INVESTIGATION OF THE FREEZE THAW DURABILITY OF ALTERNATIVE CEMENTITIOUS MATERIALS

By

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INVESTIGATION OF THE FREEZE THAW DURABILITY OF ALTERNATIVE CEMENTITIOUS MATERIALS

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Abstract: Currently there is little known about the long-term freeze thaw durability of ACMs. There are no established limits for air content or other durability parameters for most ACMs. For ACMs to be used in structural and transportation structures such as roads, buildings, and bridges with long design service lives in climates that experience freezing and thawing, limits and specifications for long-term durability must be determined. The work presented investigates the requirements for different ACMs to achieve satisfactory results in both ASTM C666 and ASTM C672. From this testing recommendations are made for minimum requirements to achieve durable concrete using ACMs.

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CHAPTER I

INTRODUCTION

Alternative cementitious materials (ACMs) have been used for decades in specialized applications and as repair material. To be considered for use in structural or paving applications long term durability is very important and must be understood. Over 60 years of research has been done on Portland cement (OPC) concrete to determine and set standards to ensure long-term durability in both bulk freezing and thawing and salt scaling. The ability to meet these durability requirements is not widely known for many of the current ACMs in use today. Testing to determine if an ACM has the ability to pass current durability standards must be determined before widespread use of these materials is allowed in critical projects exposed to freezing conditions and deicing chemicals.

Many ACMs are often referred to as fast setting cements. Depending on the intended use, this can be either a desired quality or a hindrance. Many projects require an ample amount of time for placement and finishing. To address this, citric acid was added to many of the ACMs to approximate the setting time of OPC. Modifying set time could cause strength concerns so strength data was collected to ensure that a sufficient strength could still be achieved with extended set time.

This work is part of a much larger project sponsored by the FHWA – Exploratory Advanced Research program. The focus of this project, led by Georgia Tech, The Army Corps of Engineers, Tourney Consulting, and Oklahoma State University, is to investigate these materials in a number of durability tests. This document specifically investigates the bulk freeze thaw and salt scaling performance in standardized ASTM tests. This created several new insights and developed a number of possible mechanisms.

CHAPTER II

PERFORMANCE OF ACMS IN FREEZE THAW DURABILITY TESTING

2.0 INTRODUCTION

The requirements for OPC concrete for strength and durability are well known and understood. Specifications exist to address durability and strength concerns. Strength gain rates, ultimate strength, and durability requirements such as air content and spacing factor have been widely discussed in previous publications.

To consider using an ACM for a structure with a long design life where freezing and thawing cycles are experienced then one must reexamine these parameters. Since both ACMs and OPC are hydraulic ceramics that are exposed to water and outside chemicals while experiencing freeze thaw cycles, it is logical that similar material properties can describe the performance of both. However, it is also logical that the critical values may be different as the strength, pore structure, total porosity, and possibly some other critical parameters are not the same for these materials.

To examine the freeze thaw durability of ACMs their performance in bulk freeze thaw durability (ASTM C 666) and surface scaling (ASTM C 672) was investigated. Performing these tests is done to experimentally determine what material properties are needed to provide comparable performance to OPC.

Compressive strength (ASTM C 39) gain from 2 d to 56 d is used to compare the relative strength gain between the ACMs. Not all ACMs are fast setting or high early strength. It is also helpful to understand how the increase in air content impacts the strength of these materials. This information is helpful to quantify the rate of strength gain of these different materials.

Ultimately, the work aims to make relative comparisons between all of these measurements for OPC and the ACMs in order to find if consistent freeze thaw performance can be obtained.

2.1 MATERIALS

The concrete mixtures were prepared using a Type I/II OPC, blended calcium sulphoaluminate cement (CSA2), blended calcium sulphoaluminate cement with polymer (CSA2B), blended calcium aluminate cement (CAC2), calcium aluminate cement (CAC3), and alkali activated fly ash (AA1). The oxide analysis is shown in Table 1 below.

Table 1: Chemical composition of binders with bulk XRF (% weight)

Binder type	OPC	CSA2, CSA2B	CAC2	CAC3	Fly Ash for AA1
SiO_2	17.39	14.24	14.95	5.50	38.24
Al_2O_3	4.87	14.84	12.03	45.16	17.87
Fe_2O_3	4.71	1.12	2.66	6.90	5.88
CaO	65.15	49.23	55.15	37.68	24.75
MgO	1.40	1.55	2.57	0.22	6.24
SO_3	2.51	13.55	7.72	0.07	1.56
K_2O	0.48	0.67	0.83	0.26	0.34
Na_2O	0.46	0.21	0.28	0	1.85
P_2O_5	0.13	0.11	0.14	0.09	-
TiO_2	0.39	0.70	0.51	2.11	-
Mn_2O_3	0.11	0.02	0.16	0.02	-
SrO	0.15	0.20	0.21	0.04	-
ZnO	0.03	0.01	0.11	0	-
Cr_2O_3	0.09	0.05	0.07	0.089	-
LOI	2.12	3.51	2.61	1.86	0.20

The aggregates used were a crushed granite from Georgia and natural sand from Michigan. All collaborators shared these materials and so the results are comparable. The maximum nominal aggregate size was (¾ in), and the sand had a fineness modulus of 3.04. Both the rock and sand met ASTM C 33 "Standard Specification of Concrete Aggregates". Table 2 shows the details of the mixtures investigated and Table 3 has information about the admixtures.

Table 2: SSD Mixture Proportions

Cement Type	w/cm	Cement lb/yd³	Paste Volume (%)	Coarse lb/yd³	Fine lb/yd³	Water lb/yd³	Admixtures Used
CSA2	0.42	765	35.7	1789	1121	319	SYNTH, WRA, RETA
CSA2B	0.42	765	35.7	1789	1121	319	POWDER, WRA, RETA
CSA2B .35	0.35	765	32.8	1873	1173	268	POWDER, WRA, RETA
CAC2	0.42	765	34.8	1789	1181	318	SYNTH, WRA, RETA
CAC2 .35	0.35	765	31.8	1880	1235	268	SYNTH, WRA, RETA
CAC3	0.42	765	34.8	1780	1163	319	WROS,RETB
AA1	0.22	822	31.4	1780	1354	183	SYNTH, ACT
OPC	0.42	765	34.5	1789	1163	318	SYNTH, WRA

Table 3: Admixture Descriptions

ID	Description	Application
WROS	Wood rosin	Air Entrainer
SYNTH	Synthetic chemical combination	Air Entrainer
Powder	Long-chain olefin sulphonate	Air Entrainer
WRA	Polycarboxylate	Highrange water reducer
RETA	Citric acid	Retarder
RETB	Proprietary retarder	Retarder
ACT	Alkali activator	Activator

Mixture designs with constant cement weight and three different w/cms were tested. Citric acid was used in varying dosages to give all cements approximately the same set time. The synthetic air entrainer and the high range water reducer were chosen because they worked with nearly all of the cements. CAC3 would not entrain air with a synthetic air entrainer so a wood rosin air entrainer was used instead. CSA2B used the same powdered air entrainer that is typically pre blended into the cement, but it was added separately in varying dosages to produce the desired air contents. The 0.42 w/cms was used as a baseline mixture that represented a typical concrete mixture used for bridge decks. A water reducer was added to give all mixtures approximately the same slump. The AA1 mixture uses a two part chemical activator that reacts with the fly ash in the material. Water is added to this material to increase the slump. A w/cm of 0.22 was used in AA1 as this created a comparable slump to the other mixes. Mixes for CSA2B and CAC2 were created with a 0.35 w/cm to investigate the performance of a lower water content. This will be discussed in more detail later in the document.

2.2 CONCRETE MIXTURE PROCEDURE

Aggregates are collected from outside storage piles, and brought into a temperature-controlled room at 73°F for at least 24 h before mixing. Aggregates were placed in the mixer and spun and a representative sample was taken for a moisture correction. At the time of mixing, citric acid was added to the water if used. Citric acid was used as a set retarder for these mixtures. Next, all aggregates were loaded into the mixer along with approximately half of the mixing water. This combination was mixed for three minutes to allow the aggregates to saturate and ensure that the aggregates were evenly distributed.

Next, the cement and the remaining water was added and mixed for three minutes. If an alkali activator was being used it was added to the mixture at the same time as the cement. The resulting mixture rested for two minutes while the sides and paddles of the mixing drum were scraped. After the rest period, the mixer was turned on and admixtures were introduced. If a water-reducer was used then it was incorporated into the mixture 30 s before the AEA was added. After the addition of the AEA the concrete was mixed for three minutes.

2.3 SAMPLING AND TESTING

After mixing, the material was tested for slump (ASTM C 143), unit weight (ASTM C 138), and SAM (AASHTO TP 118). Next, samples were prepared for freeze thaw durability testing (ASTM C 666), salt scaling (ASTM C 672), strength (ASTM C 39), and hardened air void analysis (ASTM C 457). For each mixture two ASTM C 666 beams, three ASTM C 672 blocks and an ASTM C 457 sample were cast. Freeze thaw prisms were cured for one day in steel molds while covered with wet burlap. The beams were then demolded and placed in saturated limewater for the remainder of the 14 d curing period, as per ASTM C666. The ASTM C 672 samples were sealed in their containers for 14 d after casting. Next, the blocks were demolded and allowed to dry at 73°F and 50% relative humidity for another 14 d.

The freeze thaw beams were then 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. The dynamic modulus, expansion, and mass change were measured every 36 cycles or before. Measurements were taken as long as dynamic modulus readings were measurable. The dynamic modulus was used to calculate the Durability Factor as per ASTM C 666. ASTM C 666 does not clearly define freeze thaw failure, but based off previous freeze thaw work an acceptable value for the durability factor of 70% was used (Ley 2007). Although many specifications are based on investigating the durability factor after 300 cycles, this work also investigated the total mass loss of the samples. This is important because it is not satisfactory for concrete to lose significant mass during freezing and thawing cycles as this would reduce the cover, member dimensions, and impact the smoothness and ride quality of a bridge deck or pavement. It was decided that one appropriate way to investigate the mass loss is to use the same mass loss criteria for ASTM C 666 that is suggested in the MTO LS-412 version of the ASTM C 672 test. The allowable scaling was determined by comparing the total mass loss to calculated surface area of the formed dimensions. This mass loss was equal to 1.5% of the sample mass. For simplicity, a specimen was determined to fail if the Durability Factor decreased below 70% at any point during the testing or noted as a concern if the mass loss exceeded 1.5%. Figure 1 shows a sample with unacceptable mass loss but a Durability Factor above 90%. This mass loss occurred on concretes made with CSA2B and occurred worse on the side of the sample that was closest to the freezing plate.

Figure 1: CSA2B Mass Loss

The salt scaling blocks were placed in a plastic container and then a silicone seal was placed between the concrete and the form. Drain holes were added to the bottom of the sample to allow any solution that leaked by the seal to drain and not saturate the sample from any side other than the surface. Sample mass, scaled mass, and visual surface ranking were performed every 5 cycles until either a rank of 5 was reached or 50 cycles had been completed. A visual ranking of 4 or higher or if the cumulative scaled mass reached .0182 oz/in² which is equivalent to the scaling threshold set in MTO LS-412 was deemed as failure.

2.4 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 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 lapping machine 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, 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 h. A second coat of black marker was then applied in the perpendicular direction to the first coat and the sample dried for 8 h. A thin layer of barium sulfate, a white powder with a particle size less than 3.94 x 10^{-5} in, 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 and ASTM C 457. 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

Figure 2. This technique is outlined in detail in (Ley 2007) and has been used by several other researchers (Jakobsen et al 2006, Carlson 2005, Peterson et al 2007).

Once the voids in the paste had been preferentially marked the contrast between the voids and the surrounding material is used 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 .0012 in 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).

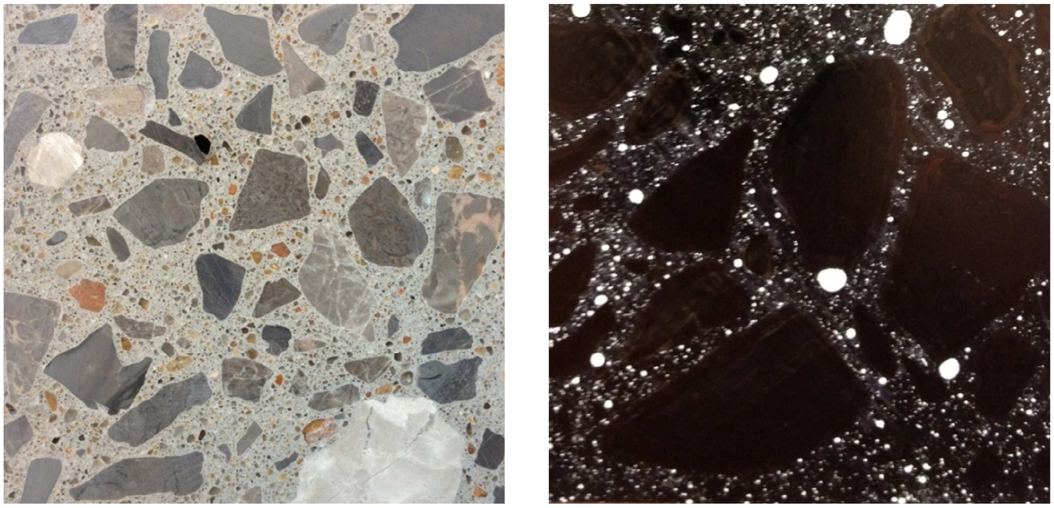

Figure 2: Satisfactory Lapped Sample and Completed Sample

2.5 RESULTS AND DISCUSSION

2.5.1 Strength

To examine the effect of air content and curing time on the compressive strength of the cements, testing was performed at four different time periods. From this testing the effects of air content and curing time on compressive strength are shown in Table 4 below. The slope of the line is the compression strength loss in psi per percent of air. The Y intercept is the theoretical maximum strength of a mixture with 0% air. The slope of the line divided by the theoretical maximum strength is also shown. This is helpful as it gives percent strength change per percent air increase. If mixtures with different theoretical maximum strengths are produced then the same relationship can be used since it is a percentage.

Table 4: Effect of Air Content on Strength

	2 Days	7 Days	28 Days	56 Days	Description
CSA2	-535	-557		-485	Psi change per % air
	9902	10338		12016	Theoretical maximum strength
	0.88	0.87		0.97	R^2
	-5.4%	-5.4%		-4.0%	% Strength change per % air increase
CSA2B	-207	-231	-152	-245	Psi change per % air
	7606	8213	9475	10467	Theoretical maximum strength
	0.95	0.54	0.90	0.83	R^2
	-2.7%	-2.8%	-1.6%	-2.3%	% Strength change per % air increase
CSA2B .35W/C		-136	-158	-194	Psi change per % air
		10180	11279	12326	Theoretical maximum strength
		0.23	0.19	0.22	R^2
		-1.3%	-1.4%	-1.6%	% Strength change per % air increase
CAC2	-144	-69	-393	-295	Psi change per % air
	3904	4786	8916	9277	Theoretical maximum strength
	0.29	0.05	0.67	0.44	R^2
	-3.7%	-1.4%	-4.4%	-3.2%	% Strength change per % air increase
CAC3	-281	-241	-222	-214	Psi change per % air
	9203	10146	10376	9851	Theoretical maximum strength
	0.87	0.64	0.46	0.43	R^2
	-3.0%	-2.4%	-2.1%	-2.2%	% Strength change per % air increase
AA1	-181	-219	-292	-275	Psi change per % air
	4153	6113	7546	7980	Theoretical maximum strength
	0.61	0.48	0.45	0.41	R^2
	-4.4%	-3.6%	-3.9%	-3.4%	% Strength change per % air increase
OPC	-196	-246		-298	Psi change per % air
	5037	6301		8014	Theoretical maximum strength
	0.99	0.99		0.97	R^2
	-3.9%	-3.9%		-3.7%	% Strength change per % air increase

Table 4 above shows that the effects of air content on compressive strength varies significantly depending on the binder. The table shows that CSA2's compressive strength was the most affected by air, while CSA2B .35W/C was the least affected by air. This is graphically shown at 2 and 56 days in Fig. 3 and 4 with each point being an average of 3 samples.

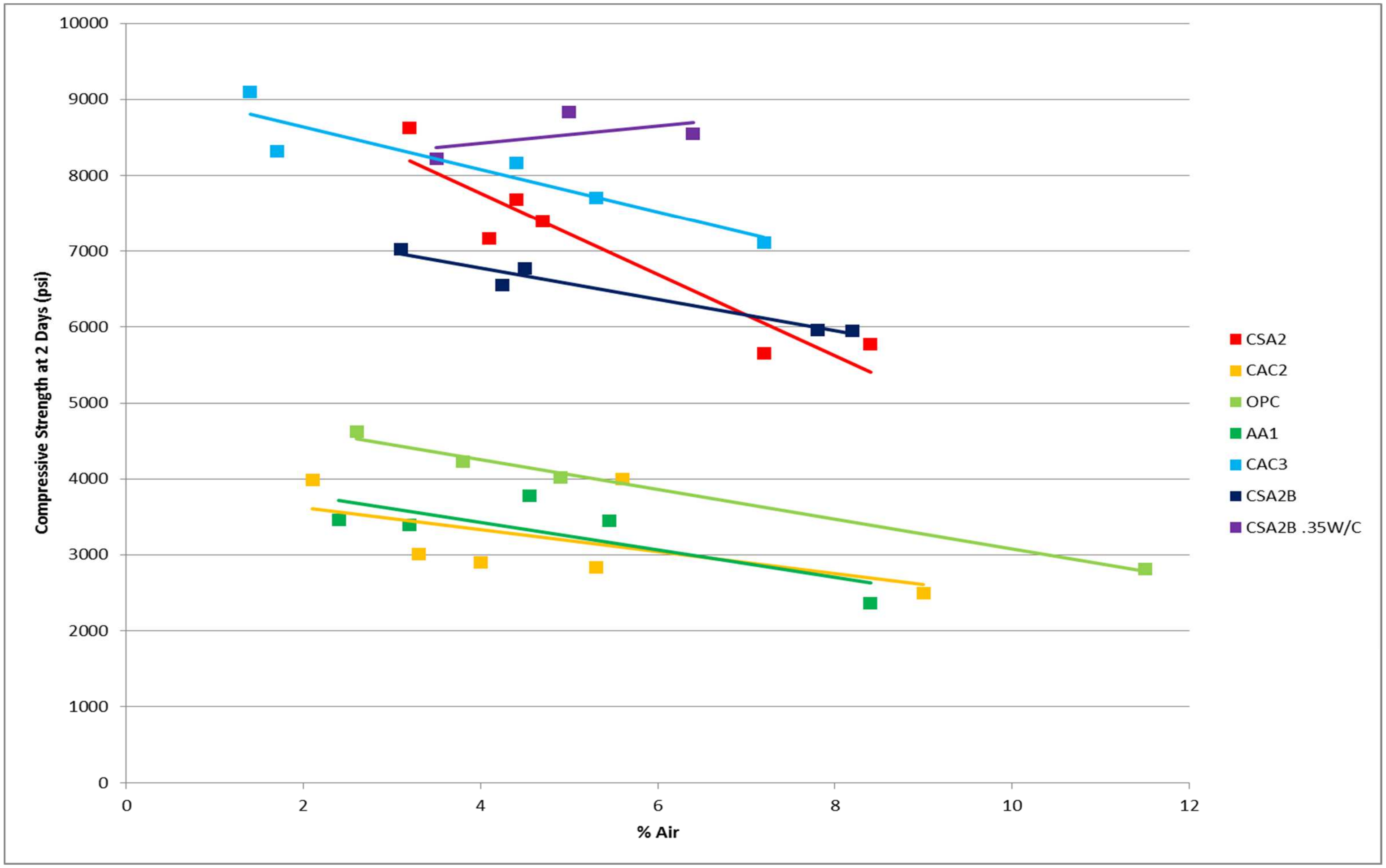

Figure 3: Compressive Strength at 2 Days

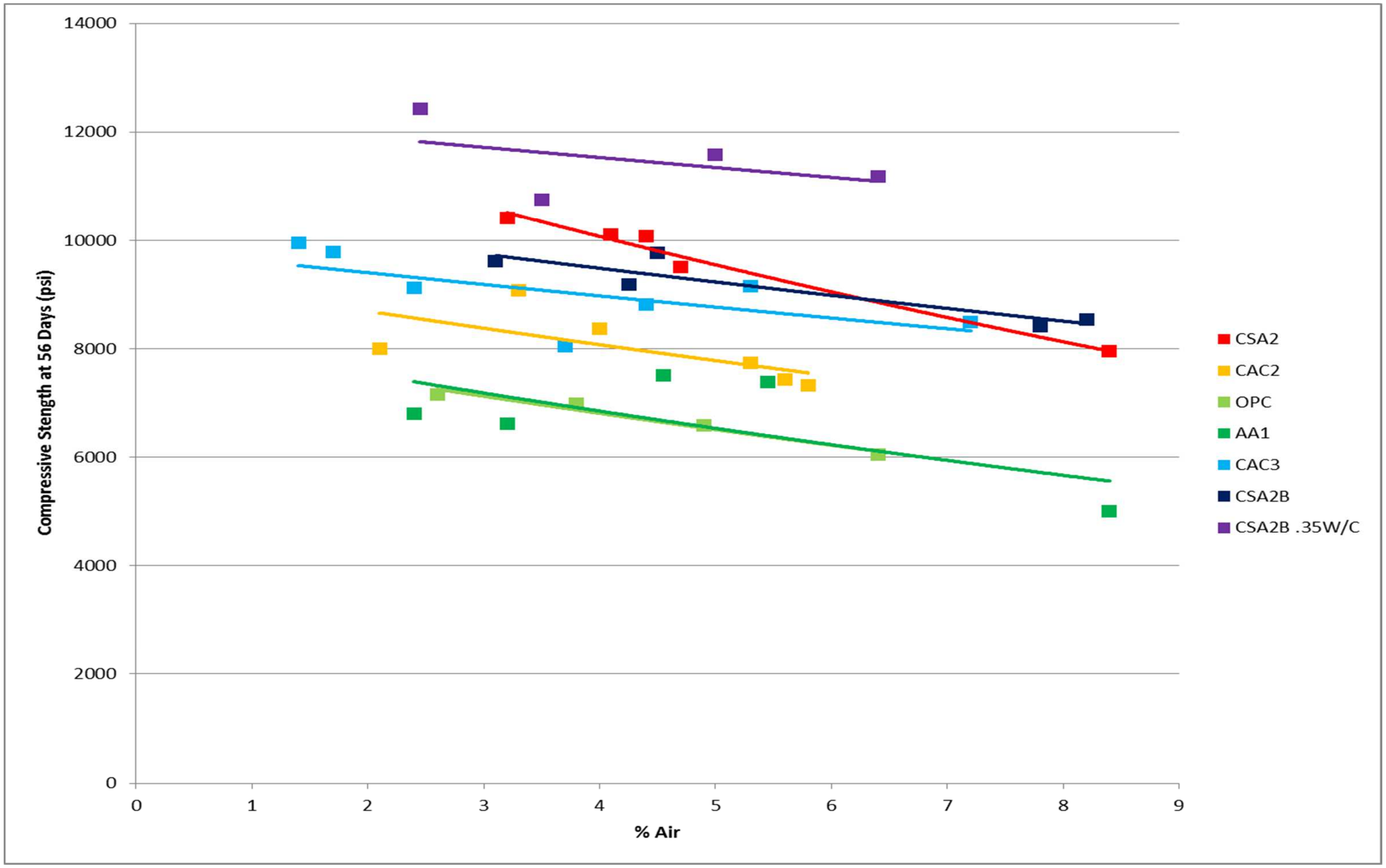

Figure 4: Compressive Strength at 56 Days

Compressive strength gain comparisons were done by finding the percent change for the 7 d, 28 d, and 56 d compressive strength compared to the 2 d compressive strength. This is shown in Figure 5. These percentages were then averaged for all the mixtures of each type of cement. Comparing each mixture to itself removes air content as a factor allowing the different cements to be compared to each other despite each cement having varying air contents. This shows that the range of 2 d strength obtained at 7 d varies from 105% for CSA2 to 155% for AA1. This means that for CSA2 there is very little strength gain from 2 d to 7 d.

The 2 d, 7 d, and 28 d compressive strengths were also compared to the 56 d compressive strength on a percent basis. This is shown in Figure 6. The results range from 42% for CAC2 to 87% for CAC3 meaning that for CAC3 nearly all the strength gain occurs within the first 2 d. The data also shows how CAC3 reaches its maximum compressive strength before 56 days and then starts to loose strength. This is likely caused by conversion, a process where hydrates transform causing a volume reduction resulting in an increase in porosity and a loss of strength.

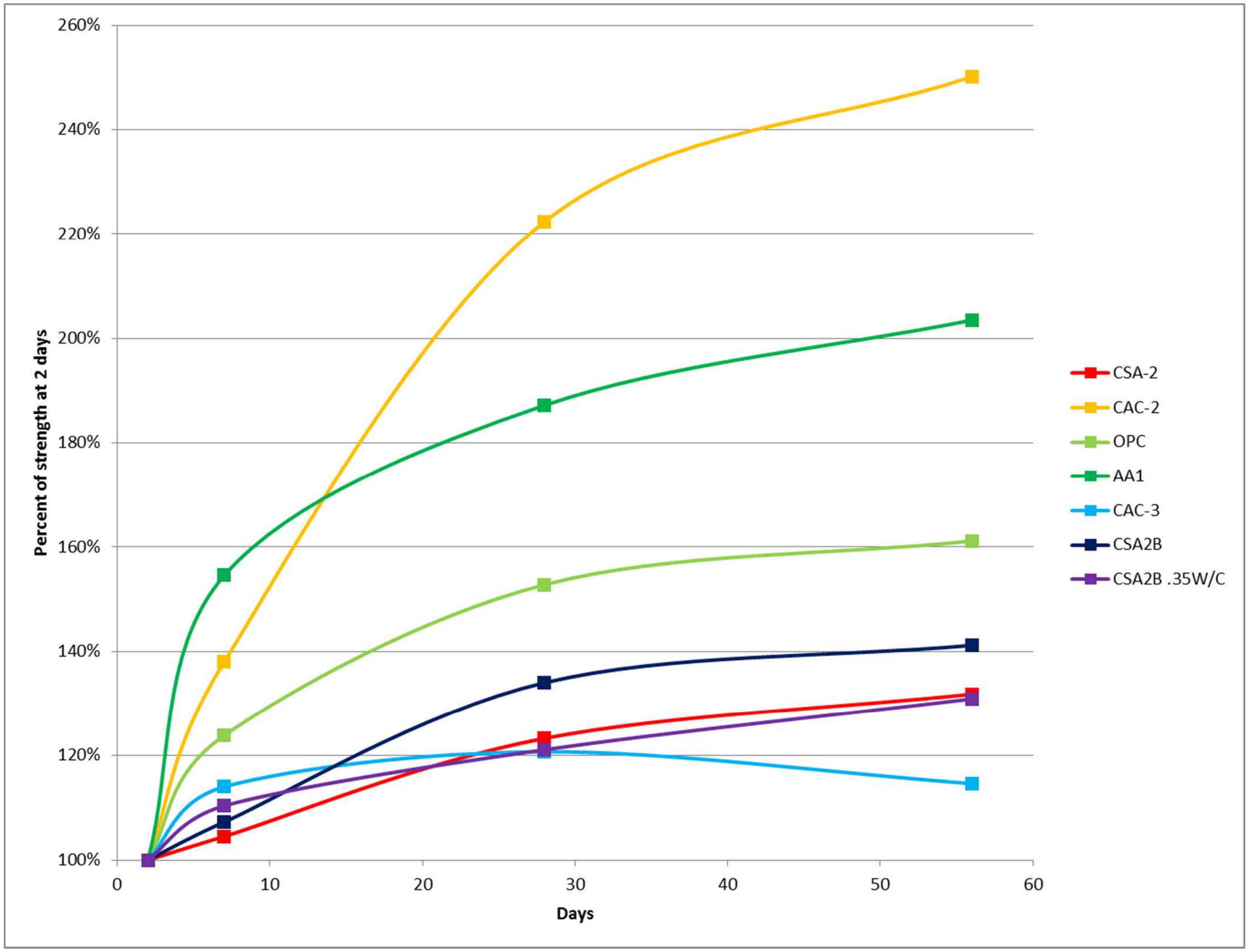

Figure 5: Percent of Strength at 2 Days

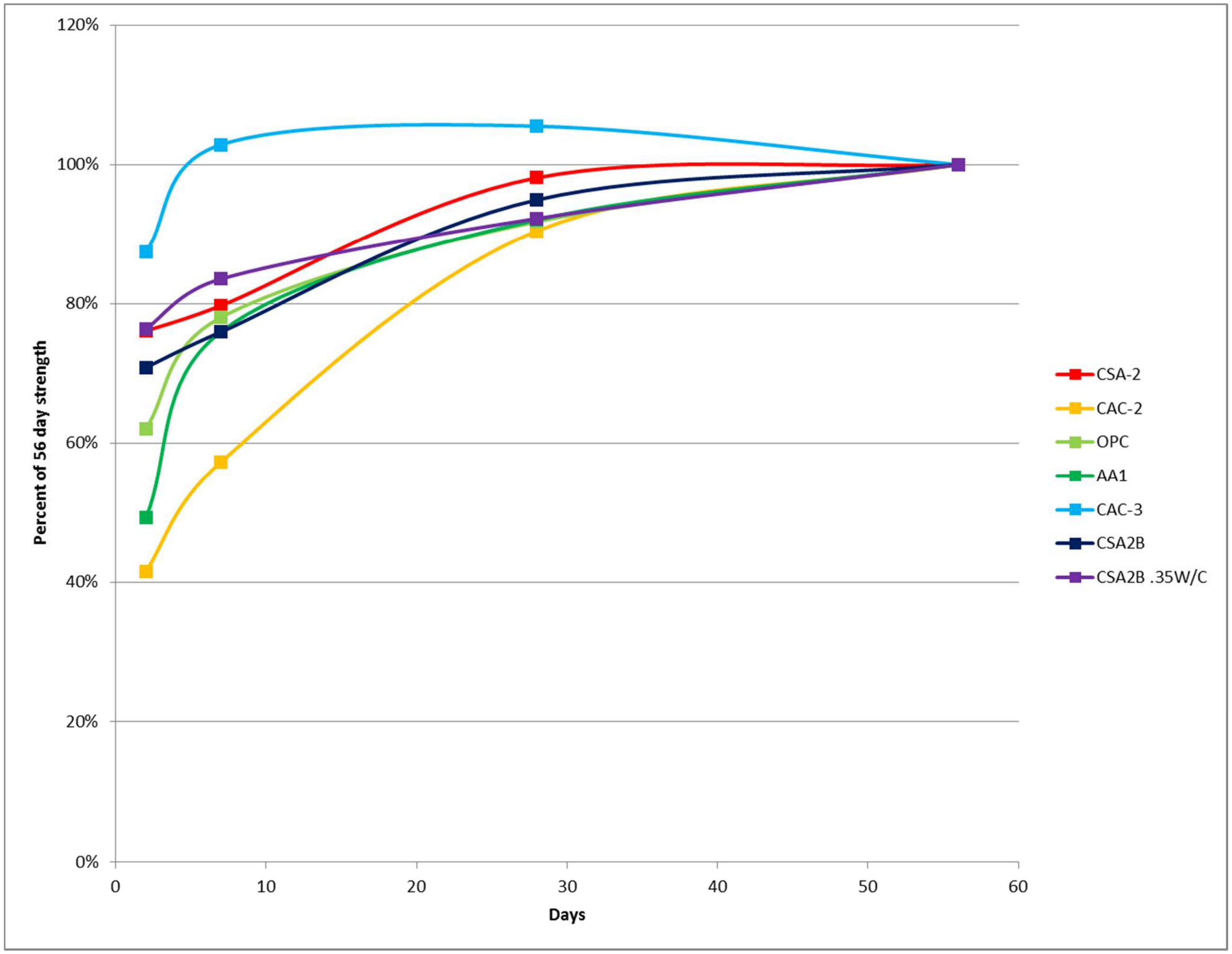

Figure 6: Percent of Strength at 56 Days

2.5.2 ASTM C672

Many of the samples did not make it to 50 cycles in the salt scaling test. For these samples the number of cycles is noted and the cumulative scaled mass loss is reported through the last completed cycle. The results are shown in Fig. 7-11. Figures 7-10 show the different forms of scaling that some of the cements experienced. Figure 11 graphically shows scaling vs air content. The results showed a surprising finding. Based on previous research for OPC, there is consistently improved performance in the salt scaling test with increased air contents. But many of the ACMs did not show this same performance. Some ACMs performed better at low air contents in the scaling test. For example CAC2 and CAC3 showed better scaling performance when the air content was below 3% and 6% respectively. Other ACMs performed better at moderate air contents. AA1 showed better performance for air contents between roughly 4% and 6% while CSA2B had the best performance between 2% and 4%. CSA2B .35W/C showed no significant difference in scaling performance over the range of air contents investigated. The w/cm of the mixture appears to be very important for some of these ACMs.

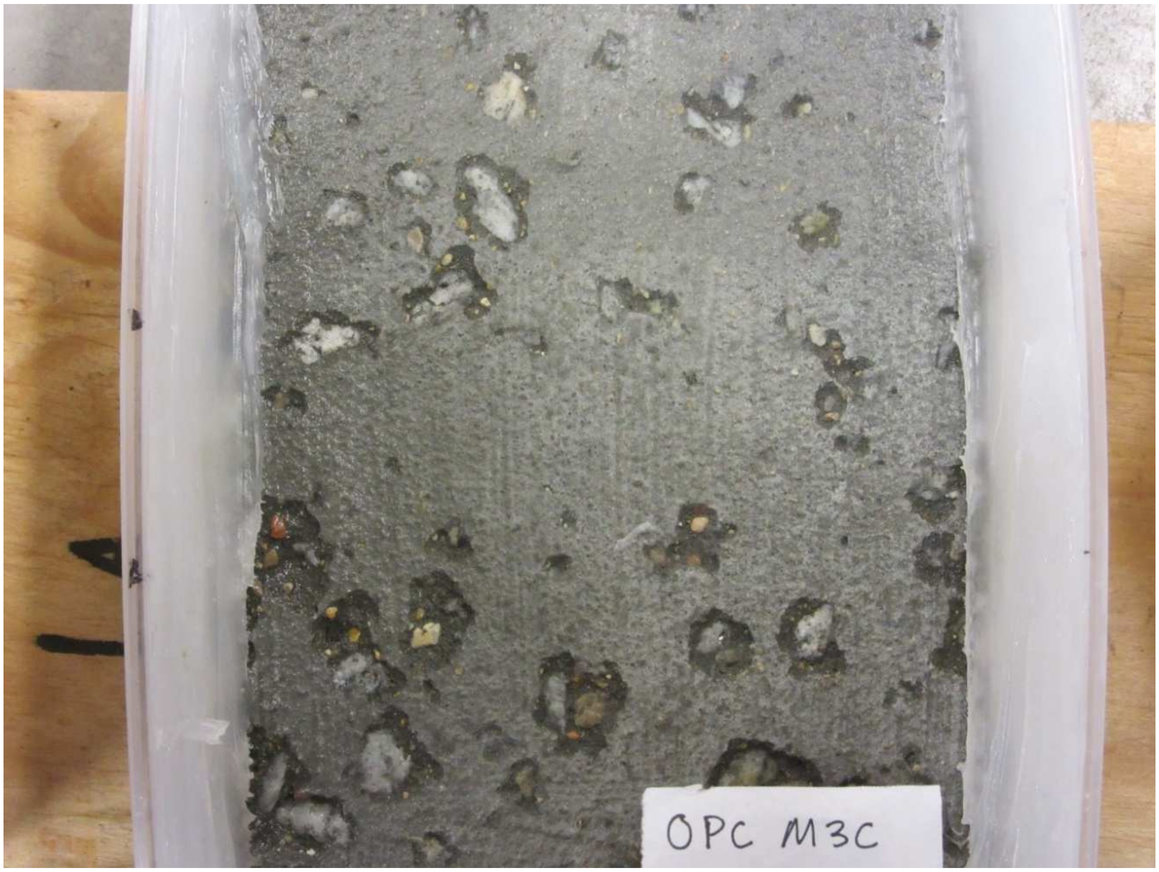

Figure 7: OPC Sample with Visual Rank of 3

OPC sample with 3.8% air after 50 cycles with a visual rank of 3. There is noticeable scaling over the coarse aggregates but a majority of the surface is still intact. This image shows how a typical sample performs, scaling mainly over coarse aggregate while showing no scaling on the rest of the surface.

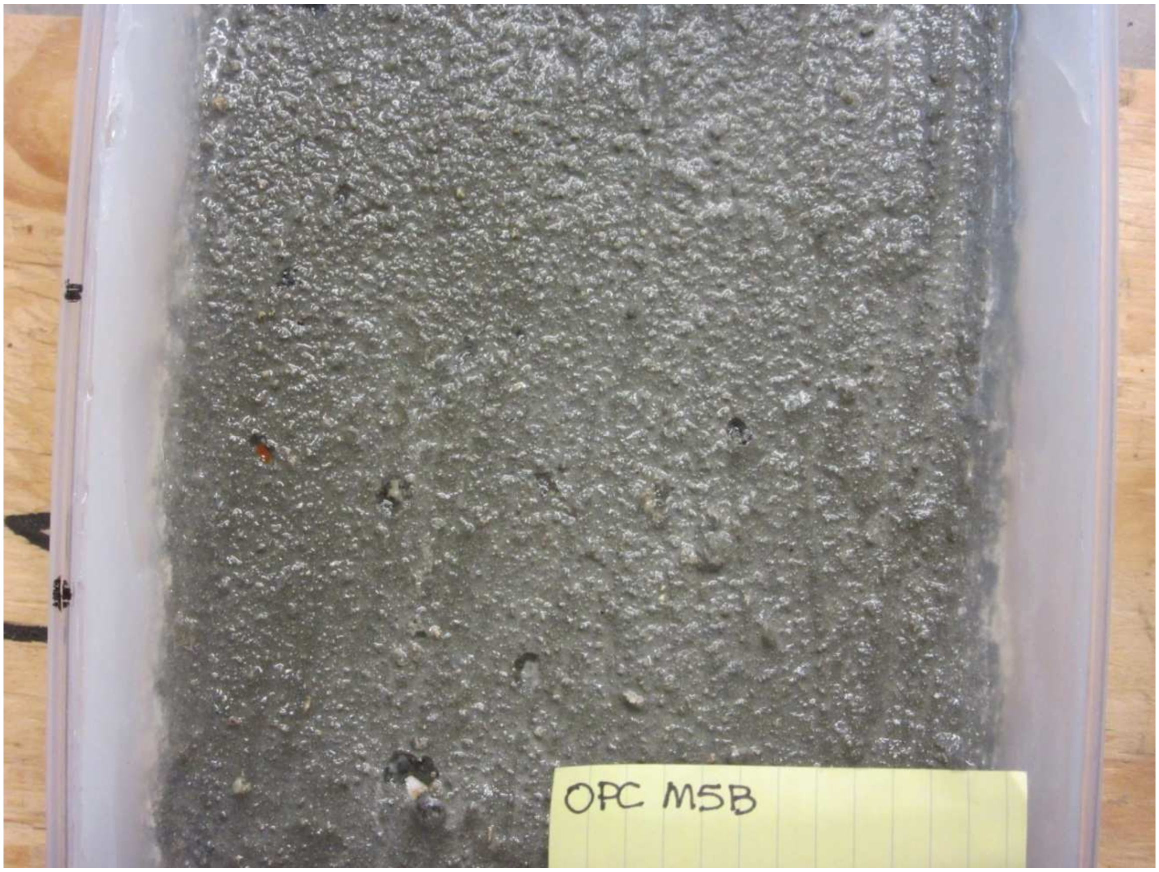

Figure 8: OPC Sample with Visual Rank of 1

OPC sample with 6.4% air after 50 cycles with a visual rank of 1. There is minor scaling over a few coarse aggregates but almost the entire surface is still intact. This image shows how a typical sample of adequate air content performs, minor scaling over coarse aggregate while showing no scaling on the rest of the surface.

Figure 9: AA1 Sample with Visual Rank of 4

AA1 sample with 8.4% air after 25 cycles with a visual rank of 4. There is scaling over nearly the entire surface but very little coarse aggregate is visible. This type of scaling is very different from OPC. Nearly the entire surface has scaled but hardly any coarse aggregate is visible. For example, the scaling example of OPC in Figure 8 has a lower visual ranking but the aggregates are exposed.

Figure 10: CSA2 Sample with Visual Rank of 5

CSA2 sample with 7.2% air after 25 cycles with a visual rank of 5. Severe scaling over the entire surface. The entire sample has coarse aggregate showing with some smaller aggregates missing and none of the original surface left.

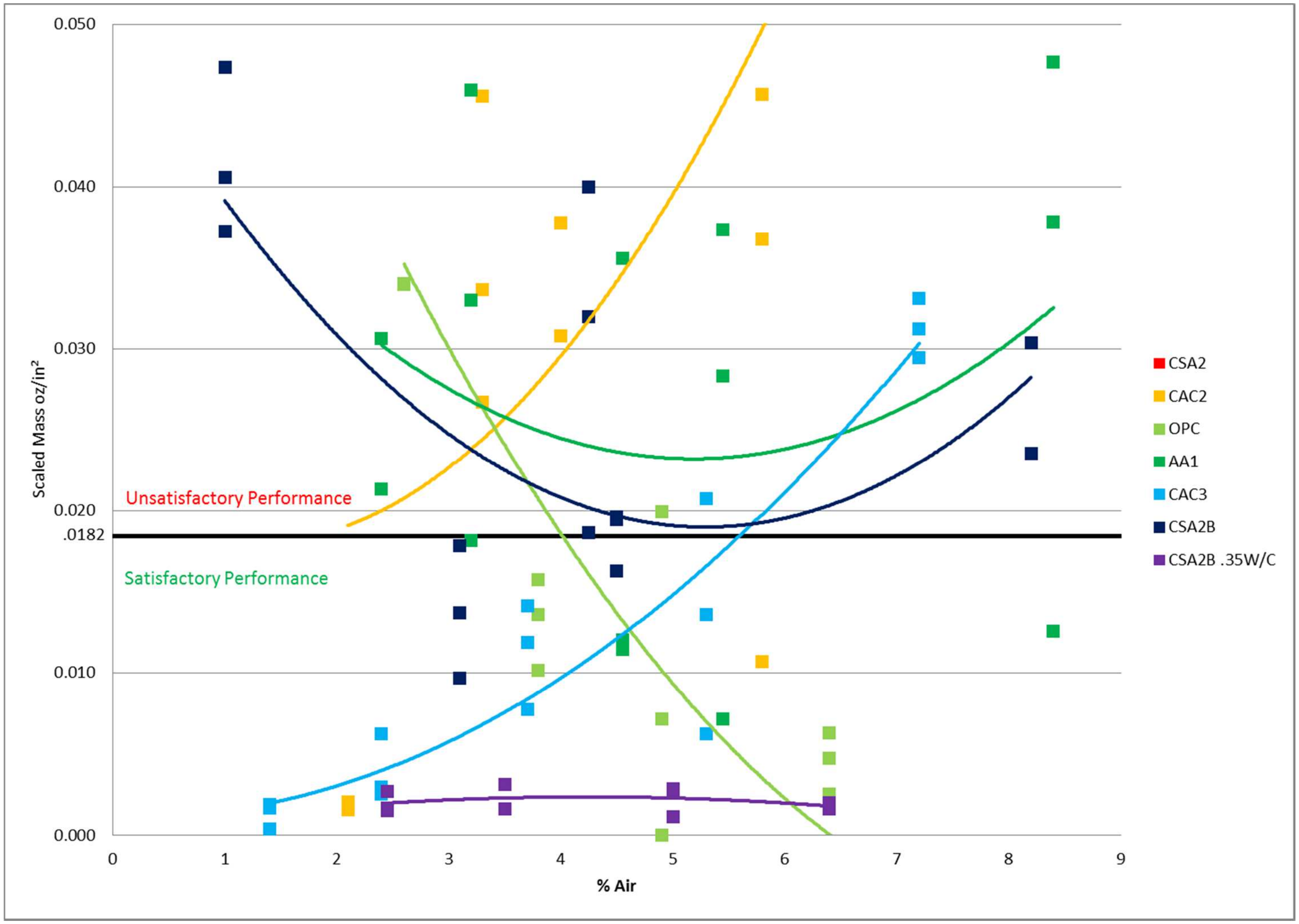

Figure 11: Scaled Mass vs. Air

2.5.3 ASTM C666

The results from the ASTM C666 testing is shown in Fig. 12 and 13. Most cements investigated needed less than 4% air to pass the ASTM C 666 test with a Durability Factor limit of 70%. CSA2 was the only cement that required 5% air content to achieve a satisfactory Durability Factor. However, CSA2B with the typical w/cm of 0.42 showed problems with surface scaling during the ASTM C666 test. When a lower w/cm of 0.35 was used then satisfactory performance for Durability Factor was found for all mixtures. Scaling was observed only in samples with air contents above 6%. CSA2B and CSA2B .35W/C were the only cements that showed severe mass loss on mixtures that had a satisfactory Durability Factor.

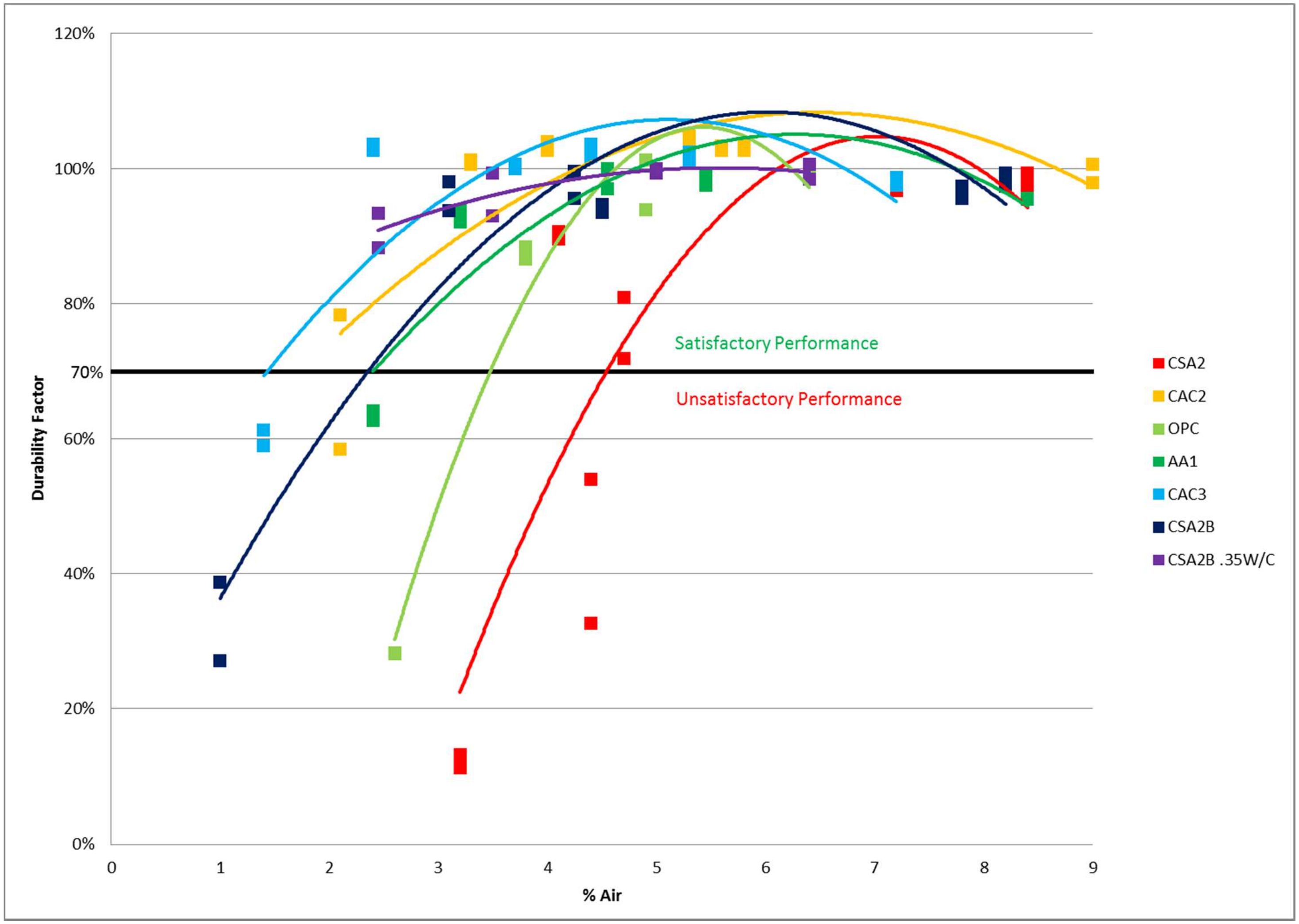

Figure 12: Durability Factor vs. Air

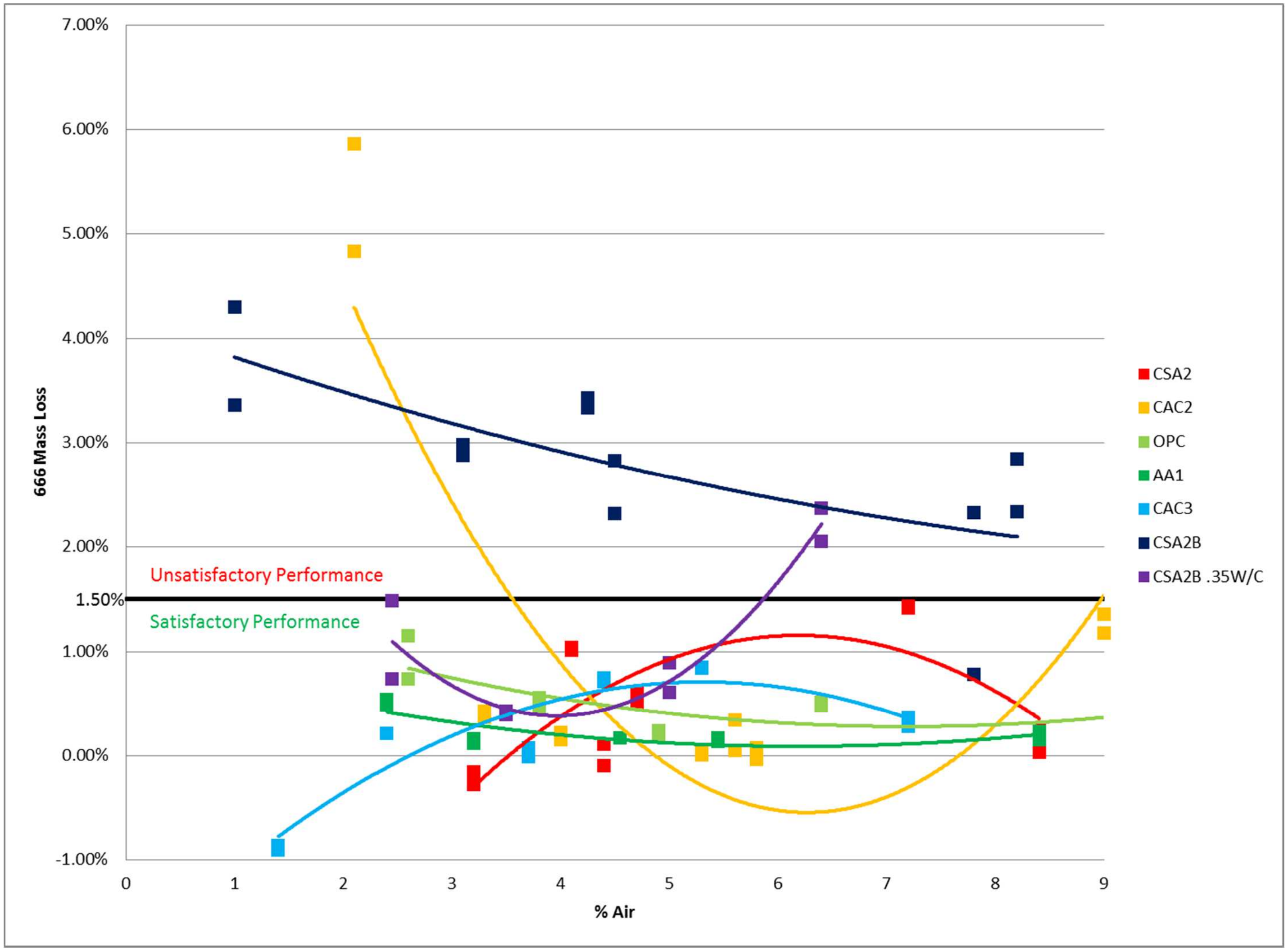

Figure 13: ASTM C666 Mass Loss vs. Air

2.6 DISCUSSION OF FINDINGS

Practitioners want cements that are durable in both bulk freeze thaw and in salt scaling. Table 5 shows the satisfactory ranges of air contents that satisfy both of these criteria. This is valuable as it shows that there is only a narrow range of air contents and in some cases, for example CSA2 and CAC2, there is not an acceptable range. This chart provides a preliminary estimate of air content ranges that are expected to show satisfactory performance in the field. Of the cements tested OPC, CAC3, and CAC2B .35W/C were the only cements that had an acceptable air content that would allow them to pass both ASTM C666 and ASTM C672. These results should be carefully interpreted as a limited number of admixture and material combinations were investigated.

Table 5: Minimum Specification Requirements

Pass	Fail	Concern
🟩	🟥	🟨

Binder	Fresh Air	Cycles	Pass or Fail Scaled Mass	Pass or Fail Visual Inspection	Pass or Fail C666 DF	Pass or Concern C666 Mass	Pass, Fail, or Concern C666 and C672	Passing Air Content Range
CSA2	4.7	20	0.0634	5	76%	-0.56%	🟥	None
	7.2	20	0.1458	5	98%	-1.43%	🟥	
	8.4	25	0.1019	5	98%	-0.14%	🟥	
CSA2B	1.0	25	0.0417	5	33%	-3.83%	🟥	3.1%
	3.1	50	0.0137	3	96%	-2.93%	🟨	
	4.3	20	0.0302	4	98%	-3.38%	🟥	
	4.5	50	0.0185	4	94%	-2.58%	🟥	
	8.2	20	0.0256	5	98%	-2.59%	🟥	
CSA2B .35W/C	2.5	50	0.0019	1	91%	-1.12%	🟩	2.5% to 6.4%
	3.5	50	0.0018	2	96%	-0.41%	🟩	
	5.0	50	0.0024	2	100%	-0.75%	🟩	
	6.4	50	0.0022	1	100%	-2.21%	🟨	
CAC2	2.1	50	0.0018	2	69%	-5.23%	🟥	None
	3.3	25	0.0354	4	101%	-0.40%	🟥	
	4.0	35	0.0450	4	103%	-0.19%	🟥	
	5.8	50	0.0310	4	103%	-0.06%	🟥	
	9.0	25	0.1141	5	99%	-1.27%	🟥	
CAC3	1.4	50	0.0013	1	60%	0.88%	🟥	2.4% to 5.3%
	2.4	50	0.0039	1	103%	-0.21%	🟩	
	3.7	50	0.0113	1	100%	-0.04%	🟩	
	5.3	50	0.0110	3	102%	-0.85%	🟩	
	7.2	50	0.0313	5	98%	-0.33%	🟥	
AA1	2.4	40	0.0260	3	63%	-0.52%	🟥	None
	3.2	25	0.0324	4	93%	-0.14%	🟥	
	4.6	50	0.0197	4	99%	-0.17%	🟥	
	5.5	25	0.0243	4	98%	-0.16%	🟥	
	8.4	25	0.0327	4	96%	-0.19%	🟥	
OPC	2.6	10	0.0400	4	27%	-0.95%	🟥	≥ 3.8%
	3.8	50	0.0132	3	87%	-0.51%	🟩	
	4.9	50	0.0136	3	98%	-0.21%	🟩	
	6.4	50	0.0045	2	99%	-0.50%	🟩	
	11.5	50	0.0018	1	98%	-0.76%	🟩	

2.7 CONCLUSION

This work has shown that all cements tested showed the ability to meet compressive strength requirements to be used in structural members. All cements were also able to meet the requirement of a durability factor of 70% in ASTM C666. For ASTM C672 only OPC, CAC3, and CSA2B .35W/C were able to have passing ranges of air contents.

Findings:

- CAC3 began to lose strength after 28 days
- CSA2 compressive strength was the most affected by increasing air content
- CSA2B w/cm 0.42 showed a concerning amount of mass loss in ASTM C666 at all air contents investigated.
- All cements were able to pass ASTM C666 with a durability factor of 70% or greater over a wide range of air contents
- Only 3 cements were able to pass ASTM C672

Recommended air contents for passing both ASTM C666 and ASTM C672:

- CSA2B .35W/C
  - Air ≥ 2.5% to 6.4%
- CAC3
  - Air 2.4% to 5.3%.
- OPC
  - Air ≥ 4.0%

CHAPTER III

INVESTIGATION OF ACM FREEZE THAW DURABILITY AGAINST THE SPACING FACTOR AND SAM NUMBER

3.0 INTRODUCTION

Hardened air void analysis (ASTM C 457) the SAM (AASHTO TP 118) were used to study the air void distribution in the different cement mixtures. It is well know that air content is not the only factor when determining how a mix will perform in bulk freeze thaw. Spacing factor is often the other parameter that is measured. This parameter was first determined by Powers (1954a, 1954b). ACI 201.2R-08 (ACI 2008) currently specifies a spacing factor of .008 in for freeze thaw durable concrete. This value was determined for OPC concrete. Every cement tested will result in different properties such as porosity and permeability. The more porous and permeable that the concrete is the easier it is for water to move through the paste. This could cause less pressure during a freezing event and so less damage. If every cement has different properties then the spacing factor required for freeze thaw durability could be different for every ACM.

It has also been shown that a SAM Number of 0.20 correlates well with a spacing factor of .008 in (Welchel, 2014). This relationship is empirical and has been determined through extensive testing on OPC concrete. Since the SAM mechanism relies on the dissolution of bubbles at given pressures in the concrete pore solution, changes in the pore solution chemistry may modify this process. This does not mean that the SAM cannot be used for ACMs. However, there may be a different correlation to the spacing factor for the different ACMs.

3.1 SAM AND HARDENED AIR RESULTS

Figure 14 shows the relationship between the air content and spacing factor. As the air content increased then the spacing factor decreased for OPC and all ACMs except CSA2B. Also, it was not possible to obtain a satisfactory polish on the AA1 samples and so the data is not reported for these mixtures. As the air content increased the spacing factor decreased until the air content was approximately 5% and then started to level out. CSA2B showed no significant change in spacing factor from 1% to 8% air. This is not expected and should be investigated in more detail.

The results for CSA2 and CAC2 most closely match the performance of OPC. This may be caused by all of these mixtures using the same air entrainer. The offset for the other curves could be caused by differences in water reducer dosage, citric acid dosages, and pore solution chemistry. All three of these could impact the quality of the air void system.

CAC3 was the most efficient air void system of the cements tested as it required only 4% air -- the lowest dosage of AEA for all ACMs -- to attain a spacing factor of .010 in. CAC3 used a different AEA that was not used in any of the other cements. This AEA may be more effective at producing a high quality air void system than the others.

CSA2B and CSA2B .35W/C had different trends from additions of AEA. Both of these mixtures used the same AEA, had the same polymer addition, used the same citric acid dosage, and the same water reducer but in different dosages. The differences could be caused by a synergistic effect between the water reducer and the AEA. It is also possible that the lower w/cm mixture also caused improved mixing and so a better air void system. This needs to be investigated in further testing.

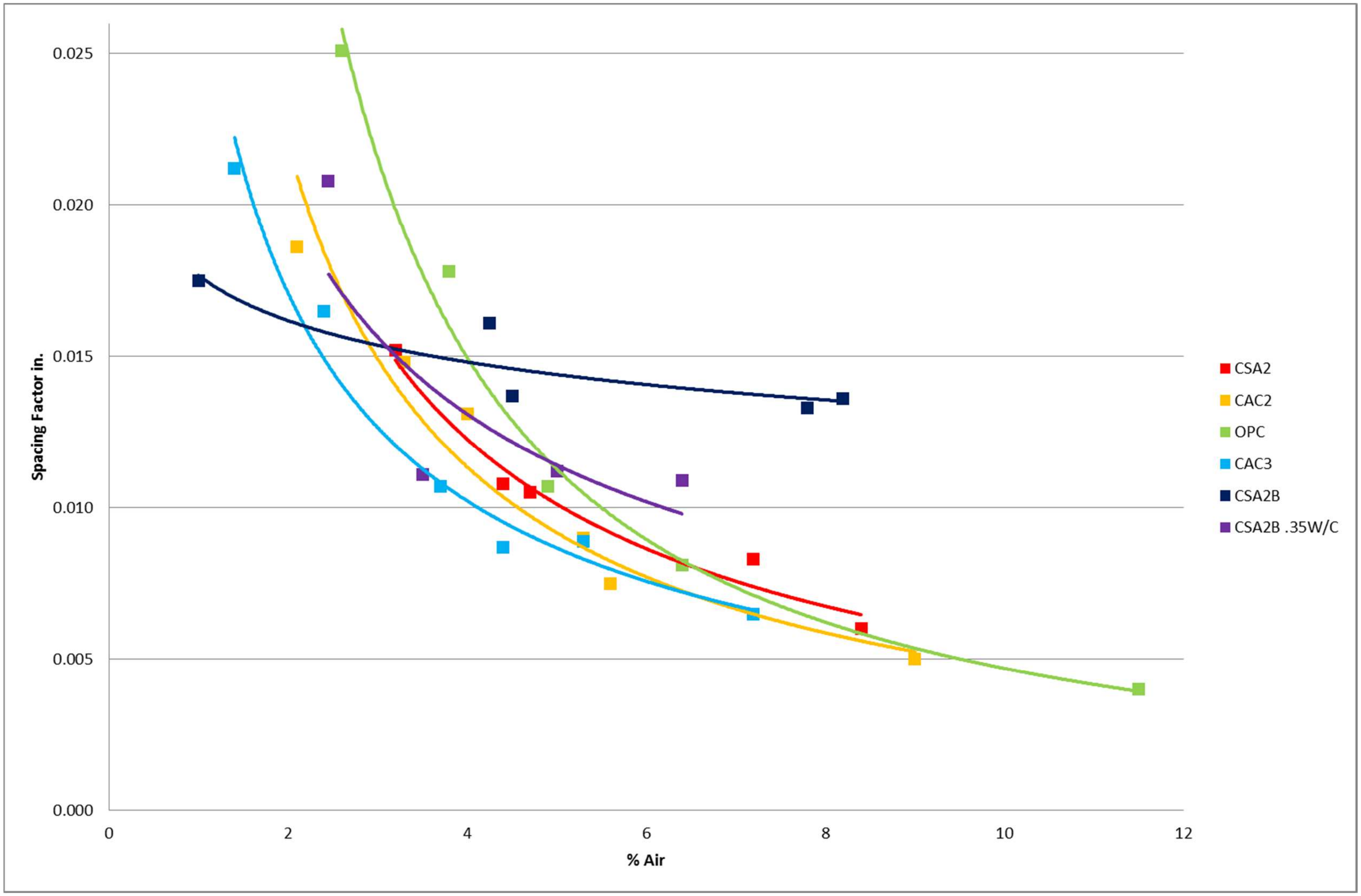

Figure 14: Spacing Factor vs. Air

The SAM versus the air content is shown in Fig. 15. CAC2, CSA2B, and CSA2B .35W/C all had similar performance to OPC. CAC3 showed a similar result but it was offset and had a lower slope. The SAM Number for this mixture did not get below 0.34 even at air contents greater than 7%. The AEA in this mixture is not the same as the others and so this could contribute to this difference in performance. The curve for CSA2 had a similar shape as CAC3 but was offset to lower SAM numbers. The AA1 results had almost no change in SAM numbers for the different air contents. This could be caused by the high concentration of chemical activator in the solution that greatly decrease the solubility of the air in the solution.

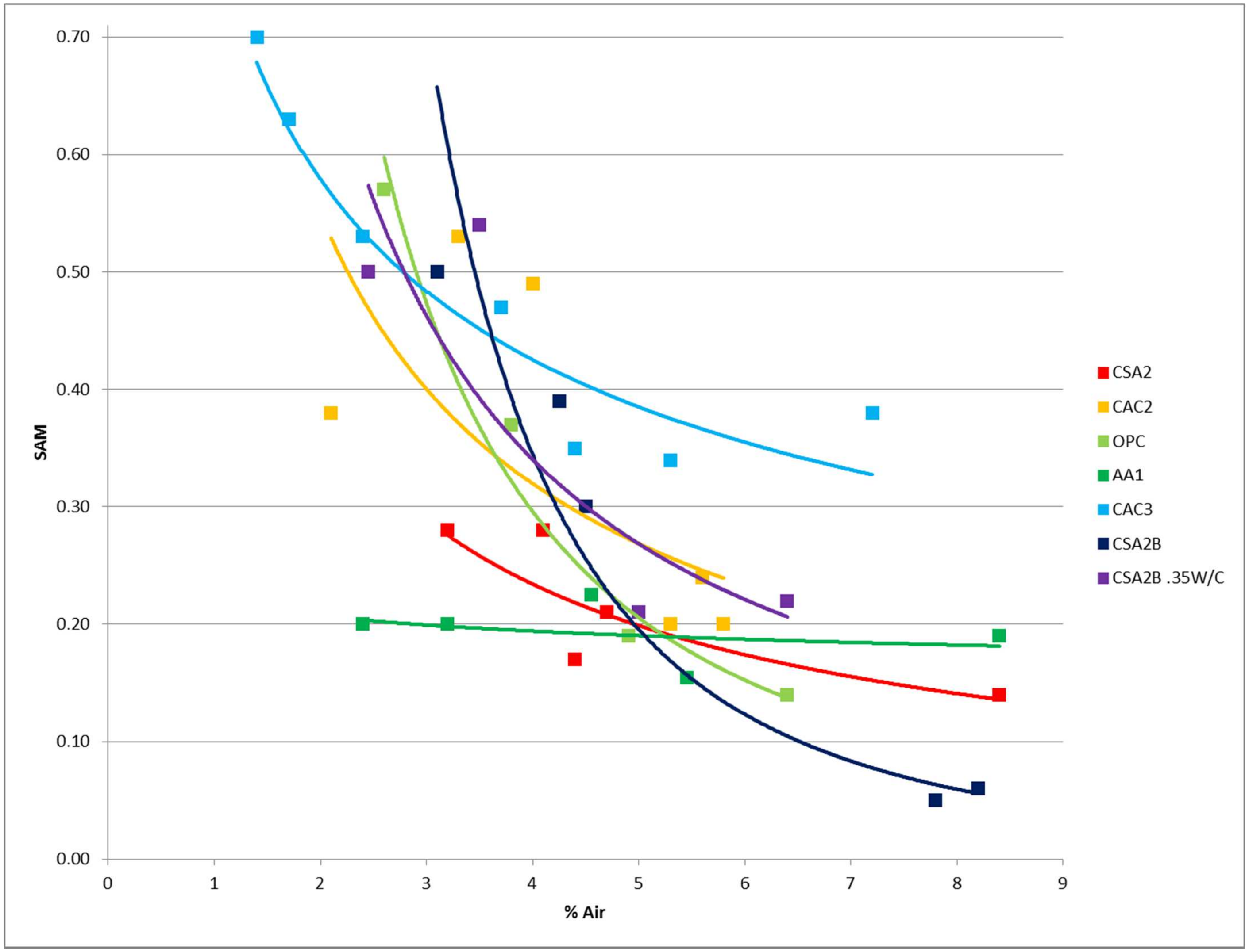

Figure 15: SAM vs. Air

The results for the SAM Number and Spacing Factor are shown in Fig. 16. For all the cements investigated, except for AA1 as it was not possible to accurately polish the samples, there was a linear relationship between SAM Number and spacing factor. This means that the SAM Number can be used for each of these cements to determine the spacing factor. However, much more work is needed to determine an accurate SAM limit between these materials and a recommended spacing factor for freeze thaw durability. Another important observation is that the response between the SAM number and spacing factor is different for the different cements. As stated previously, this is not surprising since the pore solution chemistry may be different between the different cements and this would have an impact on the solubility of the air. Point 1 and Point 2 on the graph seem to have a different response compared to the other mixtures. These samples should be repeated to see if they were measured incorrectly.

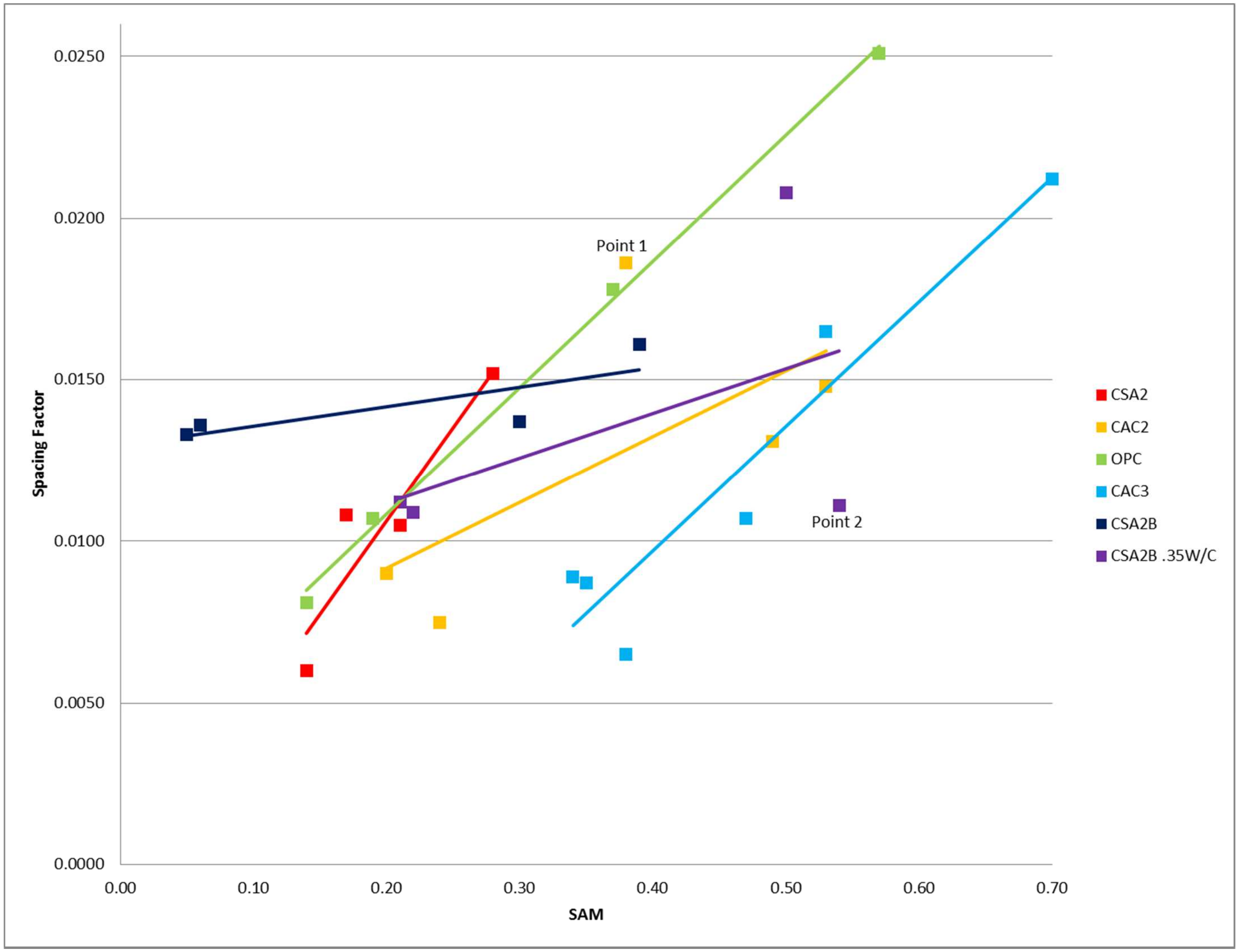

Figure 16: Spacing Factor vs. SAM

3.2 DISCUSSION

Spacing factor proved to be a useful parameter to dertermine if a mixture would be freeze thaw durable. However, the spacing factor limit is different for different cements. CAC2, CAC3, and OPC all performed nearly indentically and needed a spacing factor of aproximately 0.020 in to have a freeze thaw durability of at least 70%. CSA2 needed the lowest spacing factor of 0.0100 in for a durability factor of 70%. CSA2B did not show any clear trend as the durability factor droped sharply with only a small change in spacing factor. CSA2B .35W/C showed a slight decline in durability factor with increasing spacing factor but no mixtures had a durability factor below 91%. This mixture needed minimal air content to achieve freeze thaw durability. This is a signigicant improvement over the same mixture at a higher w/cm. Due to passing SAM numbers being much higher for many of the ACMs, many cements could not be sorted soley off of SAM number, a minumum air content was also needed due to very low air contents resulting in SAM numbers lower than expected for some mixes. This can be seen in Fig. 18 where it is hard to see the trend of SAM vs Durability Factor without taking into account that a minimum air content must be used in all mixtures.

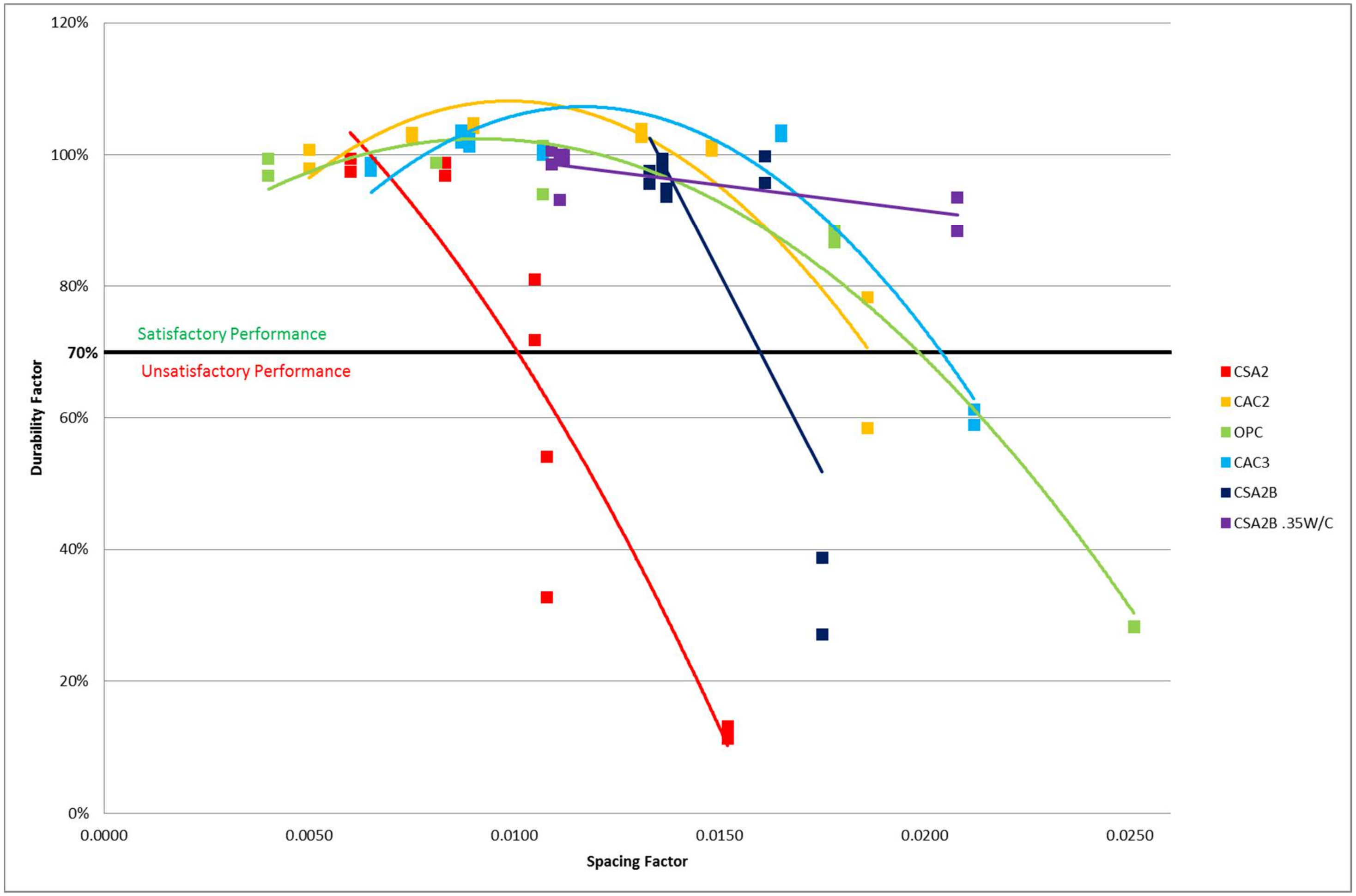

Figure 17: Durability Factor vs. Spacing Factor

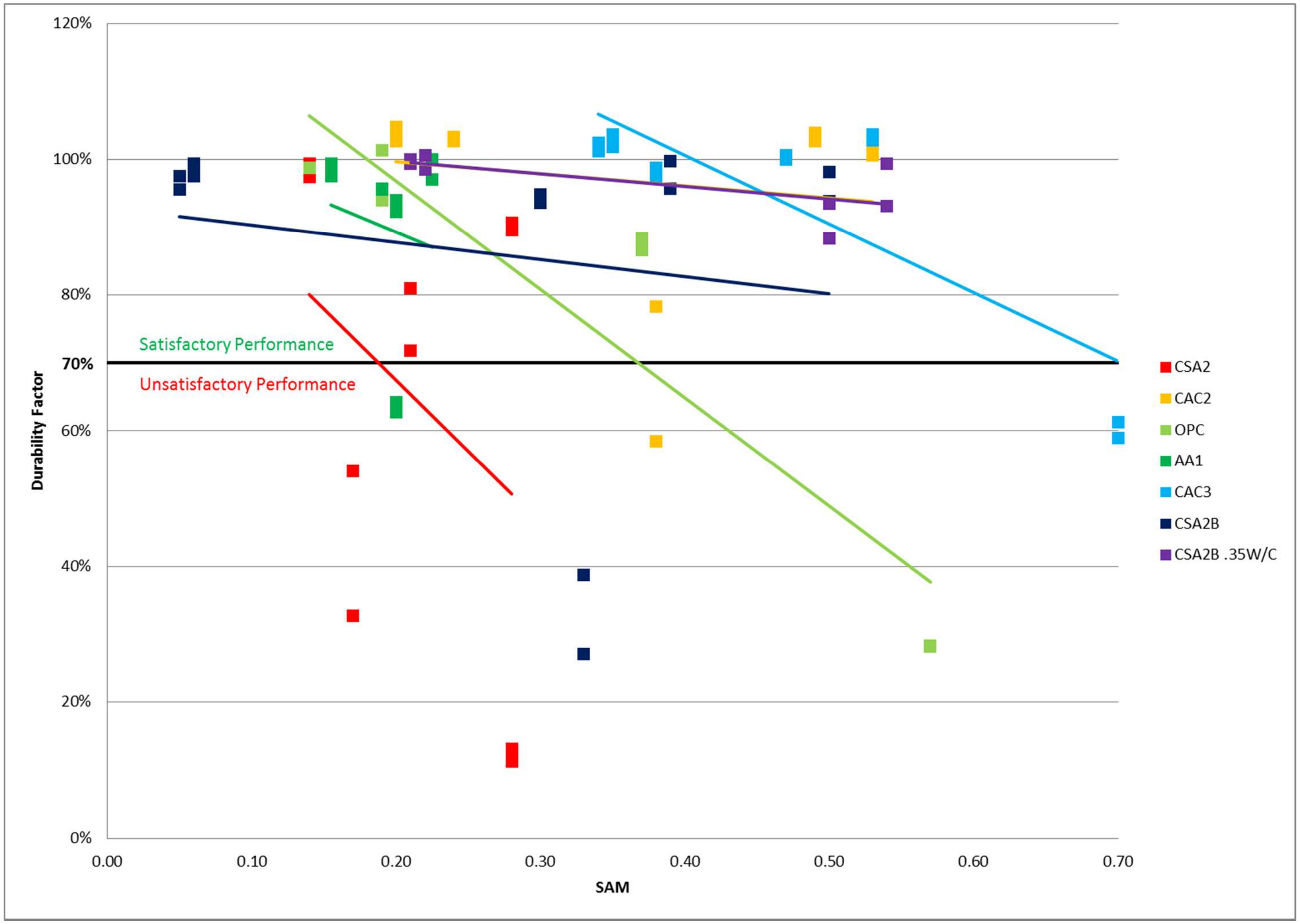

Figure 18: Durability Factor vs. SAM

The minimum values for the fresh air content, spacing factor, and SAM are given in Table 6. These values are based on performance of these mixtures in the ASTM C 666 and ASTM C 672 results. One should be careful in interrpreting these values. These values are the minimum values required for freeze thaw durability. For a specificaiton one would use a safety factor on these values to minimize the number of unsatisfactory materials provided. Furthermore, this work has investigated these materials with a limited number of admixtures, w/cm, and aggregates. This means that these recommendations may change for other mixtures. However, the amount of information in previous literature on the performance of these materials in durability tests is minimal. Furthermore, there is no known publications that have compared all of these cements in the same laboratory, with largely the same admixtures, and in the same testing. This makes this work an important contribution to the body of knowledge and could serve as a foundation for much more indepth work in the future.

Table 6: Minimum Specification Requirements

Pass 🟩	Fail 🟥	Concern 🟨

Binder	SAM Number	Fresh Air	56 Day Compressive Strength (psi)	Spacing Factor	Cycles	Pass or Fail Scaled Mass	Pass or Fail Visual Inspection	Pass or Fail C666 DF	Pass or Concern C666 Mass Change	Pass, Fail, or Concern C666 and C672	Passing Air Content Range	Minimum Requirements
CSA2	0.21	4.7	9511	0.0105	20	0.0634	5	76%	-0.56%	🟥	None	Not Recommended
	-	7.2	-	0.0083	20	0.1458	5	98%	-1.43%	🟥		
	0.14	8.4	7949	0.0060	25	0.1019	5	98%	-0.14%	🟥		
CSA2B	0.33	1.0	10045	0.0175	25	0.0417	5	33%	-3.83%	🟥	3.1%	Not Recommended
	0.50	3.1	9619	-	50	0.0137	3	96%	-2.93%	🟨		
	0.39	4.3	9182	0.0161	20	0.0302	4	98%	-3.38%	🟥		
	0.30	4.5	9768	0.0137	50	0.0185	4	94%	-2.58%	🟥		
	0.06	8.2	8546	0.0136	20	0.0256	5	98%	-2.59%	🟥		
CSA2B .35W/C	0.50	2.5	12426	0.0208	50	0.0019	1	91%	-1.12%	🟩	2.5% to 6.4%	SAM ≤ 0.50 Air ≥ 3.0% SF ≤ .0200
	0.54	3.5	10753	0.0111	50	0.0018	2	96%	-0.41%	🟩		
	0.21	5.0	11581	0.0112	50	0.0024	2	100%	-0.75%	🟩		
	0.22	6.4	11175	0.0109	50	0.0022	1	100%	-2.21%	🟨		
CAC2	0.38	2.1	8000	0.0186	50	0.0018	2	69%	-5.23%	🟥	None	Not Recommended
	0.53	3.3	9082	0.0148	25	0.0354	4	101%	-0.40%	🟥		
	0.49	4.0	8374	0.0131	35	0.0450	4	103%	-0.19%	🟥		
	0.20	5.8	7318	-	50	0.0310	4	103%	-0.06%	🟥		
	-	9.0	-	0.0050	25	0.1141	5	99%	-1.27%	🟥		
CAC3	0.70	1.4	9959	0.0212	50	0.0013	1	60%	0.88%	🟥	2.4% to 5.3%	SAM ≤ 0.50 Air 2.5% to 5.5% SF ≤ .0150
	0.53	2.4	9125	0.0165	50	0.0039	1	103%	-0.21%	🟩		
	0.47	3.7	8041	0.0107	50	0.0113	1	100%	-0.04%	🟩		
	0.34	5.3	9151	0.0089	50	0.0110	3	102%	-0.85%	🟩		
	0.38	7.2	8490	0.0065	50	0.0313	5	98%	-0.33%	🟥		
AA1	0.20	2.4	6801	-	40	0.0260	3	63%	-0.52%	🟥	None	Not Recommended
	0.20	3.2	6617	-	25	0.0324	4	93%	-0.14%	🟥		
	0.23	4.6	7502	-	50	0.0197	4	99%	-0.17%	🟥		
	0.16	5.5	7390	-	25	0.0243	4	98%	-0.16%	🟥		
	0.19	8.4	5002	-	25	0.0327	4	96%	-0.19%	🟥		
OPC	0.57	2.6	7156	0.0251	10	0.0400	4	27%	-0.95%	🟥	≥ 3.8%	SAM ≤ 0.35 Air ≥ 4.0% SF ≤ .0150
	0.37	3.8	6985	0.0178	50	0.0132	3	87%	-0.51%	🟩		
	0.19	4.9	6580	0.0107	50	0.0136	3	98%	-0.21%	🟩		
	0.14	6.4	6055	0.0081	50	0.0045	2	99%	-0.50%	🟩		
	-	11.5	-	0.0040	50	0.0018	1	98%	-0.76%	🟩		

3.3 CONCLUSION

This work has shown that there are satisfactory spacing factor and SAM Number limits for almost all the cements tested. Each cement has a different porosity, pore connectivity, strength, and pore solution chemistry and so it is not surprising that each cement shows different requirements for SAM Number and spacing factor to ensure freeze thaw durability. The following findings and recommendations have been made.

Findings

- AA1 did not show change in result in the SAM test
- CSA2B did not show a significant change in spacing factor across a 7% air content range
- With compatible air entraining admixtures every cement was able to meet the durability factor criteria of 70%

Recommendations for passing ASTM C666

- CSA2
    - Air ≥ 5.0%
    - Spacing Factor ≤ .0100 in.
- CSA2B
    - SAM ≤ 0.50
    - Air ≥ 4.0%
    - Spacing Factor ≤ .0150 in.
- CSA2B .35W/C
    - SAM ≤ 0.50
    - Air ≥ 3.0%
    - Spacing Factor ≤ .0200 in.
- CAC2
    - SAM ≤ 0.50
    - Air ≥ 4.0%
    - Spacing Factor ≤ .0150 in.
- CAC3

  - SAM ≤ 0.50
  - Air ≥ 3.0%
  - Spacing Factor ≤ .0150 in.
- AA1
  - Air ≥ 4.0%
- OPC
  - SAM ≤ 0.35
  - Air ≥ 4.0%
  - Spacing Factor ≤ .0150 in.

CHAPTER IV

CONCLUSION

This thesis addresses both the ability and the requirements for ACMs in structural or paving applications and their ability to reduce freeze thaw damage. ACMs are both chemically and structurally different from OPC. These differences can be both a positive and a negative. Many ACMs showed increased strength and higher strength gain rates than OPC but proved to have durability problems, especially in salt scaling.

AA1 showed the closest match to OPC in final strength and strength gain rate. CSA2 showed the highest strength of any ACM at the standard .42W/C mixture design nearly double the strength of OPC at 2 days. Using varying dosages of citric acid allowed for approximately the same set time to be attained for all cements tested. All of the ACMs attained more than sufficient strength to be used in structural or paving applications.

Freeze thaw durability, based on durability factor, was achieved with all cements at standard air content ranges. CSA2B showed concerning amounts of mass loss even at durability factors over 90%. Salt scale performance was not as easily attained for many cements with it being unattainable for some. Only OPC, CSA2B .35W/C, and CAC3 were able to obtain satisfactory results in both salt scale and freeze thaw at the same air contents.

Current recommendations for using an ACM in a freezing climate must result in a structure that is durable in both freeze thaw and salt scaling. A range of material properties were given for these materials where satisfactory performance was observed.

Because of the loss of compressive strength of CAC3 the use of this material may be concerning for some owners. Because of this the ACM with the most promise for freeze thaw durability would be CSA2B .35W/C with the following properties: SAM ≤ 0.50, Air ≥ 3.0%, and Spacing Factor ≤ .0200 in. This combination has shown the ability to provide durability and strength on par with or better than OPC.

Further testing is needed to examine more mixture designs with different W/C, different admixture combinations and dosages, and continued work on developing specifications so that a wider range of viable material combinations could be identified to achieve durable mixes using ACMs.

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