FROST RESISTANCE OF MODERN AIR

ENTRAINED CONCRETE

MIXTURES

By

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CHAPTER I

INTRODUCTION

Concrete is one of the most widely used construction materials in the world. Concrete can be made into almost any shape and is by nature a durable material. One mechanism that challenges the durability of concrete is the freezing and thawing of water. When water freezes it expands by approximately 9%, which can lead to intense internal stresses that cause the internal structure to deteriorate. Once significant damage has started, each cycle of freezing and thawing greatly compounds the effects of physical damage. However, this physical damage can be mitigated with the use of air entrained concretes in environments that are moist and undergo cyclic freezing and thawing. The first air entrained concretes were observed when lubricating oils used at cement processing plants accidentally made their way into newly made Portland cement. It was later observed that the concretes produced from these plants were more frost resistant.

In the 1950s Paul Klieger found the minimum volume of air required to consistently insure frost durability in a concrete mixture subjected to rapid freezing and thawing cycles. Klieger systematically changed the volume of air in the concrete mixture then, evaluated the freeze-thaw performance of the mixture. Kleiger's work predated a standard specification for freezing and thawing and was done without the aid of any hardened air void analysis. Ultimately Klieger suggested that throughout all of the mixtures investigated, frost durability was provided if 18% air was created in the concrete paste. As a result of Klieger's findings, ACI 318 has adopted these recommendations by assuming a paste volume based on the maximum nominal aggregate size and specifying a recommended volume.

T.C. Powers in 1954 examined air void systems in hardened concrete. His findings led to the first quantitative evaluation parameters for hardened air void systems, spacing factor and specific surface. Based on the findings by Powers, the ACI 201 document "Guide to Concrete Durability" suggests a spacing factor of 0.008 in and a specific surface of 600 in-1 to determine if a concrete is frost susceptible. Powers also hypothesized with the hydraulic pressure theory that the permeability and tensile strength, parameters associated with w/cm ratio, may have an impact on frost durability.

While the observations made by Klieger and Powers have proven to be excellent for the advancement in knowledge of concrete durability, the findings are more than 50 years old. During this time the materials and quality control standards have improved. Increased quality control for cements, aggregates, and durability testing combined with the development of modern air entraining agents and other admixtures have caused researchers to question if the conclusions made in the past are still valid for modern mixtures. One of the goals of this thesis is to evaluate bulk freeze thaw performance (ASTM C 666) and hardened air void systems (ASTM C 457) of concrete mixtures that use modern air entraining agents and a midrange water reducer. Part one of this work will investigate the performance of modern concretes made with state of the art admixtures at various w/cm ratios and will use methodologies that are similar in spirit to Klieger.

Work completed by Freeman in 2012 expanded on the research of modern mixtures by introducing polycarboxylates to the mixture design. Freeman observed that unconsolidated air entrained concretes treated with polycarboxylates lost significant amounts of air content with respect to time. Additionally, Freeman observed that air loss in air entrained polycarboxylate concretes could be significantly reduced by immediately consolidating the concrete after a satisfactory air content was measured. Hardened air void analysis revealed that the addition of recommended doses of polycarboxylate coarsened the air void system, and frost durability was not achieved even when air contents were near the ACI 318 recommended values. This is of concern since there is currently no adequate quality control test that can accurately measure the air void size and distribution in fresh concrete, therefore researchers have reverted to measuring the total volume and then performing hardened air void analysis to determine if a concrete is frost susceptible.

While the measuring of total volume of air is not the ideal method for determining if a concrete will be frost durable, it is currently the only method that can be used to evaluate fresh concrete. Previous research by Pigeon and Pleau in 1995 and Ley in 2007 have shown that as the volume of air increases the average spacing between voids, or the spacing factor, decreases. This decrease in spacing of voids then leads to an improvement in frost durability. The third chapter of this thesis will expand on the findings by Freeman and explore ways to regenerate air content and workability lost in transit by unconsolidated air entrained polycarboxylate concretes. The main goal will be to observe the effect remixing has on the restoration of air, especially to mixtures with polycarboxylates, and determine the impact on the void distribution and frost durability. The current air content requirements set forth by ACI 318 will be investigated to determine if the current specifications are satisfactory for concretes treated with polycarboxylates.

CHAPTER II

MODERN CONCRETE AIR REQUIREMENTS

FOR FROST DURABILITY

Concrete will suffer frost damage when saturated and subjected to freezing temperatures. Frost-durable concrete can be produced if a specialized surfactant, also known as an air-entraining admixture (AEA), is added during mixing to increase the volume of air voids. Small and well-dispersed air voids are critical to produce frost-resistant concrete. The spacing and size distribution of the bubbles are thought to be more important than the volume of air. Air void characterization is currently made in hardened concrete with ASTM C 457, "Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete". The spacing factor and specific surface are the common parameters determined from the ASTM C 457 technique. These parameters were first determined by Powers (1954a, 1954b). The ACI 201.2R-08 document, "Guide to Concrete Durability" (ACI 2008) suggests that a spacing factor of 0.008 in and a specific surface of 600 in-1 be used to determine if a concrete is frost susceptible. The Canadian Standards Association (CSA 2009) has suggested that a spacing factor from a lot can be no higher than 0.010 in as long as the average for the element is below 0.009 in. Currently there is no quality control test that can accurately measure the air void size and distribution in the fresh concrete. In the absence of an adequate test, researchers have reverted to measuring the total volume of air in a concrete mixture. Past research has shown that the average spacing between voids in the paste, or the spacing factor, decreases as the volume of air increases. (Pigeon & Pleau 1995, Ley 2007). This leads to an improvement in frost durability.

Work completed by Klieger (1952, 1956) found the minimum volume of air required to consistently insure frost durability in a concrete mixture subjected to rapid freezing and thawing cycles. These tests were carried out by systematically changing the volume of air in the concrete mixture and then evaluating the freeze thaw performance of the mixture. Kleiger's work was completed without the aid of any hardened air void analysis and ultimately suggested that throughout all of the mixtures investigated that frost durability was provided if 18% air was created in the paste. ACI 318 has adopted these recommendations by assuming a paste volume based on the maximum nominal aggregate size and specifying a recommended volume. Others commonly just specify a total volume of air such as 6% air in the concrete.

However, if one reviews the details of Klieger's past research they will realize that the characteristics of the materials Klieger investigated are not representative of modern concrete mixtures. For example in every mixture in Kleiger's research the only admixture used was a Vinsol resin AEA. At the time of the testing a Vinsol resin was the only AEA admixture widely used in concrete. Since this time several other AEAs have been introduced. Also, in modern mixtures it is common to use combinations of chemical admixtures with water reducers (WRs). Little work has been done to quantify how the interaction between AEAs and WRs impact the frost durability of the mixture (Plante et al. 1989). Furthermore, the test Klieger used to investigate frost durability does not match the modern test method to investigate bulk freeze thaw damage, ASTM C 666 "Resistance of Concrete to Rapid Freezing and Thawing". There were differences in curing, freezing and thawing rate, and failure evaluation.

Despite all of these differences these recommendations are still used. However there have been a number of workers who have suggested that these recommendations may need to change based on the large changes in materials and testing procedures (Gay 1982 & 1985, Jana et al. 2005, Ley 2007). The validity of spacing factor limits of 0.008 in. have also been challenged.

The goal of this work is to evaluate the bulk freeze thaw performance (ASTM C 666) and hardened air void systems (ASTM C 457) of modern concrete mixtures with similar methodologies as used by Klieger. This work used three different AEAs (synthetic, wood rosin, and Vinsol resin), a lignosulfonate WR, and different w/cms to evaluate performance. These findings provide many useful insights into requirements for the frost durability of modern concrete mixtures.

Materials

All of the concrete mixtures described in this paper were prepared using a typical Type I/II cement that meets the requirements of ASTM C 150. The oxide analysis is shown below in Table 2.1. The aggregates used were locally available crushed limestone and sand used in commercial concrete. The maximum nominal aggregate size was ¾ in., and both the rock and sand met ASTM C 33 “Standard Specification of Concrete Aggregates”. All admixtures met ASTM C 260 and C 494 and are described in Table 2.2.

Table 2.1 Cement oxide analysis - Type I/II cement

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	C_3S	C_2S	C_3A	C_4AF
20.1%	4.8%	2.9%	63.0%	2.0%	3.0%	0.3%	0.3%	58.0%	14.1%	7.9%	9.1%

Total Na_2O equivalent alkali content was 0.5%

A wood rosin (WROS), synthetic (SYNTH), and vinsol resin (VR) were investigated in the research. All mixtures prepared with a lignosulfonate water reducer used wood rosin as the AEA. Rapid freezing and thawing tests (ASTM C 666) and hardened air void analyses (ASTM C 457) were used to study the concrete air void systems.

Table 2.2 Admixture reference

Short Hand	Description	Application
WROS	Wood rosin	Air entrainer
SYNTH	Synthetic chemical combination	Air entrainer
VR	Vinsol resin	Air entrainer
WRA-L	Lignosulfonate	3.7oz/cwt Midrange water reducer
WRA-H	Lignosulfonate	10.2oz/cwt Midrange water reducer

Experimental Methods

Mixture Design

Mixture designs with constant cement content and varying w/cms were used for this research. The 0.41 and 0.45 w/cms mixtures were chosen as they bracket the range of typical w/cm used in low slump mixtures without the use of a water reducer. To investigate the effect of a water reducer, mixtures with a w/cm of 0.41 and 0.38 were investigated. A higher dosage of WRA, 10.2 oz/cwt, was used in the 0.38 and 0.41 w/cm mixtures. This dosage will be referred to as WRA-H. A lower dosage of 3.7 oz/cwt was used in the 0.41 w/cm mixture. This dosage will be referred to as WRA-L. Different dosages were used to simulate the different ranges of typical WRA dosages used in the field and the impact of changes in w/cm. All of these dosages were within the manufacturer recommended limits.

The addition of WRA also allowed for lower w/cms to be investigated. Powers hypothesized with hydraulic pressure theory that the permeability and tensile strength of the paste may affect freeze thaw performance (Powers 1949) Table 2.3 shows the mixture design proportions.

Table 2.3 SSD mixture proportions

w/c *ratio*	Paste Content *(%)*	Water *lb/yd* ^{3}	Cement *lb/yd* ^{3}	Coarse *lb/yd* ^{3}	Fine *lb/yd* ^{3}
0.38	26	232	611	1950	1203
0.41	28	250.5	611	1900	1129
0.45	29	275	611	1850	1203

Concrete Mixture Procedure

Aggregates are collected from outside storage piles, and brought into a temperature-controlled room at 73°F for at least 24-hours before mixing. Aggregates were placed in 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 minutes to allow the aggregates to approach the saturated surface dry (SSD) condition and ensure that the aggregates were evenly distributed.

Next, the cement and the remaining water was added and mixed for three minutes. The resulting mixture rested for two minutes while the sides of the mixing drum were scraped.

After the rest period, the mixer was turned on and charged with admixtures. The water-reducing agent was added first (if applicable) and was allowed to incorporate into the mixture for 15-30 seconds then the AEA was added. After the addition of admixtures the concrete was mixed for three minutes.

Sampling and Testing

After mixing the material was tested for slump (ASTM C 143), unit weight (ASTM C 138), and fresh concrete air content (ASTM C 231). Once the fresh properties were determined to be acceptable, samples were prepared for freeze thaw durability testing (ASTM C 666) and hardened air void analysis (ASTM C 457). For each mixture two ASTM C 666 beams and an ASTM C 457 sample was created. Freeze thaw prisms were cured for one day in steel molds while covered with wet burlap and then in saturated limewater for the remainder of the 14 day curing period, as per ASTM C666.

Next the freeze thaw beams were placed inside a temperature controlled water bath and brought to 40°F. Once the prisms were at 40°F the length, mass, and dynamic modulus were measured. The soaked prisms were then investigated in the ASTM C 666 test for 300 cycles. As per ASTM C 666 dynamic modulus, expansion, and mass change were measured every 36 cycles or before. If the durability factor decreased below 80%, dynamic modulus was no longer measured but expansion and mass measurements continued through 300 cycles with two exceptions. The 0.41 + VR and 0.38 + WROS + WRA-H specimens with target concrete air contents near 2.5% cracked down the middle in the short direction and measurement was not possible after 96 and 240 cycles respectively. Based on the trends prior to specimen failure, both the expansion and mass loss would have increased if the specimens would have continued in the test.

ASTM C 666 does not clearly define freeze thaw failure, however some guidance is given in admixture standards ASTM C 260, ASTM C 494, and ASTM C 1017. These standards recommend that the ASTM C 666 durability factor of a mixture with and without an admixture should not differ by more than 20%. If this criterion is used to evaluate the performance of a mixture in the

ASTM C 666 test then the limiting durability factor would be between 70% and 80% (Ley 2007). For this paper a specimen was determined failed if the durability factor decreased below 80% at any point during the testing cycle.

Hardened Air Sample Preparation

The hardened air samples were cut into ¾” thick slices using a self-propelled concrete saw with an 18” diameter continuous rim blade with oil based cutting fluid. The sample was cleaned with water and then dried under a fan. An equal parts mixture of lacquer and acetone was applied to harden the surface and protect the rims of the air voids. An 18 in concrete lapper with magnetically bonded diamond discs of decreasing grit size were used to prepare the samples for testing. The samples were prepared as per ASTM C 457.

After the lapping was complete each sample was inspected under a stereomicroscope to ensure aggregates and paste had been lapped to the same elevation and there was a high quality finish on the specimen. After the specimen had received an acceptable polish, then they were soaked in acetone to remove the lacquer. After soaking in acetone, the prepared sample surface was colored solid with a black permanent marker then dried for 3 hours. A second coat of black marker was then applied in the perpendicular direction to the first coat and the sample dried for 8 hours. A thin layer of barium sulfate, a white powder with a particle size less than 3.94 x 10-5 in (< 1 um), was pressed on the colored surface twice with a rubber stopper to force the white powder into the voids. This technique is described in EN 480-11. This left the surface of the concrete black and the voids stained white. Since the analysis is concerned with the voids in the paste, the voids in the aggregate must be masked. To do this the voids within the aggregate were colored with a fine permanent ink pen under a stereomicroscope. Once completed a final inspection was made of the surface to ensure that voids in the paste are white and all other areas in the sample are black. A sufficiently polished sample and a finished sample can be seen in Figures 2.1 and 2.2. This technique is outlined in detail in Ley (2007) and has been used by several other researchers (Jakobsen et al 2006, Sutter 2002, Carlson 2005, Peterson et al 2007).

Once the voids in the paste had been preferentially marked it is possible to use this contrast to determine the air void parameters of the mixture. The research team used the Rapid Air 457 from Concrete Experts, Inc. This machine completes an automated linear traverse analysis on the sample by using a CCD camera to image the surface and an automated stage for precise movement. Image analysis is then used to discern voids (white) from other portions of the sample (dark). A single threshold value of 145 was used for all of the samples that has been shown to be satisfactory with the sample preparation materials and processes used (Ley 2007). This technique requires that the volume of paste be given. This was determined from the batch weights for each concrete mixture design. For the results of the hardened air void analysis reported in this paper chords smaller than 30 µm were not included in the analysis as they are not easily detected by a human during an ASTM C 457 analysis. By excluding these chords the air void parameters determined by the hardened air void analysis are better comparable to previously reported values of ASTM C 457 results. This has been done previously by many researchers (Jakobsen et al 2006, Ley 2007, Peterson et al 2009, Ramezanianpour & Hooton 2010).

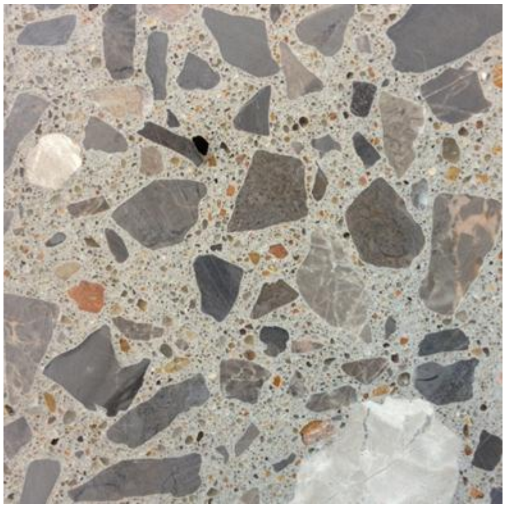

Figure 2.1 Satisfactorily lapped sample

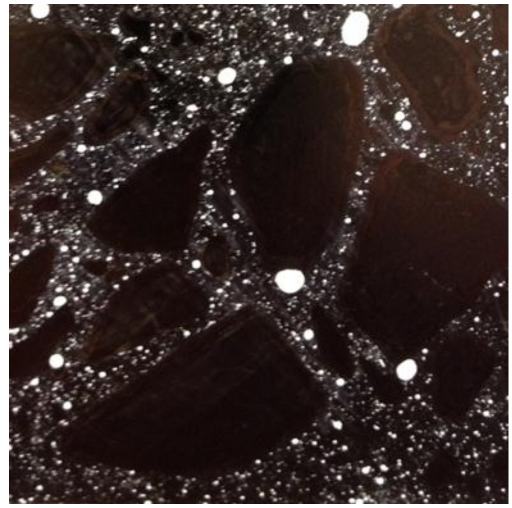

Figure 2.2 Finished sample

Results

The results have been separated in to two different groups. Table 2.4 shows the mixtures made with three types of AEAs at different w/cm ratios. Table 2.5 shows mixtures made with wood rosin AEA at different w/cms and a lignosulfonate midrange water reducer. The paste air contents were determined by using the measured air contents and the concrete batch weights. Tables 2.4 and 2.5 show C 231 and C 457 concrete air contents. In mixtures without midrange water reducer the average absolute difference in C 231 and C 457 concrete air contents is shown to be 0.47% with a standard deviation of 0.40%. In mixtures with lignosulfonate midrange water reducer the average absolute difference was 0.57% with a standard deviation of 0.35%. The C 231 concrete air content was used at the time of mixing to determine if freeze thaw beams and hardened air specimens should be made. Due to some variability in the C 231 and C 457 concrete air contents, it was decided to use the C 231 concrete air contents when preparing plots. Plots are presented to show the impact of different w/cms, AEAs, and the effect of using a midrange water reducer with wood rosin on the concrete air void systems and the performance in ASTM C 666 testing. All figures shown in this paper have closed data points for mixtures that completed 300 cycles of freezing and thawing with an average durability factor of 80% or more and open data points for those that did not.

Figures 2.3 and 2.4 show percent expansion and percent mass change for different air contents. The data point symbols indicate the w/cm with a square being 0.41, and the triangle being 0.38. A vertical line was added at 3.5% concrete air content to highlight a break in the data in frost durability. This will be discussed later in the document. Open data points indicate unsatisfactory freeze thaw performance.

Spacing factors were determined for all mixtures and can be found in Figures 2.5 and 2.6 relative to C 231 concrete air contents and calculated paste air contents. CSA recommends a limit of 0.010 in as an individual spacing factor for any given lot of concrete and is represented by a short dashed line. The ACI 201 limit on spacing factor is shown as a long dashed line at 0.008 in. The data symbols are unique to the w/cms (i.e. a diamond is for 0.45 w/cm). Open data symbols represent unsatisfactory freeze thaw performance. Lines connect the spacing factors measured at the different fresh air contents observed. A vertical line was drawn at 3.5% concrete air content and 11% paste air content to highlight a break in the data.

Specific surface values were measured for all mixtures and can be found in Figures 2.7 and 2.8 relative to C 231 concrete air contents and calculated paste air contents. ACI 201 recommends specific surface to be greater than or equal to 600 in2/in3 and is shown as a long dashed line. The data symbols are unique to the w/cms (i.e. a diamond is for 0.45 w/cm). Open data symbols represent unsatisfactory freeze thaw performance. Straight solid lines connect specific surface values measured at the different fresh air contents observed. A vertical line was drawn at 3.5% concrete air content and 11.0% paste air content to highlight a break in the data.

Table 2.4 Mixtures with different AEAs and w/cm.

Mixture	Slump C 143 (in)	Fresh Air C 231 (%)	Calculated Fresh Paste Air (%)	Concrete Air C 457 (%)	Calculated Hardened Paste Air (%)	Specific Surface (in^2/in^3)	Spacing Factor (in)	Durability Factor *
0.45 + WROS	2.5	2.1%	6.6%	2.2%	6.9%	451	0.0155	(85)
	2.25	3.1%	9.7%	3.0%	9.4%	628	0.0097	94 ± 1
	2.5	4.0%	12.6%	4.4%	13.8%	720	0.0072	82 ± 1
	2	4.3%	13.5%	3.3%	10.4%	809	0.0073	87 ± 0
0.45 + SYNTH	1	2.5%	7.8%	2.6%	8.2%	428	0.0153	(119)
	2	3.5%	11.0%	3.5%	11.0%	497	0.0116	100 ± 0
	2.25	4.2%	13.2%	4.3%	13.5%	653	0.0080	88 ± 6
0.45 + VR	2.75	2.5%	7.8%	3.0%	9.4%	574	0.0106	(300)
	3.75	3.8%	11.9%	4.8%	15.1%	605	0.0082	98 ± 1
0.41 + WROS	0.25	2.5%	7.7%	1.9%	5.9%	587	0.0125	(227)
	1	3.6%	11.1%	2.7%	8.3%	663	0.0096	100 ± 0
	1	4.5%	13.9%	5.5%	17.0%	771	0.0060	99 ± 1
0.41 + SYNTH	0.25	2.5%	7.7%	3.2%	9.9%	507	0.0116	(118)
	0.75	3.4%	10.5%	4.0%	12.3%	547	0.0097	98 ± 1
	0.50	4.3%	13.3%	3.2%	9.9%	617	0.0096	97 ± 1
0.41 + VR	1.25	2.4%	7.4%	2.6%	8.0%	464	0.0139	(68)
	1.0	3.5%	10.8%	3.4%	10.5%	551	0.0103	93 ± 1
	1.0	4.4%	13.6%	4.4%	13.6%	614	0.0083	99 ± 1

* Number in parentheses indicate freezing and thawing cycles completed when dynamic modulus was measured below 80.
Additionally a ± symbol gives the range of values seen by multiple beams of the same mixture

Table 2.5 Mixtures with a lignosulfonate (midrange) water reducer

Mixture	Midrange WRA (oz/cwt)	Slump C 143 (in)	Fresh Air C 231 (%)	Calculated Fresh Paste Air (%)	Concrete Air C 457 (%)	Calculated Hardened Paste Air (%)	Specific Surface (in^2/in^3)	Spacing Factor (in)	Durability Factor *
0.41 + WROS	-	0.25	2.5%	7.7%	1.9%	5.9%	587	0.0125	(227)
	-	1	3.6%	11.1%	2.7%	8.3%	663	0.0096	100 ± 0
	-	1	4.5%	13.9%	5.5%	17.0%	771	0.0060	99 ± 1
0.41 + WROS + WRA-L	3.7	2.5	2.6%	8.0%	1.9%	5.9%	646	0.0114	86 ± 4
	3.7	2.25	3.6%	11.1%	3.3%	10.2%	596	0.0097	83 ± 8
	3.7	2.5	4.5%	13.9%	3.4%	10.5%	659	0.0086	98 ± 2
0.41 + WROS + WRA-H	10.2	2.25	3.5%	10.8%	3.4%	10.5%	694	0.0082	(242)
	10.2	2.5	4.5%	13.9%	4.4%	13.6%	648	0.0079	98 ± 1
0.38 + WROS + WRA-H	10.2	0.75	2.4%	8.1%	2.3%	7.8%	418	0.0161	(120)
	10.2	1	3.3%	11.1%	2.6%	8.8%	745	0.0085	(300)
	10.2	1	4.5%	15.2%	3.9%	13.2%	704	0.0075	98 ± 1

* Number in parentheses indicate freezing and thawing cycles completed when dynamic modulus was measured below 80.
Additionally a ± symbol gives the range of values seen by multiple beams of the same mixture

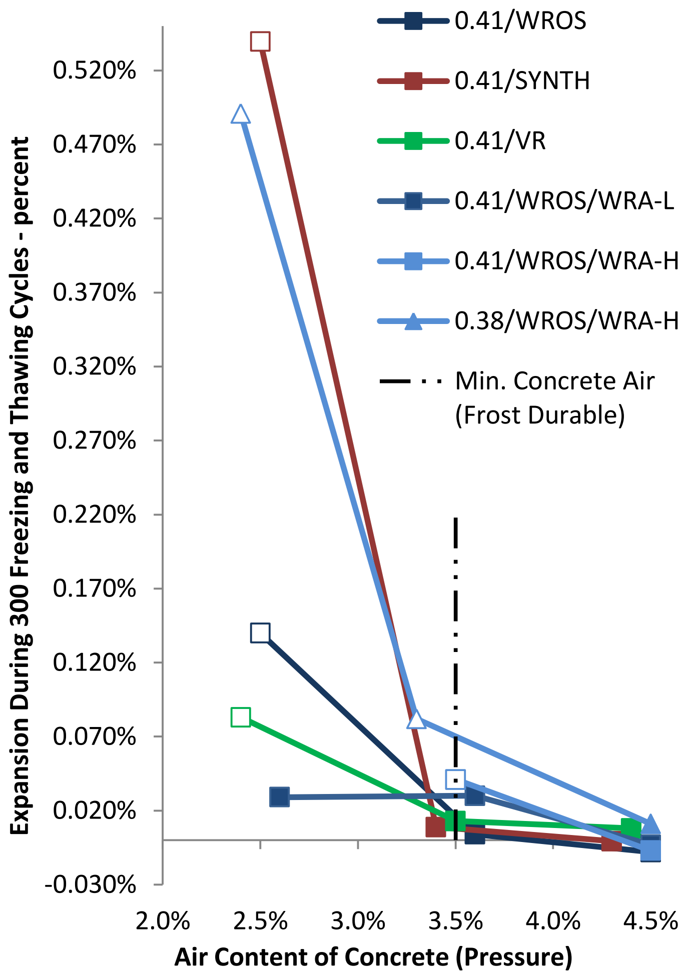

Figure 2.3 Measured percent expansions

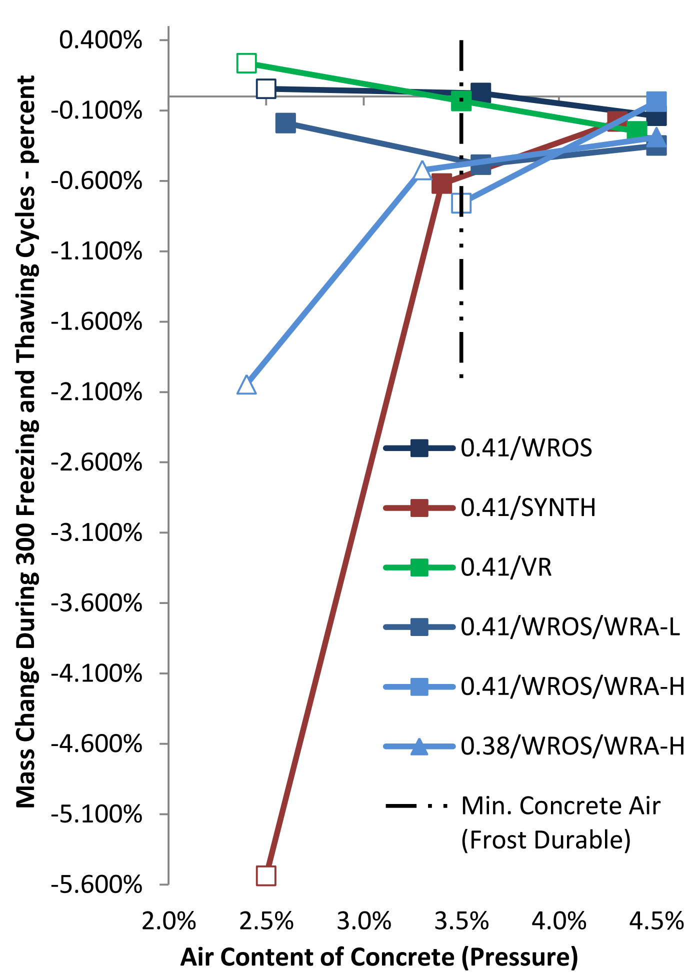

Figure 2.4 Measured percent mass change

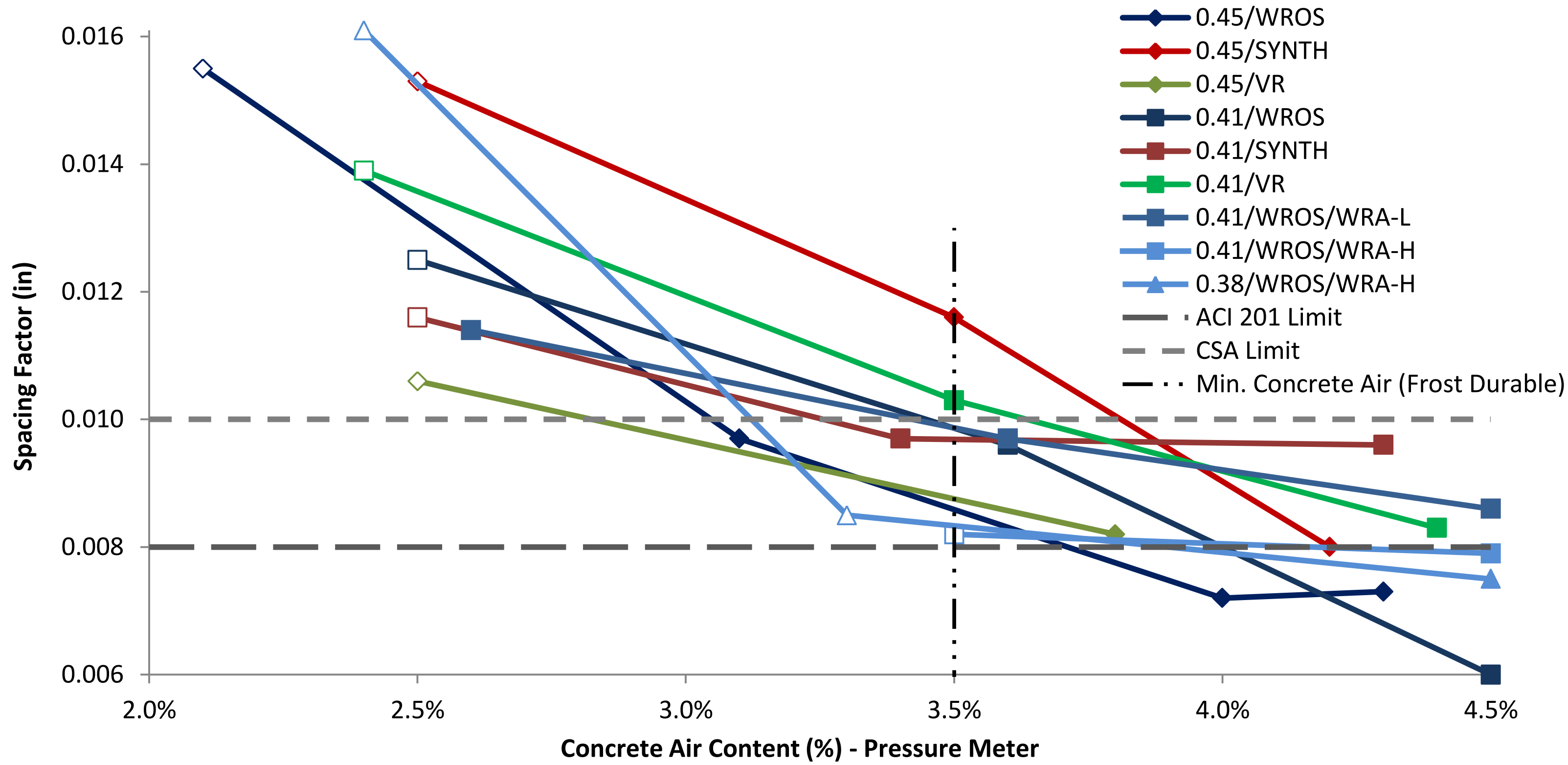

Figure 2.5 Concrete air contents measured by pressure meter and spacing factor for all mixtures

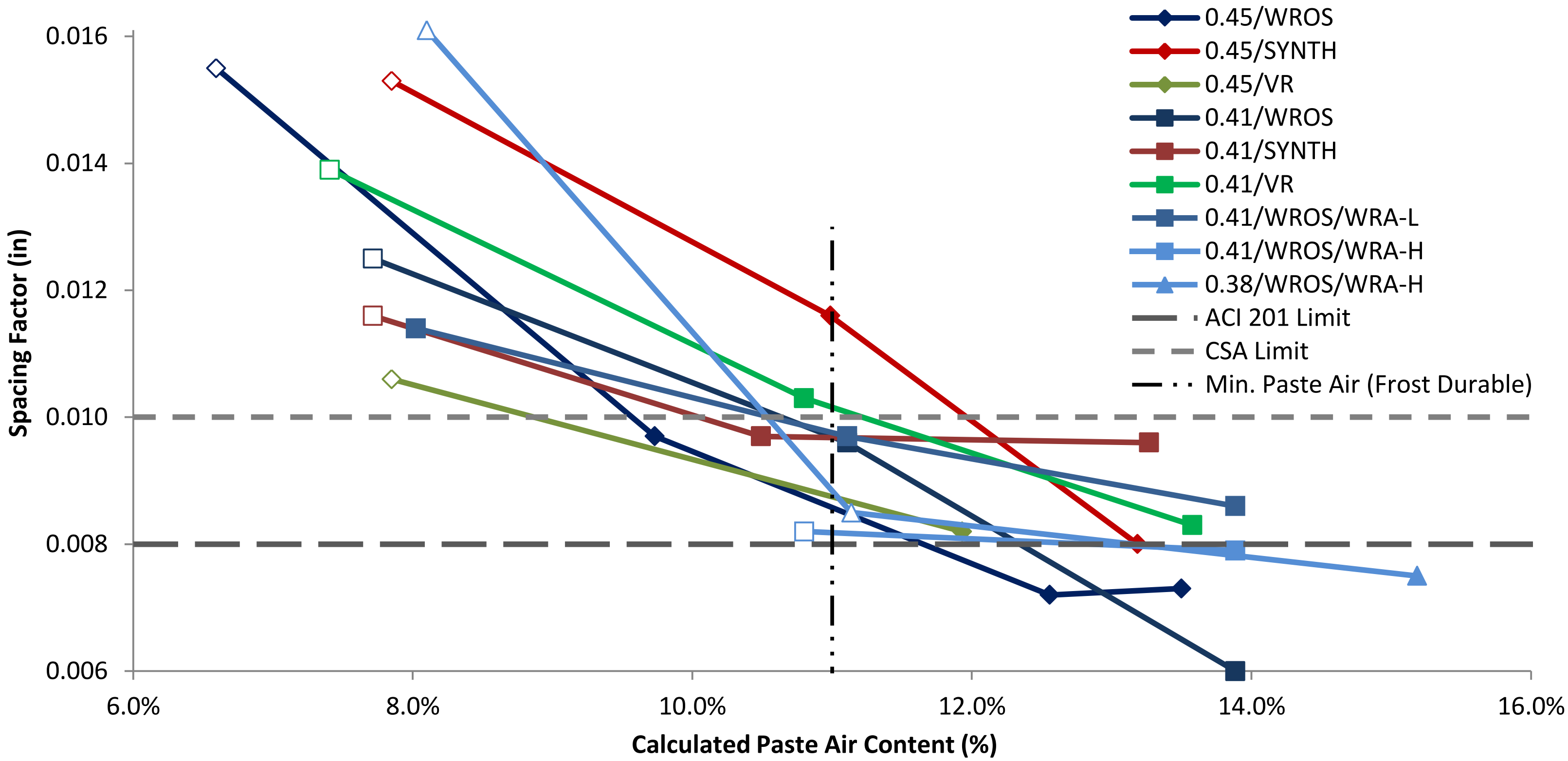

Figure 2.6 Concrete paste air contents calculated from C231 pressure meter readings and spacing factor for all mixtures

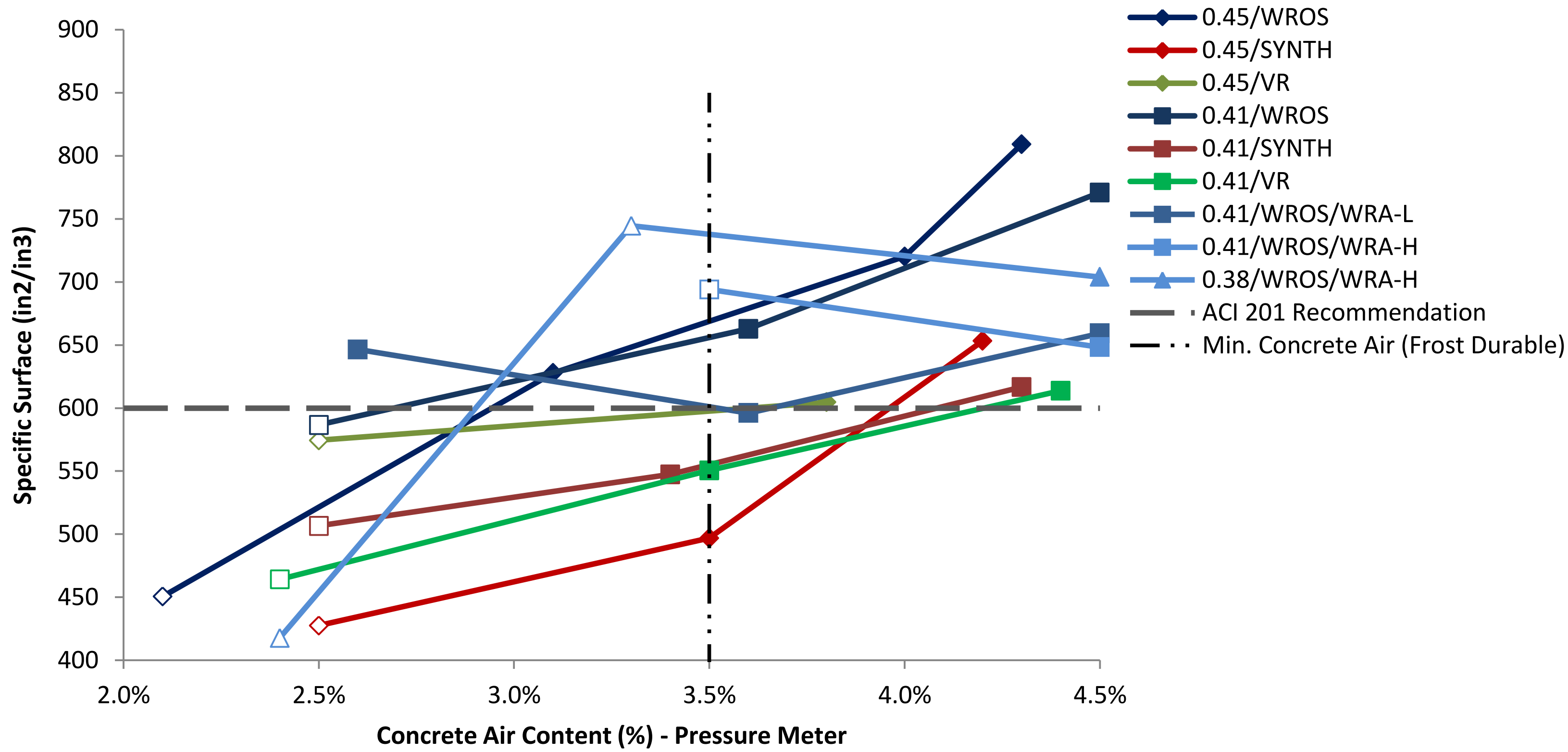

Figure 2.7 Concrete air contents measured by pressure meter and specific surface for all mixtures

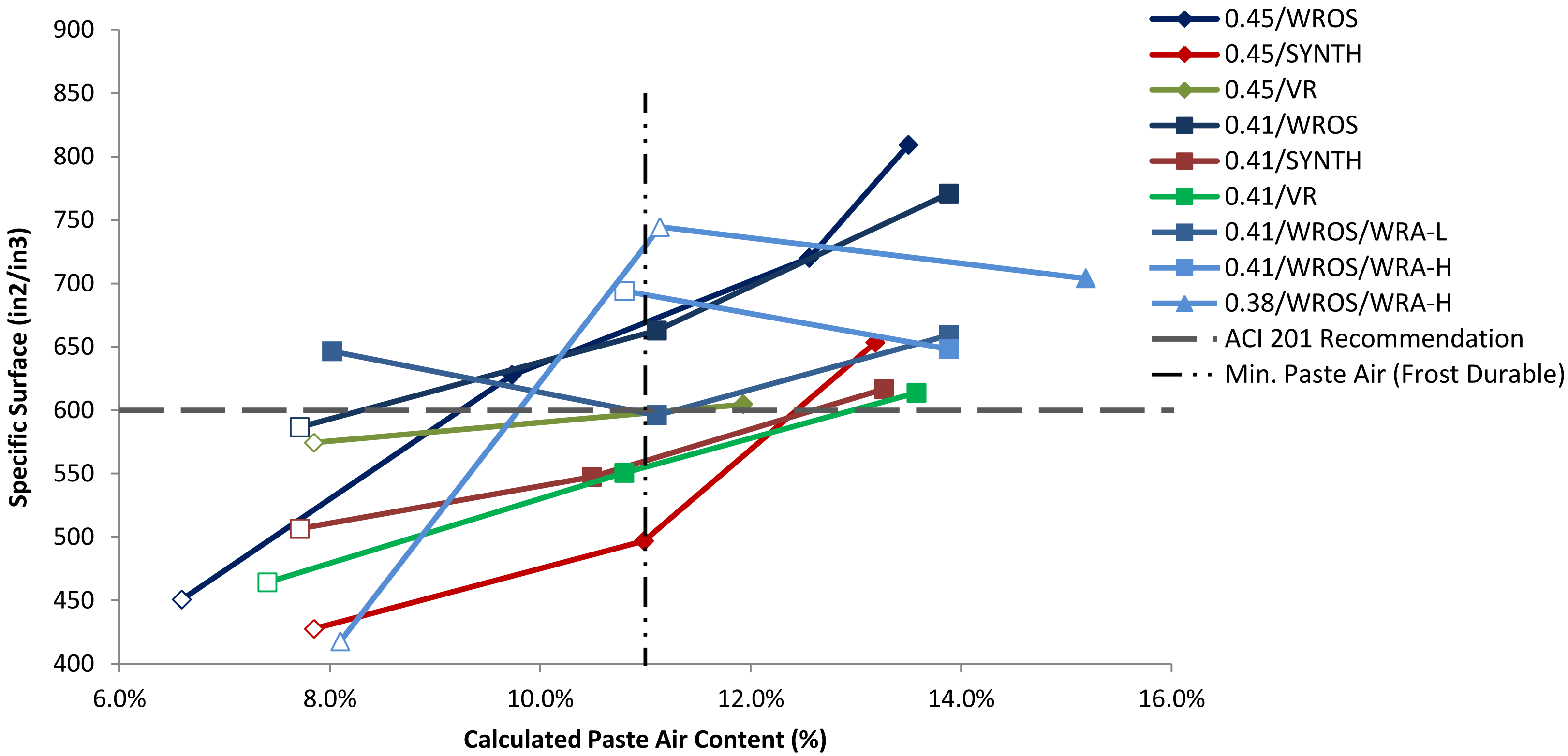

Figure 2.8 Concrete paste air contents calculated from C231 pressure meter readings and specific surface for all mixtures

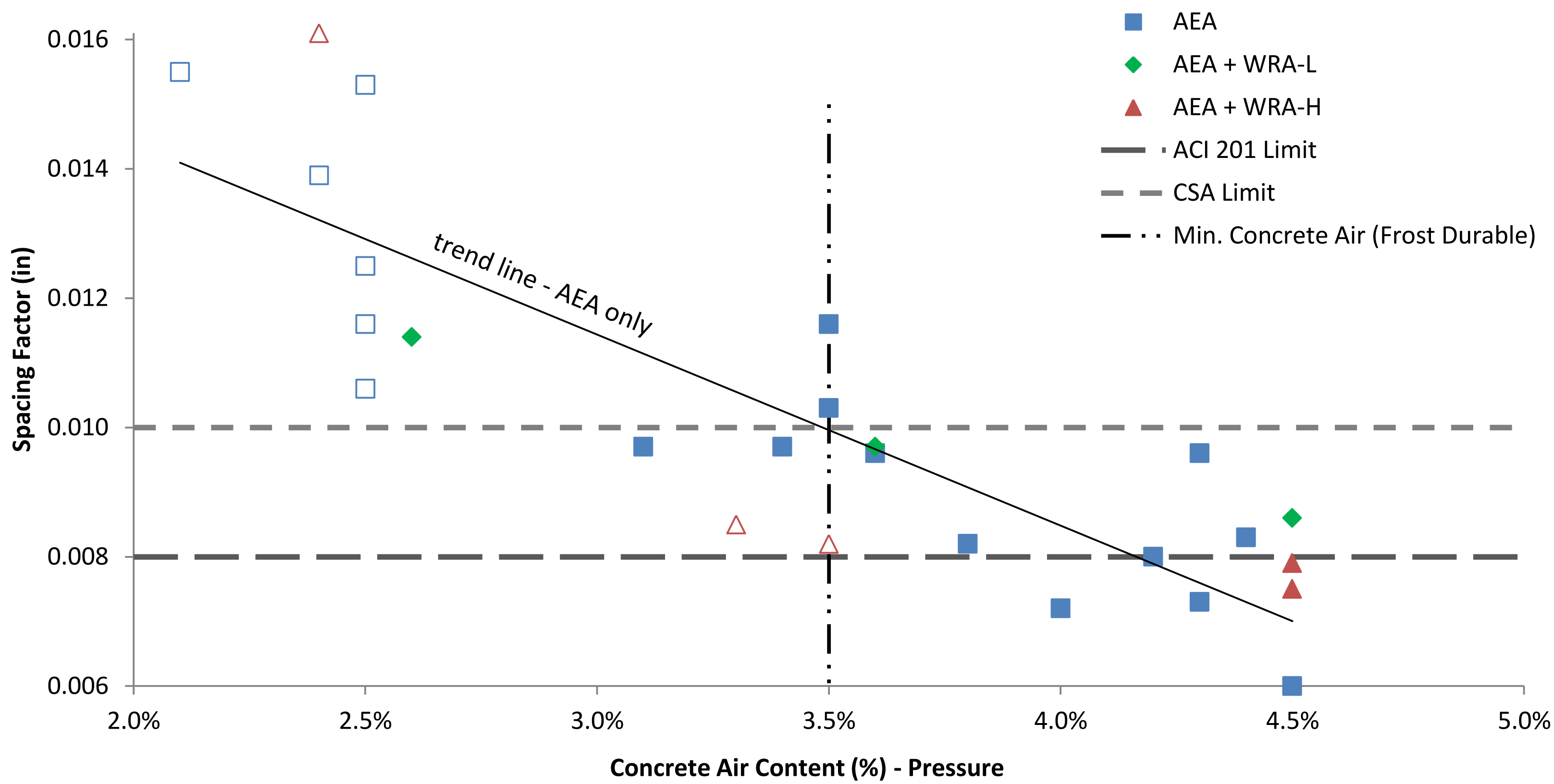

Figure 2.9 Spacing factors versus C 231 concrete air contents for mixtures with and without water reducer

Mixtures made with and without water reducer are shown in Figure 2.9. The square, diamond, and triangle symbols represent the mixtures made as part of this study. Open data points represent unsatisfactory freeze thaw performance. The CSA recommendation of 0.010in as an individual spacing factor for any given lot of concrete and is represented by a short dashed line and the ACI 201 limit on spacing factor is shown as a long dashed line at 0.008 in. A vertical line was drawn at 3.5% concrete air content to highlight a break in the data. A trend line is shown, for mixtures that contain only AEA.

Discussion

Required Air Content for Frost Durable Concrete

Figures 2.3 through 2.8 shows satisfactory performance in ASTM C 666 was achieved when air contents were near or above 3.5% in the concrete or 11% air in the paste and spacing factors were below 0.010 in for mixtures without lignosulfonate WR. A linear trend line drawn for AEA mixtures without WR highlights this finding. This observation was true regardless of the AEA used in the mixture. For mixtures that used lignosulfonate WR at 3.7 oz/cwt and wood rosin AEA this same air content seems to be satisfactory. However, for mixtures that contain 10.2 oz/cwt of lignosulfonate WR and wood rosin AEA, 1% more air was needed in the concrete or 3% more in the paste for satisfactory performance in ASTM C 666.

Impact of Admixtures on Spacing Factor

Based on work by Gay (1982 & 1985) and Jana et al (2005) it was expected that synthetic AEAs would provide a smaller bubble distribution and therefore lower spacing factor and higher specific surface than the other AEAs for a given volume of air. If this was true then Figures 2.5 and 2.6 would show that the synthetic AEA would contain a lower spacing factor and Figures 2.7

and 2.8 a higher specific surface for the same volume of air. This was not observed with the mixtures and materials used in this research. While there may be some differences in the quality of air void system at a given air volume, the experiments found that regardless of AEA type that 3.5% air volume or 11% air in the paste provided satisfactory frost durability as evaluated by ASTM C 666 testing.

Spacing Factor Limits

As shown in Figure 2.9 all mixtures containing only an AEA or lignosulfonate with 3.7 oz/cwt and wood rosin AEA were found to be frost durable when the spacing factor was at or below 0.010 in. This matches the suggested values for the CSA limits. However mixtures that contained 10.2 oz/cwt of lignosulfonate and a wood rosin AEA required a spacing factor of 0.008 in for frost durability. This matches the suggestions of ACI 201. Based on the limited data, it appears the CSA recommendations of using a spacing factor below 0.010 in was not conservative for the mixtures expected to pass the ASTM C 666 test that contain higher dosages of lignosulfonate.

This is clear from Figure 2.9 by comparing the samples with a 3.5% volume of air. The mixtures with 10.2 oz/cwt of lignosulfonate (triangles shown in Figure 2.9) have similar air volumes, improved spacing factors, but different frost durability than the other mixtures investigated. This suggests that other important parameters besides volume of air and spacing factor are critical to frost durability performance for these mixtures. One of these possible differences may be changes in the hydration shell immediately around the surface of the air void in concrete containing AEAs. The porosity of this shell has been speculated as being important to frost durability by Scherer and Valenza (2005). This shell has been observed to change based on the mixture ingredients by others (Rashad & Williamson 1991a and 1991b, Ley et al. 2009a, Ley et al. 2009 b).

Varying w/cms and Frost Durability

For the mixtures and methods investigated it was found that there was no difference in the minimum air content required for satisfactory performance in ASTM C 666 or a significant impact on the spacing factors for mixtures with a w/cm of 0.45 or 0.41. Since w/cm has been shown to impact both the tensile strength and porosity of concrete it would be expected that as w/cm decreases an air void system of lower quality may be acceptable for frost durability. This phenomenon may be observable if more mixtures with air contents between 2.5% and 3.5% are investigated or perhaps lower w/cms are needed.

Practical Implications

Current measuring techniques do not allow for the size or spacing of the air voids to be measured, instead it is common to specify the total volume of air in the concrete. Current recommendations for air content as outlined in ACI 318 are based on work done by Klieger (1952 and 1956) with assumptions for paste contents. As discussed previously the mixtures investigated by Klieger are quite different than modern mixtures. The most notable difference is that only a Vinsol resin AEA was used with no other admixtures. Work in this paper suggests that for the three AEAs investigated (synthetic, wood rosin, and Vinsol resin) all showed satisfactory performance in ASTM C 666 at the same minimum air contents (3.5% by volume in the concrete or 11% in the paste). This supports the use of a single air volume specification for modern AEAs.

However these recommendations do not hold for mixtures that contain high dosages of lignosulfonates. For the mixtures and materials investigated it is recommended that a minimum air content of 4.5% is required in the concrete or 14% in the paste to produce concrete that should adequately perform in ASTM C 666. For use in a specification a safety factor should be used to account for air lost in transit, placement, finishing and material variability. With the current recommendations in ACI 318 for ¾ in maximum nominal size aggregate and a 1% air content reduction for strengths above 5,000 psi this would provide an 11% overdesign or a safety factor of 1.11. While these findings were satisfactory for the mixtures and materials investigated they have been found to be too liberal for other combinations of AEA and admixtures or different mixing procedures. Publications are in preparation. This highlights the need to more clearly define the interaction of admixtures and their impact on frost durability.

For the mixtures investigated a spacing factor of 0.008 in was necessary and is suggested to be required for a mixture to obtain frost durability. This finding matches suggestions in ACI 201 and is more rigorous than the CSA guidelines. While void volume is currently easier to measure in fresh concrete, the spacing factor measurement was able to predict frost durability. Even though mixtures without lignosulfonate were shown to be frost durable with spacing factors up to 0.010 in, it is challenging to monitor what admixtures will be used in a concrete mixture. Because of this it is recommended to require a spacing factor of 0.008 in if the concrete would be expected to pass the ASTM C 666 test.

It is widely accepted that the environments and freezing rates of the ASTM C 666 test are more aggressive then field exposure of concrete (Pigeon and Pleau 1995). However the ASTM C 666 test is the most widely specified test method to evaluate the bulk frost durability of a concrete mixture. Satisfactory performance in ASTM C 666 should lead to satisfactory performance in almost all field applications.

Conclusions

Concrete mixtures were prepared with different modern AEAs with and without lignosulfonate WRs at different air contents. Hardened air void analysis and freezing and thawing tests as per

ASTM C 666 were used to investigate their performance. Based on the data presented the following have been found:

- A minimum air content of 3.5% in the concrete and 11.0% in the paste should yield concrete durable in the ASTM C 666 with modern AEAs and low (3.7oz/cwt) or no lignosulfonate WRs. This minimum air content was the same for a synthetic, wood rosin, and Vinsol resin AEA.

- Limited data suggests that mixtures with a higher dosage of lignosulfonate will need about 1% more air in the concrete or 3% more air in the paste for the materials and procedures used.

- Despite similar air void volume and better spacing factors there were differences in performance in ASTM C 666 for mixtures with a high dosage (10.2 oz/cwt) of lignosulfonate and those without. This suggests that there are other critical parameters besides air void volume and spacing that govern performance in ASTM C 666.

- A spacing factor of 0.008 in was found to be necessary to provide frost durability for the mixtures investigated.

- There was no noticeable difference in performance in ASTM C 666 or changes in the quality of the air void system as measured by ASTM C 457 for mixtures with a w/cm of 0.45 or 0.41 with the AEAs investigated.

While the methods and materials were limited several useful and very practical observations were made that address the volume and spacing factor required for modern AEAs. Furthermore, this work provides great insight for several unknowns in the literature. Findings also highlight a need for greater understanding of the interactions between AEAs and other admixtures on performance in freezing and thawing environments.

CHAPTER III

REGENERATION OF AIR CONTENT IN MODERN CONCRETE MIXTURES BY REMIXING

Concrete subjected to moisture and subsequent freezing and thawing cycles is susceptible to frost damage. The primary way to create frost durable concrete is to stabilize air voids in the fresh concrete during mixing with specialized surfactants called air entraining agents (AEAs). Since the total air content and other air void characteristics depend on the stage in the mixing, transport, placement, and consolidation processes at which measurements are taken (Hover 1994), it is very challenging to consistently create and stabilize an adequate air void system.

Whiting and Stark (1983), Whiting and Dziedzic (1989), Plante et al (1989), Pigeon et al (1990), and Whiting and Stark (1992) observed that interactions of other admixtures such as lignosulfonate and naphthalene water reducers and superplasticizers have been observed to cause the distribution, volume, and stability of air content to change in concrete mixtures.

The interactions of AEAs with polycarboxylate superplasticizers (PCs) are especially concerning as these chemicals have become essential tools for the concrete industry to improve the sustainability, durability, strength, and constructability of modern concrete mixtures. As documented in patents by Zhang et al (U.S. Patent No. 6,858,661 B2), Shendy et al (U.S. Patent

No. 8,088,842 B2), and Kuo (U.S. Patent No. 8,187,376) it is common for PCs to contain defoaming chemicals in combination with the surface active agents These defoaming agents are used to reduce the volume of air created by the surface active agents within a PC. This combination of admixtures that are added to simultaneously create, and destroy bubbles is significantly different than a concrete mixture where AEAs are solely used. There has been little work done to investigate these interactions of AEAs and PCs.

Work completed by Freeman (2012) investigated combinations of five superplasticizers and three modern air entraining agents. Freeman found that air and slump loss occurred simultaneously over time in unconsolidated concrete mixtures. These losses were increased for mixtures treated with PCs, but once the concrete was consolidated the volume of air in the concrete changed little with respect to time. A coarsening of the air void system was also observed and showed that higher volumes of air in the concrete (approximately 8%) would be required for frost durability.

Remixing with water is a common field practice used to increase the workability of a concrete mixture. It is preferred that if the addition of water is anticipated, that a set amount of remixing water be held back to be later added on site. The procedure aims to restore some workability without modifying the mixture design, however it is difficult to measure the amount of water added on site. This tactic is typically focused on improving workability. Some work has been done, most of which predates the use of PCs, to observe the impacts adding water has on air void systems. Langan and Ward (1976) performed laboratory studies to observe the effects of adding water or water plus AEAs and found that retempering with water or water plus AEA increased air contents and reduced spacing factors. Burg (1983) sampled and tested freshly mixed concrete from fifteen trucks, he observed a decrease in air content and slump from the “as-mixed” value when tested at the jobsite before retempering and found by adding approximately 10 lbs/cy of water the air content could be increased by an average of 0.56 percent. Pigeon et al. (1990) investigated the effects of retempering field and laboratory concrete mixtures with water or water and AEA. The concrete was retempered 45 minutes after initial mixing with an amount of water that increased the w/cm from 0.45 to 0.48. Test results showed that the added water increased the air contents slightly but had no significant effect on the spacing factor or the specific surface.

The goal of this study is to expand on the findings of Freeman (2012) by showing various remixing strategies and the influence each strategy has on bulk frost durability (ASTM C 666) and air void systems (ASTM C 457) of mixtures with PCs. The intent of this research is to determine what can be done after air loss has occurred to regenerate it to levels needed for frost durability; therefore the majority of the focus will be on samples taken after air has been lost in a mixture and then restored through mixing or retempering. This work will use a carboxylated polyether superplasticizer (PC1) and a wood rosin AEA. These findings will provide useful insights to strategies that may be applied to field concretes that are expected to be frost susceptible and experience air loss in transit or on site before consolidation.

Materials

The concrete mixtures described in this paper were prepared using a Type I/II cement that meets the requirements of ASTM C 150 “Standard Specification for Portland Cement”. The oxide analysis is shown in Table 3.1. The aggregates used were locally available crushed limestone and sand used commercially in concrete. The maximum nominal aggregate size was ¾ in, and both the rock and sand met ASTM C 33 “Standard Specification of Concrete Aggregates”. All admixtures met ASTM C 260 “Air-Entraining Admixtures for Concrete” and ASTM C 494 “Chemical Admixtures for Concrete” and are described in Table 3.2.

Table 3.1 Cement oxide analysis - Type I/II cement

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	C_3S	C_2S	C_3A	C_4AF
20.1%	4.8%	2.9%	63.0%	2.0%	3.0%	0.3%	0.3%	58.0%	14.1%	7.9%	9.1%

Total Na_2O equivalent alkali content was 0.5%

A wood rosin (WROS) was used as the AEA for all mixtures. In mixtures where the effects of superplasticizer were of interest a carboxylated polyether (PC1) was used. This work builds on previous work done by Freeman (2012) and therefore uses similar procedures, materials, and notation.

Table 3.2 Admixture reference

Short Hand	Description	Application
WROS	Wood rosin	Air entrainer
PC1	Carboxylated polyether	Superplasticizer

Experimental Methods

Mixture Design

A single mixture design was used in this testing. The materials and mixture matches those used by Freeman (2012). Table 3.3 shows the mixture proportions used before remixing with water or admixtures to increase the workability of the mixture. A w/cm of 0.45 was chosen as it is typical of modern low slump concrete mixtures. The PC1 was used in these mixtures to increase the slumps to eight or nine inches. Dosages of admixtures were used within the manufacturer recommended limits. When water was used to reconstitute the mixture the w/cm was increased to 0.47.

Table 3.3 SSD Mixture Proportions

w/cm ratio	Paste Content (%)	Water lb/yd^3	Cement lb/yd^3	Coarse lb/yd^3	Fine lb/yd^3
0.45	29	275	611	1850	1203

Concrete Mixture Procedure

Aggregates are collected from outside storage piles, and brought into a temperature-controlled room at 73°F (23°C) for at least 24-hours before mixing. Aggregates were placed in mixer and spun and a representative sample was taken for a moisture correction. At the time of mixing all aggregates were loaded into the mixer along with approximately two-thirds of the mixing water. This combination was mixed for three minutes to allow the aggregates to approach the saturated surface dry (SSD) condition and ensure that the aggregates were evenly distributed.

Next, the cement and the remaining water was added and mixed for three minutes. The resulting mixture rested for two minutes while the sides of the mixing drum were scraped.

After the rest period, the mixer was turned on and charged with admixtures. PC1 was added first (if applicable) and was allowed to incorporate into the mixture for 15-20 seconds then the AEA was added. After the addition of admixtures the concrete was mixed for three minutes.

Sampling and Testing

Sampling and testing for slump (ASTM C 143), unit weight (ASTM C 138), fresh air content (ASTM C 231), freeze thaw specimens (ASTM C 666), and hardened air specimens (ASTM C 457) were taken over 120 minutes. In addition to the typical pressure meter (ASTM C 231), a modified pressure meter sample was prepared. The modified pressure meter was prepared and measured the same way as a typical pressure meter. The only variation in testing was that immediately after mixing, the modified pressure meter sample was consolidated and then tested after sitting statically for 120 minutes. It is called the modified pressure meter because the aluminum bucket was lined with a four-gallon plastic trash bag to prevent the concrete from reacting with the aluminum. This test procedure has been used to previously show that the air content in the sample will be stabilized when the concrete is consolidated (Freeman 2012). More measurements were taken in this paper to further investigate this phenomenon. All times were measured after the initial mixing was completed. Concrete not used for testing and sampling remained in the mixing drum. Concrete used for slump and unit weight testing was returned to one side of the mixing drum to minimize the disturbance of the un-sampled concrete. Undisturbed concrete was used for each test. Freeman (2012) showed that the disturbance caused by testing, sampling, and returning slump and unit weight concrete had little effect on the subsequent measurements. After allowing the mixture to sit for 50 min, the mixture was "remixed" or reconstituted to restore the slump of the concrete. This consisted of repowering the concrete mixer for three minutes and if applicable, retempering with water or PC1. This process was done to simulate restoring the slump of a concrete mixture in the field after a long haul or static period of time in a ready-mix truck. Figure 3.1 shows some normalized slump values to illustrate the effects remixing had on the slump of the concrete mixtures observed. When the mixture was reconstituted one of the following was done: 3 min additional mixing time, 3 min additional mixing time plus 12 lbs/cy of water, or 3 min additional mixing time plus 1.03 oz/cwt of PC1. The amount of water or PC1 chosen was determined based on what was needed to return the slump of the mixture to approximately the "0 min" or initial slump value.

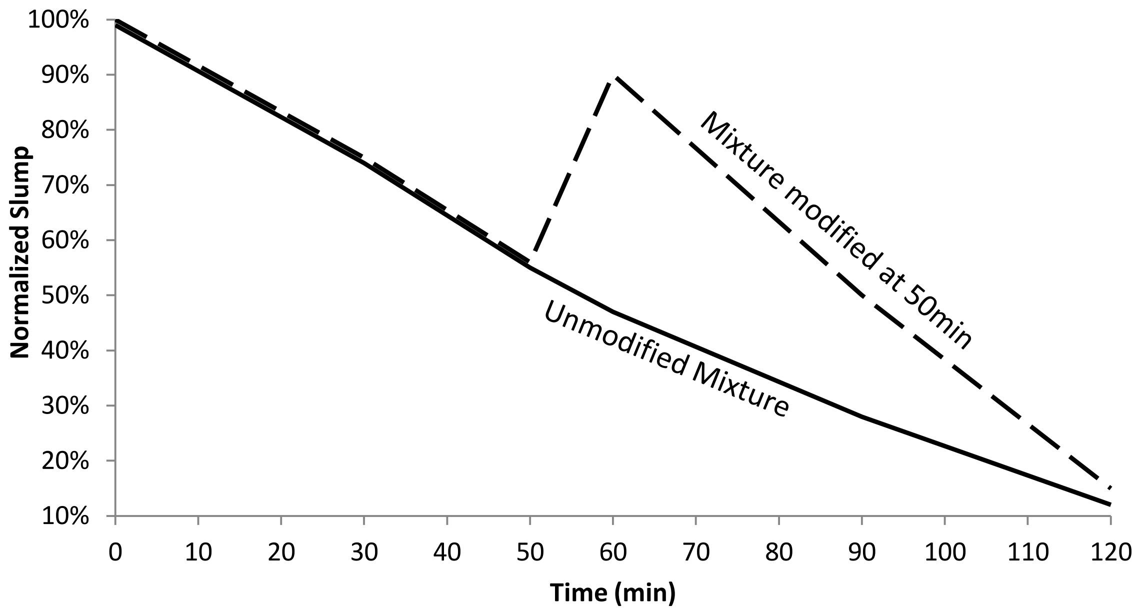

Figure 3.1 Idealized slump change in mixtures modified and unmodified by remixing during 120 min.

Each mixture has a unique mixture identification that highlights the details of the mixture and indicates the remixing method used to restore the slump. Consistent symbols and line types have been used throughout the document. Also mixtures that did not contain PC1 were shown in black; while mixtures that did are shown in gray. Mixtures with no remixing are shown as solid lines and mixtures with remixing are shown as dashed lines. One replicate mixture that contained PC1 and additional mixing time was prepared and is shown in a different shade of gray to highlight the difference in air content. Also, a replicate PC1 mixture with additional PC1 was prepared with a similar air content. Both replicates were done to verify the observations and ensure repeatability. A summary of the mixture identification, symbols, line types, color, and a detailed description for all types of mixtures investigated are shown in Table 3.4.

Table 3.4 Detailed mixture descriptions

Mixture ID	Symbol	Detailed description
WROS		wood rosin added to initial mixture and the mixture remained static for the sampling period
WROS+Mix		wood rosin added to initial mixture; three min of mixing after the mixture had been sitting for 50 min
WROS+Water		wood rosin added to initial mixture; 12 lbs/cy of water was added and three min of mixing after the mixture had been sitting for 50 min
PC1+WROS		wood rosin plus 4.96 oz/cwt superplasticizer added to initial mixture; the mixture remained static for the sampling period
PC1+WROS+Mix-1		wood rosin plus 4.96 oz/cwt superplasticizer added to initial mixture; three min of mixing after the mixture had been sitting for 50 min
PC1+WROS+Mix-2		wood rosin plus 4.96 oz/cwt superplasticizer added to initial mixture; three min of mixing after the mixture had been sitting for 50 min
PC1+WROS+Water		wood rosin plus 4.96 oz/cwt superplasticizer added to initial mixture; 12 lbs/cy of water was added and three min of mixing after the mixture had been sitting for 50 min
PC1+WROS+PC1*		wood rosin plus 4.96 oz/cwt superplasticizer added to initial mixture; 1.03 oz/cwt of superplasticizer was added and three min of ximing after the mixture had been sitting for 50 min

* A second PC1+WROS+PC1 mixture was made to validate observations and is represented with the same symbol notation as the original mixture since the intial properties and behavior over time were observed to be similar

Table 3.5 summarizes the testing and sampling performed. After the initial mixing period, 0 min, the mixture was tested for slump (ASTM C 143), unit weight (ASTM C 138), and fresh air content (ASTM C 231). The modified pressure meter specimen was consolidated simultaneously with the C 231 sample to ensure the 0 min air content can be accurately compared to the 120 min modified pressure meter measurement. Care was taken during sampling not to disturb more than the necessary amount of concrete while it remained in the mixer. Freeze thaw and hardened air samples were collected and consolidated as per ASTM C 666 at 0 and 60 min and samples for hardened air void analysis (ASTM C 457) at 0, 30, 60, 90, and 120 min. After sampling, a plastic cover was used to minimize evaporation.

Immediately after mixing was complete the mixture was tested for slump, unit weight, and fresh air content. ASTM C 666 and ASTM C 457 samples were made and the modified C 231 sample was consolidated and set aside. After 30 min from stopping the mixer, the mixture was tested for slump and unit weight. A hardened air void sample was collected as per ASTM C 457.

In mixtures where remixing was done, the mixture was tested for slump and unit weight at 50 min. No samples were collected at this time. The concrete was mixed for an additional three minutes and reconstituted with water or PC1. After the testing and additional mixing, the mixture was tested for slump, unit weight, and fresh air content, freeze thaw, and hardened air void analysis. This testing was completed at approximately 60 min after initial mixing.

After sitting statically for an additional 30 min, or 90 min after the initial mixing time, the mixture was tested for slump and unit weight. Samples were collected for hardened air void analysis as per ASTM C 457. After 120 min had elapsed from the initial mixing the concrete was tested for slump, unit weight, and fresh air content. A sample was collected for hardened air void analysis as per ASTM C 457 and the modified pressure meter was tested.

Table 3.5 Sampling and testing summary

ASTM	Description	Time after initial mixing					
		0min	30min	50min	60min	90min	120min
C 143	Slump	X	X	X	X	X	X
C 138	Unit Weight	X	X	X	X	X	X
C 231	Pressure Meter	X			X		X
N/A	Modified Pressure Meter	(+)					X
C 666	Freeze Thaw	X			X		
C 457	Hardened Air	X	X		X	X	X

(+) indicates the sample was consolidated as per ASTM C 231 and set aside for testing at 120min

Freeze thaw performance criteria

ASTM C 666 does not clearly define freeze thaw failure, however some guidance is given in admixture standards ASTM C 260, ASTM C 494, and ASTM C 1017. These standards recommend that the reduction in the ASTM C 666 durability factor of a mixture with and without an admixture should not differ by more than 20%. If this criterion is used to evaluate the performance of a mixture in the ASTM C 666 test then the limiting durability factor would be between 70% and 80% (Ley 2007). For this paper a specimen was determined to fail freeze thaw testing when the durability factor decreased below 80% at any point during the testing cycle. A ± symbol represents the range of durability factors seen by concretes of the same mixture proportions, admixtures, and mixing procedure.

Hardened Air Sample Preparation

The hardened air samples were cut into ¾ in thick slices using a self-propelled concrete saw with an 18 in diameter continuous rim blade with oil based cutting fluid. The sample was cleaned with water and then dried under a fan. An equal parts mixture of lacquer and acetone was applied to harden the surface and protect the rims of the air voids. An 18 in concrete lapper with magnetically bonded diamond discs of decreasing grit size were used to prepare the samples for testing. The samples were prepared as per ASTM C 457.

After the lapping was complete each sample was inspected under a stereomicroscope to ensure aggregates and paste were lapped to the same elevation and there was a high quality finish on the specimen. After the specimen had received an acceptable polish then they were soaked in acetone to remove the lacquer. After drying the prepared sample surface was colored black with a permanent marker then dried for 3 hours. A second coat of black marker was then applied in the perpendicular direction to the first coat and the sample dried for 8 hours. A thin layer of barium sulfate, a white powder with a particle size less than 3.94 x 10-5 in (< 1 um), was pressed on the colored surface twice with a rubber stopper to force the white powder into the voids. This technique is described in EN 480-11. This left the surface of the concrete black and the voids stained white. Since the analysis is concerned with the voids in the paste, the voids in the aggregate must be masked. To do this the voids within the aggregate were colored with a fine permanent ink pen under a stereomicroscope. Once completed a final inspection is made of the surface to ensure that voids in the paste are white and all other areas in the sample are black. A sufficiently polished sample and a finished sample can be seen in Felice (2012). This technique is outlined in detail in Ley (2007) and has been used by several other researchers (Jakobsen et al 2006, Sutter 2002, Carlson 2005, Peterson et al 2007).

Once the voids in the paste have been preferentially marked it is possible to use this contrast to determine the air void parameters of the mixture. The research team used the Rapid Air 457 from Concrete Experts, Inc. This machine completes an automated linear traverse analysis on the sample by using a CCD camera to image the surface and an automated stage for precise movement. Image analysis is then used to discern voids (white) from other portions of the sample (dark). A single threshold value of 145 was used for all of the samples that has been shown to be satisfactory with the sample preparation materials and processes used (Ley 2007). This technique requires that the volume of paste be given. This was determined from the batch weights from the concrete mixture design. For the results of the hardened air void parameters, spacing factor and specific surface, reported in this paper chords smaller than 30 µm were not included in the analysis as they are not easily detected by a human during the ASTM C 457. By excluding these chords the air void parameters determined by the hardened air void analysis are better comparable to other past reported values of ASTM C 457 results. This has been done previously by many researchers (Jakobsen et al 2006, Ley 2007, Peterson et al 2009, Ramezanianpour & Hooton 2010).

Results

The findings of this research are summarized in the following tables and figures. Tables 3.6, 3.7A, and 3.7B show fresh and hardened concrete properties with respect to sampling time and results from ASTM C 666 rapid freezing and thawing tests. In addition to the measured fresh and hardened properties, the total change and normalized change are supplied in Tables 3.6, 3.7A, and 3.7B to show how the fresh and hardened properties change over a 120 min period. The total change is simply the difference of the 120 min value relative to the 0 min value, and normalized change is the total change divided by the 0 min value. These values provide a comparison of the properties after the two hour time period for mixtures modified by remixing and those that are unmodified. Table 3.6 represents mixtures made without PC1. Table 3.7A and 3.7B show mixtures that have the carboxylated polyether (PC1) superplasticizer.

Two mixtures, PC1+WROS+Mix and PC1+WROS+PC1, were repeated to confirm that the results and observations were repeatable. The mixture with additional mixing time had a higher initial air content but had a similar loss in air and slump over time. Due to the higher initial air content, hardened air void samples were made. The mixture remixed with PC1 had almost identical behavior in all aspects of the fresh testing but had a lower initial air content than the original PC1+WROS+PC1, so only the fresh properties were observed for consistent behavior. The obtained results of the repeated PC1 mixtures can be found in Tables 3.7A and 3.7B.

Plots were made to show the effects of each remixing modification and how it impacted the concrete properties, specifically the air void system and the frost durability. Based on the results of work by Freeman (2012), it is hypothesized that voids less than 150 μm are very influential to frost durability.

Table 3.6 Mixtures made without carboxylated polyether (PC1)

Mixture ID	w/cm*	Admix. WRA/AEA (oz/cwt)	Tests		Time After Initial Mixing						Total 120 min Change	Normalized 120 min Change
					0 min	30 min	50 min	60 min	90 min	120 min		
WROS	0.45	0/1.49	Slump, in (C143)		2.5	1.75	-	1.25	0.75	0.375	-2.125	-85%
			Unit Wt., lbs/cf (C138)		145.2	145.2	-	146.0	146.6	147.9	2.72	2%
			Air Content	Gravimetric (C138)	4.5%	4.5%	-	3.9%	3.5%	2.7%	-1.8%	-40%
				Pressure Meter (C231)	4.6%	-	-	4.2%	-	3.3%	-1.3%	-28%
				Modified C231	-	-		-	-	4.4%	-0.2%	-4%
			Remixing (After 50 min)		none							
			Rapid Freeze/Thaw (C666)		99 ± 1%	-	-	97 ± 3%	-	-	-	-
			ASTM C 457	Hardened Air Content	4.1%	-	-	3.3%	-	3.7%	-0.4%	-9%
				Spacing Factor (in)	0.0075	-	-	0.0079	-	0.0087	0.0012	16%
				Specific Surface (in^{-1})	711	-	-	741	-	643	-68.2	-10%
WROS +Mix	0.45	0/1.92	Slump, in (C143)		2	2	1.75	2.125	1.625	1	-1	-50%
			Unit Wt., lbs/cf (C138)		144.2	144.7	145.0	144.2	144.6	145.8	1.64	1%
			Air Content	Gravimetric (C138)	5.1%	4.8%	4.6%	5.1%	4.9%	4.0%	-1.1%	-21%
				Pressure Meter (C231)	5.5%	-	-	5.4%	-	4.4%	-1.1%	-20%
				Modified C231	-	-		-	-	5.2%	-0.3%	-5%
			Remixing (After 50 min)		3 min mixing time							
			Rapid Freeze/Thaw (C666)		98 ± 2%	-	-	97 ± 1%	-	-	-	-
			ASTM C 457	Hardened Air Content	5.1%	3.8%	-	4.1%	3.0%	3.1%	-2.0%	-39%
				Spacing Factor (in)	0.0065	0.0073	-	0.0065	0.0080	0.0079	0.0014	22%
				Specific Surface (in^{-1})	740	758	-	824	771	763	23	3%
WROS +Water	0.45 (0.47)	0/2.13	Slump, in (C143)		2.375	2.25	1.75	3.75	3.25	2.375	0	0%
			Unit Wt., lbs/cf (C138)		144.2	144.3	144.8	139.9	140.4	141.8	-2.48	-2%
			Air Content	Gravimetric (C138)	5.1%	5.0%	4.7%	7.5%	7.2%	6.3%	1.2%	24%
				Pressure Meter (C231)	5.6%	-	-	7.8%	-	6.6%	1.0%	18%
				Modified C231	-	-		-	-	5.2%	-0.4%	-7%
			Remixing (After 50 min)		12 lbs water/cy + 3 min mixing time							
			Rapid Freeze/Thaw (C666)		99 ± 1%	-	-	100 ± 0%	-	-	-	-
			ASTM C 457	Hardened Air Content	5.4%	5.3%	-	7.7%	6.1%	5.2%	-0.2%	-4%
				Spacing Factor (in)	0.0061	0.0057	-	0.0054	0.0054	0.0067	0.0006	10%
				Specific Surface (in^{-1})	772	833	-	678	824	718	-54.3	-7%

*w/cm ratio in parenthesis represents w/cm after the addition of remix water (12 Lbs/CY)

Table 3.7A Mixtures made with carboxylated polyether (PC1)

Mixture ID	w/cm	Admix. WRA/AEA (oz/cwt)	Tests		Time After Initial Mixing						Total 120 min Change	Normalized 120 min Change
					0 min	30 min	50 min	60 min	90 min	120 min		
PC1 +WROS	0.45	4.96/0.32	Slump, in (C143)		8.75	3.5	-	2.5	1.5	1.5	-7.25	-83%
			Unit Wt., lbs/cf (C138)		145.5	149.6	-	149.4	150.4	150.0	4.48	3%
			Air Content	Gravimetric (C138)	4.2%	1.6%	-	1.7%	1.0%	1.3%	-2.9%	-69%
				Pressure Meter (C231)	4.5%	-	-	2.4%	-	2.4%	-2.1%	-47%
				Modified C231	-	-	-	-	-	4.1%	-0.4%	-9%
			Remixing (After 50 min)		none							
			Rapid Freeze/Thaw (C666)		(108)	-	-	(72)	-	-	-	-
			ASTM C 457	Hardened Air Content	5.3%	-	-	2.5%	-	-	-2.7%	-52%
				Spacing Factor (in)	0.0130	-	-	0.0135	-	-	0.0005	4%
				Specific Surface (in^{-1})	366	-	-	488	-	-	122	33%
PC1 +WROS +Mix-1	0.45	4.96/0.54	Slump, in (C143)		9.625	7	5.25	6	3.75	1.75	-7.875	-82%
			Unit Wt., lbs/cf (C138)		143.4	147.1	148.2	138.6	144.2	147.2	3.84	3%
			Air Content	Gravimetric (C138)	5.7%	3.2%	2.5%	8.8%	5.1%	3.1%	-2.5%	-45%
				Pressure Meter (C231)	5.8%	-	-	9.5%	-	4.0%	-1.8%	-31%
				Modified C231	-	-		-	-	5.7%	-0.1%	-2%
			Remixing (After 50 min)		3 min mixing time							
			Rapid Freeze/Thaw (C666)		(180)	-	-	100 ± 0%	-	-	-	-
			ASTM C 457	Hardened Air Content	5.7%	4.7%	-	8.6%	5.7%	3.9%	-1.8%	-32%
				Spacing Factor (in)	0.0107	0.0094	-	0.0068	0.0099	0.0105	-0.0002	-2%
				Specific Surface (in^{-1})	427	491	-	475	463	519	91.9	22%
PC1 +WROS + Mix-2	0.45	4.96/0.64	Slump, in (C143)		9.75	7.875	6.25	7.875	4.5	2.75	-7	-72%
			Unit Wt., lbs/cf (C138)		141.2	143.0	145.9	133.7	140.6	145.2	4	3%
			Air Content	Gravimetric (C138)	7.1%	5.9%	4.0%	12.0%	7.5%	4.5%	-2.6%	-37%
				Pressure Meter (C231)	7.2%	-	-	12.0%	-	5.2%	-2.0%	-28%
				Modified C231	-	-		-	-	7.1%	-0.1%	-1%
			Remixing (After 50 min)		3 min mixing time							
			Rapid Freeze/Thaw (C666)		n.m.	-	-	n.m.	-	-	-	-
			ASTM C 457	Hardened Air Content	7.0%	5.8%	-	9.6%	6.1%	5.1%	-1.9%	-27%
				Spacing Factor (in)	0.0090	0.0092	-	0.0067	0.0088	0.0086	-0.0004	-4%
				Specific Surface (in^{-1})	441	491	-	435	505	563	121.6	28%

n.m. = not measured

Table 3.7B Mixtures made with carboxylated polyether (PC1)

Mixture ID	w/cm*	Admix. WRA/AEA (oz/cwt)	Tests		0 min	30 min	50 min	60 min	90 min	120 min	Total 120 min Change	Normalized 120 min Change
PC1 +WROS +Water	0.45 (0.47)	4.96/0.75	Slump, in (C143)		9.25	5.5	4.75	8.25	5.25	3.875	-5.375	-58%
			Unit Wt., lbs/cf (C138)		142.6	144.9	146.6	134.6	139.3	145.5	2.96	2%
			Air Content	Gravimetric (C138)	6.2%	4.7%	3.5%	11.0%	7.9%	3.8%	-2.4%	-38%
				Pressure Meter (C231)	6.0%	-	-	11.0%	-	4.8%	-1.2%	-20%
				Modified C231	-	-		-	-	5.8%	-0.2%	-3%
			Remixing (After 50 min)		12 lbs water/cy + 3 min mixing time							
			Rapid Freeze/Thaw (C666)		(260)	-	-	98 ± 0%	-	-	-	-
			ASTM C 457	Hardened Air Content	5.2%	4.3%	-	9.7%	5.1%	4.4%	-0.8%	-16%
				Spacing Factor (in)	0.0091	0.0097	-	0.0053	0.0087	0.0094	0.0003	3%
				Specific Surface (in^{-1})	524	535	-	550	561	554	29.8	6%
PC1 +WROS +PC1	0.45	4.96/0.87 4.96/0.73	Slump, in (C143)		9.5	8.125	6.75	9.75	5.5	2.5	-7	-74%
			Slump, in (C143)		8.875	7	5	9.5	3.75	1.875	-7	-79%
			Unit Wt., lbs/cf (C138)		141.5	145.0	147.0	145.8	149.0	149.9	8.4	6%
			Unit Wt., lbs/cf (C138)		142.3	146.2	148.4	146.8	151.4	151.5	9.2	6%
			Air Content	Gravimetric (C138)	6.9%	4.6%	3.3%	4.0%	2.0%	1.3%	-5.5%	-80%
				Gravimetric (C138)	6.4%	3.8%	2.3%	3.4%	0.4%	0.3%	-6.1%	-95%
				Pressure Meter (C231)	6.6%	-	-	5.2%	-	2.5%	-4.1%	-62%
				Pressure Meter (C231)	5.8%	-	-	3.8%	-	1.9%	-3.9%	-67%
				Modified C231	-	-		-	-	6.4%	-0.2%	-3%
				Modified C231	-	-	-	-	-	5.0%	-0.8%	-14%
			Remixing (After 50 min)		1.03 oz/cwt PC1 + 3 min mixing time							
			Rapid Freeze/Thaw (C666)		(234)	-	-	(162)	-	-	-	-
			ASTM C 457	Hardened Air Content	6.0%	5.0%	-	5.5%	3.2%	3.7%	-2.3%	-38%
				Spacing Factor (in)	0.0105	0.0101	-	0.0108	0.0108	0.0110	0.0005	5%
				Specific Surface (in^{-1})	424	482	-	430	553	504	79.6	19%

*w/cm ratio in parenthesis represents w/cm after the addition of remix water (12 Lbs/CY)

The average absolute difference between ASTM C 231 and ASTM C 457 air contents was found to be 0.59% with a standard deviation of 0.40%. Due to this small variability the ASTM C 231 and ASTM C 457 concrete air contents will both be used in plots.

Figures 3.2, 3.3 and 3.4 show normalized change in slump, hardened air content and spacing factor respectively over the 120 min sampling period. Hardened air void volumes were used as they correlated well with fresh measurements and more samples were taken from the mixture. Changes in air content versus time were assumed to be linear up until 50 min. The dotted lines connecting 50 and 60 min show the changes caused by remixing.

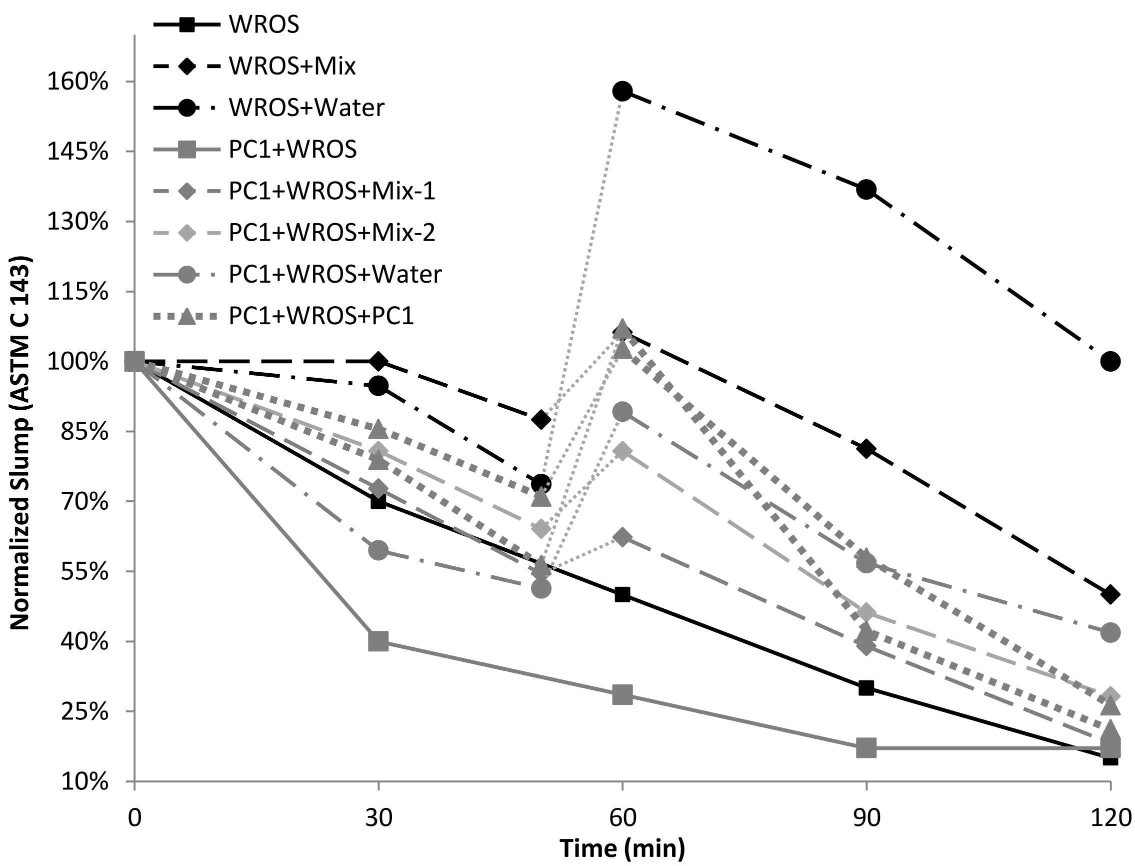

Figure 3.2 Normalized slump change over the 120 min sampling period

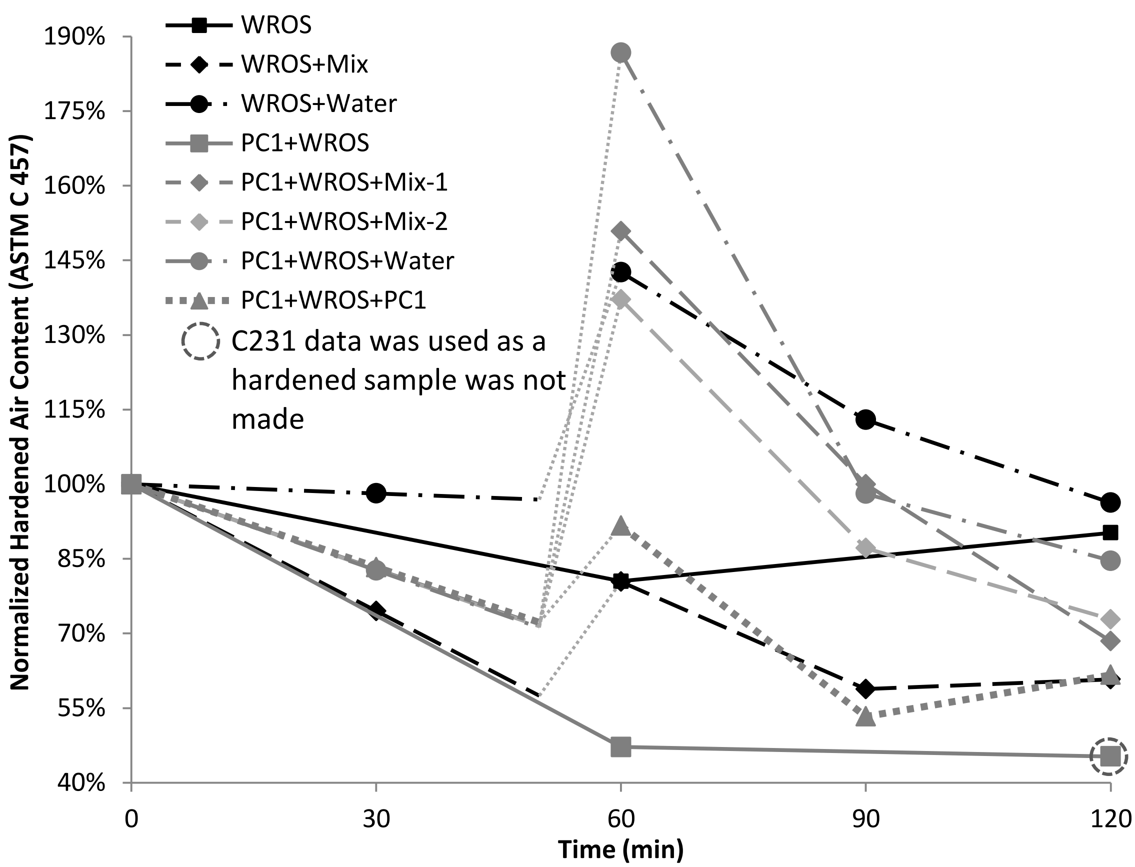

Figure 3.3 Normalized hardened air change over the 120 minute sampling period

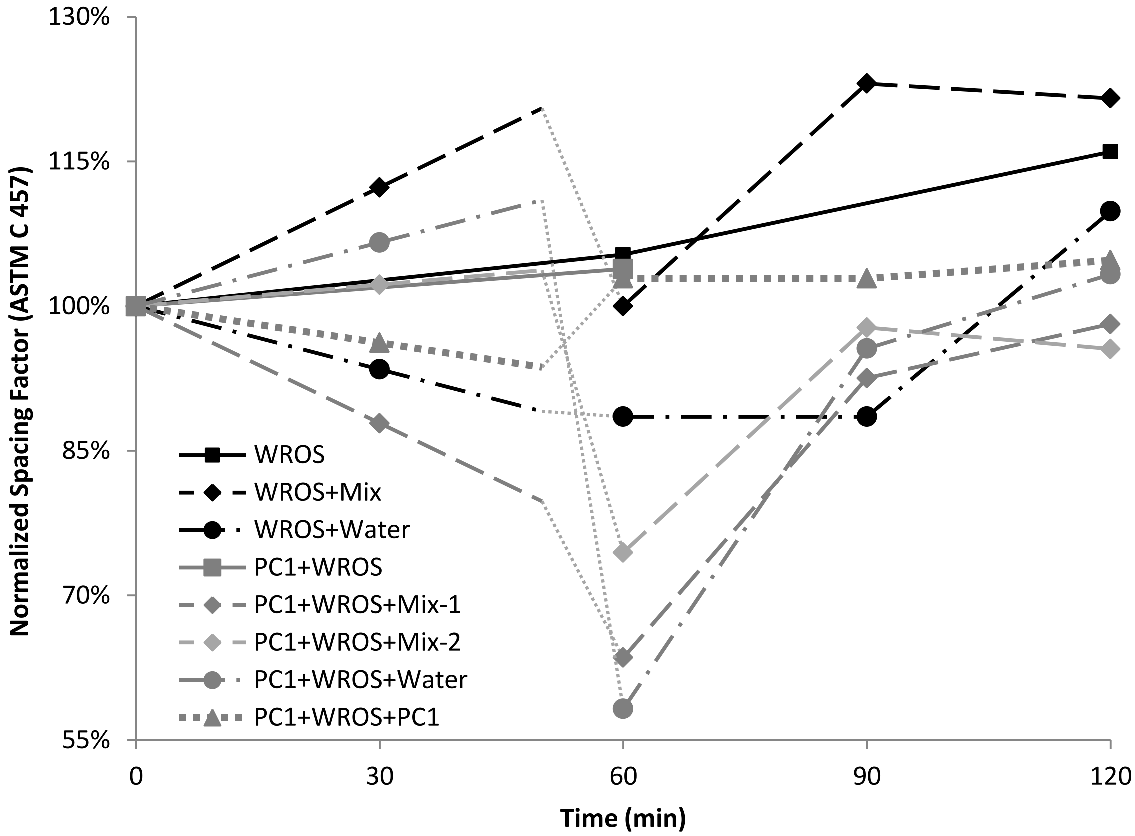

Figure 3.4 Normalized spacing factor change over the 120 minute sampling period

Figure 3.5 shows the amount of wood rosin used (oz/cwt) to achieve initial, 0 min, concrete air contents using the pressure meter for mixtures with AEA only and for mixtures with 4.96 oz/cwt of PC1 in the initial mixture. The points on the figure are prior to any remixing modifications.

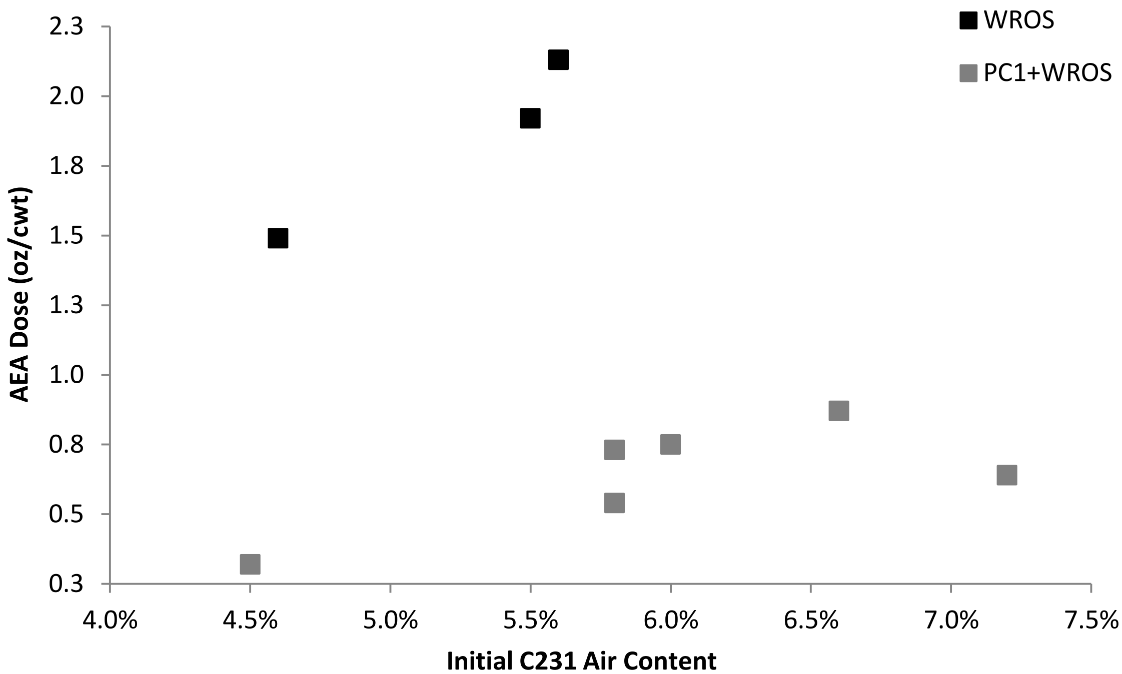

Figure 3.5 AEA dose (oz/cwt) used to achieve initial concrete air contents

Table 3.8 summarizes the change in slump (ASTM C 143), hardened air content and spacing factor (ASTM C 457) before and after remixing modifications. The percentage change due to remixing shows the change in slump, hardened air content and spacing factor that occurred as a result of remixing. The rate of change is also provided in Table 3.8 and is simply the average slope taken from Figures 3.2, 3.3 and 3.4 before and after remixing expressed as the change per minute. In mixtures where no remixing modification was made the average rate of change over 120 min is provided except for the change in spacing factor in the PC1+WROS mixture, where the change in spacing factor is calculated over 60 min since no hardened sample was taken at 120 min.

Table 3.8 Slump, hardened air and spacing factor rate of change before and after remixing

		Percent change due to remixing	Rate of change per minute before remixing	Rate of change per minute after remixing
WROS	Slump	-	- 0.71%*	
	Hardened Air	-	- 0.08%*	
	Spacing Factor	-	0.13%*	
WROS+Mix	Slump	19%	- 0.25%	- 0.94%
	Hardened Air	23%	- 0.85%	- 0.33%
	Spacing Factor	- 21%	0.42%	0.37%
WROS+Water	Slump	84%	- 0.53%	- 0.97%
	Hardened Air	46%	- 0.06%	- 0.77%
	Spacing Factor	0%	- 0.22%	0.35%
PC1+WROS	Slump	-	- 0.69%*	
	Hardened Air	-	- 0.46%*	
	Spacing Factor	-	0.07%**	
PC1+WROS+Mix-1	Slump	8%	- 0.91%	- 0.74%
	Hardened Air	79%	- 0.57%	- 1.38%
	Spacing Factor	- 16%	- 0.40%	0.57%
PC1+WROS+Mix-2	Slump	17%	- 0.72%	- 0.88%
	Hardened Air	66%	- 0.57%	- 1.07%
	Spacing Factor	- 30%	0.08%	0.37%
PC1+WROS+Water	Slump	38%	- 0.97%	- 0.79%
	Hardened Air	116%	- 0.58%	- 1.70%
	Spacing Factor	- 53%	0.22%	0.75%
PC1+WROS+PC1	Slump	32%	- 0.58%	- 1.27%
	Hardened Air	20%	- 0.56%	- 0.50%
	Spacing Factor	9%	- 0.12%	0.03%

* change over 120 min since remixing modification was not implemented

** change over 60 min

Figures 3.6 and 3.7 show the results of hardened air void analysis for samples taken immediately after remixing (60 min). Figure 3.6 shows the normalized hardened air content, and Figure 3.7 shows the normalized cumulative hardened air content. Black lines represent mixtures without PC1 and gray lines represent mixtures with PC1 in the mixture. The line types (noted in Table 3.4) represent the various remixing modifications used.

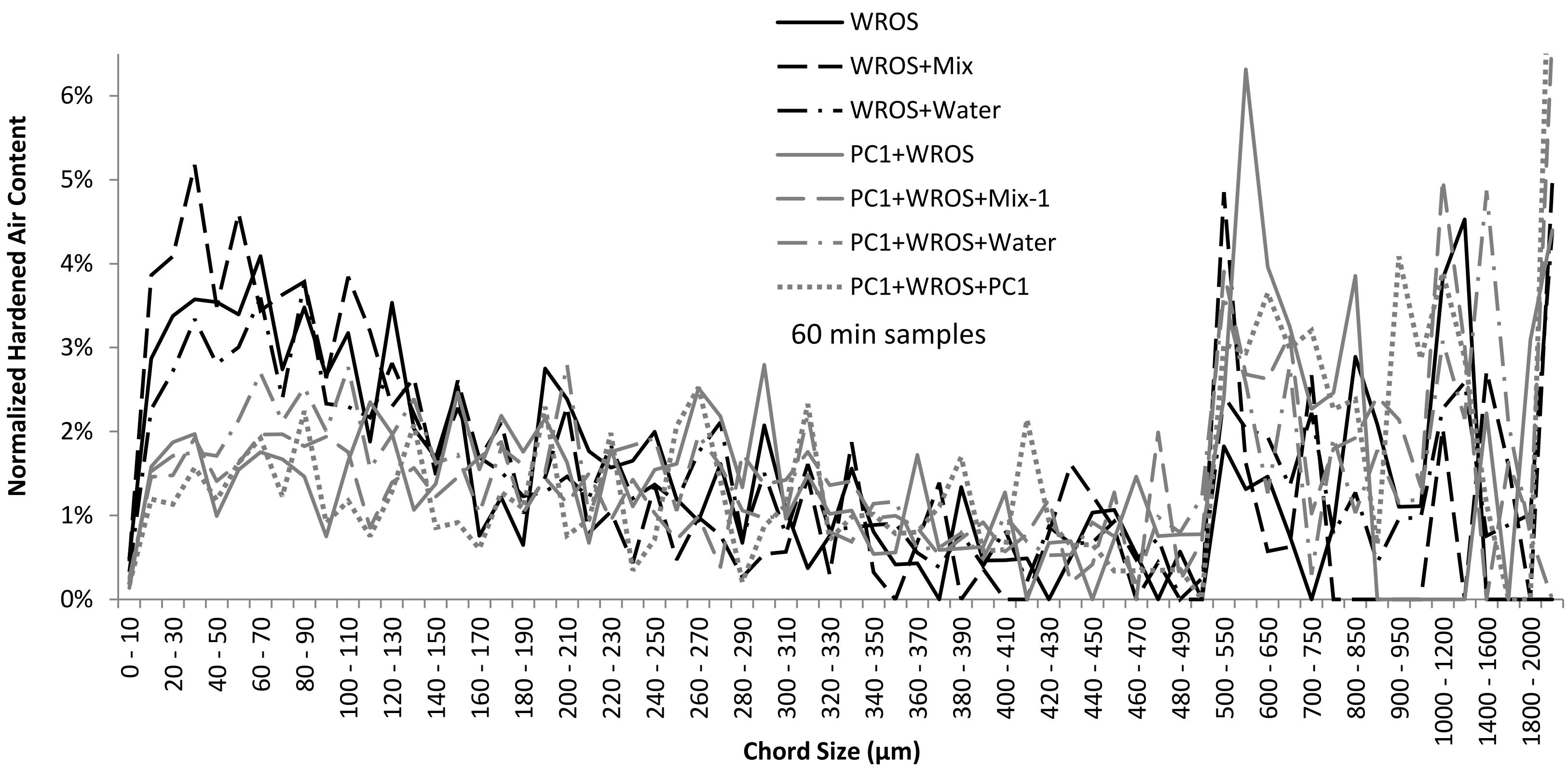

Figure 3.6 Normalized hardened air content for various chord sizes

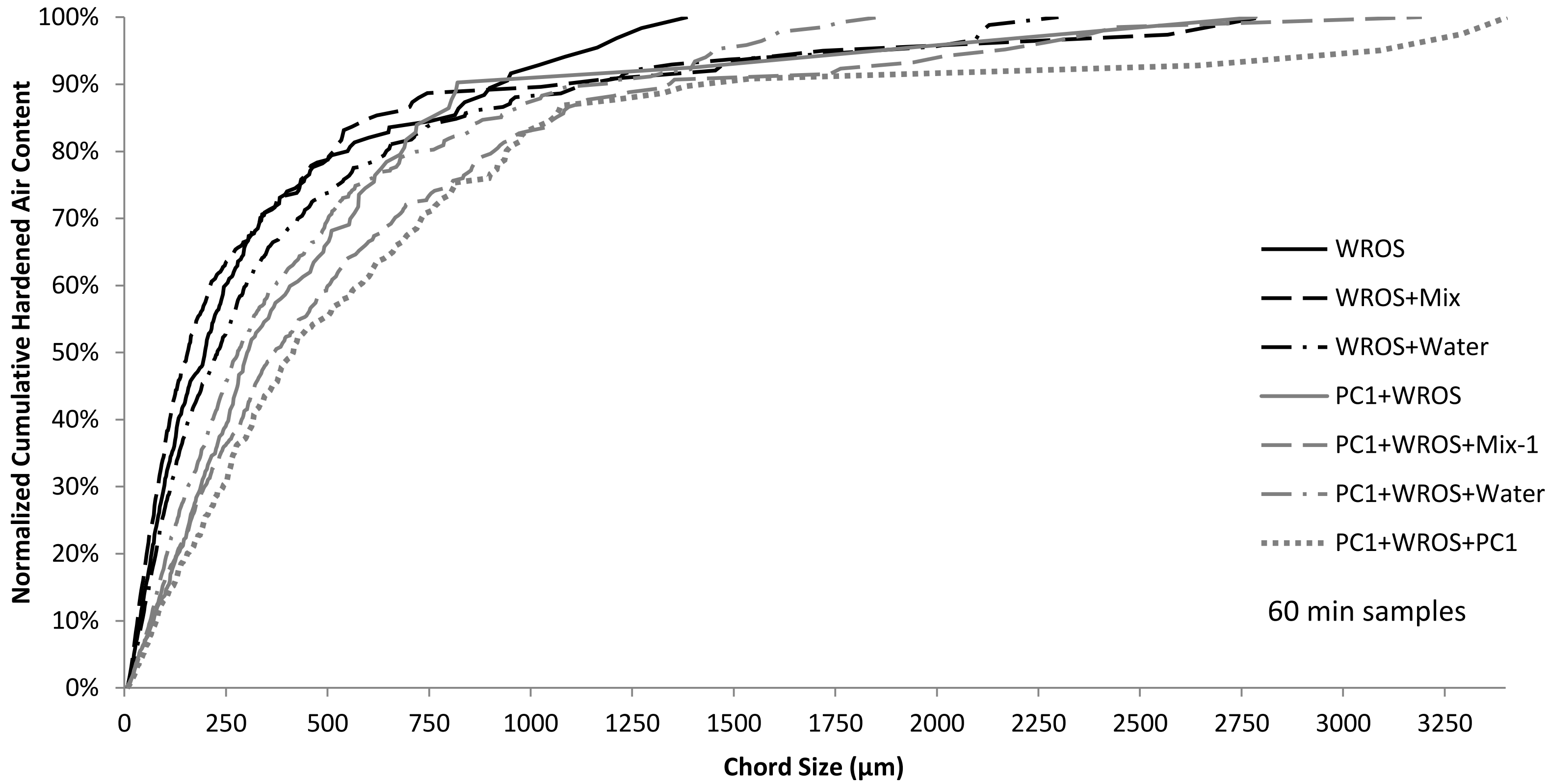

Figure 3.7 Cumulative hardened air content for various chord sizes

Shown in Figure 3.8 are the spacing factors and concrete air contents observed in mixtures with and without remixing modifications. The ASTM C 666 results are shown for each mixture by using a filled data point for a specimen that satisfactorily completed the test and an open data point for ones that failed. The ACI 201 spacing factor limit is shown as well as trend lines for mixtures with and without PC1. The WROS – trend line is used for wood rosin mixtures with air contents of 6% or less, and the PC1 + WROS – trend line is drawn for mixtures with air contents up to 9%. As anticipated non-linear behavior between spacing factor and air content occurs at higher air contents. Previous work by Felice (2012) showed satisfactory frost durability with combinations of admixtures when the spacing factors were below the ACI 201 limit of 0.008 in. To achieve spacing factors below 0.008 in it was found that an ASTM C 231 air content of 7.7% in the concrete was needed for the mixtures containing PC1 and an air content of 4.2% was needed for the mixtures with wood rosin as the only admixture.

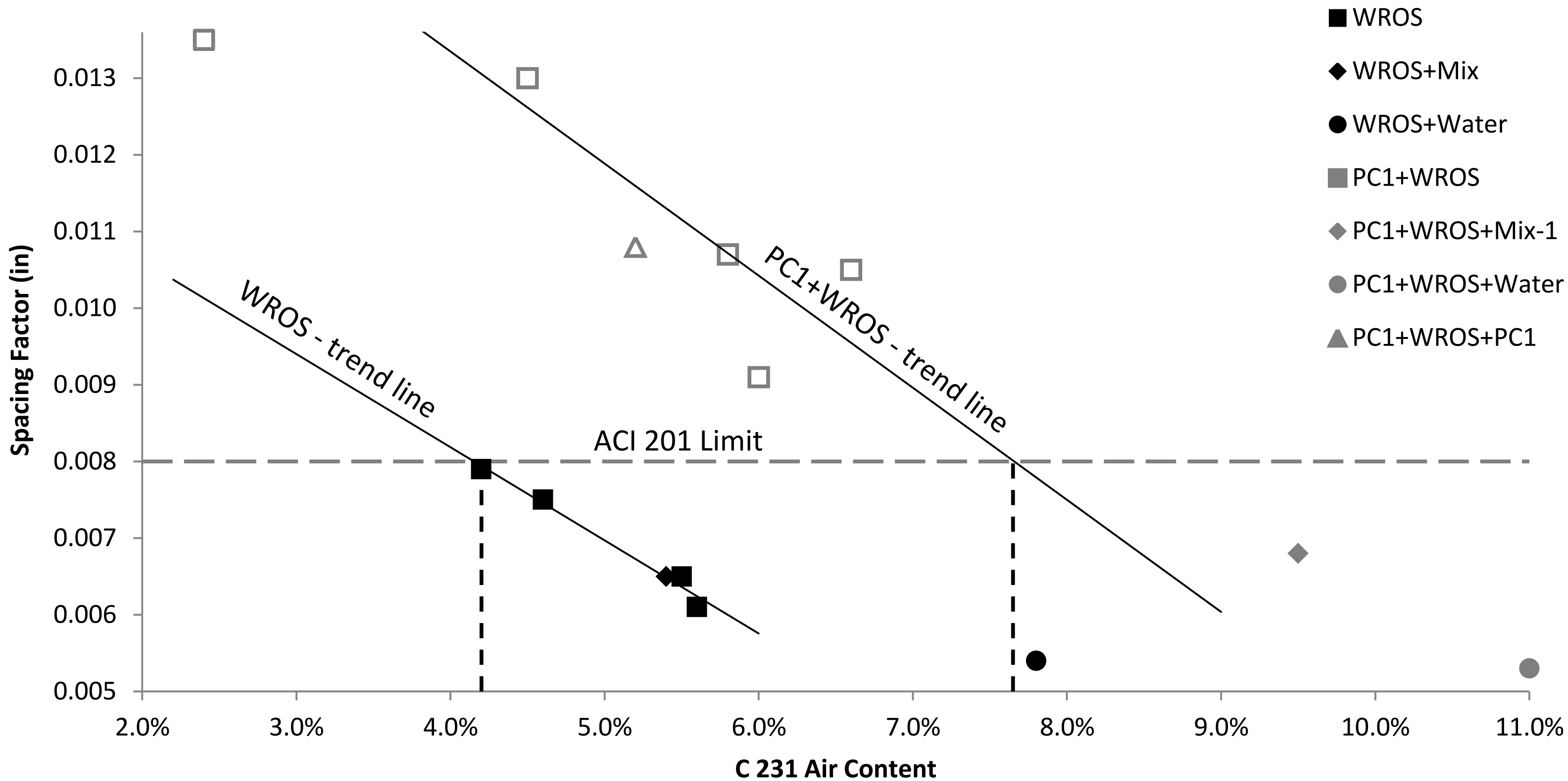

Figure3.8 Spacing factors and frost performance relative to measured fresh concrete air contents. Solid markers represent mixtures that passed ASTM C 666 and open does those that failed.

Figure 3.9 shows the number of chords per inch smaller than 150 µm observed in the concrete mixtures. This parameter has been suggested by Freeman (2012) to provide greater insight into frost durability than the spacing factor. A dashed horizontal continuous line at approximately 7 chords smaller than 150 µm per inch highlights the minimum number chords smaller than 150 µm per inch that led to satisfactory frost durability in the mixtures made for this study. Open data markers represent concretes that had unsatisfactory freeze thaw performance, while filled data markers represent satisfactory performance. Each mixture is labeled with either 0 min or 60 min which shows if the sample was made after initial mixing (0 min) or after remixing (60 min).

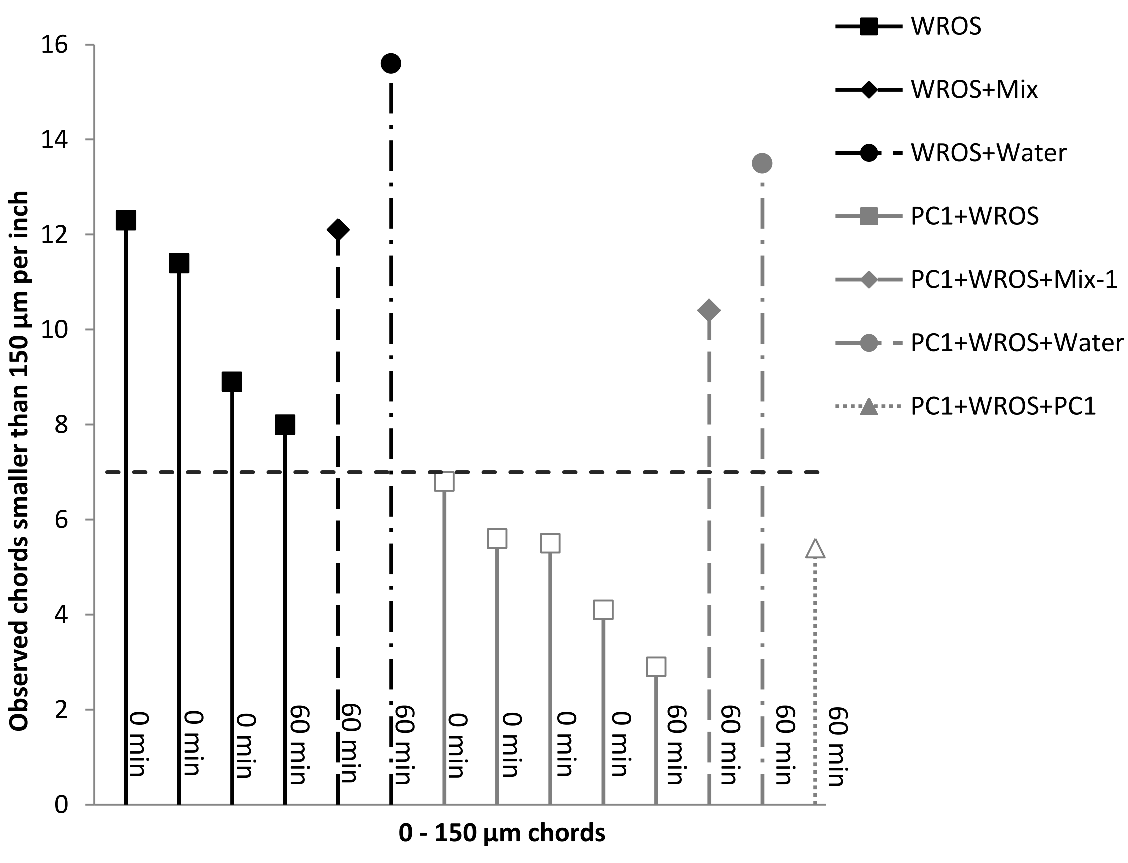

Figure 3.9 Number of 0-150 µm chords observed per inch of traverse

Discussion

The use of PCs to achieve high levels of workability without modifying the rate of hydration is a great asset to the concrete industry; however care must be taken to ensure that satisfactory void distributions are provided in these mixtures. This work focuses on investigating different practical methods to restore the workability and air void system to fresh concrete after it has been lost over time. The findings in this work reinforce the unstable slump and air contents observed by Freeman (2012) with mixtures that contain AEA and PCs.

General observations

Figures 3.2 and 3.3 show the slump and air content changes caused by remixing as well as the change experienced by mixtures without remixing. Figure 3.4 shows the change in spacing prior to and after remixing. Figure 3.5 highlights the dosage of AEA used to achieve the 0 min air contents. In mixtures where AEA was the only admixture, approximately 3.5 times more AEA was used than that used for PC1 mixtures to obtain similar air contents. In general, mixtures made with only WROS AEA did not experience dramatic air loss over time. When WROS was the only admixture used without remixing, a reduction of the slump by 85% and air volume by 0.4% and an increase in spacing factor of 0.0012 in was observed over the 120 minute testing period. Mixtures with PC1 and no remixing had a similar reduction of slump of 83% but a 53% reduction in the air volume over 120 minutes, and a 0.0005 in increase in spacing factor over 60 minutes. The decrease over 60 minutes was used as the 120 minute sample was not taken. These findings are similar to those observed by Freeman (2012). When mixtures were remixed in order to increase the slump the air content was observed to increase to different degrees as shown in Table 3.8 and Figures 3.2 and 3.3. Based on the observations a strong relationship relating increase in slump to an increase in air content was not observed. A relationship between voids less than 150 µm and frost durability was observed and is of focus in the discussion below.

Impact of PC1 on the air void system

Figure 3.6 shows normalized hardened air contents for all voids observed. A vertical offset between the PC1 and WROS only mixtures, most notably in the 0-150 µm range, suggests a coarsening of the air void system when PC1 is introduced. Figure 3.7 provides an alternative way of showing this finding with normalized cumulative hardened air content, where a similar noticeable offset can be seen between PC1 and non-PC1 mixtures. The steeper the slope and further to the left the normalized cumulative air content line is for a mixture the higher percentage of total air content is retained in smaller voids. On average the mixtures made with wood rosin as the only admixture had approximately 50% of the total air content from voids less than 150 µm. When PC1 was used only 25-30% of the total air content was contained in voids less than 150 µm. These findings confirm work presented by Freeman (2012) for a number of PCs.

Minimum concrete air content for satisfactory frost durability

Figure 3.6 and 3.7 shows that remixing clearly regenerates lost air content in the concrete. However, the void distribution (or shape of the curves) and increases in normalized hardened air content seen for all types of remixing for either WROS only or PC1+WROS mixtures is not significantly different. This implies that the act of remixing, whether by remixing alone or with water or with more PC1, does not substantially change chord distributions. In the case of remixing or adding water and remixing the total volume of air was increased and so the spacing factors decreased. In the case of additions of PC1 the air content did not increase after remixing. This is not beneficial to the long term frost durability.

It appears from the PC1+WROS - trend line in Figure 3.8 that a minimum threshold concrete air content for frost durability for mixtures made with PC1 exists at 7.7%, and when the concrete air content above this threshold is obtained then spacing factors less 0.008 in would be expected. Freeman (2012) observed a similar minimum threshold concrete air content, greater than 7.5%, to achieve spacing factors below 0.008 in with different combinations of PCs and AEAs. This work also shows evidence that if spacing factors are below the ACI 201 limit of 0.008 in frost durability should be expected. However, as shown in Tables 3.7A and 3.7B, when PC1 concretes were found to be frost durable after remixing, the specific surface values were all below the ACI 201 recommended 600 in-1. For the mixtures investigated the ACI 201 limit on spacing factor appears to accurately predict frost durability, while the recommended value for specific surface does not.

Remixing

Remixing alone was an effective technique to restore air contents and increase workability. The best impact was seen in the WROS only mixtures for the regeneration of air voids less than 150 µm. Remixing alone did regenerate voids smaller than 150 µm in PC1 concrete mixtures compared to the PC1 mixture without remixing, but as Figures 3.6 and 3.7 show the increase was not as much as was seen for other remixing modifications. Slump increased by 19%, hardened air content increased by 23% and spacing factor decreased by 21% for the WROS+Mix mixture. In the two PC1 mixtures with additional mixing time an 8 and 17% increase in slumps, 79 and 66% increase in hardened air content and a 16 and 30% decrease in spacing factor was observed. The percentage increase in slumps were similar in concretes with and without PC1 that were remixed for 3 min. However, the increase in air content was substantially more for the high slump PC1 mixtures. Additionally, with the increase in air content caused by remixing the spacing factors were substantially reduced.

Remixing with water

The WROS+Water mixture experienced an increase in slump of 84%, a 46% increase in hardened air content and no change in spacing factor. In the mixtures with AEA as the only admixture a non-linear relationship between air content and spacing factor, seen in Figure 3.8, appeared to occur once air contents approached 5.5%. This behavior is expected and is likely the cause of little or no change in spacing factor for the WROS+Water mixture that had a hardened air content of 5.3% before remixing and 7.7% after remixing. The PC1 mixture that was remixed at 50 min with 12 lbs/cy of water experienced a 38% increase in slump, a 116% increase in hardened air content and a 53% decrease in spacing factor. Increased slumps for mixtures with and without PC1 were substantial and support the idea that even though a direct relationship of slump and air content cannot be made with these mixtures, the two properties are tied. Although the percentage increase in slump observed in the PC1+WROS+Water was 46% less than the WROS+Water mixture, the increase in air content was 70% more in the mixture with PC1. This follows the observed trend of higher slumps improving the ability for a mixture to entrain air.

Even though the shapes of the normalized chord distribution curves support that no remixing modification significantly alters the air void distribution; Figures 3.6 and 3.7 show that of all the PC1 mixtures observed, the PC1 mixture remixed with water experienced the most increase in normalized volume of hardened air content in voids less than 150 μm compared to the other remixed PC1 mixtures. Table 3.7B shows that the use of water plus remixing significantly increases the total volume of air, in this case to 9.7% total measured hardened air content and significantly decreases the spacing factor to a value of 0.0053 in, which is well below the ACI 201 limit. This performance is intriguing as the specific surface value is 550 in-1 which does not meet the ACI 201 recommendations of 600 in-1.

Remixing with more PC1

The mixture that was remixed with PC1 had a 32% increase in slump, a 20% increase in hardened air content and a 9% increase in spacing factor. This was by far the lowest increase in hardened air content seen by remixed PC1 mixtures. Figure 3.5 shows the spacing factor increased as a result of remixing with more PC1; spacing factors in the rest of the mixtures that were remixed decreased as a result of remixing. Figure 3.6 shows the normalized hardened air void distribution of the PC1+WROS+PC1 and PC1+WROS (no remixing) are very similar. Figure 3.9 shows the PC1+WROS+PC1 mixture as having the least amount of observed chords in the 0-150 µm range, and Figure 3.7 shows 50% of its air content comes from voids greater than 400 µm. Table 3.7B shows that the use of more PC1 plus remixing had very little impact on the total volume of hardened air content, and did not decrease the spacing factor. This type of behavior to remixing was only seen when additional PC1 was added, all other remixed mixtures experienced increases in hardened air contents corresponding to decreased spacing factors after remixing.

Impact of consolidation

The modified C 231 test results found in Tables 3.6, 3.7A, and 3.7B when compared to the ASTM C 231 air content at 120 min show air loss can be significantly reduced by immediately consolidating the concrete. This finding is well documented by Freeman (2012) in which the loss of air and slumps for fifteen mixtures are shown over 120 min in concretes treated with five different PCs and three different AEA combinations. Of the fifteen mixtures the average 120 min air loss observed in unconsolidated concrete was 48.4% and this loss was reduced to 11.9% air loss for consolidated concrete. Figures 3.2 and 3.3 show trends of air and slump loss in the first 50 min, before remixing, similar to those observed by Freeman. This study did observe similar results as Freeman when comparing modified C 231 values for concrete that sat consolidated for 120 min, where an average air loss of 0.3% occurred for the nine mixtures observed. These combined results support consolidation of concretes with adequate air contents as a means to mitigate air losses.

Stability of air content after remixing

Commercial PCs are known to contain defoaming agents that decrease the volume of air created during mixing. This is done because PCs by themselves are known to increase the air content in a mixture to such a degree that they can be detrimental to the strength of concrete. As shown by the rate of decrease of hardened air content in Table 3.8, three of the four remixed PC1 mixtures and one of the two remixed WROS only mixtures experienced higher rates of air loss after remixing. Of these the highest losses occurred in PC1 concretes. The WROS+Water mixture, while not as rapid, experienced a faster rate of air loss compared to the other WROS mixtures after the addition of water and remixing. While it is possible to regenerate air from remixing it appears that it is imperative that a concrete with PC, be consolidated as quickly as possible after an adequate air content is measured in order to retain the volume of air that is deemed necessary for frost resistance, especially if the adequate air content is achieved by remixing. Additionally, it is recommended to consolidate mixtures without PC as quickly as possible, especially those that have been remixed with water as the rate of air loss is shown to increase after remixing.

Frost durability

Figure 3.8 shows the spacing factors and air contents of all mixtures that were evaluated in freezing and thawing tests. This data set suggests that mixtures made with wood rosin as the only admixture had spacing factors below 0.008 in and satisfactory frost durability when the concrete air content was 4.2% or higher. This finding is similar to the frost durability results in wood rosin mixtures made by Felice (2012). For PC1 mixtures it was found that a minimum air content of 7.7% was needed before the spacing factor was below 0.008 in or frost durability was achieved. The addition of more AEA was not used in this research as a means to regenerate air content, however based on the behavior of the mixtures observed, it would be expected that the addition of more AEA would lead to higher air contents, lower spacing factors, and adequate frost performance.

The use of more PC1 to significantly regenerate air content was not successful. The use of more PC1 increased the hardened air content by only 20% compared to the 66-79% from remixing alone and 116% increase with the addition of water in PC1 mixtures. Since the air content was not increased above the minimum threshold concrete air content, spacing factors remained well above the ACI 201 limit of 0.008 in and unsatisfactory freeze thaw performance was observed. The air content likely did not increase because of the presence of defoamer within the PC1.

The ACI 201 limit on spacing factor predicted frost performance for the mixtures investigated. Since the hardened air void distribution was not significantly impacted by remixing, it was observed that as long as the volume of air is increased enough to give the mixture a spacing factor less than 0.008 in than satisfactory frost performance can be expected. To ensure this criteria is met it is suggested that the ACI 318 recommendation to ensure frost durable concrete with ¾ in nominal maximum aggregate size for class F2 and F3 (severe exposure) be increased from 6% to at least 8% concrete air content for mixtures containing a PC. Based on the findings of this research, PCs should not be used to regenerate air content or workability in concrete mixtures susceptible to freezing and thawing.

Number of 150 µm or smaller chords per inch to predict frost durability

Figure 3.9 highlights the correlation between the number of chords less than 150 µm observed per inch of traverse and frost durability. As stated before the 0-150 µm size voids appear to play a significant role in frost performance. For the mixtures made in this study it was observed that when the concrete had more than 7 chords (less than 150 µm) per inch it proved to be frost durable. Freeman (2012) observed a similar value near 6.5 chords (less than 150 µm) per inch with different admixture combinations. It is visible from Figure 3.9 that the only remixing modification that did not increase the number of 0-150 µm chords per inch to more than 7 chords per inch was the addition of more PC1. This reinforces the findings relating the addition of more PC1 to unsatisfactory frost performance shown in Figure 3.8. While the ACI 201 limit for spacing factor predicted frost durability for these mixtures, it is a calculated value that requires the input of the specific surface value. The chords less than 150 µm per inch calculation in the future may provide another way to define the quality of an air void system.

Practical Implications

The spacing factor and total air content were dependent on one another in the mixtures investigated. It is recognized that different concrete mixtures will require different volumes of air and will have different minimum threshold air contents for frost durability. As suggested previously, the threshold value observed for the mixtures without PC1 provided frost durability at an air content of 4.2% or higher and 7.7% or more for mixtures containing PC1. These findings imply that the addition of PC1 coarsens the air void system and a higher total volume of air is required to provide the necessary small voids to provide frost durability.

If air is lost during the hauling of concrete made with a PC one would prefer to try remixing alone and only add water if water was held back from the original mixture design. The addition of more AEA would likely also be an acceptable means of improving the slump and increasing the air content as observed by Langan (1976); however, this was not investigated in this study. PCs should not be added to field concretes to increase slump without first checking the air requirements of the fresh concrete.

In this work it was observed that consolidating a concrete mixture stops the loss of air from the fresh concrete when compared to the material left unconsolidated. This confirms work by Freeman (2012). Although not tested it would be expected that slow agitation of concrete would also promote the loss of air in the fresh concrete.

In most cases when remixing was used to increase slump and regenerate lost air, the rate of slump and air loss was greater after remixing than before. This means that after a satisfactory volume of air in the concrete is achieved, especially if the volume is increased by a remixing modification, it is recommended that the concrete be consolidated as quickly as possible to reduce further air losses.

Currently ACI 318 suggests 6% air content for concretes subjected to exposure classes F2 and F3 with ¾ in nominal maximum aggregate size and an air content of 5% is allowed if the f'c is greater than 5,000 psi. From the results in this work it appears that these recommendations are not conservative for mixtures with the PC investigated in this work. Instead an air content of 8% is recommended for frost durability of the mixtures in this paper or a hardened air void analysis that shows the spacing factor is below 0.008 in. It should be noted that this recommendation may not be the same for all combinations of materials and PCs. These recommendations were based on performance of the mixtures in the ASTM C 666. This test has been shown to be extreme but is commonly used to evaluate the frost durability of modern concrete mixtures. These recommendations support similar findings observed by Freeman (2012) with multiple PCs and AEAs.

Typically strengths do not control modern mixture designs. However, if the concrete design is strength controlled then alterations to the mixture may be needed to compensate for the loss in strength from this recommended increase in air content of PC mixtures that are to be frost durable. These alterations may require more cement to be used in the mixture which can impact the sustainability, economy, and durability of the concrete mixture.

Conclusions

Concrete mixtures with wood rosin as the only admixture, and wood rosin with a polycarboxylate (PC1) were prepared for this study. Mixtures with PC1 were shown to lose slump and air over time more rapidly than the mixtures that did not contain PC1. Methods to regenerate the air including: remixing, adding water and remixing, or adding more PC1 and remixing were investigated. Slump and air contents were measured over a 120 min sampling period to investigate the effects of remixing on air void systems and frost durability. Hardened air void analysis as per ASTM C 457 and freezing and thawing tests as per ASTM C 666 were used to investigate their performance. Based on the data presented the following have been found:

- Concrete mixtures made with PC1 were observed to simultaneous lose slump and air content over time faster than mixtures that did not contain PC1.

- Approximately 3.5 times more AEA (oz/cwt) was needed in mixtures with AEA as the only admixture to achieve similar air contents as those made with PC1 and AEA.
- The rate of slump and air loss were typically higher for mixtures after remixing.
- Consolidation was observed to mitigate air losses over time in all mixtures made.
- A strong relationship was found between a spacing factor and the total volume of air for a given mixture. When air volume increased spacing factors decreased. Spacing factors for these mixtures below 0.008 in provided satisfactory frost performance.
- For the mixtures observed, remixing modifications did not appear to significantly alter void distributions, therefore spacing factors were reduced by increasing the volume of air in the mixtures.
- Coarsening of the air void system was observed with the addition of PC1. For the mixtures made in this study, when wood rosin was the only admixture, 4.2% total concrete air content was required to achieve spacing factors below 0.008 in and satisfactory frost durability. PC1 mixtures required approximately 8% concrete air contents to achieve spacing factors below 0.008in.
- Voids smaller than 150 µm appear to have a significant influence on frost durability. The data presented for these mixtures shows that the concrete mixtures with at least 7 chords smaller than 150 µm per inch of traverse were observed to perform satisfactorily in freeze thaw testing. This criterion was met in PC1 mixtures when remixing generated air contents above the minimum threshold concrete air content of 7.7%.

- Remixing alone and remixing with water increased air contents above the minimum threshold air content and decreased spacing factors in wood rosin mixtures with and without PC1.

- It is recommended that one would try remixing alone first to achieve adequate slump and air contents. However water can be used to increase the workability and air volume to desirable levels. However this water must be withheld up front in the mixing water and consolidation should be done as soon as possible after remixing as the rate of slump and air loss was observed to increase.

- While remixing and retempering with PC1 was able to increase the slump to the initial level it was shown to not increase the air content in the mixture as much as remixing alone. Due to these findings it is not recommended that additional PC1 be used in restoring concrete slumps and air contents of concretes susceptible to freezing and thawing, unless the use of hardened air void analysis can prove the air void system is adequate.

- Modern specifications should change for concrete mixtures that use PCs to require more air content or to require a hardened air void analysis to insure that a satisfactory spacing factor has been achieved.

Despite losing air in a mixture over time this work shows that it is possible to regenerate concrete air contents to levels that are able to provide satisfactory frost durability. Since these remixing procedures do not seem to impact the fundamental void distribution, these techniques can be used on the job site to increase the slump and air content of the mixture to provide frost durability.

CHAPTER IV

CONCLUSIONS

This thesis is composed of two studies that investigated modern concrete mixtures. The first study was modeled after work by Paul Klieger (1952 and 1956) and consisted of mixtures prepared with different modern air entraining admixtures (AEAs) with and without lignosulfonate WRs at different air contents. The second study expanded upon work completed by Freeman (2012). In this work concrete mixtures with wood rosin as the only admixture as well as mixtures with wood rosin and a polycarboxylate superplasticizer (PC1) were prepared. This study goal was to regenerate air content that has been lost, specifically in PC concretes, to frost durable levels. To do this three types of remixing tactics were utilized. Remixing of the concrete with no additives, adding water and remixing, or adding more PC1 and remixing. Hardened air void analysis as per ASTM C 457 and freezing and thawing tests as per ASTM C 666 were used in both studies to investigate their performance. Based on the data presented by the two investigations, the following conclusions were found:

Mixtures with varying w/cm, three modern AEAs, and a lignosulfonate WR

- A minimum air content of 3.5% in the concrete and 11.0% in the paste should yield concrete durable in the ASTM C 666 with modern AEAs and low (3.7oz/cwt) or no lignosulfonate WRs. This minimum air content was the same for a synthetic, wood rosin, and Vinsol resin AEA.

- Limited data suggests that mixtures with a higher dosage of lignosulfonate will need about 1% more air in the concrete or 3% more air in the paste for the materials and procedures used.

- Despite similar air void volume and better spacing factors there were differences in performance in ASTM C 666 for mixtures with a high dosage (10.2 oz/cwt) of lignosulfonate and those without. This suggests that there are other critical parameters besides air void volume and spacing that govern performance in ASTM C 666.

- A spacing factor of 0.008 in was found to be necessary to provide frost durability for the mixtures investigated.

- There was no noticeable difference in performance in ASTM C 666 or changes in the quality of the air void system as measured by ASTM C 457 for mixtures with a w/cm of 0.45 or 0.41 with the AEAs investigated.

Stability and regeneration of air content lost in PC concretes

- Concrete mixtures made with PC1 were observed to simultaneous lose slump and air content over time faster than mixtures that did not contain PC1.

- Approximately 3.5 times more AEA was used per percent of air content when AEA was the only admixture, compared to PC1 mixtures.

- The rate of slump and air loss were typically higher for mixtures after remixing at 50 min.

- Consolidation was observed to mitigate air losses over time in all mixtures made.

- A strong relationship was found between a spacing factor and the total volume of air for a given mixture. When air volume increased spacing factors decreased. Spacing factors for these mixtures below 0.008 in provided satisfactory frost performance.

- Remixing modifications did not appear to significantly alter void distributions; therefore higher volumes of air were required to achieve spacing factors below 0.008 in.

- Coarsening of the air void system was observed with the addition of PC1. For the mixtures made in this study, when wood rosin was the only admixture, 4.2% total concrete air content was required to achieve spacing factors below 0.008 in and satisfactory frost durability. PC1 mixtures required approximately 7.7% concrete air contents to achieve spacing factors below 0.008in.

- Voids smaller than 150 µm appear to have a significant influence on frost durability. The data presented for these mixtures shows that the concrete mixtures with at least 7 chords smaller than 150 µm per inch of traverse were observed to perform satisfactorily in freeze thaw testing. This criterion was met in PC1 mixtures when remixing generated air contents above the minimum threshold concrete air content of 7.7%.

- Remixing alone and remixing with water increased air contents above the minimum threshold air content and decreased spacing factors in wood rosin mixtures with and without PC1.

- It is recommended that one would try remixing alone first to achieve adequate slump and air contents. However water can be used to increase the workability and air volume to desirable levels. However this water must be withheld up front in the mixing water and consolidation should be done as soon as possible after remixing as the rate of slump and air loss was observed to increase. While remixing and retempering with PC1 was able to increase the slump to the initial level it was shown to not increase the air content in the mixture as much as remixing alone. Due to these findings it is not recommended that additional PC1 be used in restoring concrete slumps and air contents of concretes susceptible to freezing and thawing, unless the use of hardened air void analysis can prove the air void system is adequate.

- Modern specifications should change with concrete mixtures that use PCs to require more air content or to require a hardened air void analysis to insure that a satisfactory spacing factor has been achieved.

Future work in these areas could include repeating experiments to verify results and add additional data points. The use of other types of water reducers with modern AEAs should be investigated to improve the strength of the observations found with a lignosulfonate and wood rosin concrete mixture. The use of different cements with different alkali contents could be investigated since it is known that different admixtures behave differently depending on the cement used. The interaction between stabilized air in concrete and the impacts defoamers have on this air should be examined, as well as additional admixtures that alter concrete behavior and performance. The need for less AEA in mixtures with PCs should be investigated to see if the reduced amount of AEA in PC mixtures has an impact on air void system stability and quality. Studies should be done to observe the impacts of the remixing modifications and how the air void systems respond to field concretes subjected to long hauls that have been treated with polycarboxylates.

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Scope and Method of Study:

This work consists of two main studies. The first study is to observe the impact of w/cm, three types of air entraining admixtures (AEA), and a midrange (lignosulfonate) water reducing admixture (WRA) have on concrete air void systems and frost performance. The second study focused on a 0.45 w/cm concrete that used wood rosin AEA and a polycarboxylate super plasticizer (PC). The second study looks to investigate how to regenerate fresh air content and workability lost in concrete treated with a PC to achieve satisfactory frost performance. Concrete mixtures for this study were made in a climate controlled laboratory environment. Mixtures were prepared using locally available aggregates used in commercial concretes (ASTM C 33), a Type I/II cement (ASTM C 150), and admixtures (ASTM C 260 and C 494). A standard mixing procedure was used and is discussed in the respective experimental methods sections corresponding to each study. To investigate the hardened concrete parameters the surface of samples were prepared in accordance to ASTM C 457, and scanned under a CCD camera.

Findings and Conclusions:

The first study determined that a concrete air content of 3.5% was needed for satisfactory frost performance in mixtures where AEA was the only admixture. It was observed that in concretes prepared with higher doses of midrange water reducer, 4.5% concrete air contents were needed for satisfactory frost performance. The second study found that the current guidelines used for air contents are not conservative for concretes treated with PCs. The addition of PC was shown to severely coarsen the air void system requiring concrete air contents near 8% for frost durability. Remixing modifications (remixing alone, remixing plus water, and remixing plus more polycarboxylate) were used to regenerate lost air contents. Frost durability was seen in these mixtures by remixing alone and remixing with water. Remixing with additional PC did not provide frost durable mixtures for this study.

ADVISER'S APPROVAL: Dr. Tyler Ley