
Concrete Pavement Mixture Design and Analysis (MDA): Assessment of Air Void System Requirements for Durable Concrete

National Concrete Pavement
Technology Center



Technical Report
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16. Abstract 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 stabilize microscopic air voids. Small and well-dispersed air voids are critical to produce frost-resistant concrete. Work completed by Klieger in 1952 found the minimum volume of air required to consistently ensure frost durability in a concrete mixture subjected to rapid freezing and thawing cycles. He suggested that frost durability was provided if 18 percent air was created in the paste. This is the basis of current practice despite the tests being conducted on materials that are no longer available using tests that are different from those in use today. Based on the data presented, it was found that a minimum air content of 3.5 percent in the concrete and 11.0 percent in the paste should yield concrete durable in the ASTM C 666 with modern AEAs and low or no lignosulfonate water reducers (WRs). Limited data suggests that mixtures with a higher dosage of lignosulfonate will need about 1 percent more air in the concrete or 3 percent more air in the paste for the materials and procedures used. A spacing factor of 0.008 in. was still found to be necessary to provide frost durability for the mixtures investigated.					
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CONCRETE PAVEMENT MIXTURE DESIGN AND ANALYSIS (MDA): ASSESSMENT OF AIR VOID SYSTEM REQUIREMENTS FOR DURABLE CONCRETE

**Technical Report
June 2012**

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INTRODUCTION

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 stabilize microscopic 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 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 as the volume of air increases, the average spacing between voids in the paste, or the spacing factor, decreases (Pigeon and 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 ensure 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. Klieger's work was completed without the aid of any hardened air void analysis and ultimately suggested that throughout all of the mixtures investigated, 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 Klieger'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 and 1985, Jana et al. 2005, Ley 2007). The validity of spacing factor limits of 0.008 in. has 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 report 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^o1.

Table 1. Cement Oxide Analysis - Type I/II Cement

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
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 Na₂O equivalent alkali content was 0.5%

The aggregates used were locally available crushed limestone and sand used in commercial concrete. The maximum nominal aggregate size was 3/4 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.

Table 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

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.

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 3 shows the mixture design proportions.

Table 3. SSD Mixture Proportions

w/c ratio	Paste Content (%)	Water lb/yd³	Cement lb/yd³	Coarse lb/yd³	Fine lb/yd³
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, and 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 were 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 3/4 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 had been lapped to the same elevation, and that there was a high quality finish on the specimen. After the specimen had received an acceptable polish, 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 three hours. A second coat of black marker was then applied in the perpendicular direction to the first coat and the sample dried for eight hours. A thin layer of barium sulfate, a white powder with a particle size less than 3.94×10^{-5} in. ($< 1 \mu\text{m}$), 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 were white and all other areas in the sample were black. A sufficiently polished sample and a finished sample can be seen in Figures 1 and 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).

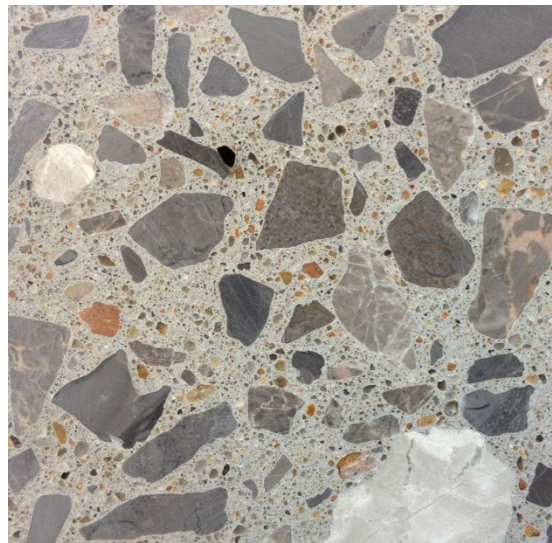


Figure 1. Satisfactorily lapped sample

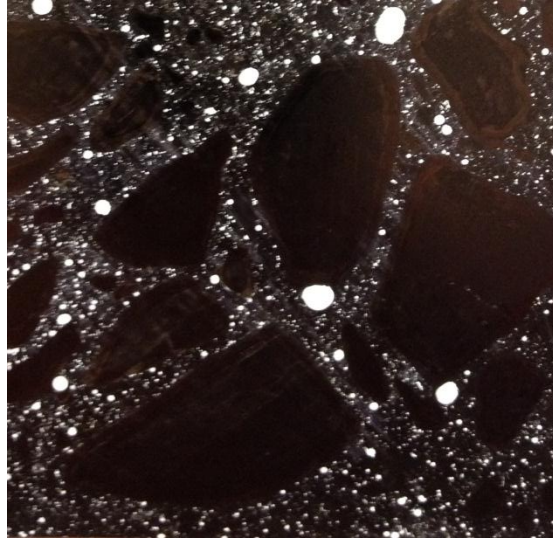


Figure 2. Finished sample

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, which 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, Ramezani pour and Hooton 2010).

RESULTS

The results have been separated into two different groups. Table 4 shows the mixtures made with three types of AEAs at different w/cm ratios. Table 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 4 and 5 show C 231 and C 457 concrete air contents.

Table 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

* Numbers in parentheses indicate freezing and thawing cycles completed when dynamic modulus was measured below 80

± symbol gives the range of values seen by multiple beams of the same mixture

Table 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

* Numbers in parentheses indicate freezing and thawing cycles completed when dynamic modulus was measured below 80

± symbol gives the range of values seen by multiple beams of the same mixture

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 3 and 4 show percent expansion and percent mass change for different air contents.

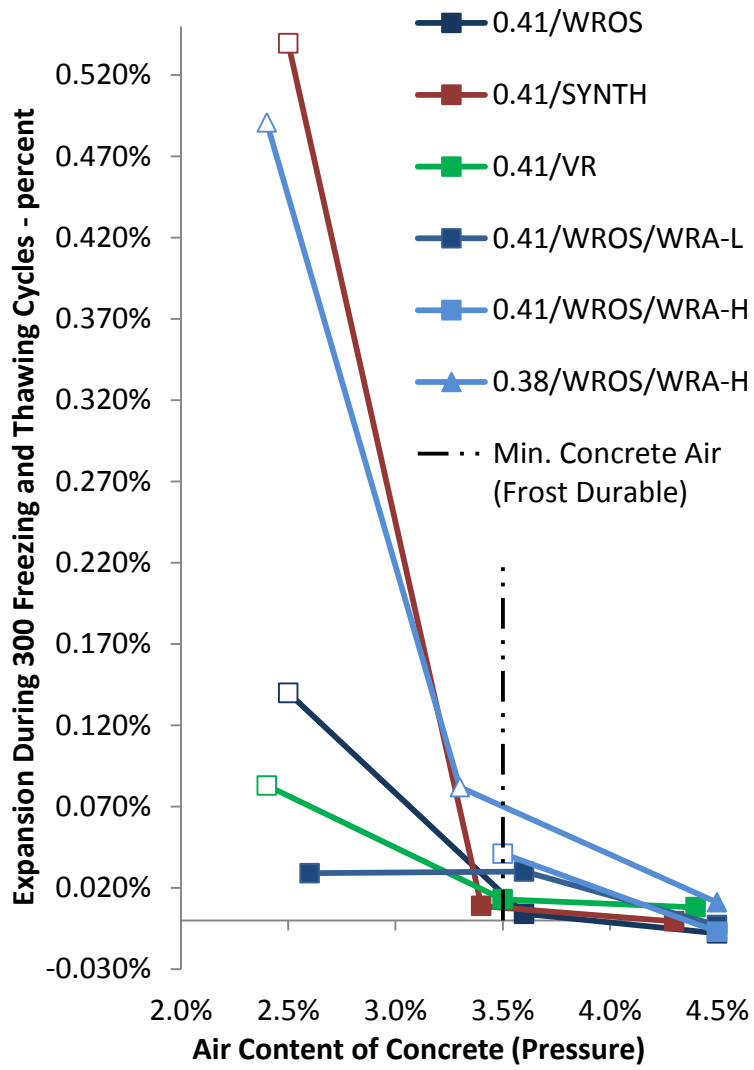


Figure 3. Measured percent expansions

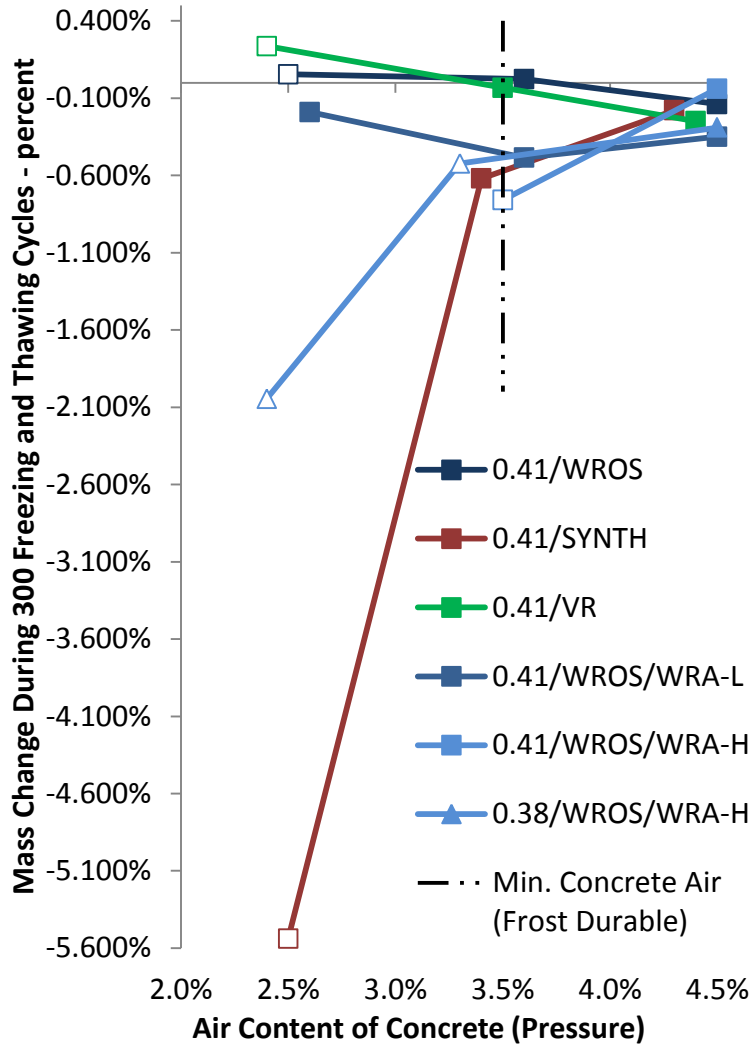


Figure 4. Measured percent mass change

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 5 and 6 relative to C 231 concrete air contents and calculated paste air contents.

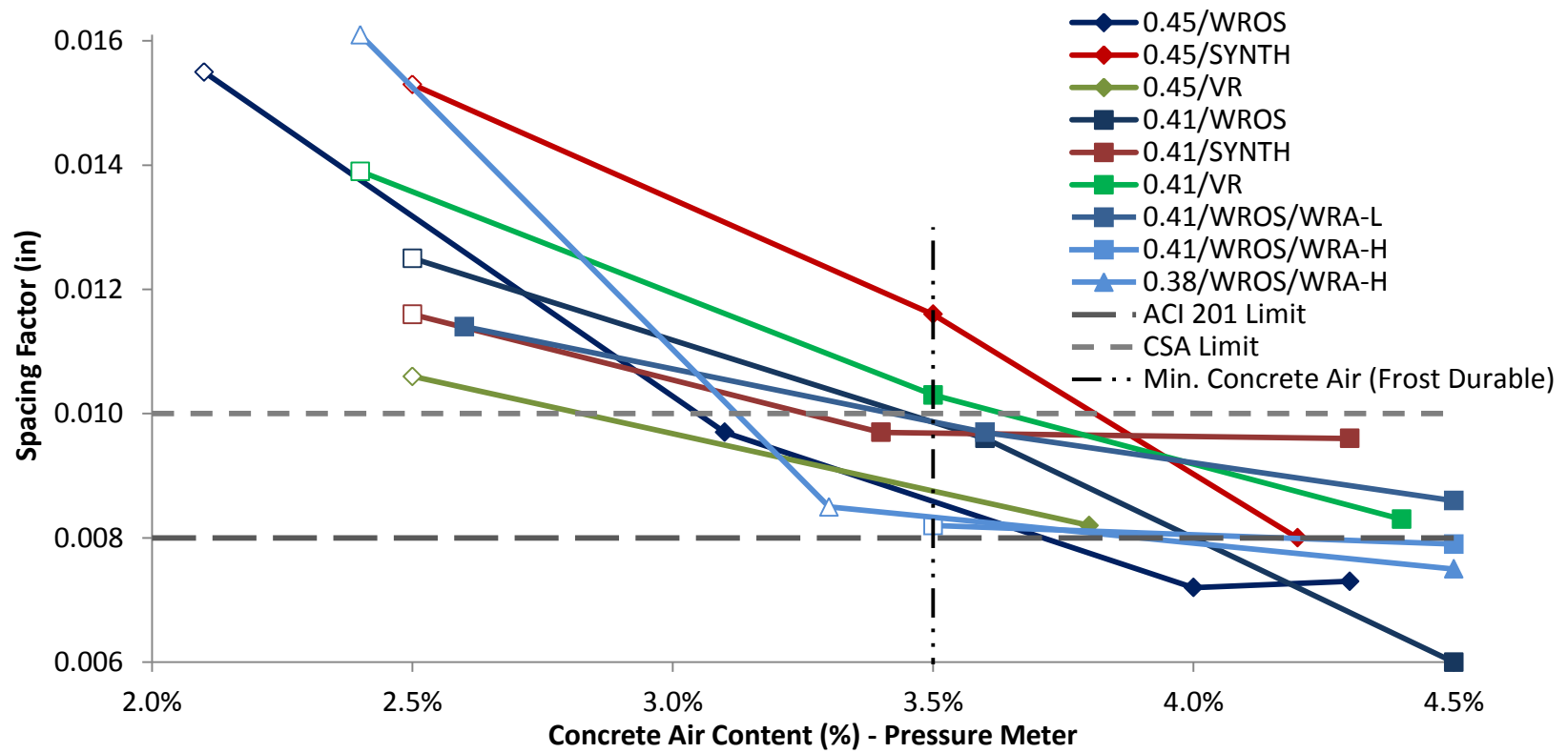


Figure 5. Concrete air contents measured by pressure meter and spacing factor for all mixtures

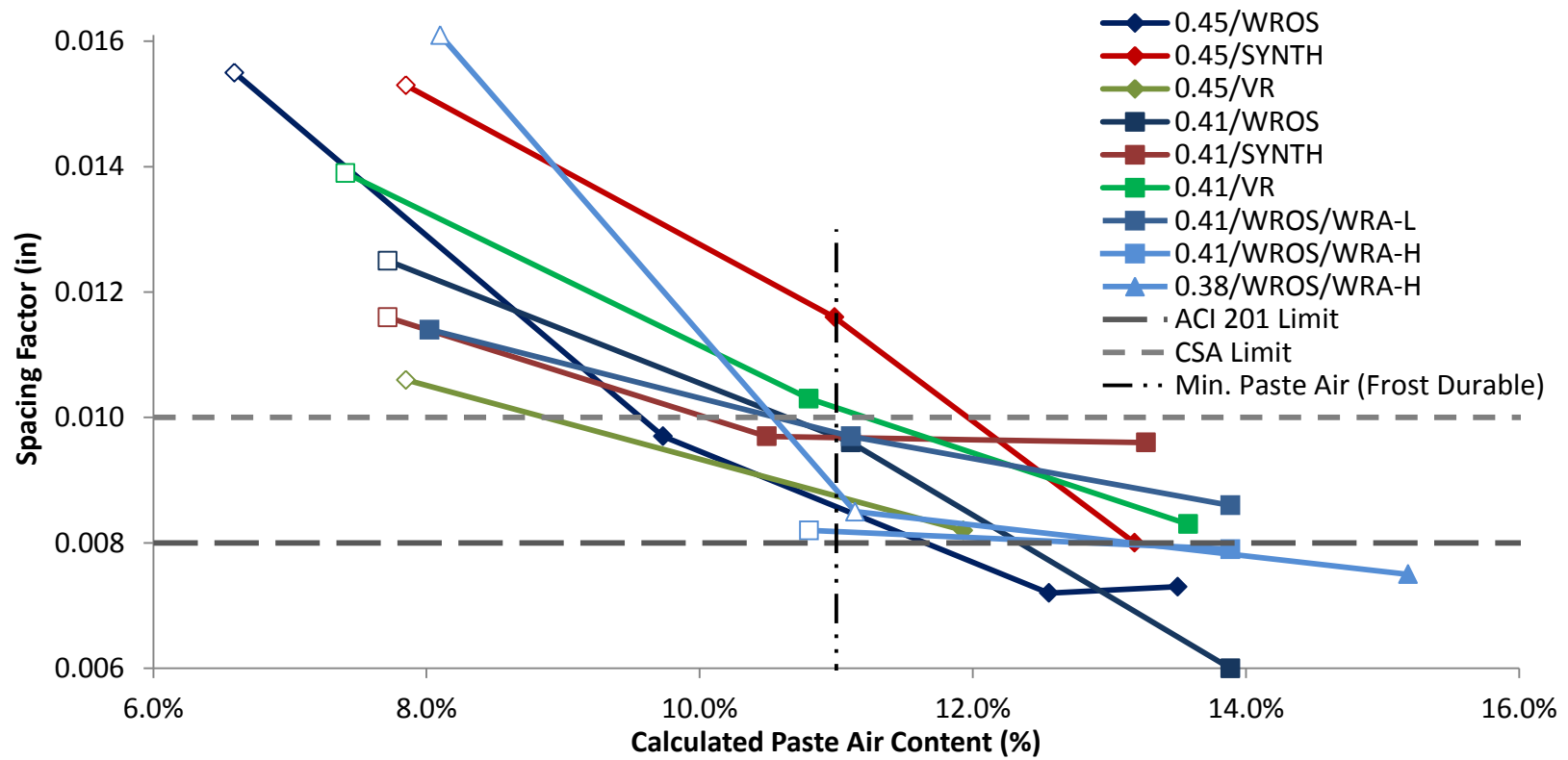


Figure 6. Concrete paste air contents calculated from C231 pressure meter readings and spacing factor for all mixtures

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 7 and 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 $600 \text{ in.}^2/\text{in.}^3$ 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.

Mixtures made with and without water reducer are shown in Figure 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 0.010 in. 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.

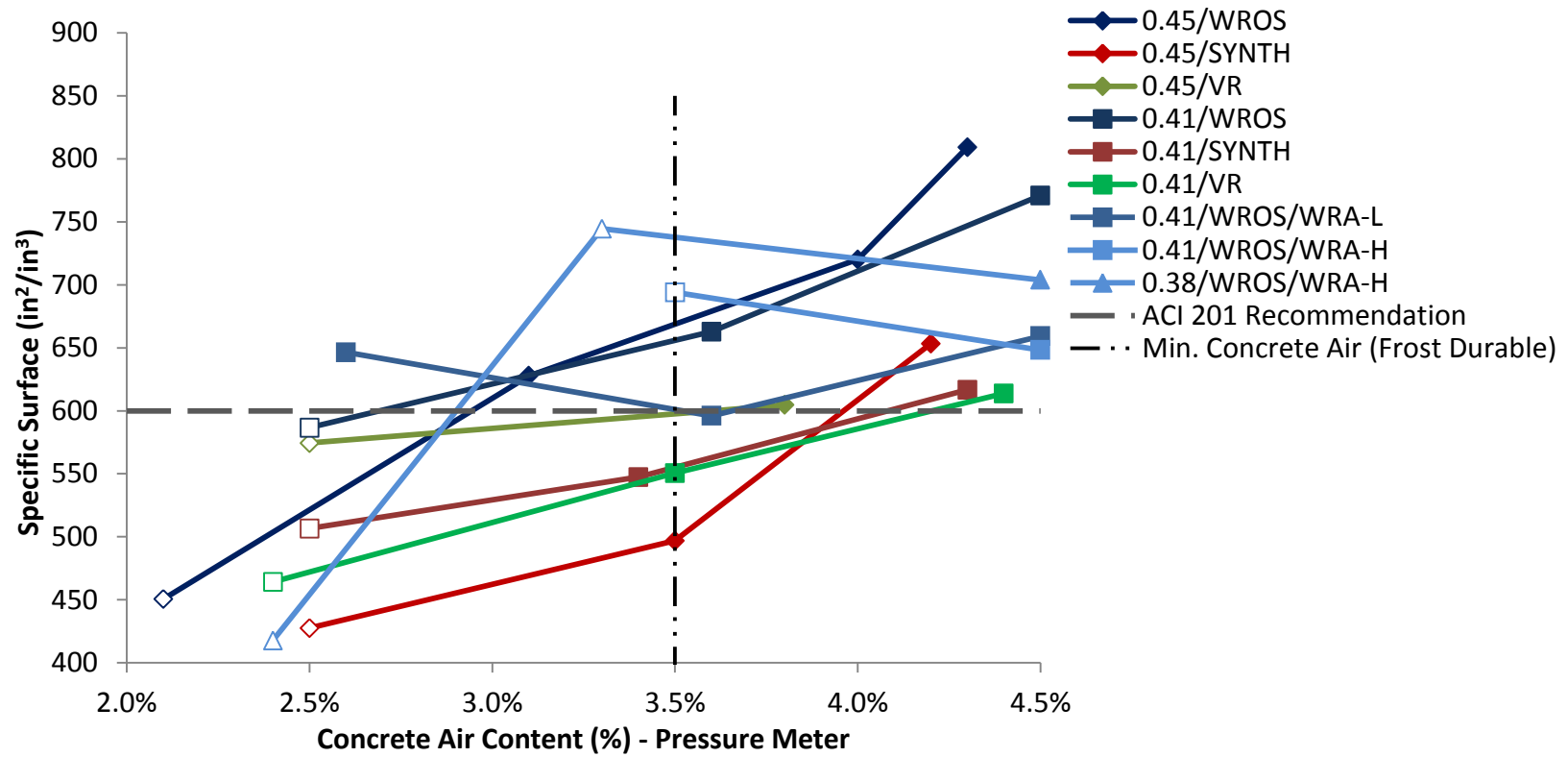


Figure 7. Concrete air contents measured by pressure meter and specific surface for all mixtures

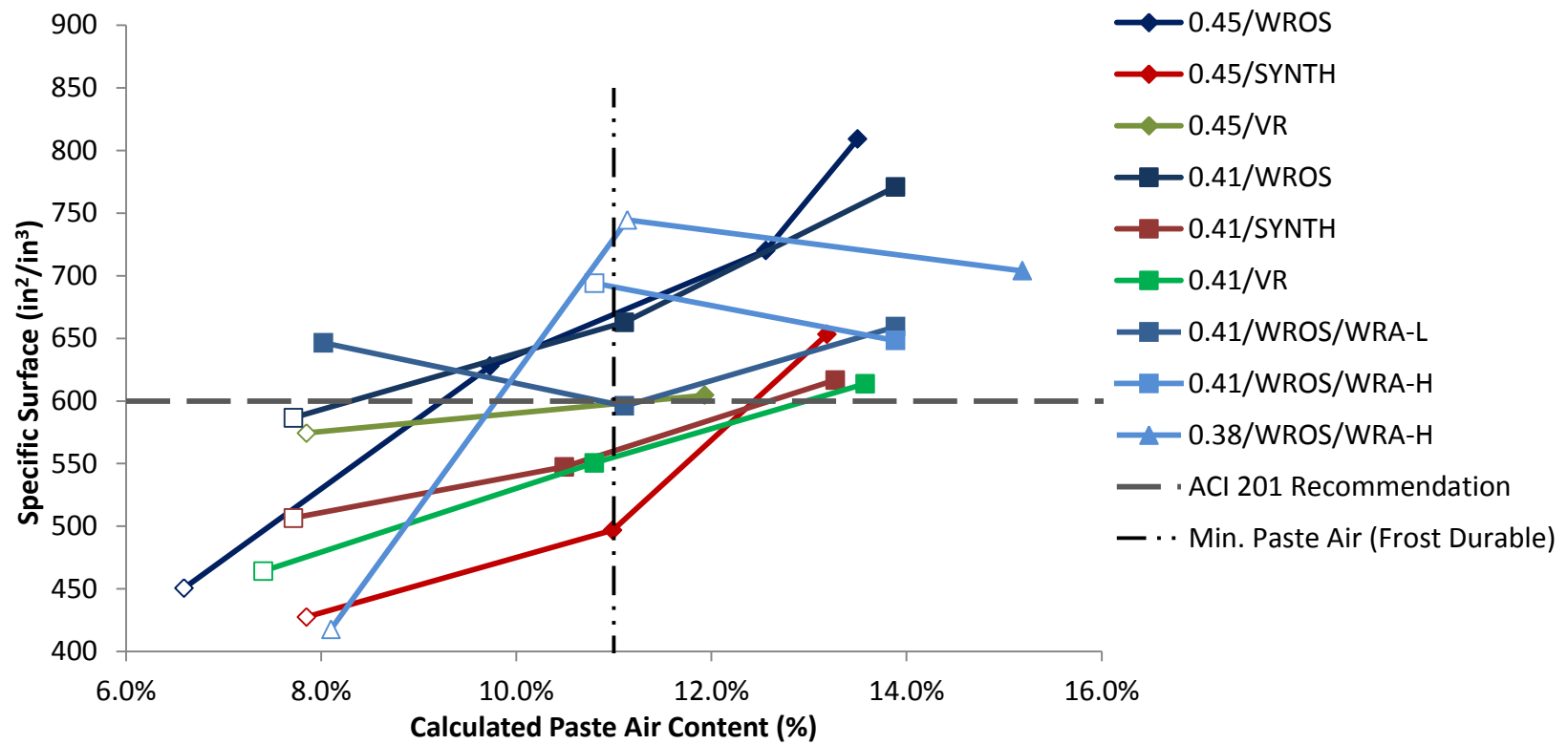


Figure 8. Concrete paste air contents calculated from C231 pressure meter readings and specific surface for all mixtures

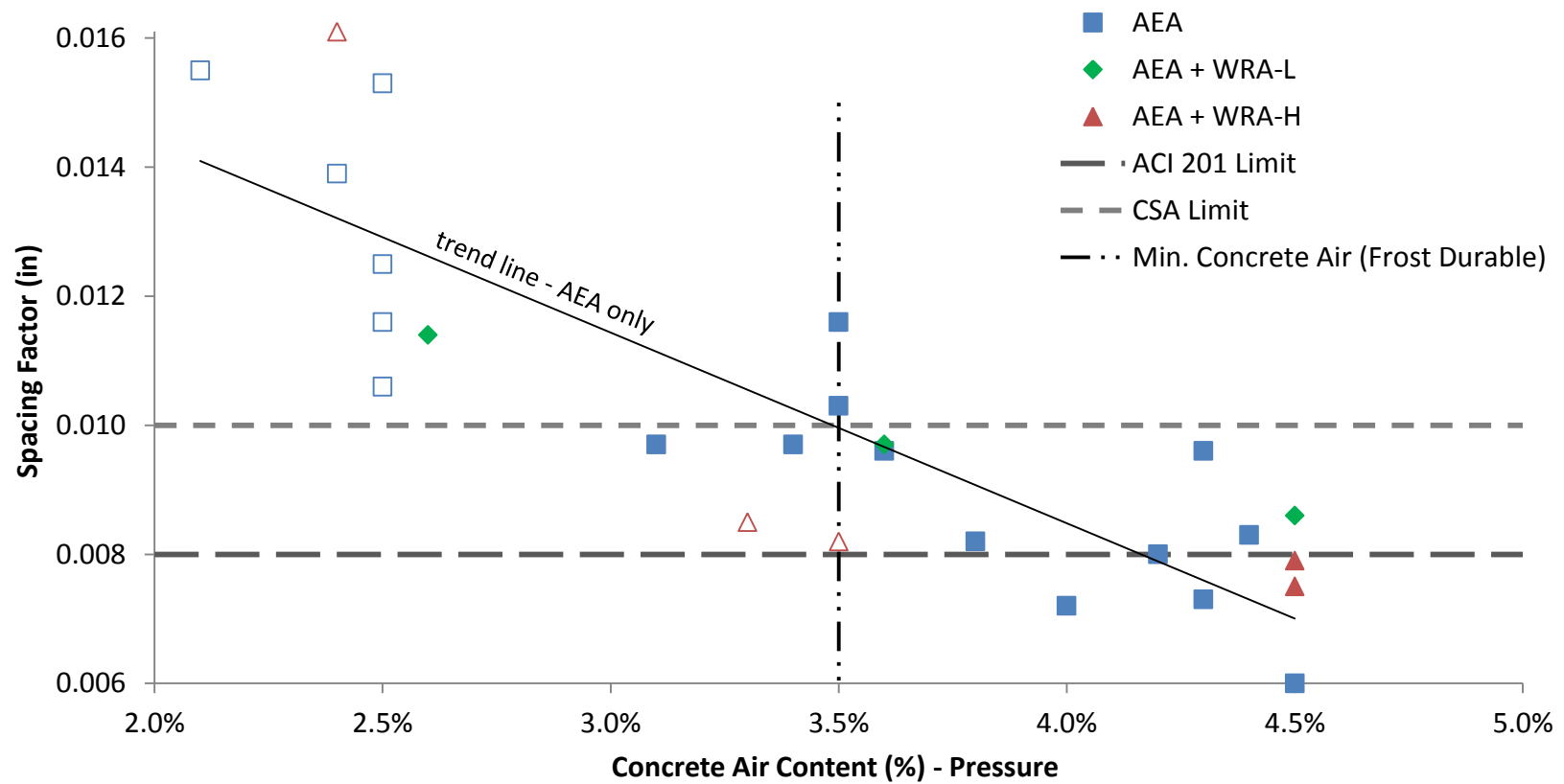


Figure 9. Spacing factors versus C 231 concrete air contents for mixtures with and without water reducer

DISCUSSION

Required Air Content for Frost Durable Concrete

Figures 3 through 8 show 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 and 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, Figures 5 and 6 would show that the synthetic AEA would contain a lower spacing factor, and Figures 7 and 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, 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 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 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 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 and 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 3/4 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 than 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.

REFERENCES

- Carlson, J., Sutter, L., Peterson, K., Van Dam, T. "An update on application of a flat-bed scanner for performing ASTM C 457," Proceedings of the 27th International Conference on Cement Microscopy, Victoria, British Columbia, Canada, 2005.
- Gay, F. T., "A Factor Which May Affect Differences in the Determined Air Content of Plastic and Hardened Air-Entrained Concrete," Proceedings of the Fourth International Conference on Cement Microscopy, Las Vegas, Int. Cem. Microscopy Assoc., 286-292, 1982.
- Gay, F.T., "The Effect of Mix Temperature on Air Content and Spacing Factors of Hardened Concrete Mixes with Standardized Additions of Air-Entraining Agent," Proceedings, Seventh International Conference on Cement Microscopy, Fort Worth, International Cement Microscopy Assoc., Duncanville, TX, pp. 305-315, 1985.
- Jakobsen, U. H., Pade, C., Thaulow, N., Brown, D., Sahu, S., Magnusson, O., De Buck, S., and De Schutter, G., "Automated air void analysis of hardened concrete - a round robin study," Cement and Concrete Research, Vol. 36, pp. 1444-1452, 2006.
- Jana, D., Erlin, B., Pistilli, M., "A Closer Look at Entrained Air in Concrete," Concrete International, V27, 07, July 2005.
- Klieger, P., "Effect of Entrained Air on Strength and Durability of Concrete Made with Various Maximum Sizes of Aggregate," Proceedings, Highway Research Board, Vol. 31, 1952, pp. 177-201; Bulletin No. 40, Research and Development Laboratory, Portland Cement Association, Skokie, IL.
- Klieger, P., "Further Studies on the Effect of Entrained Air on Strength and Durability of Concrete with Various Sizes of Aggregate," Proceedings, Highway Research Board, 1956, pp. 16-17; Bulletin No. 77, Research and Development Laboratory, Portland Cement Association, Skokie, IL.
- Ley, M. T., "The Effects of Fly Ash on the Ability to Entrain and Stabilize Air in Concrete," Dissertation, University of Texas at Austin, August 2007a.
- Ley, M. T., Folliard, K. J., Hover, K.C., "Observations of Air-Bubbles Escaped from Fresh Cement Paste," Cement Concrete Research, doi:10.1016/j.cemconres.2009.01.019, 2009a.
- Ley, M. T, Chancey, R., Juenger, M., and Folliard, K. J., "The Physical and Chemical Characteristics of the Shell of Air-Entrained Bubbles in Cement Paste," Cement Concrete Research, doi:10.1016/j.cemconres.2009.01.018, 2009b.
- Peterson, K., Sutter, L. and Radlinski, M., "The Practical Application of a Flatbed Scanner for Air-Void Characterization of Hardened Concrete," J. ASTM Intl., Vol. 6, No. 9. doi:10.1520/JAI102446.
- Peterson, K. W., Thaulow, N., Baumgart, C. W., Furuichi, H., and Jana, D., "A Round Robin Test on Measurements of Air Void Parameters in Hardened Concrete by Various Automated Image Analyses and ASTM C 457 Methods," Proceedings of the twenty-ninth conference on cement microscopy, Quebec City, PQ, Canada, 2007.
- Pigeon, M., and Pleau, R., "Durability of Concrete in Cold Climates," London; New York: E & FN Spon, 1995.
- Plante, P., Pigeon, M., and Saucier, F. (1989). "Air-Void Stability, Part ii: Influence of Superplasticizers and Cement," *ACI Materials Journal*, 581-589.

- Powers, T. C., with discussion by Willis, T. F., "The Air Requirement of Frost Resistant Concrete," Proceedings, Highway Research Board, vol. 29, 1949, pp. 184–211; Bulletin No. 33, Research and Developments Laboratories of the Portland Cement Association.
- Powers, T. C., "Void Spacing as a Basis for Producing Air-Entrained Concrete," ACI Journal, Proc. V. 50, (1954) 741-760.
- Powers, T. C., "Void Spacing as a Basis for Producing Air-Entrained Concrete," ACI Journal, Part 2, Proc. V. 50 (1954) 760-6-760-15.
- Ramezaniapour, A. M., and Hooton, R. D. (2010). "Evaluation of Two Automated Methods for Air-Void Analysis of Hardened Concrete. Journal of ASTM International, 7(2), http://www.astm.org/DIGITAL_LIBRARY/JOURNALS/JAI/PAGES/JAI102476.htm.
- Rashed, A. I., Williamson, R.B., "Microstructure of Entrained Air Voids in Concrete: Part I," J. Mater. Res. 6 (9) (1991a) pp. 2004-2012.
- Rashed, A. I., Williamson, R. B., "Microstructure of Entrained Air Voids in Concrete: Part II," J. Mater. Res. 6 (11) (1991b) pp. 2474-2483.
- Scherer, G. W., and Valenza II, J. J., "Mechanisms of Frost Damage," pp. 209-246 in Materials Science of Concrete, Vol. VII, eds. J. Skalny and F. Young (American Ceramic Society, 2005).

Standards and Specifications

- American Concrete Institute (ACI) (2008). "Guide to Durable Concrete." ACI Manual of Concrete Practice—Part 1. ACI 201.2R-08. American Concrete Institute, Farmington Hills, MI.
- ASTM C 33-03, "Standard Specification for Concrete Aggregates," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 138, "Standard Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 143/C 143M-03, "Standard Test Method for Slump of Hydraulic Cement Concrete," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 150, "Standard Specification for Portland Cement, American Society for Testing and Materials," West Conshohocken, Pennsylvania.
- ASTM C 231-04, "Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 494-05, "Standard Specification for Chemical Admixtures for Concrete," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 666-03, "Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- ASTM C 1017-07, "Standard Specification for Chemical Admixtures for Use in Producing Flowing Concrete," American Society for Testing and Materials, West Conshohocken, Pennsylvania.
- CSA A23.2-09 "Test Methods and Standard Practices for Concrete," Canadian Standards Association, Toronto, Canada. 2009.
- European Standard, EN 480-11, "Admixtures for Concrete, Mortar and Grout – Test Methods – Part 11: Determination of Air Void Characteristics in Hardened Concrete."