freeze thaw durability, but does not allow for adjustments to be made to the concrete mixture before placement [3, 4]. This work uses these tools to investigate specific concrete mixtures.

Laboratory test results from two different data sets containing various admixtures are used to analyze the usefulness of the SAM test method.

This work aims to show the usefulness of the SAM to investigate a series of different concrete mixtures with superplasticizers and AEAs, SRAs and AEAs, and mixtures with just AEAs. Data from the SAM will be used to show how these different mixtures impact the air void size and spacing in the concrete and how this impacts the freeze thaw durability of the concrete.

3.1 EXPERIMENTAL METHODS

3.1.1 Laboratory Materials

All of the laboratory concrete mixtures in this research used a Type I cement that met the requirements of ASTM C150. Both the oxide analysis and Bogue calculations for this cement used is shown in Table 3-1. The aggregates used were locally available crushed limestone and natural sand used in commercial concrete. The crushed limestone had a maximum nominal aggregate size of 19 mm (3/4"). One mixture contained a blend of the coarse and intermediate aggregate as well. Both the crushed limestone and the sand met ASTM C33 specifications. All the admixtures used are described in Table 3-2, which met the requirements of ASTM C260 and ASTM C494.

Table 3-1 – Type I cement oxide analysis

Oxide (%)	SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	P_2O_5	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Cement	21.1	4.7	2.6	62.1	2.4	3.2	0.2	0.3	-	-	56.7	17.8	8.2	7.8
Fly Ash	38.7	18.8	5.8	23.1	5.6	1.2	1.8	0.6	1.5	0.4	-	-	-	-

Table 3-2 – Admixture references

Short Hand	Description	Application
WROS	Wood Rosin	Air-entraining agent
PC	Polycarboxylate	Superplasticizer
WR	Triethanolamine	Water reducer
SRA	Glycol Ethers	Shrinkage reducer

The wood rosin (WROS) AEA is a popular commercial AEA. Eight different mixture designs were investigated, and are shown in Table 3-3. A subset of mixtures was investigated with either a polycarboxylate (PC) superplaticizer meeting ASTM C1017, a midrange water reducer (WR) meeting ASTM C494, or a shrinkage reducer (SRA) meeting ASTM C494. A dose of between 60 and 200 mL/100 kg was used for the superplasticizer to increase the slump of the mixture between 50 mm to 200 mm. Between four and seven dosages of AEA were investigated for each mixture to achieve a range of air contents from 2% to 10%. An ASTM C618 Class C fly ash was used in several of the mixtures with a 20% cement replacement by weight.

Table 3-3 – SSD Mixture proportions

w/cm	Cement kg/m ³	Fly-Ash kg/m ³	Paste Volume (%)	Coarse kg/m ³	Fine kg/m ³	Water kg/m ³	Admixture Used
0.40	362	0	28	1098	742	145	WROS
0.40	362	0	28	1098	742	145	WROS+PC1
0.45	335	0	27	1142	742	151	WROS
0.45	335	0	27	1142	742	151	WROS+PC1
0.50	335	0	29	1115	724	167	WROS
0.50	335	0	29	1115	724	167	WROS+PC1
0.45	268	67	27	1106	792	151	WROS+WR
0.45	268	67	27	1106	792	151	WROS+WR+SRA

3.1.2 Concrete Mixture Procedure and Testing

Aggregates were collected from outside storage piles, and brought into a temperaturecontrolled room at 23°C for at least 24 hours before mixing. Aggregates were placed in the mixer and spun and a representative sample was taken for a moisture correction. At the time of mixing all aggregate was loaded into the mixer along with approximately two thirds of the mixing water. This combination was mixed for three min to allow the aggregates to approach the saturated surface dry (SSD) condition and ensure that the aggregates were evenly distributed.

Next, the cement, fly ash (if used), and the remaining water was added and mixed for three min. The resulting mixture rested for two min while the sides of the mixing drum were scraped. After the rest period, the mixer was started and the admixtures were added. If the PC, WR, or SRA was used then it was added first and allowed to mix for 15 seconds to 30 seconds then the AEA was added. After the admixtures were added, the concrete was mixed for three minutes.

Samples were made for hardened air-void analysis (ASTM C457). Two 7 L samples were tested with the SAM. These two samples were investigated simultaneously by different operators to determine the average SAM value of a concrete mixture.

3.1.2.1 Mixtures with Superplasticizer

Seventeen concrete mixtures with w/cms of 0.40, 0.45, and 0.50 with and without Superplasticizer (PC1). These mixtures were compared to concrete mixtures with the same w/cm and various amounts of the PC1 in order to reach a target slump of 200 mm. This caused different amounts of PC1 to be used with mixtures of different w/cm. For instance,

all of the 0.50 w/cm mixtures contained 155 mL/100 kg of PC1, while all of the 0.40 w/cm mixtures contained 390 mL/100 kg grams of PC1.

3.1.2.2 Shrinkage Reducing Admixture

Seven concrete mixtures with a set amount of WR and a set amount of the SRA were tested using a range of AEA amounts. A dose of 185 mL/100 kg was used for the water reducer. The SRA dosage of 4752 mL/100 kg was chosen to reduce drying shrinkage of the concrete. One control mixture without SRA was used as a comparison. Two SAM tests were performed immediately following the completion of the mixture and two were performed 60 minutes after the first set of tests were complete. This was done to investigate the stability of the air void system.

3.1.3 Sequential Air Method

The device used to complete the SAM resembles an ASTM C231 Type B pressure meter with some modifications. The meter uses a digital pressure gauge and six restraining clamps instead of the typical four. These additional clamps are required because of the increased pressures during the SAM test. A picture of an initial version of the device is shown in Figure 3-1.



Figure 3-1 – The device used to complete the SAM.

The different components of the meter are shown in Figure 3-1 and are referenced throughout the procedure. The first step in the method is to fill, consolidate, and level fresh concrete in the bottom chamber according to ASTM C231. A plate is used to level the concrete. Next, the rim and seal between the lid and bottom chamber is cleaned. The lid is then secured to the bottom chamber by the clamps. Water is then added through the petcocks to fill the area between the concrete and the lid. Next, the top chamber is pressurized to 100 kPa \pm .7 kPa (14.5 psi \pm 0.05 psi) and allowed to stabilize. The petcocks are then closed, and the lever is pressed to bring the two chambers to equilibrium while the bottom chamber is hit on all sides with a rubber mallet. This lever is held for at least 10 s to allow the two chambers to reach equilibrium. The value is recorded and used to calculate the volume of the air in the concrete [8, 9]. Without opening the petcocks, the top chamber is pressurized to 207 kPa \pm .7 kPa (30 psi \pm 0.05 psi). The lever is then pressed for 10 s to

bring the two chambers to equilibrium while the bottom chamber is hit on all sides. The top chamber is then pressurized to $310 \text{ kPa} \pm .7 \text{ kPa}$ (45 psi $\pm 0.05 \text{ psi}$) without opening the petcocks. The lever is then pressed for 10 s and the sides of the bottom chamber are again hit with a rubber mallet. This value should be recorded and will be known as Pc₁. The petcocks are then opened to release the pressure on the bottom chamber. Without removing the lid, water is then added to the bottom chamber to fill the area between the lid and the consolidated concrete and the procedure is repeated. The equilibrium pressure after completing the 310 kPa pressure is recorded as Pc₂. The test takes between eight to ten min by an experienced user to complete. Figure 2-3 shows a typical data set and a video of the test is available [10].



Figure 3-2 - A graphical representation of the pressures in the top and bottom chamber in the SAM.

The device in this paper is an improved version over previous publications [11]. The previous version used five pressure steps with a maximum of 517 kPa (75 psi). This test uses three pressure steps with a maximum pressure of 310 kPa (45 psi) and a more sensitive gauge. These changes increase the speed, accuracy, and create new correlations to air-void quality in the test results.

3.1.3.1 SAM Number Calculations

From the results in Figure 2-3 it can be seen that the two pressure curves are not exactly the same. To quantify these differences a term called the SAM Number is used. This can be expressed mathematically as:

SAM Number = $(Pc_2 - Pc_1)/c$

Where Pc₂ is the second equilibrium pressure at 310 kPa (45 psi) and Pc1 is the first equilibrium pressure at 310 kPa (45 psi). The value c is a constant that is 1.45 if the units are in kPa and 1.0 if the units are in psi. SAM Numbers in the 303 mixtures investigated ranged from 0.03 to 0.83. The SAM Number is an empirical number that will be correlated to other parameters such as Spacing Factor and Durability Factor. The SAM Number is reported as a unitless value because it does not have a physical meaning and is only used as a correlative number.

3.1.3.2 Air Content and Aggregate Correction

The volume of air in the concrete can be determined by using Boyle's Law from the first equilibrium pressure at 100 kPa (14.5 psi). This procedure is discussed in other publications [8, 9, 12] and matches the same method and procedure used in the conventional pressure meter (ASTM C231). Past experiments with similar equipment have shown that the air

content determined by the SAM closely matched results from the ASTM C231 pressure method [7, 11, 12]. Because the procedures are the same and shown to be equivalent, this is not investigated further in this work.

The calculated air volume with the procedure does not include the aggregate correction factor caused by air contained within the aggregate. The procedure to find the aggregate correction factor is outlined in ASTM C231. Since the SAM Number compares the difference between two sequential pressures, any impact caused by the aggregate on the response to pressure should be removed by subtracting the two pressure responses from each other. The application of this procedure on lightweight aggregates is an area of future research.

3.1.3.3 Void Size Estimation

Concrete mixtures that contain large air bubbles have been shown to not provide a stable air-void system and not be as effective at providing freeze thaw durability as mixtures with smaller bubbles [13, 14]. The industry would benefit from a method that provides immediate feedback so that mixtures could be quickly evaluated to determine the current size of their bubbles and how different variables affect the size of the bubbles.

One way to determine the average size or quality of the air-void system in concrete is to look at the combination of the volume of air and the SAM Number in the concrete. Since the SAM provides both of these numbers after completing a test, this information could be used to rapidly determine the air-void size distribution in concrete mixtures. For a given air volume, the mixtures with a higher SAM Number have bubbles that are on average larger than mixtures with a smaller SAM Number. However, a user does not always realize if the SAM Number that they are investigating is a large or small value for the air content found. Historic data could be used to provide this guidance.

To do this a quantile regression method was used. A quantile regression takes a set of data and estimates the upper or lower bound of the data. For example, the 50th quantile separates 50% of the data for two different variables. The 85th quantile gives a line where 15% of the data is above and 85% of the data is below. For this work, quantile lines of 85% and 15% provide useful guidance for users to understand where the SAM Number falls in relation to the air content found.

This analysis is useful, as it uses the air content and SAM Number to produce a graph that shows where a typical mixture falls along with mixtures that have on average larger and smaller air-voids. This can be helpful for a user make an immediate evaluation of the average void size of a mixture as both the air content and SAM Number can be measured in the fresh concrete. This immediate feedback can allow users to learn how different ingredients or construction procedures impact the quality of the bubble size and spacing in the concrete.

More details are given in the previous chapter. This chapter aims to show the usefulness of these tools by applying them to investigate concrete mixtures with different admixture combinations.

3.1.4 Hardened Air Sample Preparation

Samples were cut into 19 mm thick slabs, the surface was treated with an acetone and lacquer mixture to harden the surface, and then the samples were lapped with sequentially finer grits. The prepared surface was then inspected under a stereo microscope. After a

satisfactory surface was obtained, the hardener was removed with acetone. The sample was then blackened with black permanent marker, the voids were filled with less than 1 μ m white barium sulfate powder, and the voids within the aggregates were blackened under a stereo microscope. This process left the surface of the concrete sample black and the voids within the paste white. Sample preparation details can be found in other publications [12, 15]. The surface was then investigated with ASTM C457 method C by using the Rapid Air 457 from Concrete Experts, Inc. A single threshold value of 185 was used for all samples in this research and the results do not include chords smaller than 30 μ m. These settings have been shown to provide satisfactory results with the materials and instrument used and match the practices by others [15-17].

3.2 RESULTS

3.2.1 Mixtures With and Without a Superplasticizer

Concrete mixtures with three different w/cm with and without a superplasticizer have been compared to show how the SAM relates to Spacing Factor, air content, and Durability Factor. A variety of air contents ranging from 2% to 9% was studied to see how PC1 would affect the quality of the air-void system.

Figure 3-3 shows the relationship between air content and Spacing Factor for three w/cm with and without PC1. Trend lines are shown to differentiate the two sets of mixtures. The data shows that the Spacing Factor is different between the two types of mixtures at similar air contents. As the trend lines show, the mixtures without PC1 require 5% air to reach a Spacing Factor of 200 μ m while the mixtures containing PC1 require nearly 7% air. This relationship between air volume and Spacing Factor makes it difficult for the quality of the air-void system to be determined in the fresh concrete by only using air content. While this

can be observed with the Spacing Factor, it is not possible to get this information before the concrete has hardened.



Figure 3-3 – Air Content versus Spacing Factor for 0.40, 0.45, and 0.50 w/cm concrete mixtures with and without PC1.

In Figure 3-4, the same set of mixtures is shown for the SAM Number compared to the Spacing Factor. The results show that the SAM Number of 0.20 was able to identify correctly if the Spacing Factor was above or below 200 µm for 27 out of 30 mixtures for 90% of the data. This shows that the SAM Number correlates better to the Spacing Factor or quality of air-void system for these two sets of mixtures than the air content shows in Figure 3-3. The limitations of the Spacing Factor process taking a significant amount of time slows down the process of accepting or rejecting a concrete mixture. The industry needs quicker results like the SAM test to adjust fresh concrete mixtures before placement.



Figure 3-4 – SAM Number versus Spacing Factor for 0.40, 0.45, and 0.50 w/cm concrete mixtures with and without PC1.

The comparison between air content and SAM Number for the three different w/cms is shown in Figure 3-5. The differences between mixtures with and without PC1 have similar results despite the differing w/cms. All three w/cm show that when PC1 is added to the mixture, a minimum of 6% air is needed to provide a 0.20 SAM Number while a minimum of 4% air is needed for the mixtures without PC1. This means that the PC1 coarsens the air-void distribution. This can be confirmed by using the results from the quantile analysis.

In Figure 3-5, the overall trends in mixtures with and without PC1 are shown. The mixtures containing PC1 above 6% air fall near the 15th quantile line, which means they have a fine air-void distribution and pass a 0.20 SAM Number. Once the mixtures fall below 6% air

content, they move toward the 85th quantile line, which means they have a coarse air-void distribution and do not pass the SAM Number of 0.20. The mixtures without PC1 fall near the 15th quantile line starting at 4% air, which means they have a fine air-void distribution. No matter the w/cm, the mixtures without PC1 seem to have a better air-void distribution starting at a lower air content. Knowing that the superplasticizer affects the air content makes it helpful to have the SAM Number measurement that provides insight into the quality of the air-void system of any mixture.



Figure 3-5 – Air content versus SAM Number for 0.40, 0.45, and 0.50 w/cm concrete mixtures with and without PC1.

These same mixtures are shown in Figure 3-6 and Figure 3-7. The relationship between air content and SAM Number are compared to the Durability Factor. Polynomial trend lines

are shown in both figures. However, in Figure 3-6 the air content is shown to be very different for the two sets of mixtures to reach a Durability Factor of 70%. The mixtures containing PC1 need nearly 5% air to pass while the mixtures without it need a minimum of 3% air. This agrees with previous observations that the volume of air is difficult to use for determining the air-void system quality within these concrete mixtures, which also makes it difficult to determine for freeze thaw durability measurements.



Figure 3-6 – Air Content versus Durability Factor for 0.40, 0.45, and 0.50 w/cm concrete mixtures with and without PC1.

The relationship between the SAM Number and the Durability Factor is shown in Figure 3-7. The trend lines for each set of mixtures are shown to nearly overlap, which means the SAM Number seems to be a more reasonable form of measurement when comparing freeze thaw durability performance. Previous studies have shown that a SAM Number of 0.32

correlates with a Durability Factor of 70% [4]. In Figure 3-7, 87% of the data agrees with the recommended SAM Number and Durability Factor shown.



Figure 3-7 – SAM Number versus Durability Factor for 0.40, 0.45, and 0.50 w/cm concrete mixtures with and without PC1.

By comparing the SAM Number and air content to Spacing Factor and Durability Factor, the data shows that the volume of air within a concrete mixture does not provide enough information to determine a set air content for every mixture to pass all specifications. The SAM Number has shown to provide insight into both hardened air-void analysis and freeze thaw durability before the concrete is placed. This type of information before the concrete hardens allows the mixture to be adjusted to meet necessary requirements to make longer lasting concrete infrastructure.

3.2.2 Mixture with Shrinkage Reducing Admixture

Concrete mixtures with a WR and SRA are compared to mixture with just a WR. A variety of air contents ranging from 2% to 10% was studied to see how the SRA would affect the quality of the air-void system. One mixture did not contain the SRA to act as a control. Two SAM tests were performed immediately following the completion of the mixture and two were performed 60 minutes after the first set of tests were complete. This helps investigate the air void stability of the concrete mixture.

The comparison between air content and Spacing Factor is shown in Figure 3-8. For each mixture, the 0 min data point and the 60 min data point are shown with an arrow between to help understand how a given mixture moves. The Spacing Factor is shown to change over 60 min for air contents starting at 6% containing SRA. To compare, the mixture with SRA needs approximately 7% air to reach a Spacing Factor of 200 μ m after 60 min, while the control mixture stays constantly below 200 μ m from 5.5% air at 0 min to 4.5% air at 60 min. This change in the two Spacing Factors after one hour shows that concrete mixtures containing SRA need to be designed for a higher air content than desired on site.



Figure 3-8 – Air content versus Spacing Factor for six laboratory mixtures with SRA and one control mixture without SRA.

The comparison between SAM Number and Spacing Factor is shown in Figure 3-9. The change in arrows for 0 min and 60 min show that the Spacing Factor typically increases after 60 min for mixtures with SRA, but stays steady for the control mix without SRA. The agreement between quadrants displays a correlation between the SAM Number and Spacing Factor. The SAM Number shows a more accurate representation of air-void quality in fresh concrete than the air volume comparison. Because the air volume measurement needed changes from mixtures with and without SRA, the SAM Number helps identify which mixtures will meet specifications and which ones will not.



Figure 3-9 – SAM Number versus Spacing Factor for six laboratory mixtures with SRA and one control mixture without SRA.

The relationship between the air content and SAM Number is shown in Figure 3-10. Seven concrete mixtures with various air contents at 0 min are shown to lose air 60 min after time of mix. The higher the initial air content, the larger the air loss seems to be after 60 min. To compare, the mixture starting with 10% air lost 3% over 60 min, and the mixture starting at 4% air lost 1% in 60 min. The control mix without SRA had a 19% air loss within an hour of mixing. The SRA mixtures lost an average of 32% initial air within an hour of mixing. This study has shown for mixtures containing SRA, approximately 7% of air is needed initially to obtain a passing SAM Number of 0.20 after 60 min. Refer Table 3-4 for an outline of air loss depending on initial air content.



Figure 3-10 – Air content versus SAM Number for seven laboratory mixtures with SRA and one control mixture without SRA.

Mix #	Initial Air Content	Air Content at 60 min	Air Loss	% Loss
SRA Mix 1	4.1%	3.0%	1.1%	27%
SRA Mix 2	5.0%	4.1%	1.0%	19%
SRA Mix 3	6.3%	4.1%	2.3%	36%
SRA Mix 4	6.8%	4.5%	2.3%	34%
SRA Mix 5	8.3%	4.9%	3.4%	41%
SRA Mix 6	9.0%	5.7%	3.3%	37%
SRA Mix 7	10.1%	7.1%	3.0%	30%
			Average	32%
			Standard Deviation	7%
No SRA	5.7%	4.6%	1.1%	19%

1 auto 3 - 4 - 3 KA All Lus	Table
-----------------------------	-------

These same mixtures were investigated to determine the relationship between air volume and SAM Number and the Durability Factor. Figure 3-11 shows that the mixtures that passed at 0 min failed at 60 min. For the mixtures with SRA, the air content needed to start at 9% air for a Durability Factor to be above 70% to be reached after 60 min. This shows the challenges with using the volume of air to predict the air-void quality in concrete mixtures containing SRA and in turn the freeze thaw durability of the mixture.



Figure 3-11 – Air content versus Durability Factor for seven laboratory mixtures with SRA and one control mixture without SRA.



Figure 3-12 – Spacing Factor versus Durability Factor for six laboratory mixtures with SRA and one control mixture without SRA.

In Figure 3-13, the SAM Number versus Durability Factor is shown. The data shows that mixtures containing SRA could require a lower SAM Number to pass freeze thaw performance at 70%. The data shows that a SAM Number of less than 0.15 or possibly 0.10 may be necessary. This SAM Number relates to the 9% air content needed in Figure 3-11 to pass freeze thaw durability. This is a much lower SAM Number than what has been observed in previous testing. It is currently unknown as to what mechanism causes these results.



Figure 3-13 – SAM Number versus Durability Factor for seven laboratory mixtures with SRA and one control mixture without SRA.

3.3 DISCUSSION

This work studies the implementation of a new method of investigating concrete mixtures with different admixtures with SAM. This analysis shows that the SAM Number has the ability to give immediate insight into the freeze thaw durability of the mixture despite the various w/cm. The drastic loss in air in the SRA mixtures over 60 min shows how the SAM Number can be used to predict the air-void quality within the fresh concrete.

The quantile lines on the air content versus SAM Number figures show the fine and coarse air-void distributions to provide guidelines for studying new admixtures. These curves will help users to understand where their concrete mixture stands in relation to a variety of other concrete mixtures in terms of freeze thaw durability. The two data sets studied show the ability of the SAM test to study new admixtures to determine how various materials affect the quality of the air-void distribution. Producers, contractors, and suppliers can all benefit from testing fresh concrete mixtures with various admixtures to ensure proper freeze thaw durable concrete.

3.4 SUMMARY

This work investigated concrete mixtures with different admixtures to determine the usefulness of the SAM to predict the air-void size distribution and their performance in freeze thaw testing.

These specific findings have been made:

- The 0.40, 0.45, and 0.50 w/cm studied show that when PC1 is added, a minimum of 6% air is needed to pass a 0.20 SAM Number while a minimum of 4% air is needed for the mixtures without PC1.
- The PC1 mixtures required 5% air to reach a Spacing Factor of 200 μm while the mixtures containing PC1 require nearly 7% air.
- The mixtures containing PC1 need nearly 5% air to reach a Durability Factor of 70% while the mixtures without it need a minimum of 3% air.
- The SRA mixtures studied lost an average of 32% initial air within an hour of mixing.
- This study has shown for mixtures containing SRA, approximately 7% of air is needed initially to obtain a passing SAM Number of 0.20 after 60 min.

- The mixtures with SRA need approximately 7% air to reach a Spacing Factor of 200 µm after 60 min, while the mixture without SRA stays constantly below 200 µm from 5.5% air at 0 min to 4.5% air at 60 min.
- The Spacing Factor typically increased after 60 min for the mixtures studied with SRA.
- For the mixtures with SRA, the air content needed to start at 9% air for a Durability Factor above 70% to be reached after 60 min.

These studies show the usefulness of the SAM to investigate their performance. This continues to show the promise of this test to serve as a useful tool to measure the air void size and distribution in fresh concrete.

CHAPTER IV

CONCLUSION

4.0 SUMMARY

This thesis was composed of three main studies to verify the SAM test method: a largescale study of 227 laboratory mixtures, a large-scale study of 231 field mixtures, and laboratory mixtures with various admixtures. The diversity among the data shown determines the reliability of the SAM test method and gives guidance to field users.

Cubic quantile lines based on the laboratory data provide helpful insight into the average air-void size in fresh concrete mixtures for users to know whether the mixture will meet specifications or not. The 15th and 85th quantile lines are shown as fine and coarse air-void distribution curves. These curves act as guidelines for field users to relate to data from a wide variety of other mixtures.

The overall study has shown promising results for the SAM method to provide a positive impact in the concrete industry. Nearly immediate feedback from fresh concrete mixtures is test method that could benefit each phase within the concrete industry for the better, from materials to producers to construction implementation. The following conclusions have been drawn from Chapter 2:

- For 227 laboratory mixtures, the correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μm agrees with 84% of the laboratory data comparisons.
- For 231 field mixtures, the correlation between a SAM Number of 0.20 and a Spacing Factor of 200 μm agrees with 71% of the field data comparisons.
- SAM Number versus Spacing Factor data for both laboratory and field concrete mixtures show that the SAM Number and Spacing Factor are correlated and there is agreement for a 0.20 SAM Number and a 200µm Spacing Factor.
- Cubic 85% and 15% quantile lines based on the laboratory data provides a useful tool to evaluate the average void size in fresh concrete mixtures. This can be a useful tool for a user to gain immediate feedback on how their concrete mixtures, material changes, and construction practices impact the average void size in their concrete.

Conclusions from Chapter 3:

- The 0.40, 0.45, and 0.50 w/cm studied show that when PC1 is added, a minimum of 6% air is needed to pass a 0.20 SAM Number while a minimum of 4% air is needed for the mixtures without PC1.
- The PC1 mixtures required 5% air to reach a Spacing Factor of 200 µm while the mixtures containing PC1 require nearly 7% air.
- The mixtures containing PC1 need nearly 5% air to reach a Durability Factor of 70% while the mixtures without it need a minimum of 3% air.

- The SRA mixtures studied lost an average of 32% initial air within an hour of mixing.
- This study has shown for mixtures containing SRA, approximately 7% of air is needed initially to obtain a passing SAM Number of 0.20 after 60 min.
- The mixtures with SRA need approximately 7% air to reach a Spacing Factor of 200 µm after 60 min, while the mixture without SRA stays constantly below 200 µm from 5.5% air at 0 min to 4.5% air at 60 min.
- The Spacing Factor typically increased after 60 min for the mixtures studied with SRA.
- For the mixtures with SRA, the air content needed to start at 9% air for a Durability Factor above 70% to be reached after 60 min.

REFERENCES

- 1. Ley, M.T., *The Effects of Fly Ash on the Ability to Entrain and Stabilize Air in Concrete* in *Civil, Architectural, and Environmental Engineering*. 2007, University of Texas at Austin
- 2. Backstrom, J., et al., *Void spacing as a basis for producing air-entrained concrete*. ACI Journ., 1954. 4: p. 760-761.
- 3. Powers, T.C. and T. Willis. *The air requirement of frost resistant concrete*. in *Highway Research Board Proceedings*. 1950.
- Ley, M.T., et al., *Determining the Air-Void Distribution in Fresh Concrete with the Sequential Air Method*. Construction and Building Materials, 2017. 150: p. 723-737.
- Pigeon, M. and R. Pleau, *Durability of concrete in cold climates*. 1995: CRC Press.
- Scherer, G.W. and J. Valenza, *Mechanisms of frost damage*. Materials science of concrete, 2005. 7(60): p. 209-246.
- Tanesi, J., et al., Super Air Meter for Assessing Air-Void System of Fresh Concrete. Advances in Civil Engineering Materials, 2016. 5(2): p. 22-37.
- 8. Hover, K.C., *Analytical investigation of the influence of air bubble size on the determination of the air content of freshly mixed concrete.* Cement, concrete and aggregates, 1988. 10(1): p. 29-34.
- 9. Klein, W. and S. Walker. A method for direct measurement of entrained air in concrete. in Journal Proceedings. 1946.
- 10. LeFlore, J. *Super Air Meter Test Video*. 2016; Available from: https://www.youtube.com/watch?v=xAcHqMz_m3I.
- 11. Ley, M.T. and B. Tabb. A test method to measure the freeze thaw durability of fresh concrete using overpressure. in T&DI Congress 2014: Planes, Trains, and Automobiles. 2014.

- Welchel, D., *Determining the Size and Spacing of Air Bubbles in Fresh Concrete*.
 2014, Oklahoma State University.
- 13. Felice, R., J.M. Freeman, and M.T. Ley, *Durable Concrete with Modern Air-Entraining Admixtures*. Concrete international, 2014. 36(8): p. 37-45.
- 14. Freeman, J.M., *Stability and quality of air void systems in concretes with superplasticizers*. 2012, Oklahoma State University.
- 15. Ley, M.T., *The effects of fly ash on the ability to entrain and stabilize air in concrete*. 2007: ProQuest.
- 16. Jakobsen, U., et al., *Automated air void analysis of hardened concrete—a Round Robin study*. Cement and Concrete Research, 2006. 36(8): p. 1444-1452.
- Peterson, K., L. Sutter, and M. Radlinski, *The practical application of a flatbed scanner for air-void characterization of hardened concrete*, in *Recent Advancement in Concrete Freezing-Thawing (FT) Durability*. 2010, ASTM International.
- 18. 201.2R, A.C. *Guide to Durable Concrete*. 2016. American Concrete Institute.

APPENDICES

The raw data from the mixtures are presented below.

Ain Contont	SAM Number							
All Content	15th Quantile	85th Quantile						
2%	0.37	0.68						
3%	0.27	0.55						
4%	0.19	0.42						
5%	0.13	0.31						
6%	0.10	0.22						
7%	0.08	0.16						
8%	0.08	0.14						

Table A-1 – SAM Quantile Curve Values

Mixture	a.		SAM]	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
	(mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
	76	0.11	0.33		0.22	3.9	3.7	4.0	206	27				
Mixture SI (1) WROS .45	89	0.16	0.10		0.13	5.1	4.6	5.5	178	27				
	114	0.19	0.15		0.17	8.5	8.6	5.6	147	32				
	64	0.19	0.17		0.18	4.1	3.7	4.0	244	22				
	89	0.19	0.26		0.23	3.7	2.9	3.7	211	27				
	76	0.24	0.36		0.30	3.1	2.3	3.7	246	23				
	64	0.53	0.58		0.56	2.2	2.2	2.3	368	19				
	64	0.60	0.56		0.58	2.5	2.3	2.2	325	22				
	44	0.54	0.65		0.59	2.5	2.6	3.4	333	18				
	76	0.61	0.70		0.66	2.0	1.5	2.8	368	18				
	83	0.67	0.76		0.72	2.4	1.5	3.7	262	22				
	76	0.33	0.10	0.13	0.19	4.5	4.2	4.3	203	26				
	76	0.16	0.15		0.15	6.0	5.6	4.3	196	27				
	108	0.09	0.23		0.16	5.2	5.2	4.5	150	35				
	76	0.19	0.19		0.19	5.8	5.8	5.3	193	25				
	89	0.42	0.29	0.22	0.31	3.7	3.1	3.5	229	26				
SVNTH	89	0.28	0.25	0.35	0.29	3.0	2.3	2.2	295	24				
51N111 15	79	0.28	0.26	0.35	0.30	2.7	3.1	2.1	340	21				
.43	67	0.42	0.28		0.35	2.1	2.1	3.5	249	23				
	70	0.31	0.35		0.33	2.8	3.0	2.8	335	19				
	114	0.31			0.31	2.8	3.4	1.7	307	26				
	83	0.34	0.38		0.36	3.6	3.5	2.3	302	23				
	89	0.30	0.36		0.33	3.4	2.1	2.5	234	29				
	83	0.40	0.37		0.38	2.9	3.1	2.5	353	19				

Table A-2 – Oklahor	na State Universit	v Concrete M	lixture Design and	l Testing Data
		2	()	•

	<i>c</i>		SAM]	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666				
Mixture	(mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)		
CANTEL	83	0.47			0.47	2.2	2.4	1.8	467	17					
51N1H 45	76	0.07	0.33		0.20	3.9		4.4	198	27					
.45	89	0.31	0.45		0.38	4.2		4.2	191	28					
	216	0.12			0.12	8.6	8.4	7.0	155	29					
	229	0.17	0.12		0.15	7.9	7.8	8.1	142	28					
	229	0.17	0.10		0.14	6.2	5.8	6.3	188	25					
WROS	229	0.20	0.22		0.21	6.0	5.8	6.7	185	25					
.53	216	0.25	0.25		0.25	5.5	5.4	6.2	198	24					
	229	0.46	0.63		0.54	4.4	3.9	5.5	241	21					
	229	0.43	0.63		0.53	3.6	3.3	4.1	244	23					
	216	0.76	0.70		0.73	2.7	2.7	3.5	320	19					
	38	0.32	0.16		0.24	3.5	3.2	3.5	244	23					
	51	0.19			0.19	5.7	5.8	5.7	191	24					
	44	0.20	0.29	0.22	0.24	4.5	4.2	3.5	188	30					
	38	0.19	0.19		0.19	5.1	4.9	5.1	170	28					
	44	0.15	0.20		0.17	3.8	3.3	3.1	287	21					
WROS	51	0.36	0.21	0.13	0.23	3.6	3.3	4.5	229	22					
.41	38	0.55	0.32		0.44	3.1	2.8	3.0	292	21					
	44	0.60	0.50		0.55	2.7	2.7	2.1	297	24					
	54	0.60	0.40		0.50	2.0	1.8	1.1	417	23					
	44	0.29	0.55	0.54	0.46	2.2	2.3	1.5	361	23					
	29	0.56	0.67		0.61	2.7	2.4	2.9	320	20					
	29	0.63	0.60		0.61	2.5	2.2	2.5	338	20					
WROS	13	0.17	0.13		0.15	4.3	3.4	4.7	226	22					
30	19	0.12	0.19		0.15	6.1	6.0	7.3	127	29					
.53	19	0.19	0.26		0.23	3.7	3.2	4.0	269	20					

	CI.		SAM]	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
Mixture	(mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
	19	0.51	0.50		0.50	2.8	2.9	3.8	292	19				
	25		0.60		0.60	2.7	2.2	4.4	259	20				
WROS	19	0.61	0.54		0.57	2.6	2.5	3.3	264	22				
	19	0.48	0.61		0.55	2.5	2.3	2.9	483	13				
.39	25	0.58	0.70		0.64	2.2	1.7	3.1	264	22				
	19	0.19			0.19	3.3		2.2	381	18				
	25	0.28	0.25	0.29	0.27	4.9		3.8	213	26				
	254	0.11	0.04		0.07	8.0	7.5	8.9	163	20				
	229	0.09	0.14		0.12	10.5	10.1	7.3	155	26				
	241	0.16	0.12		0.14	7.2	6.2	7.3	180	22				
	241	0.14	0.24	0.22	0.20	6.3	7.0	5.4	277	17				
	241	0.31	0.25		0.28	5.5	5.3	5.0	366	14				
WROS	229	0.49	0.31	0.17	0.32	3.1	2.9	3.9	406	14				
+ PC1	235	0.30	0.23		0.27	6.2	5.9	6.8	361	12				
.45	235	0.55	0.38	0.40	0.44	5.3	5.2	8.0	257	14				
	216	0.39	0.37		0.38	2.7	3.1	3.7	338	17				
	241	0.41	0.25	0.42	0.36	5.2	5.0	6.2	302	15				
	248	0.40	0.39		0.39	2.3	2.6	3.0	409	15				
	229	0.44	0.27		0.35	3.8	3.7	4.3	340	16				
	241	0.44	0.39	0.40	0.41	3.8	3.5	4.0	361	15				
	216	0.06	0.12	0.09	0.09	8.5	7.3	6.2	147	31				
SVNTU	235	0.15	0.05		0.10	5.6	5.3	4.4	191	28				
	229	0.14	0.15		0.15	7.1	6.9	5.6	157	30				
	229	0.43	0.39	0.18	0.33	3.5	3.0	2.5	274	25				
.45	210	0.58	0.23	0.20	0.34	5.0	4.7	3.6	292	20				
	229	0.36	0.21	0.11	0.23	4.6	4.2	4.6	277	19				

	<i>a</i>		SAM	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
Mixture	(mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
CVNITH	229	0.51	0.38	0.46	0.45	3.4	3.1	5.1	267	18				
	235	0.37	0.45	0.37	0.40	2.7	2.7	2.4	432	16				
.45	216	0.46	0.54	0.50	0.50	2.9	2.4	3.0	353	18				
.43	216	0.46	0.62	0.50	0.53	3.6	3.5	3.6	297	19				
	178	0.10			0.10	8.0	7.5	7.5	178	22				
	165	0.15	0.45		0.30	6.7	6.3	8.2	130	28				
WROS	191	0.16	0.25		0.21	5.6	5.2	4.2	198	27				
20% Fly	172	0.23	0.18		0.21	6.1	5.4	6.0	175	26				
Ash .45	165	0.27	0.20		0.23	3.4	3.0	3.4	262	23				
	165	0.76	0.77		0.76	2.4	2.0	3.4	284	21				
	140	0.82	0.71		0.77	2.3	1.9	2.7	282	23				
	64	0.36	0.40	0.40	0.39	2.9		3.0	282	22				
	51	0.16	0.18	0.20	0.18	3.7		3.2	257	24				
	64	0.54	0.52		0.53	2.9		2.8	373	17				
	70	0.14	0.16		0.15	4.6		4.4	183	29				
	89	0.36	0.24	0.32	0.31	2.9		5.4	208	23				
	95	0.07	0.12		0.10	5.2		4.3	152	35				
TEMP	76	0.71	0.66	0.35	0.68	2.6		2.6	226	29				
MIXES	70	0.13	0.14	0.16	0.14	4.0		4.7	173	30				
MIALS	64	0.20	0.14	0.19	0.18	3.2		3.7	262	22				
	44	0.19	0.17	0.14	0.17	3.7		5.1	180	27				
	64	0.10	0.05	0.22	0.14	3.3		3.6	216	27				
	83	0.15	0.09	0.14	0.15	5.2		6.1	155	29				
	95	0.15	0.09	0.14	0.13	4.1		5.4	173	28				
	89	0.04	0.25	0.26	0.19	2.7		4.0	221	25				
	89	0.15	0.13	0.10	0.13	5.3		4.8	173	30				
	<i>c</i> ,		SAM	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
------------	---------------	------------	------------	------------	---------	---------------------------	------------------------	-----------------	---------------------------	--	-----------------------------	-------------------------	-----------------------	--
Mixture	Slump (mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
TEMP	140	0.96	0.54	0.53	0.68	2.0		2.6	284	24				
MIXES	114	0.41			0.41	3.3		2.9	201	30				
MIALS	127	0.12	0.25	0.13	0.17	5.0		6.8	145	29				
	203	0.12	0.12	0.19	0.14	5.5	5.0	5.2	191	26	87	-0.20	0.03	
WROS	165	0.23	0.28	0.21	0.24	3.9	3.7	4.5	224	24	96	-0.15	0.02	
$\pm 20\%$	140	0.28	0.34		0.31	3.0	2.6	3.1	236	26	94	-0.18	0.03	
Flv Δsh	152	0.23	0.16	0.16	0.18	4.9	4.3	5.1	198	25	97	-0.23	0.03	
.45	152	0.06	0.12	0.17	0.12	6.6	5.9	6.9	150	29	98	-0.21	-0.14	
	146	0.34	0.39	0.37	0.37	1.3	1.1	2.0	338	22	5	0.25	0.23	
	152	0.10	0.11	0.16	0.12	5.8	5.3	5.0	183	28	96	-0.09	-0.35	
	70	0.56	0.43		0.50	2.0	1.7	3.6	356	16	2	-0.59	0.14	
	64	0.40			0.40	2.6	2.1	2.5	368	18	84	-0.04	0.06	
WROS	70	0.20	0.19		0.20	3.0	2.4	4.5	208	25	42	0.24	0.08	
+ 20%	64	0.21	0.19	0.30	0.23	3.3	2.9	3.5	272	21	94	0.08	0.02	
Fly Ash	76	0.20	0.08		0.14	3.7	3.1	3.4	216	27	97	0.05	0.03	
.40	64	0.10	0.15	0.18	0.14	4.4	4.2	6.0	185	24	95	0.12	0.03	
	70	0.19	0.11		0.15	4.9	4.4	4.2	163	32	94	0.04	0.04	
	76	0.12	0.12		0.12	5.5	5.0	3.4	201	29	92	-0.06	-0.58	
	165	0.35			0.35	2.4	2.1	1.6	820	10	7	0.36	0.18	
	216	0.38	0.39		0.39	4.3	4.2	4.6	356	14	19	-0.38	0.14	
WROS	229	0.34	0.39		0.36	4.7	4.8	4.6	315	16	28	-0.08	0.17	
$\pm PC1$	191	0.49	0.46		0.47	4.9	5.1	5.0	373	13	13	0.14	0.35	
	216	0.24	0.35		0.30	5.7	5.6	5.2	246	19	82	-0.27	0.03	
.40	191	0.17	0.16		0.16	6.7	6.4	8.0	183	19	85	-0.43	0.05	
	216	0.16	0.18		0.17	7.2	6.8	5.8	251	18	67	0.11	0.07	
	216	0.15	0.15		0.15	7.3	6.8	7.3	203	19	86	-0.16	-0.14	

	<i>c</i> r		SAM]	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
Mixture	Slump (mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
WROS														
+ PC1	203	0.11	0.18		0.15	7.5	7.1	8.4	196	17	90	-0.19	0.04	
.40														
	184	0.29	0.25		0.27	2.5	2.4	2.5	488	13	12	0.36	1.66	
	165	0.32	0.29		0.31	3.6	3.4	4.3	399	13	6	0.46	0.21	
	64	0.42	0.43		0.43	2.9	2.0	3.4	295	19	21	0.33	0.47	
WROS	64	0.34	0.30	0.41	0.35	4.0	3.5	4.5	264	19	58	0.30	0.21	
+ PC1	133		0.23		0.23	6.0	5.4	4.5	396	13	87	0.33	0.05	
.35	222	0.14	0.17		0.15	9.2	8.9	8.5	140	22	95	0.10	0.03	
	83	0.20	0.20	0.33	0.24	5.1	4.5	4.9	239	20	75	0.11	0.12	
	114	0.12			0.12	5.5	5.2	4.3	211	24	97	0.03	0.02	
	76	0.13	0.08		0.11	5.3	5.1	6.1	226	19	96	-0.11	0.03	
	51	0.39	0.53		0.46	2.4	2.0	3.6	396	14	25	0.28	0.04	
	76	0.13	0.11		0.12	7.5	6.7	7.0	124	32	100	0.05	0.01	
WROS	64	0.13	0.13		0.13	5.8	5.1	4.4	165	31	98	0.00	0.02	
+ PC2	127	0.49	0.52		0.50	2.4	2.5	2.8	315	20	60	-0.93	0.12	
.40	114	0.32			0.32	3.0	2.7	2.6	348	19	81	-0.41	0.11	
•••	64	0.26	0.22		0.24	3.3	2.9	2.9	320	20	93	-0.04	0.03	
	70	0.07	0.11		0.09	4.7	4.1	5.8	163	28	98	-0.07	0.07	
	76	0.54	0.43		0.48	2.5	1.8	1.9	640	12	30	-0.41	0.25	
WROS	114	0.23	0.19		0.21	3.8	3.3	3.3	239	25	95	-0.26	0.05	
+ PC3	102	0.11	0.07		0.09	5.0	5.1	3.6	193	29	99	-0.24	0.04	
.40	114	0.02	0.15		0.09	5.1	4.5	4.5	175	29	96	-0.22	0.03	
	121	0.07	0.12		0.10	6.8	6.3	5.8	147	31	96	-0.78	0.18	
WROS	203	0.23	0.36		0.29	3.8	3.5	2.1	460	16	85	0.02	0.08	
+ PC4	241	0.03	0.07		0.05	6.8	5.8	5.6	178	26	95	0.07	0.03	
.40	229	0.04	0.08		0.06	5.6	4.4	5.5	170	27	95	0.02	0.04	

		SAM Number				Air from	ASTM C138		ASTM C45	57	ASTM C666			
Mixture	Slump (mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
	83	0.16	0.57		0.36	3.5	2.8	2.8	188	34	94	-0.66	0.03	
WROS	102	0.12	0.23		0.18	5.3	4.8	4.7	137	36	93	-0.93	0.01	
+ WR	64	0.48	0.27		0.38	3.1	2.7	3.3	236	25	87	-0.16	0.09	
.40	76	0.19	0.25		0.22	3.7	3.3	3.9	211	26	96	-0.14	0.03	
	76	0.50	0.74		0.62	2	1.7	1.4	653	13	10	0.37	0.2	
WROS	203	0.12	0.07		0.10	5.6	5.7	5.5	191	24	84	0.02	0.06	
+ PC5	197	0.09	0.08		0.08	8.7	9.3	8.2	155	22	97	-0.25	0.02	
40	121	0.35	0.57		0.46	2.9	2.8	2.7	340	19	41	0.05	0.3	
•••	165	0.15	0.41		0.28	4	4.1	4.0	224	24	71	-0.09	0.16	
	19	0.11	0.11		0.11	5.0		5.6	201	23	100	0.34	-0.06	
	13		0.20		0.20	4.1		3.0	249	25	99	0.02	-0.19	
0.40	19	0.09	0.11		0.10	5.8		9.5	122	24	98	0.04	-0.03	
WROS	13	0.28	0.27		0.28	3.5		3.4	409	14	93	0.03	-0.88	
	13	0.13	0.15		0.13	4.5		4.9	201	24	100	0.03	-0.78	
	13	0.48	0.47		0.48	2.8		3.2	361	17	49			
	216	0.11	0.13		0.12	6.5		8.3	193	17	100	0.02	0.32	
0.40	216	0.40	0.38		0.39	4.9		5.4	262	18	89	0.01	-0.06	
WROS+	216	0.24	0.40		0.32	5.2		6.4	239	18	95	0.05	-0.36	
PC1	216	0.48	0.53		0.50	3.7		4.2	315	17	55	0.07	-0.23	
	229	0.13	0.12		0.13	6.9		9.3	150	20	100	0.03	-0.47	
	32	0.57	0.54		0.56	2.5		4.0	302	18	8			
	25	0.71	0.66		0.68	3.0		4.6	254	20	34			
0.45	13	0.35	0.31		0.33	3.5		3.5	394	14	94	0.04	-1.06	
WROS	25	0.14	0.26		0.20	4.2		5.5	188	24	97	0.03	-0.50	
	38	0.11	0.14		0.13	5.7		5.1	165	29	99	0.03	-0.23	
	38	0.11	0.12		0.11	6.3		4.7	185	26	100	0.00	0.10	

			SAM]	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
Mixture	Slump (mm)	Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
0.45	191	0.41	0.47		0.44	4.0		3.5	343	17	25			
U.45 WROS+	203	0.27	0.33		0.30	5.1		4.9	323	15	84	0.05	-1.20	
PC1	203	0.20	0.22		0.21	6.2		4.5	244	21	89	-0.06	-1.04	
	216	0.10	0.10		0.10	6.8		9.5	170	17	94	0.03	-1.08	
	102	0.16	0.16		0.16	6.4		7.0	137	30	100	0.01	0.13	
0.50	76	0.40	0.47		0.44	2.6		3.3	284	21	11			
U.SU WROS	51	0.51	0.43		0.47	3.5		4.2	229	23	92	0.04	-1.52	
WROS	146	0.09	0.10		0.09	7.7		8.9	109	29	100	-0.03	-0.79	
	76	0.19	0.19		0.19	4.6		6.1	201	22	100	0.05	-0.40	
0.50	203	0.05	0.06		0.06	9.0		7.0	99	21	100	-0.06	-1.13	
U.SU WROS+	203	0.32	0.36		0.34	2.7		3.7	373	15	8			
PC1	203	0.34	0.32		0.33	5.3		6.1	269	17	89	0.05	-2.00	
	203	0.14	0.16		0.15	6.5		7.6	234	16	94	0.03	-1.85	
0.45 WROS+	89	0.16	0.21		0.18	5.7		4.9	196	25	100	-0.11	0.02	
WR		0.28	0.21		0.25	4.6		4.5	191	26	100	-0.14	0.02	
	171	0.40	0.41		0.41	4.1		3.5	295	19	0			
		0.38	0.49		0.44	3.0		3.9	361	15	0			
0.45	178	0.30	0.42		0.36	5.0		6.1	208	21	0			
		0.35	0.57		0.46	4.1		3.8	363	15	0			
	203	0.08	0.06		0.07	10.1		11.9	104	21	100	-1.26	0.01	
SRA		0.14	0.18		0.16	7.1		6.2	178	24	99	-1.29	0.01	
	222	0.05			0.05	9.0		13.7	109	18	98	-1.44	0.02	
		0.10	0.10		0.10	5.7		5.7	191	23	77	-2.16	0.06	
	216	0.10	0.08		0.09	6.8		8.1	185	18	20			

Mixture	Slump (mm)		SAM	Number		Air from	ASTM C138		ASTM C45	57	ASTM C666			
		Meter A	Meter B	Meter C	Average	Super Air Meter (%)	Gravimetric Air (%)	Hard Air (%)	Spacing Factor (µm)	Specific Surface (mm ⁻¹)	Durability Factor (%)	Length Change (%)	Mass Change (%)	
0.45		0.19	0.15		0.16	4.5		6.9	198	20	41			
	191	0.15			0.15	6.3		8.3	104	31	69			
		0.24	0.23		0.23	4.1		4.9	239	20	15			
SRA	229	0.09	0.13		0.11	8.3					93	-1.06	0.04	
JIA		0.09	0.16		0.13	4.9		5.0	191	25	19			

VITA

Hope Hall

Candidate for the Degree of

Master of Science

Thesis: IMPLEMENTATION OF THE SEQUENTIAL AIR METHOD IN

LABORATORY AND FIELD STUDIES

Biographical:

Education:

Completed the requirements for the Master of Science in Civil Engineering at Oklahoma State University, Stillwater, Oklahoma in December, 2017.

Completed the requirements for the Bachelor of Science in Architectural Engineering at Oklahoma State University, Stillwater, Oklahoma in May, 2015.