

# Freeze Thaw Durability of Modern Concrete Mixtures



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# Acknowledgements

- Oklahoma Transportation Center
- CP Tech Center

# Summary

- The basics...
- Why do we add air to concrete?
- What do we want in an air-void system?
- Current measuring techniques
- How much air do we need in our concrete?
- Ongoing work
- Conclusion



What is...

Concrete



PCA Photo

What is...

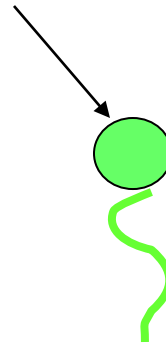
Air-entrained concrete



# Air-Entrained Concrete

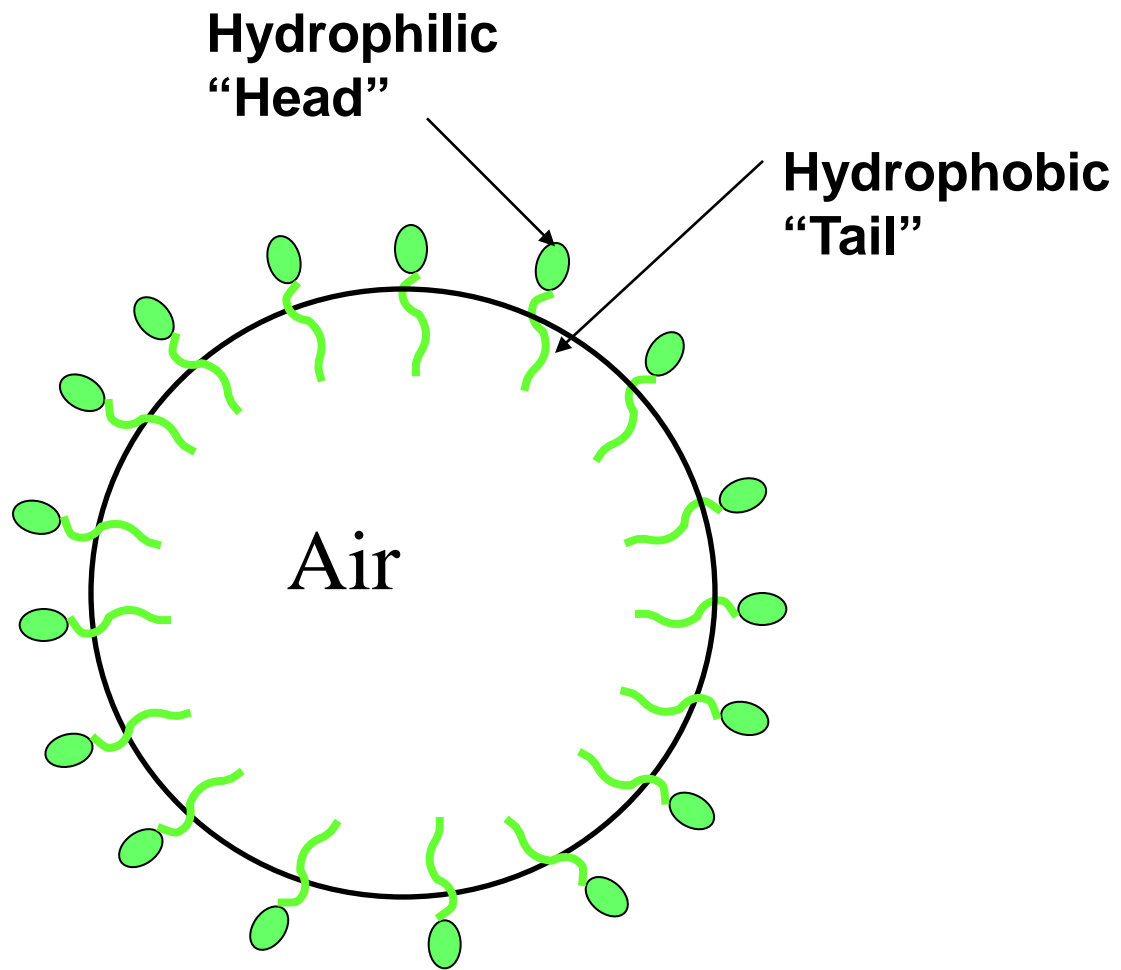
- A surfactant is used to stabilize the air bubbles created during mixing

**Hydrophilic  
“Head”**



**Hydrophobic  
“Tail”**

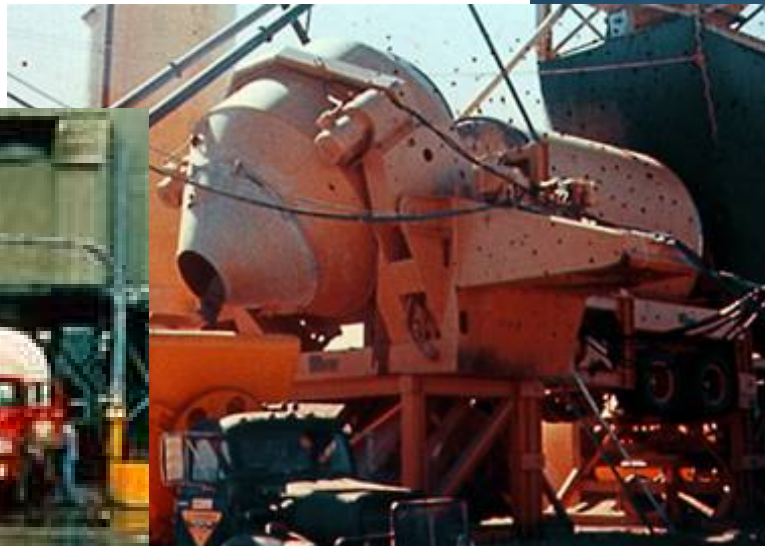
Water





# How...

- Air is Stabilized in the Concrete During the Mixing Process





# Why Do We Add Air to Concrete?

- Air-entrained bubbles are the key to the freeze-thaw resistance of concrete
- Smaller bubbles are more effective in providing freeze-thaw resistance than larger bubbles





2  
EVIDENCE MARKER  
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# What Affects Air-Entrainment...





# What Affects Air-Entrainment...

**Everything!**

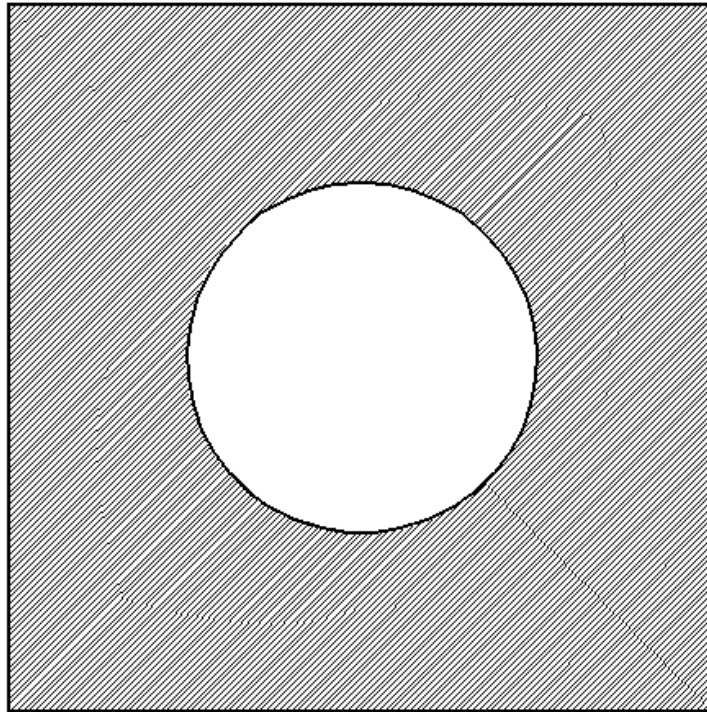
# What Do You Want in an Air-Void System?



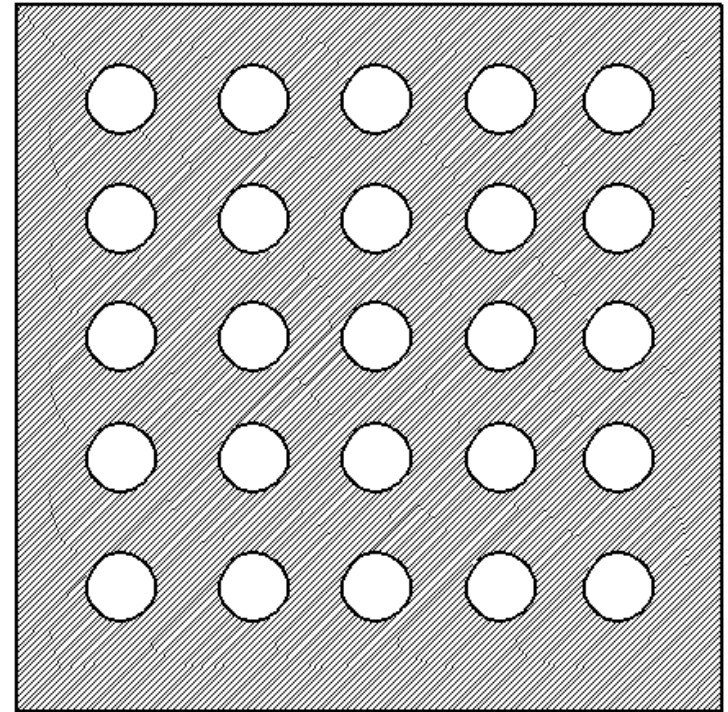


# What Do You Want in an Air-Void System?

A



B

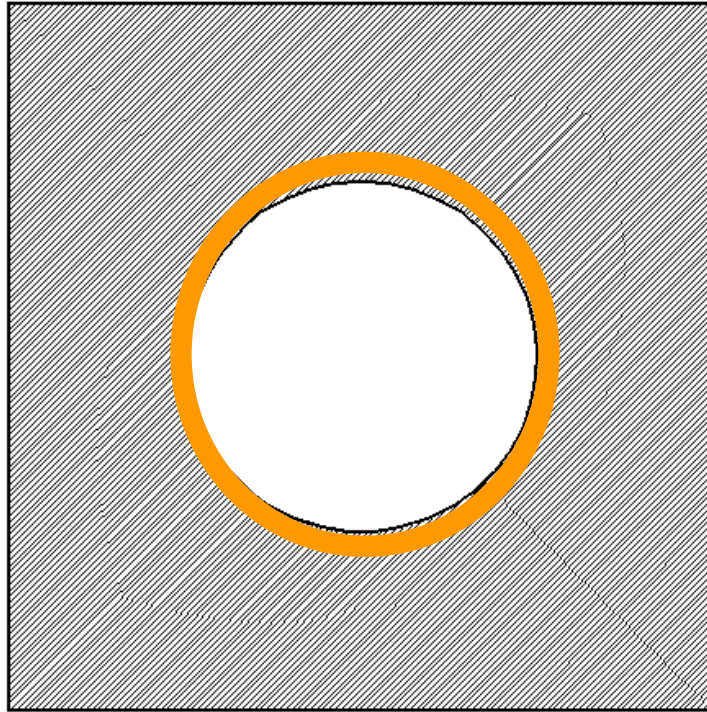


- Volume of air provided is the same for both circumstances.
- Case B has a lower spacing factor and a higher specific surface.

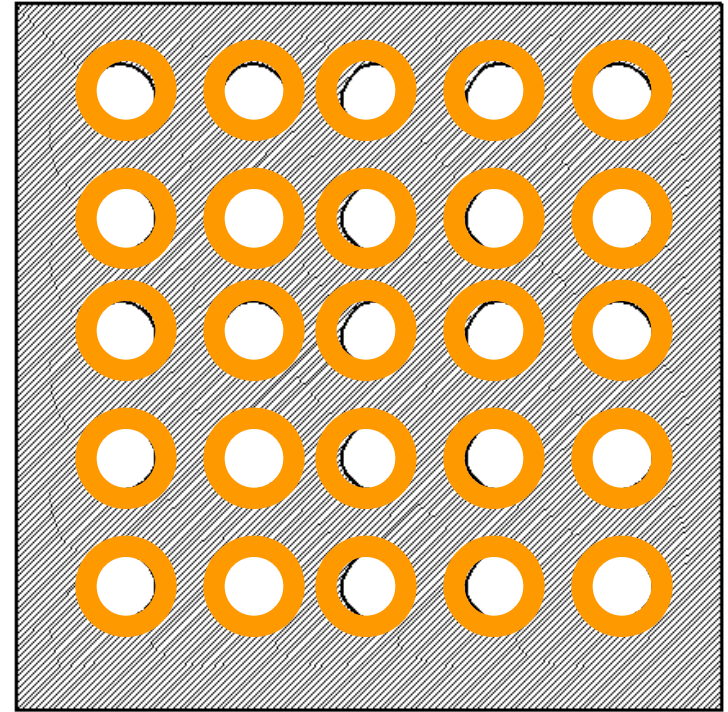


# What Do You Want in an Air-Void System?

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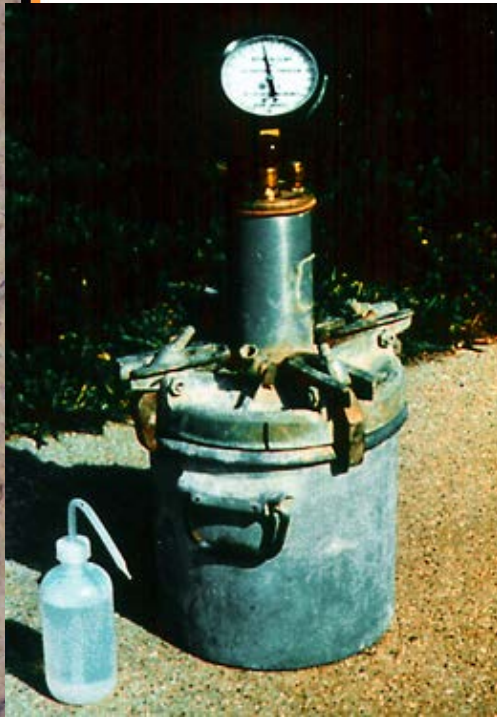
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# Current Measuring Techniques





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PCA photo

**ASTM C 231**



PCA photo

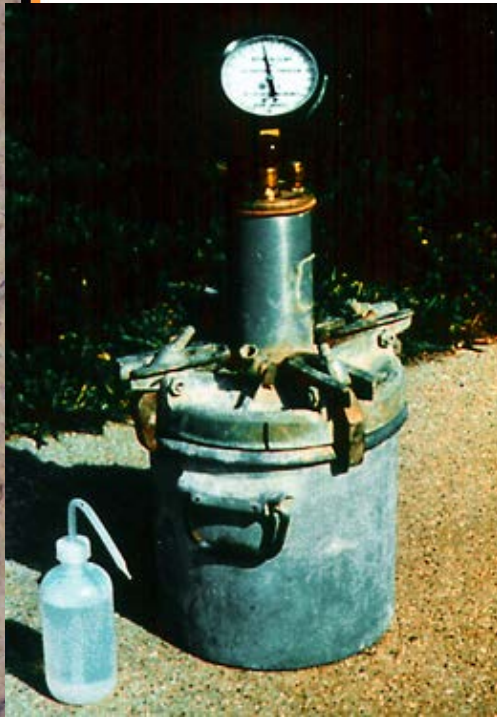
**ASTM C 173**



**ASTM C 138**



# Current Measuring Techniques



PCA photo

**ASTM C 231**



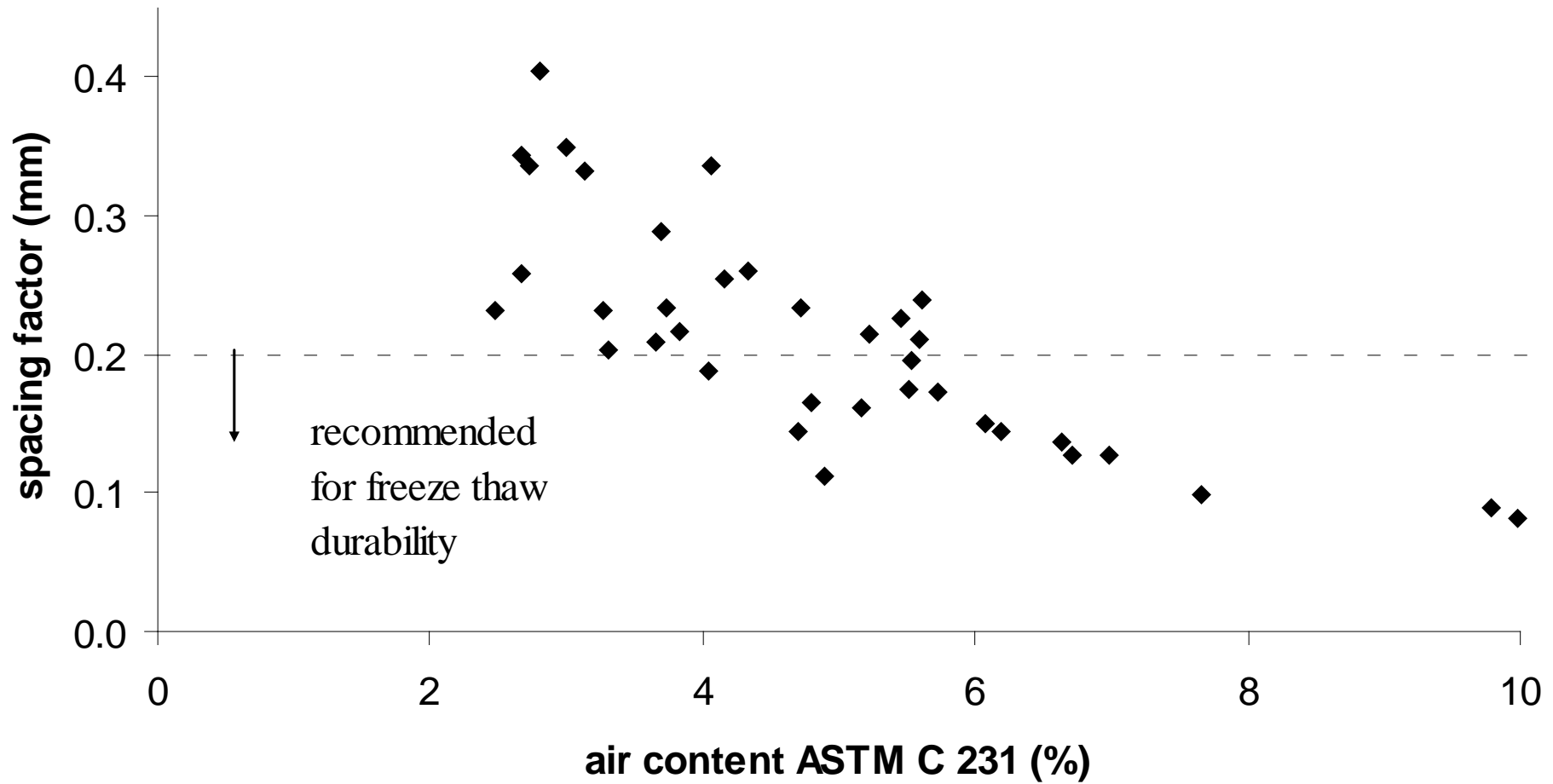
PCA photo

**ASTM C 173**



**ASTM C 138**

**These only measure volume!!!**





# How Much Air Do We Need?



**TABLE 4.4.1 — TOTAL AIR CONTENT FOR CONCRETE EXPOSED TO CYCLES OF FREEZING AND THAWING**

Increased paste content



Nominal maximum aggregate size, in.*	Air content, percent	
	Exposure Class F1	Exposure Classes F2 and F3
3/8	6	7.5
1/2	5.5	7
3/4	5	6
1	4.5	6
1-1/2	4.5	5.5
2 <sup>†</sup>	4	5
3 <sup>†</sup>	3.5	4.5

\*See ASTM C33 for tolerance on oversize for various nominal maximum size designations.

<sup>†</sup>Air contents apply to total mixture. When testing concretes, however, aggregate particles larger than 1-1/2 in. are removed by sieving and air content is measured on the sieved fraction (tolerance on air content as delivered applies to this value). Air content of total mixture is computed from value measured on the sieved fraction passing the 1-1/2 in. sieve in accordance with ASTM C231.

If  $f'_c > 5,000$  psi then these recommendations can be reduced by 1%



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As max nom. Aggregate size ↓ paste ↑

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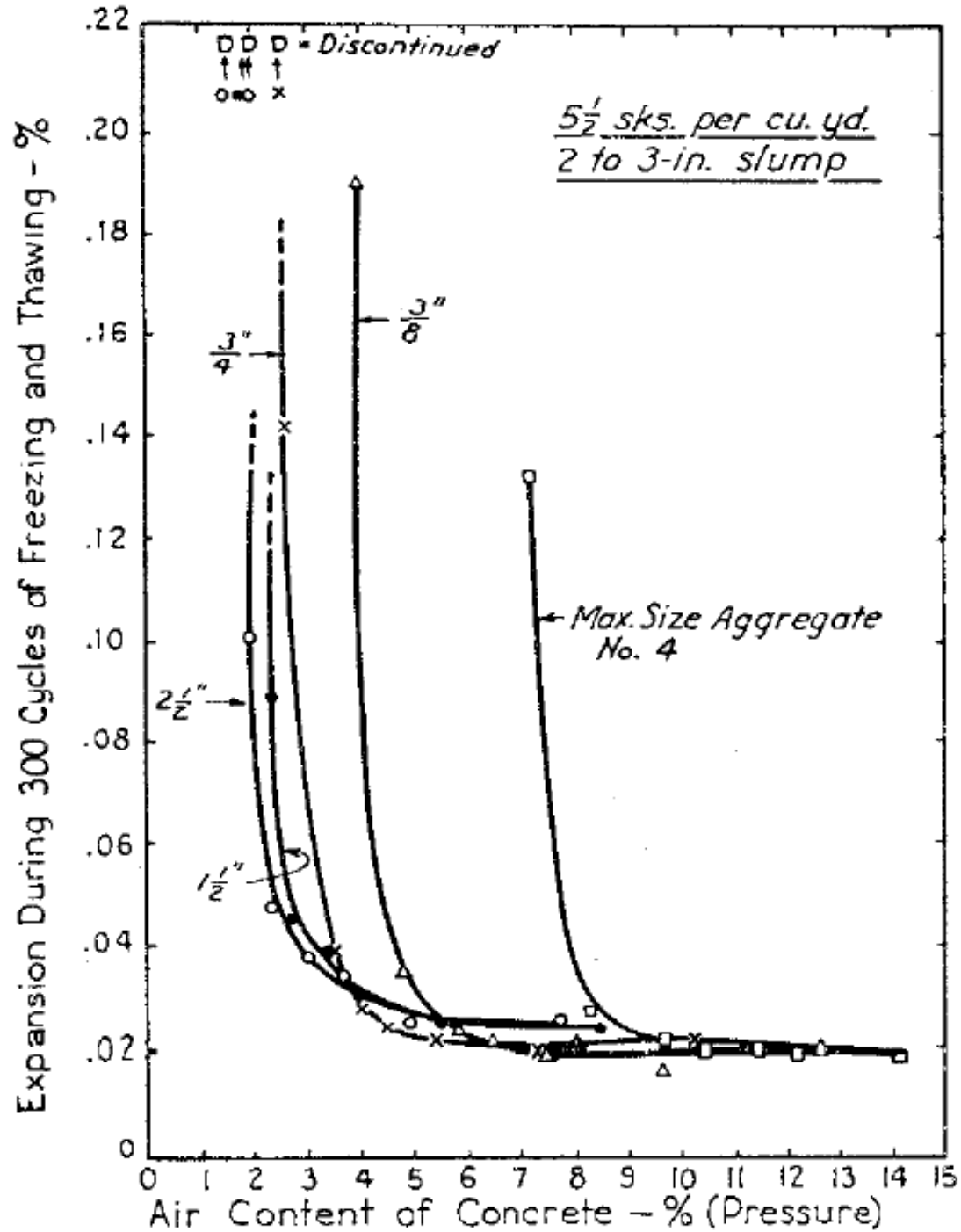
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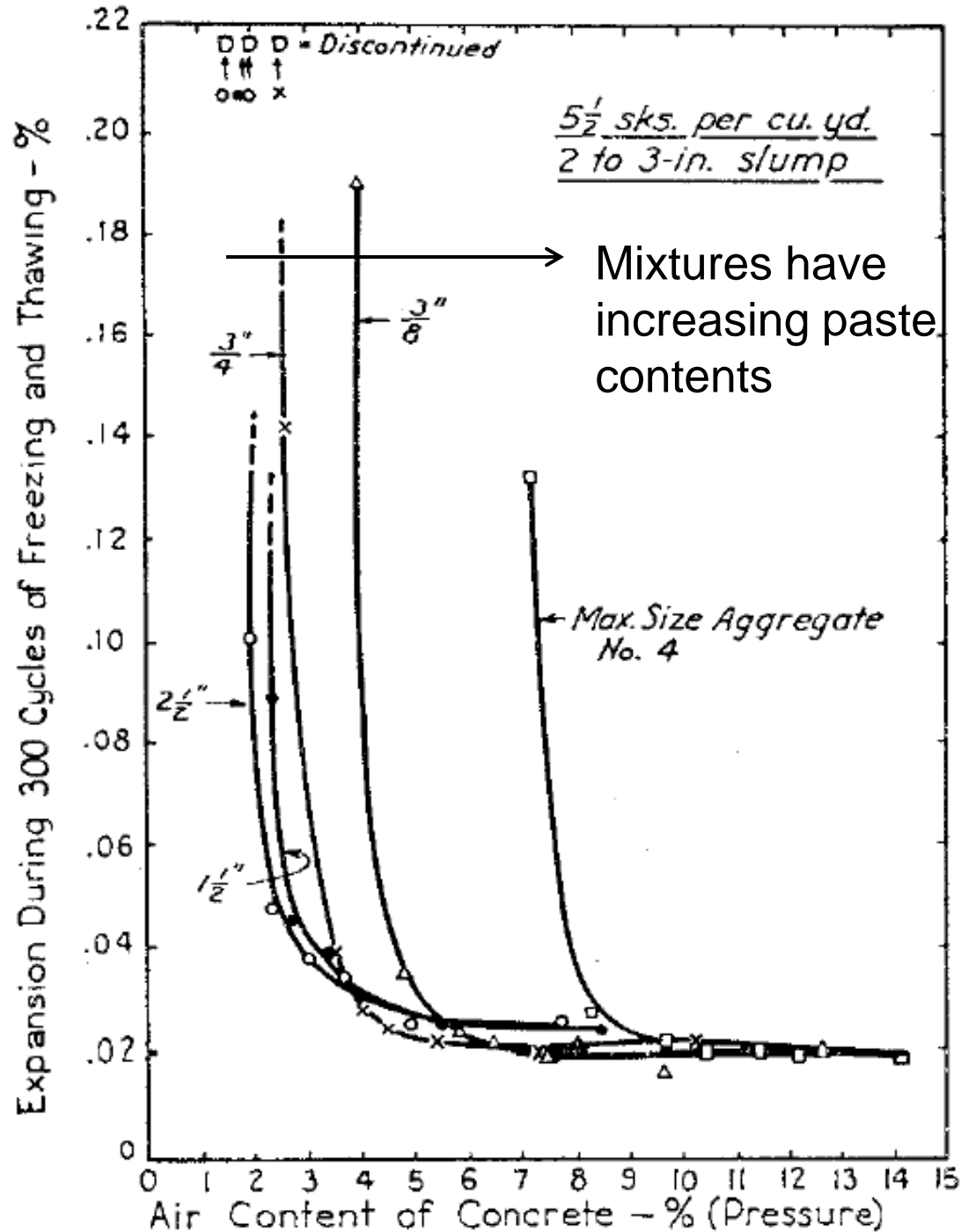
# Klieger

- There was no standard freeze-thaw testing method at the time
- To investigate the freeze thaw durability specimens were manually wheeled in and out of freezers for 300 cycles!!!
- The length change of the prisms were used to measure deterioration
- His team investigated over 1000 different mixtures



Klieger 1952





Klieger 1952

# The Back Story...

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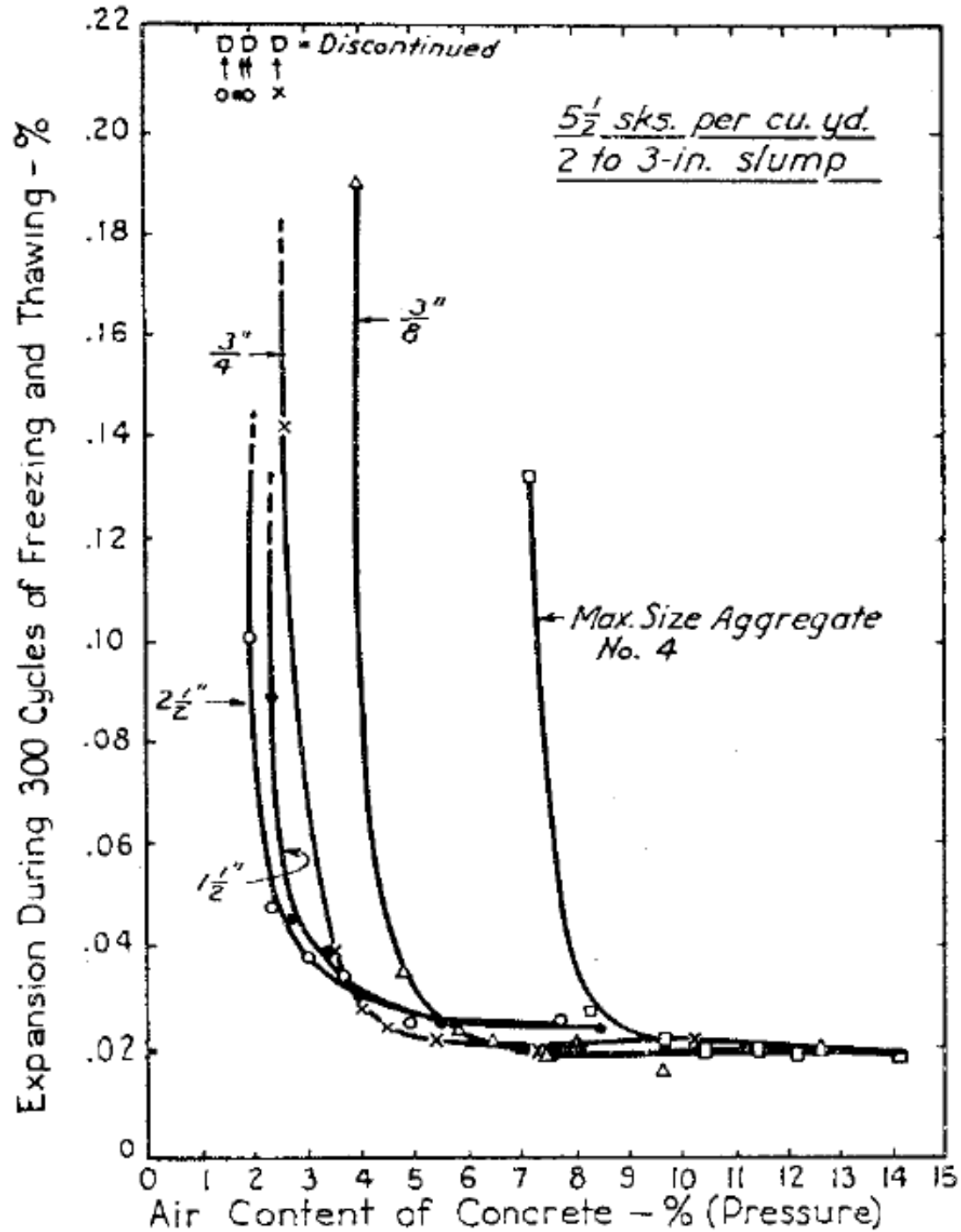
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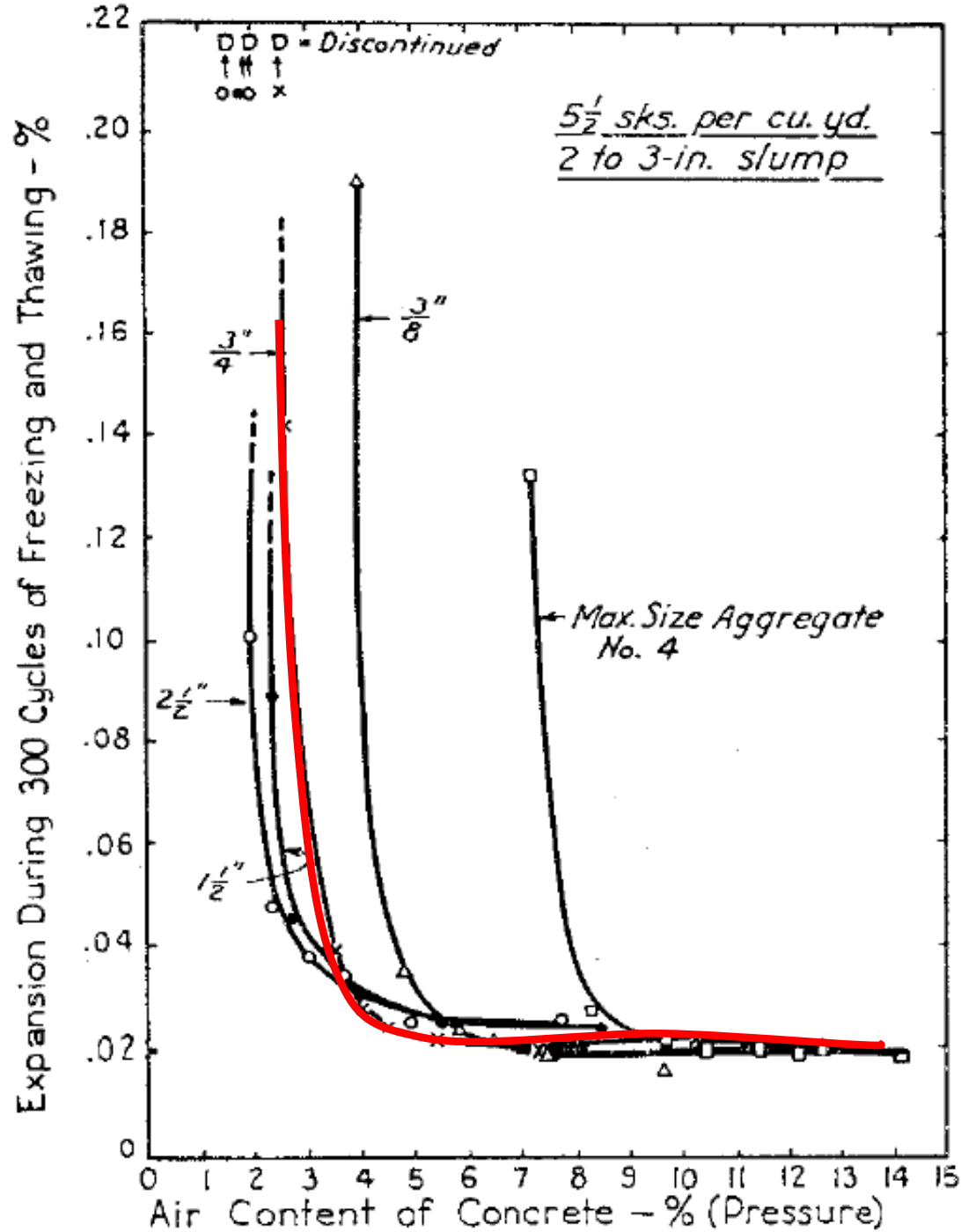
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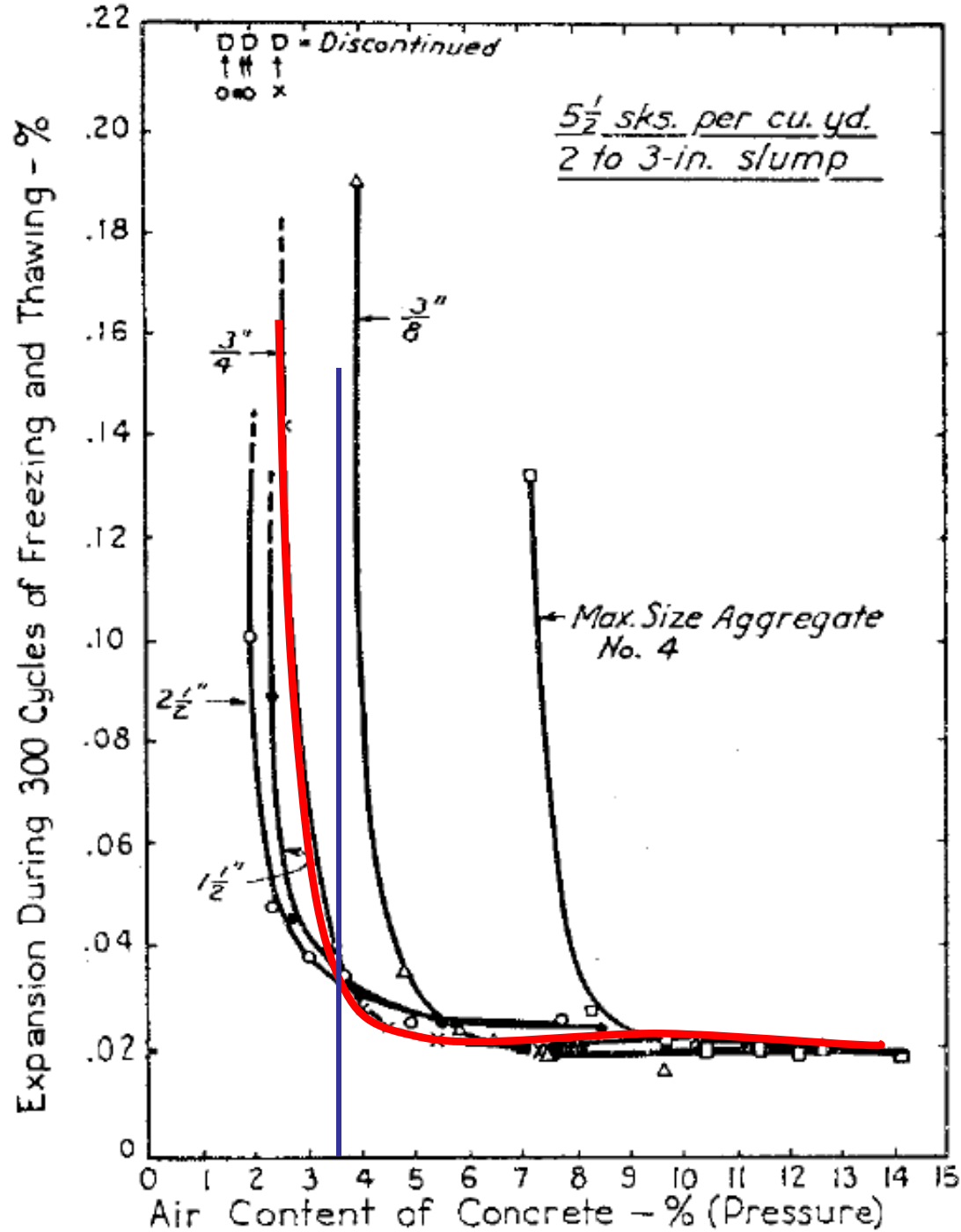


Klieger 1952

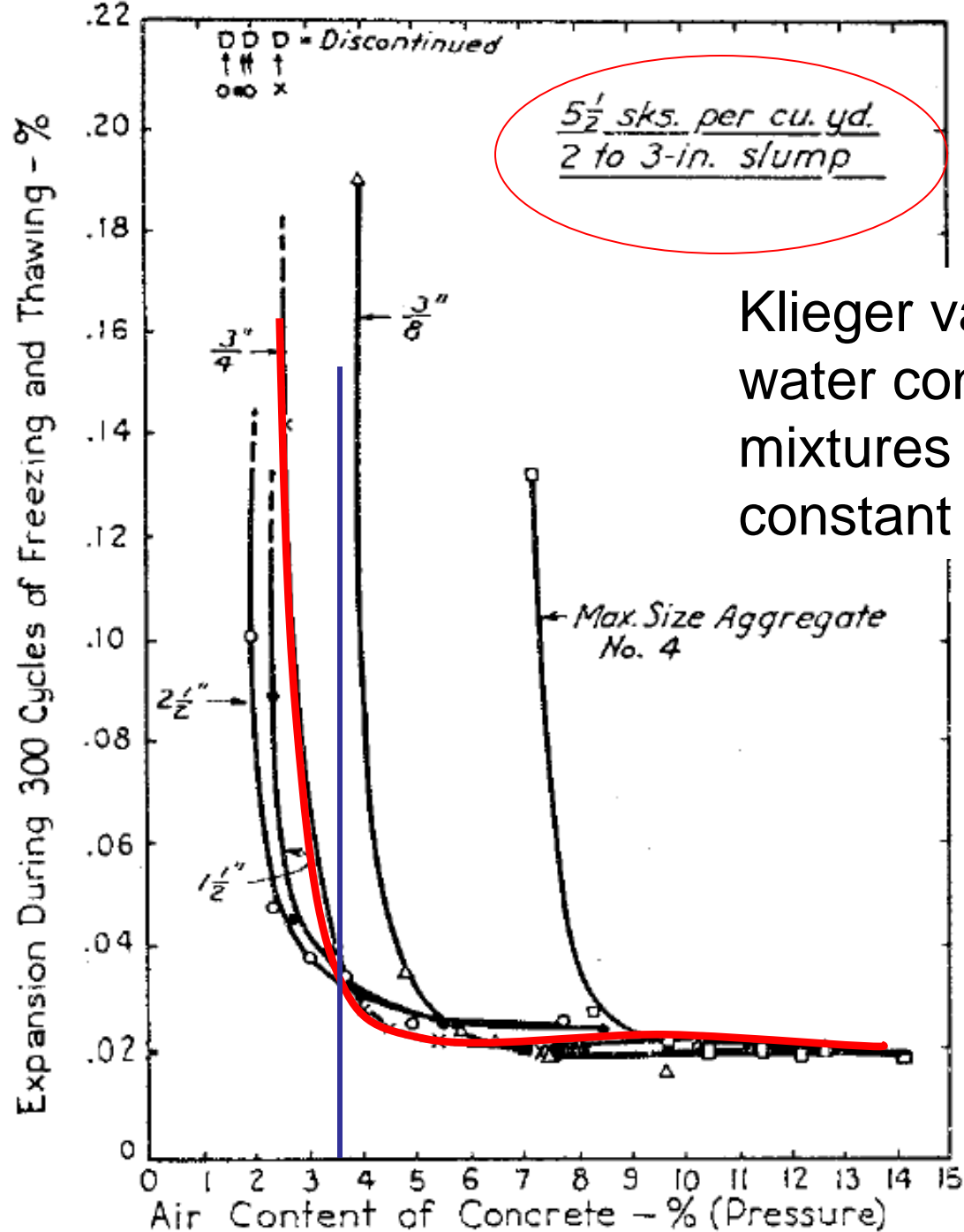


Klieger 1952





Klieger 1952



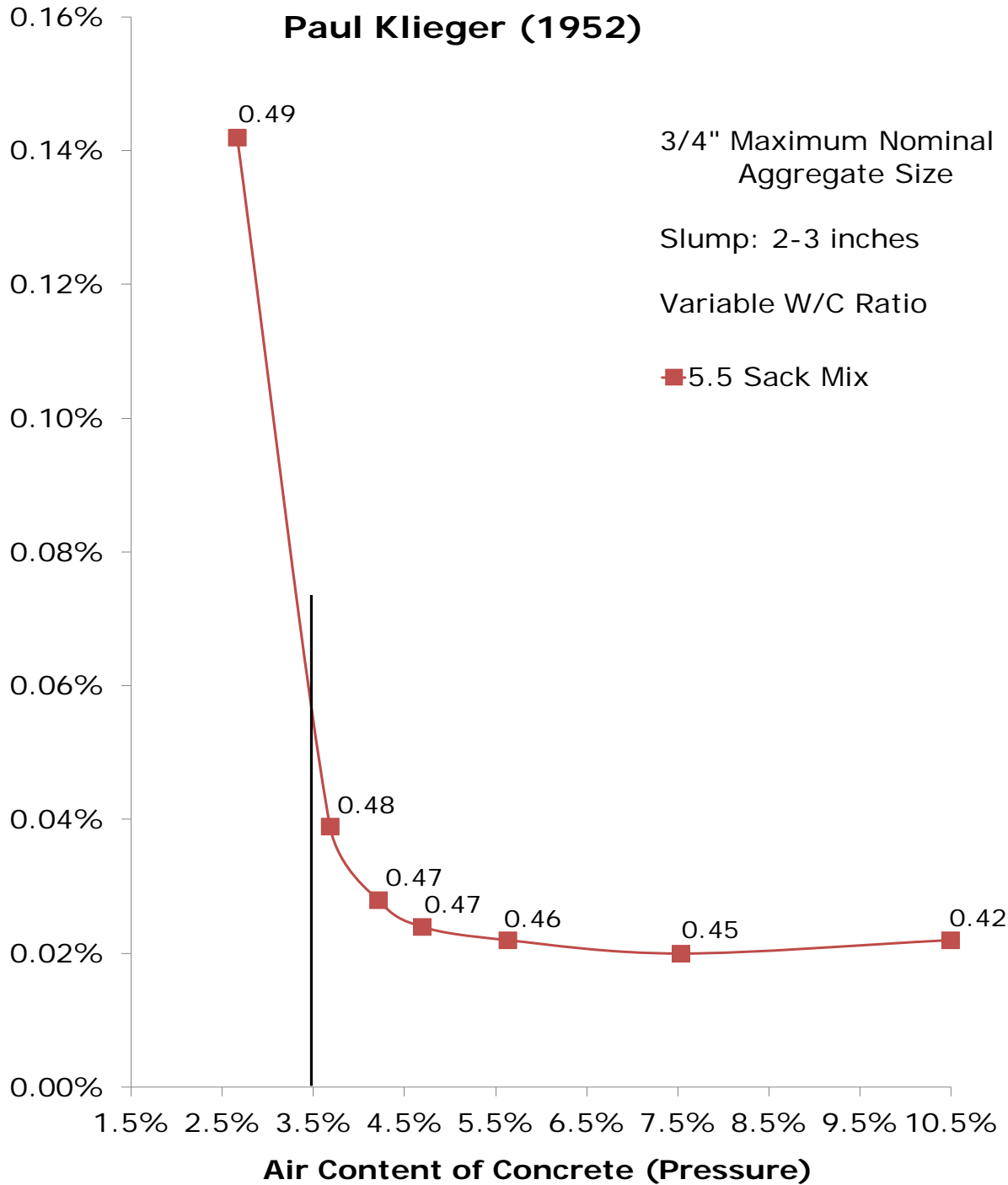
Klieger varied the water content of the mixtures to get a constant slump

Klieger 1952



# Paul Klieger (1952)

Expansion During 300 Cycles of Freezing and Thawing - percent



# Klieger

- The testing was NOT the same as the modern ASTM C 666
- There were differences in:
  - curing
  - freezing rate
  - measurement techniques
- Only one type of cement, one AEA (Vinsol resin), no midranges or supers, no SCMs



# Laboratory work at OSU

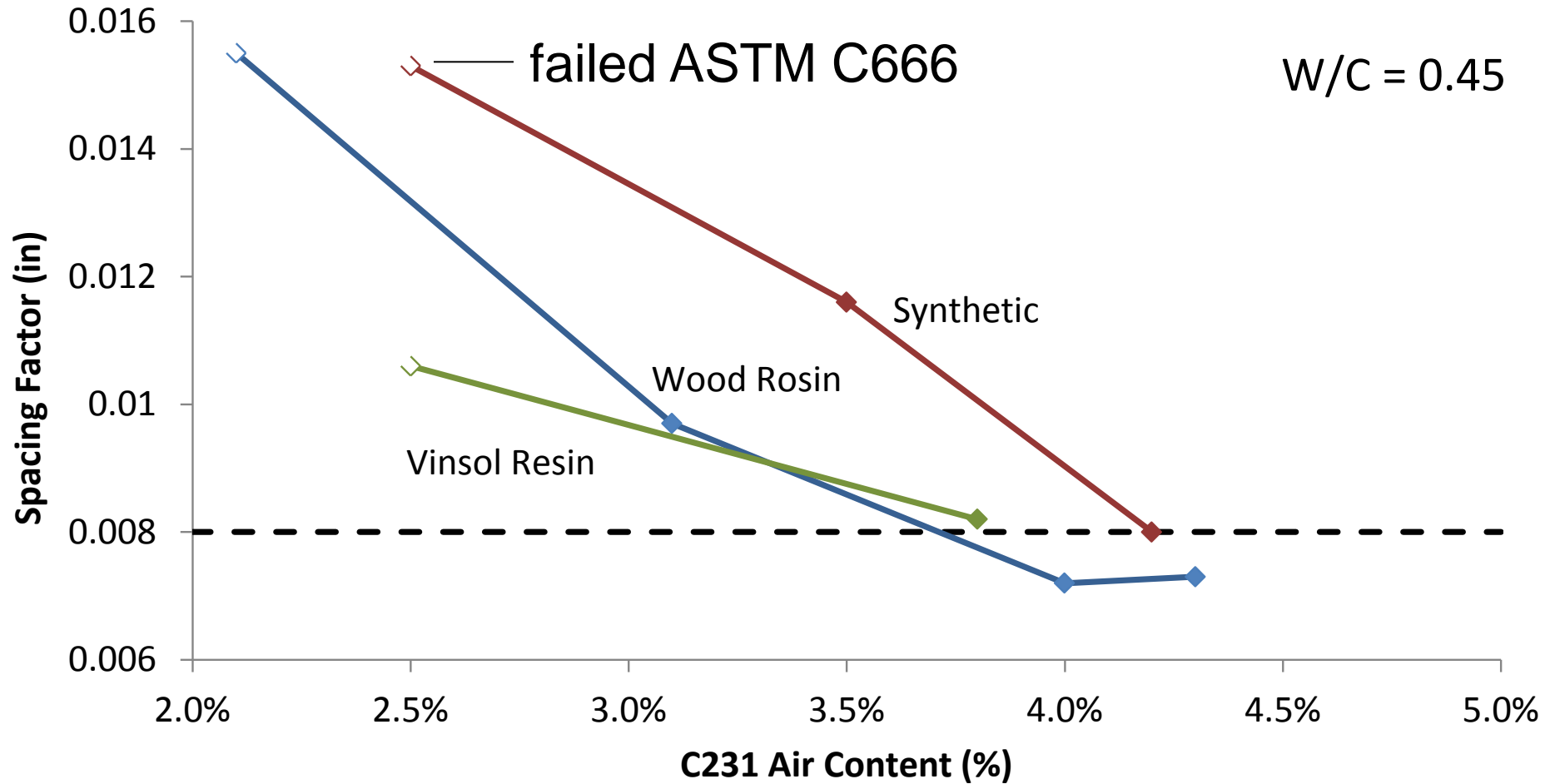
- Concrete mixtures were investigated with ASTM C666 testing to look at the following variables:
  - Vinsol resin, wood rosin, and synthetic AEAs at different air contents at 0.45 and 0.41 w/cm
  - Combinations of mid range WR and wood rosin AEA at 0.41 and 0.38 w/cm

<b>w/c ratio</b>	<b>Cement <i>lb/yd</i><sup>3</sup></b>	<b>Coarse <i>lb/yd</i><sup>3</sup></b>	<b>Fine <i>lb/yd</i><sup>3</sup></b>	<b>Water <i>lb/yd</i><sup>3</sup></b>
0.38	611	1950	1203	232
0.41	611	1900	1129	250.5
0.45	611	1850	1203	275

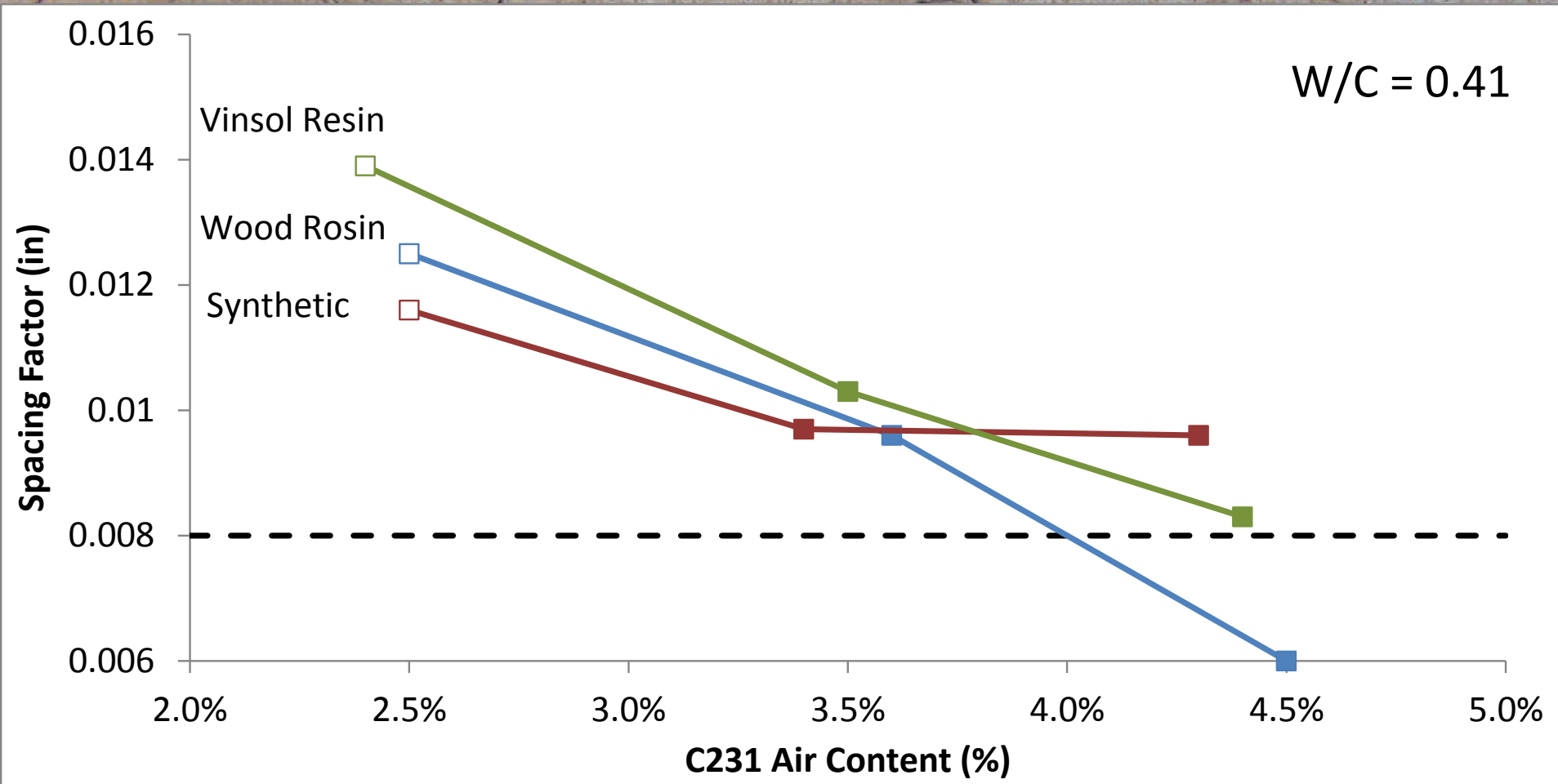


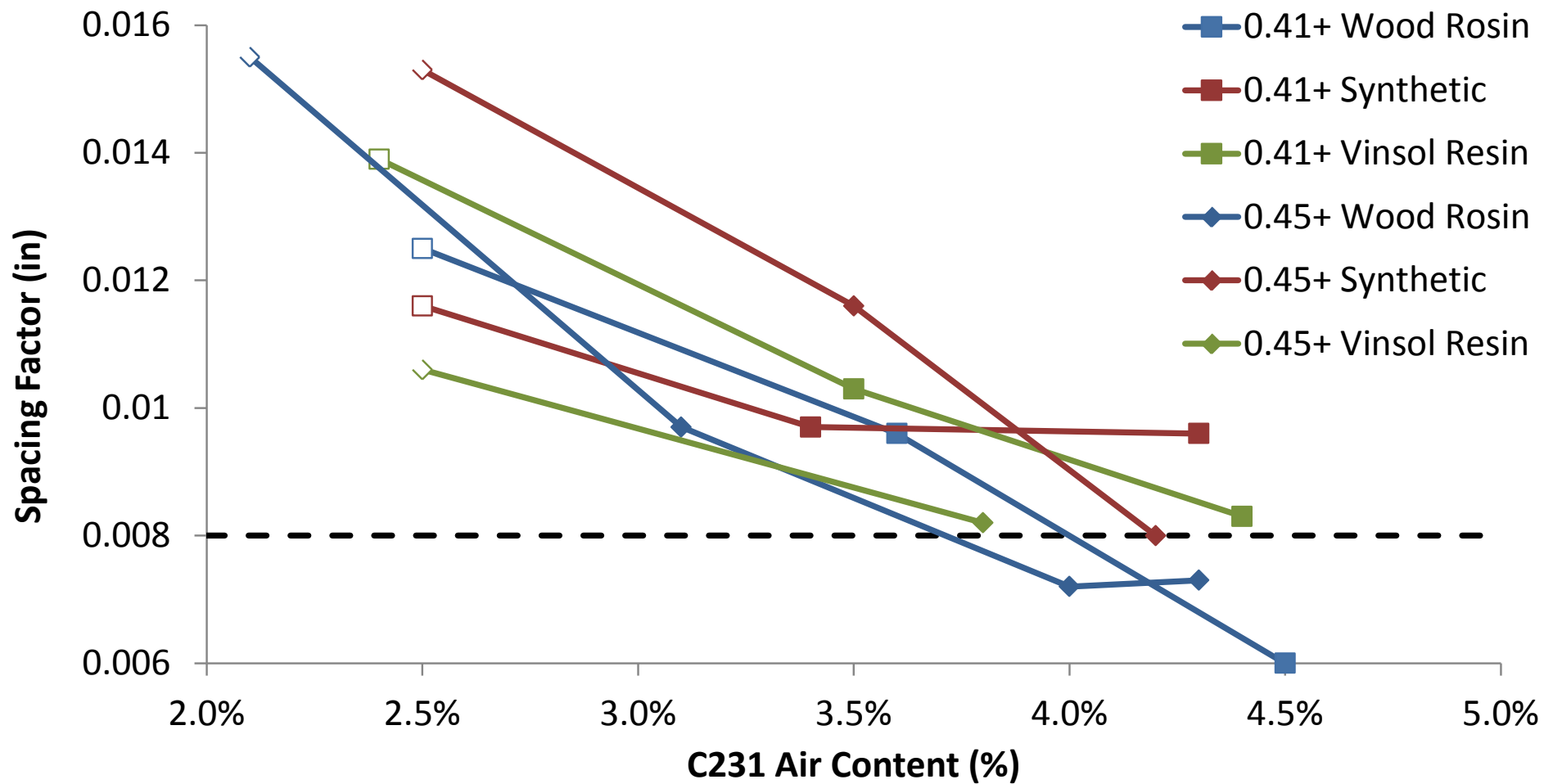
# Laboratory work at OSU

- The following were measured:
  - Slump
  - Unit weight
  - Pressure meter air
  - Strength
  - Hardened air
    - spacing factor
    - specific surface
    - total air

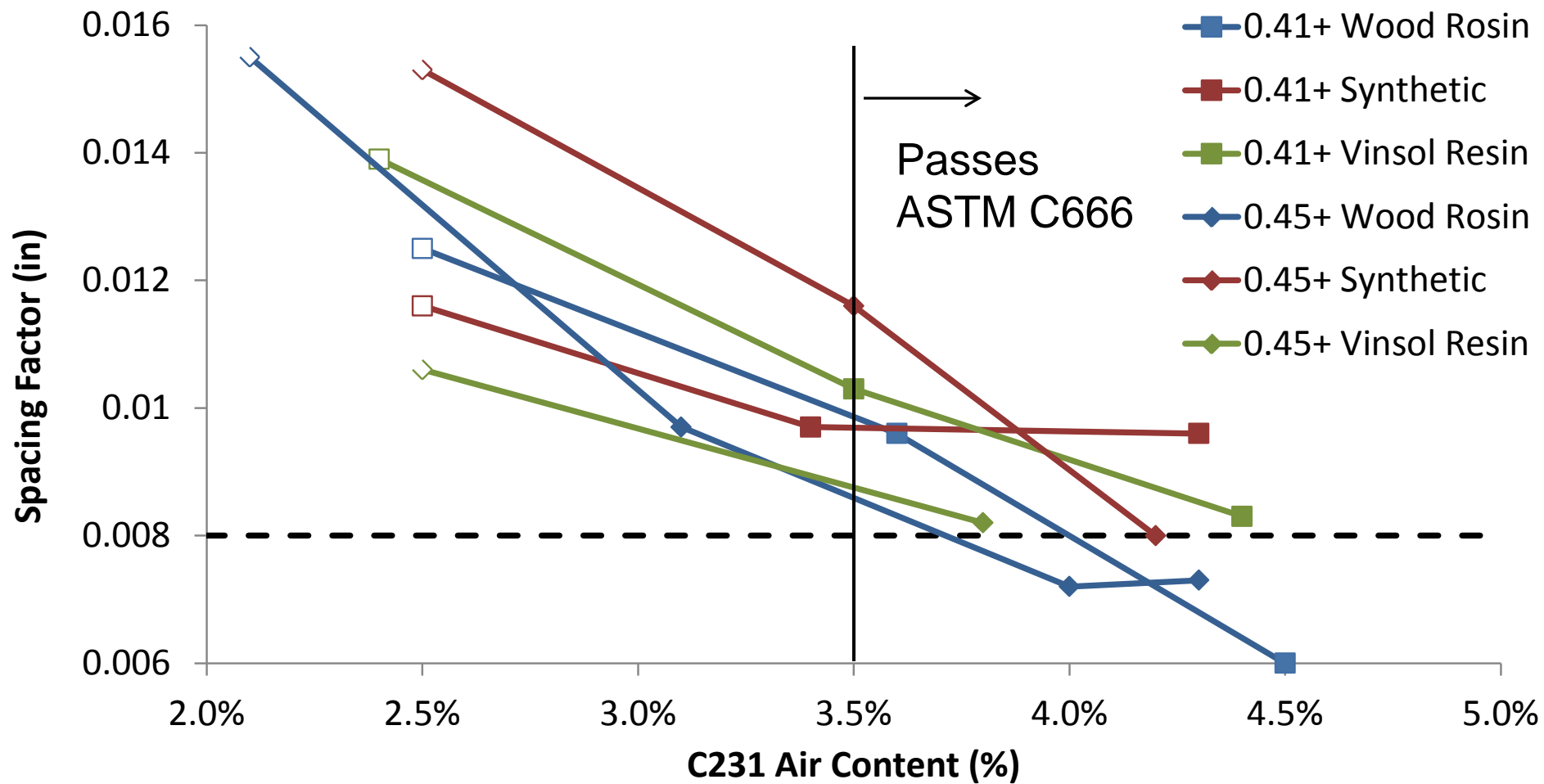


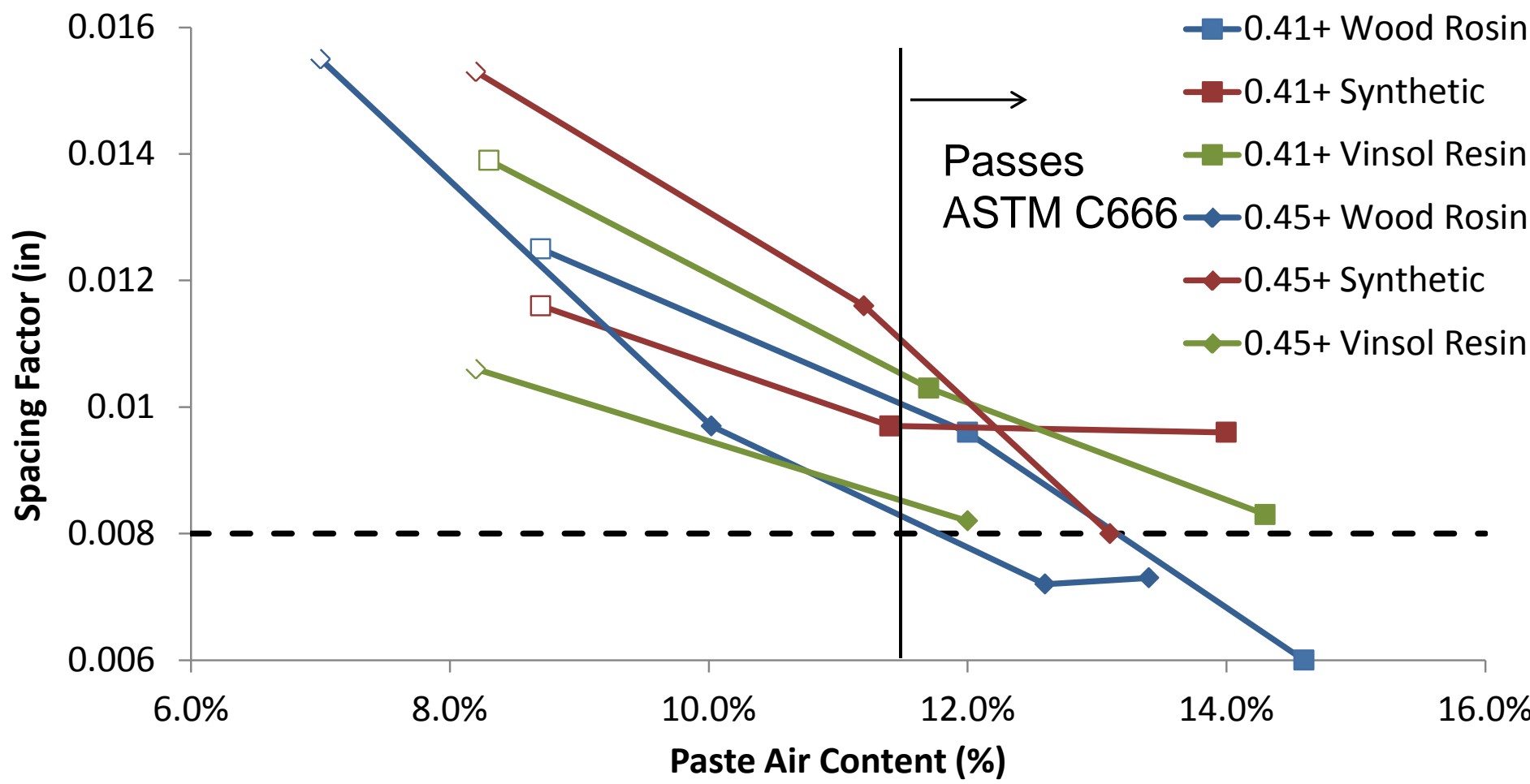


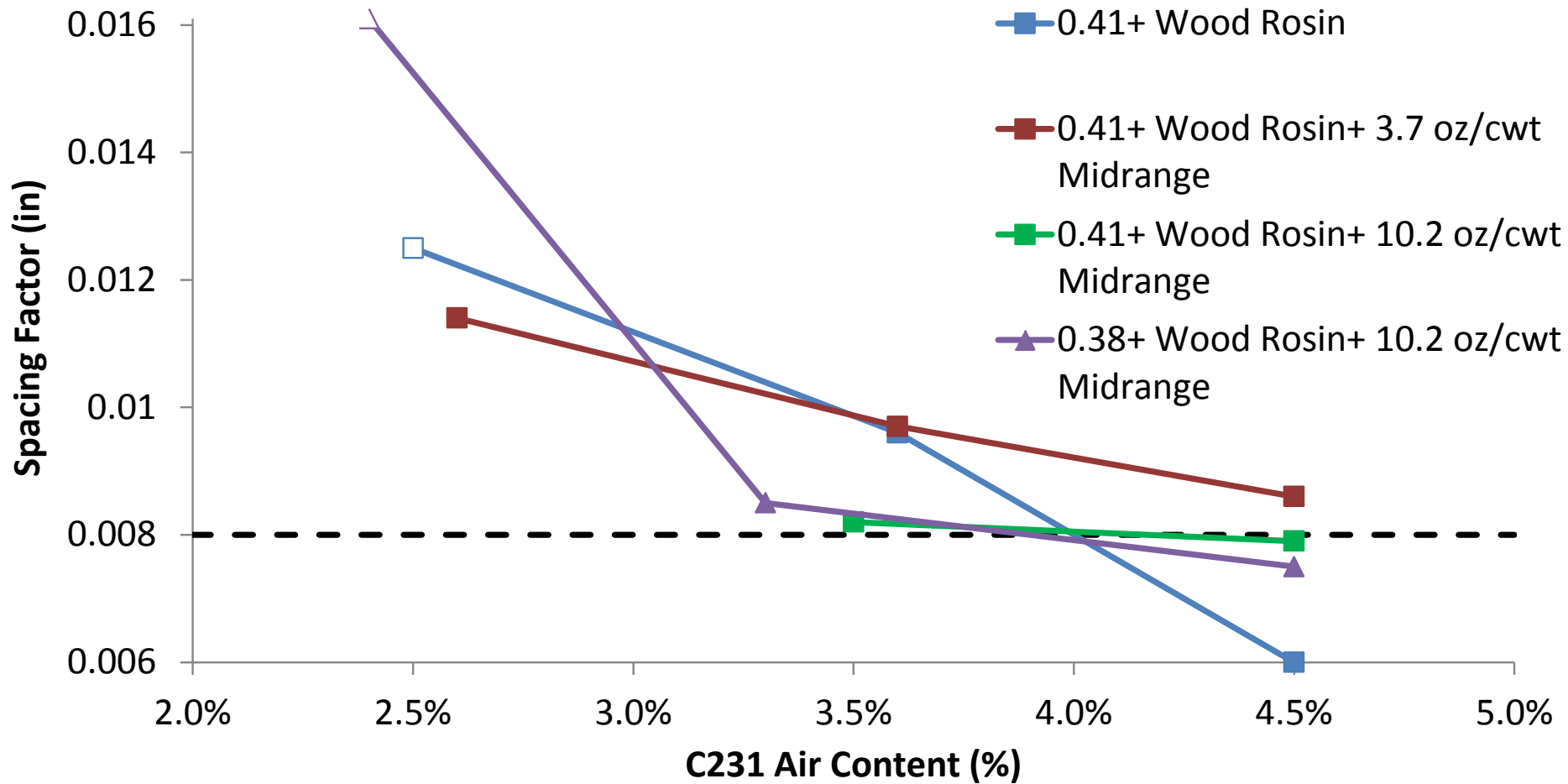




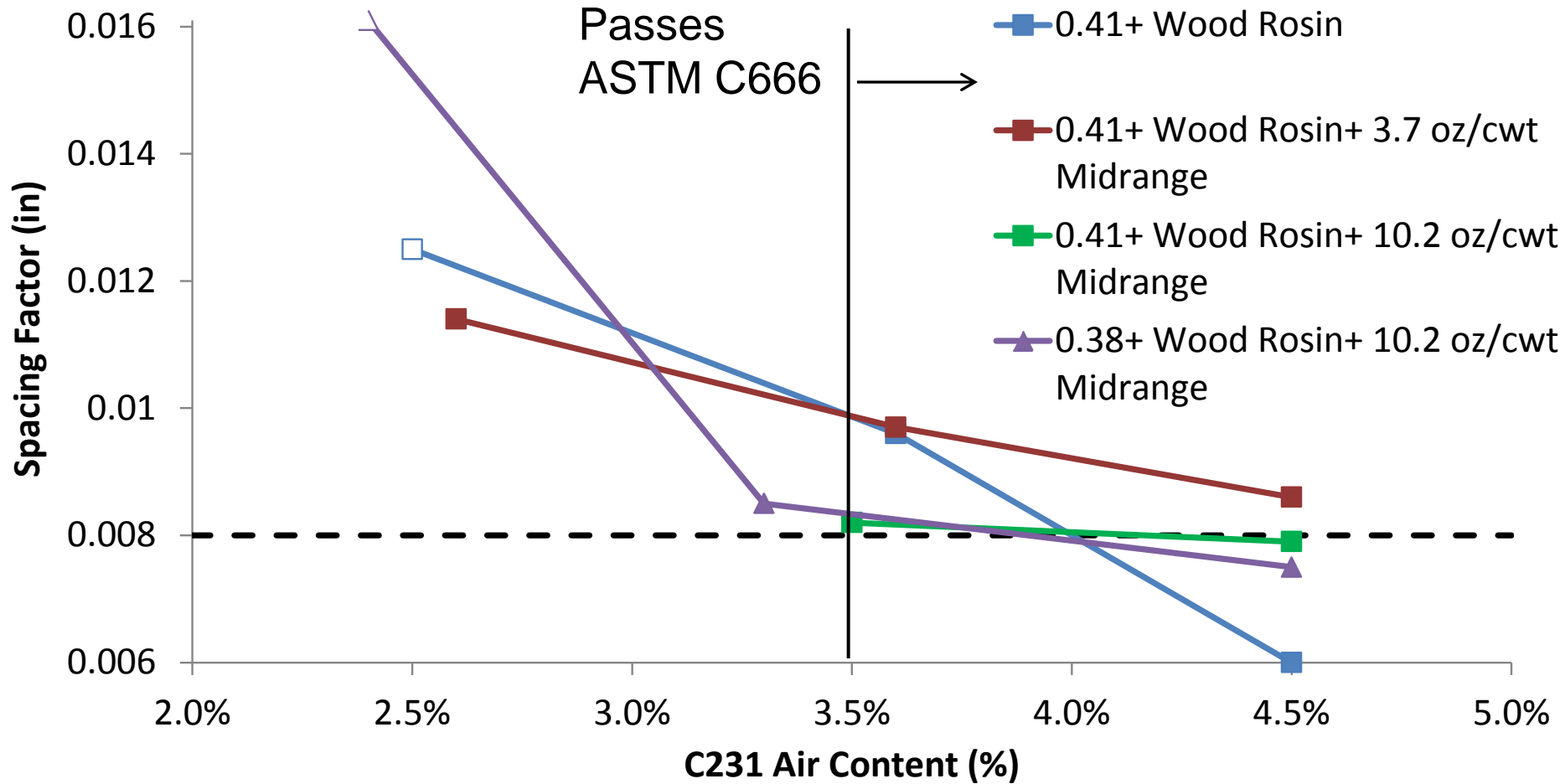


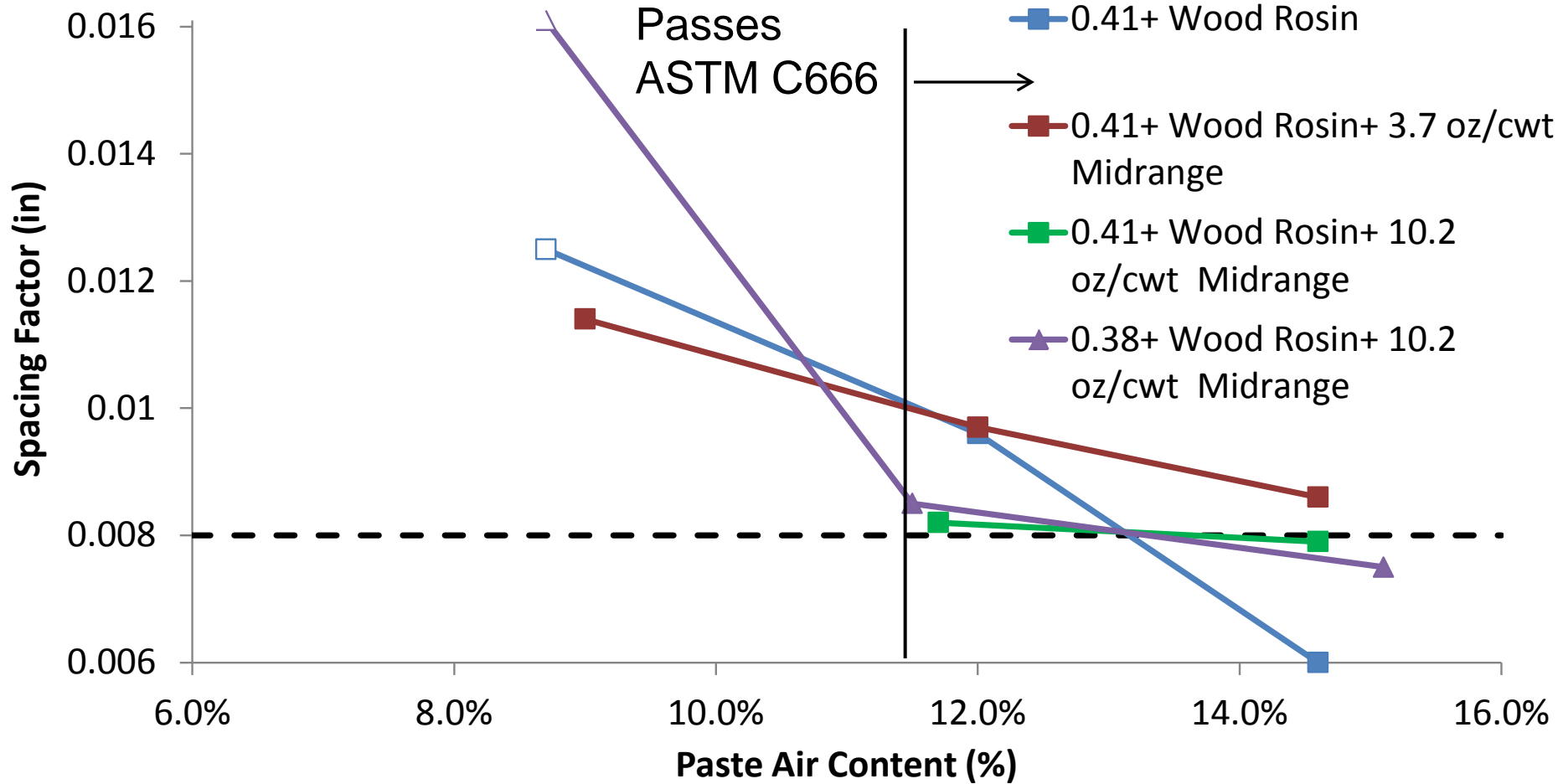


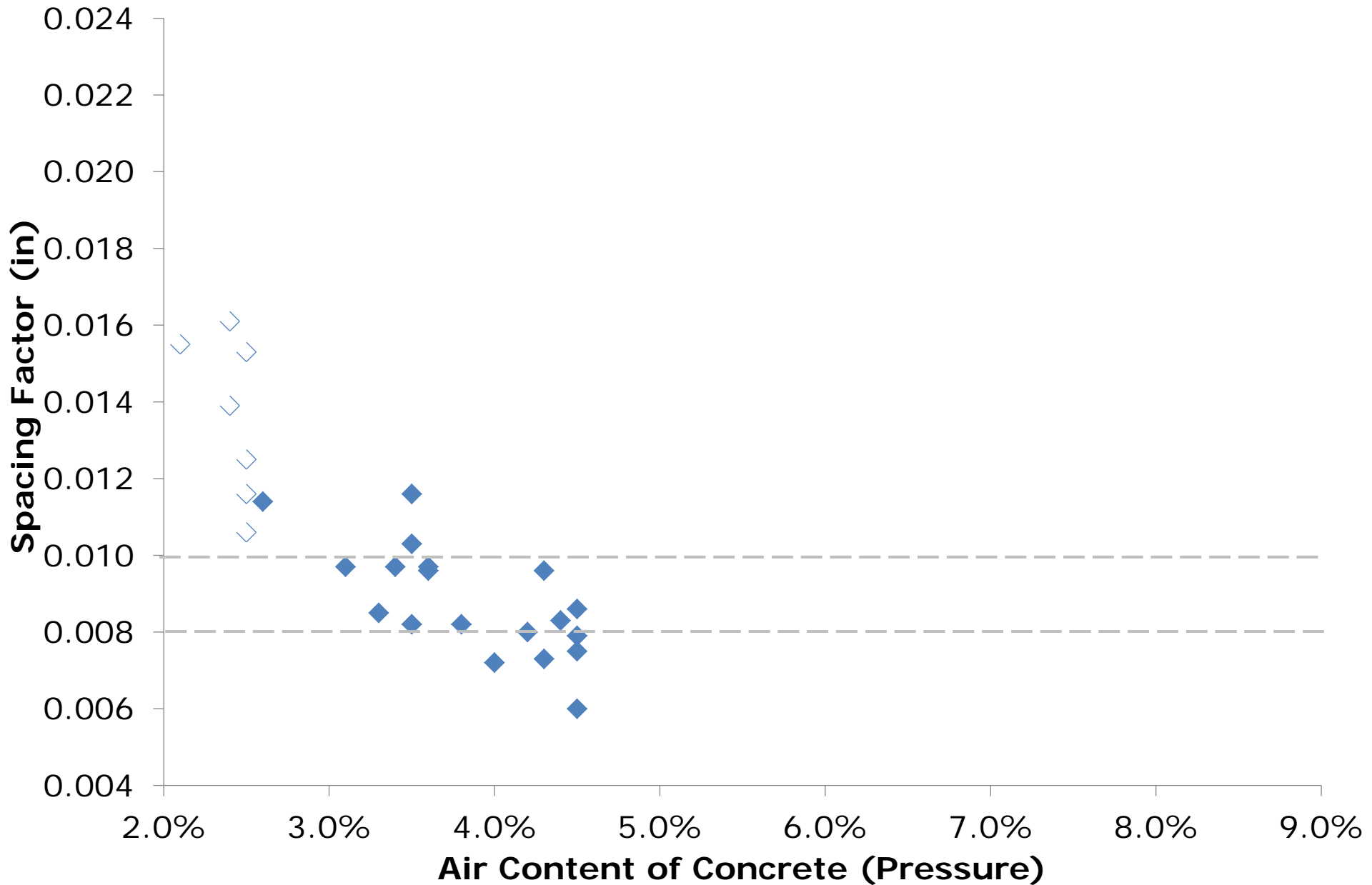




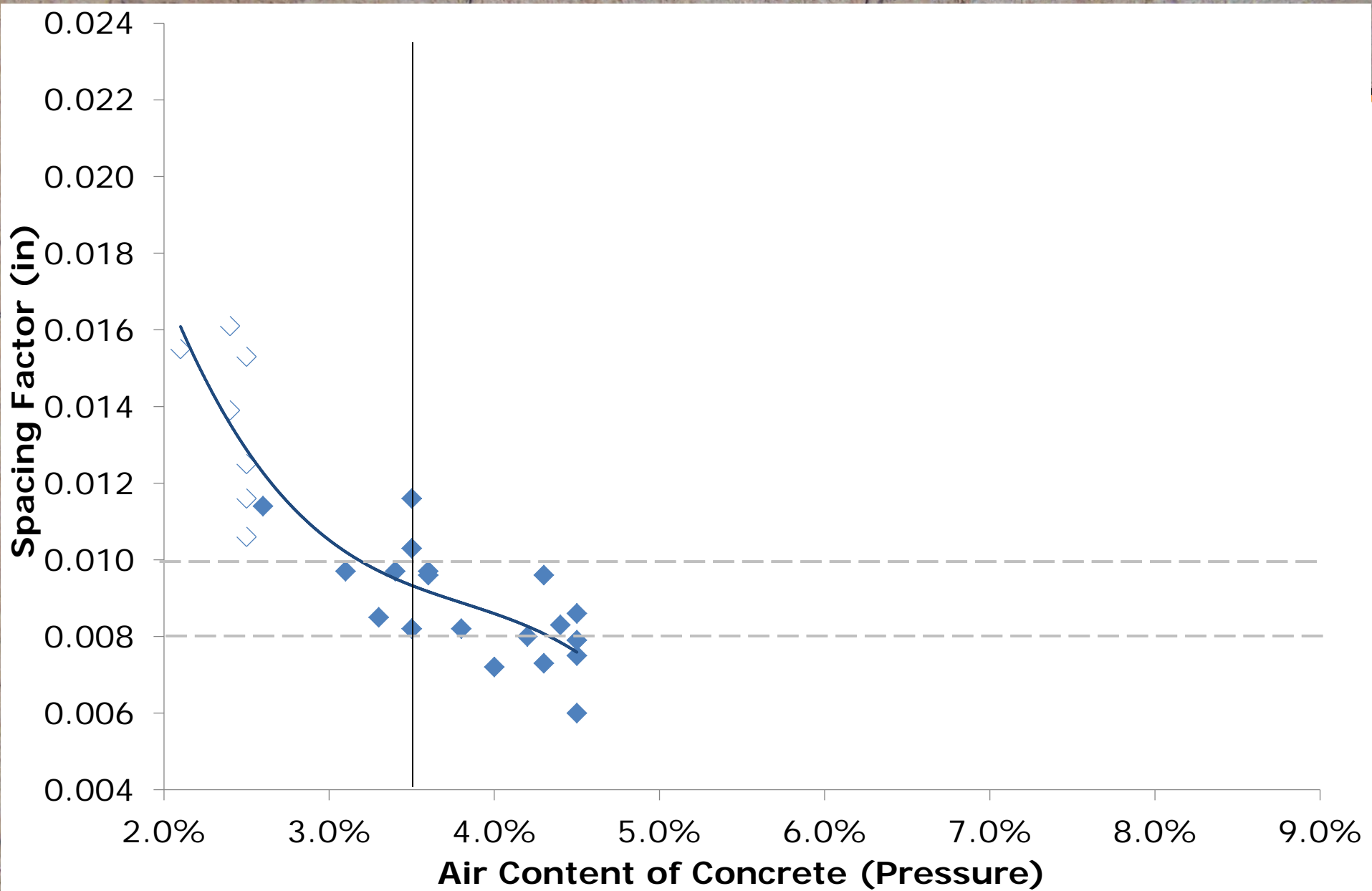












# What does this mean???

- Based on the testing presented it appears that concrete with an air content greater than 3.5% or a paste air content of 11.5% should be frost durable for the mixtures shown.
- A safety factor should be expected to be placed on this value for field usage

# What does this mean???

- From the limited laboratory data investigated it appears that Klieger's recommendations (and therefore the modern AEA specifications) are appropriate for the modern mixtures presented
- Care needs to be taken that the required air content is obtained in the hardened concrete!!!!



# What does this mean???

- Although it is common to specify .008” as a limit for the required spacing factor in hardened concrete the data presented suggests that .010” and occasionally .012” have been found to provide satisfactory performance in the ASTM C666 test for the materials and mixing procedures used.
- This finding is in line with the Canadian Standard Specification

# Questions???



**Greetings Concrete Fans and Welcome to HydrationTheater.com!**

# High Range WR and AEAs

- If things were only that easy...



# Polycarboxylate Super Ps

- PCEs are the latest generation of water reducers
- They are very effective WRs that have very little impact on setting
- They are the only way to effectively obtain very high water reduction
- There is a trend to make both midrange and HRWRs based on PCEs

# Polycarboxylate Super Ps

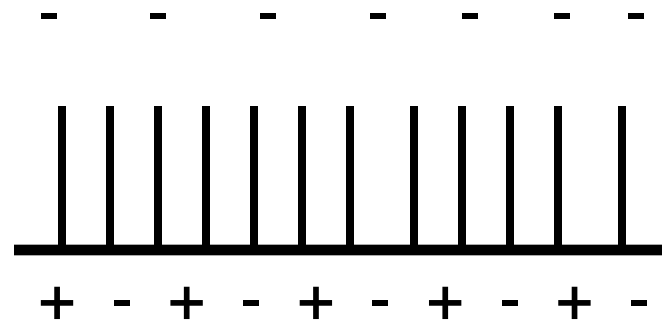
- PCEs are a very important part of the industry
- They allow for great improvements in economy, durability, sustainability, and performance of concrete

# Polycarboxylate Super Ps

- PCEs are comb shaped surfactants that are attracted to the surface of cement grains.
- Through steric hindrance they push the grains away from one another
- This allows the mixture to flow



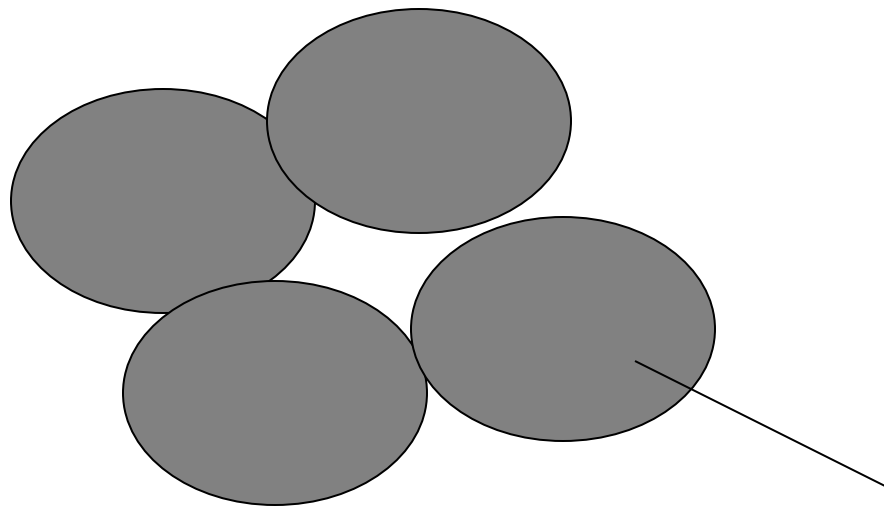
PCE molecule



Negative charges

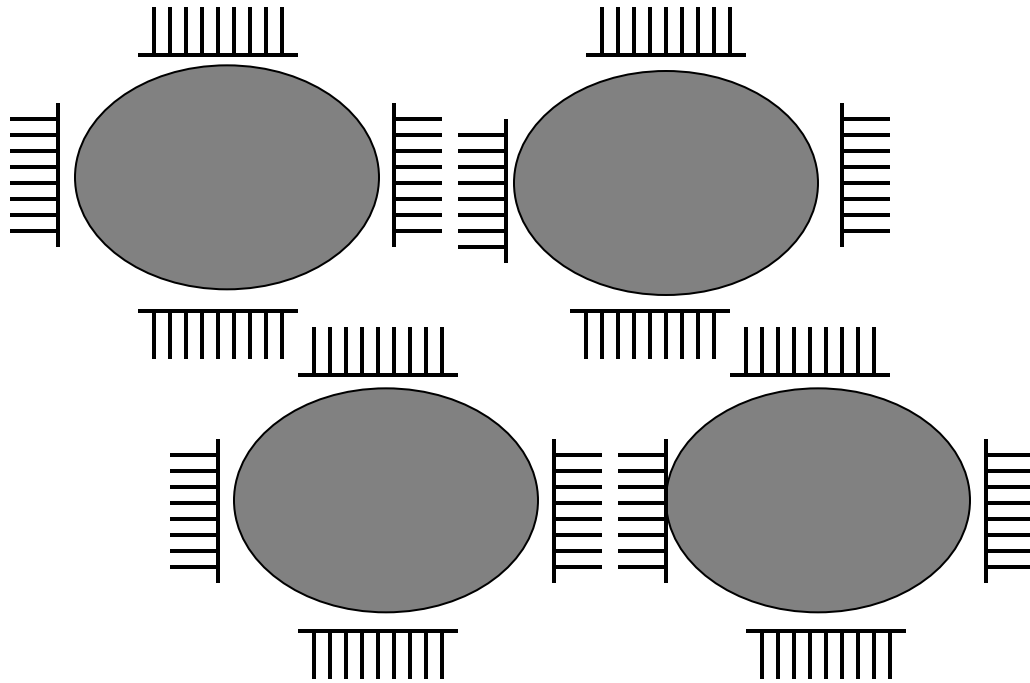
Positive and negative charges

No PCE



cement grain

with PCE





# Polycarboxylate Super Ps

- While this ability to repel cement grains creates great water reduction it causes some challenges with air entrainment

# Polycarboxylate Super Ps

- PCEs are surfactants!
- These comb shaped molecules will entrain air
- This air is commonly coarse and there is A LOT of it
- To lower the air content it is common to add a defoamer to a PCE
- Different PCE and defoamer packages are used

# Polycarboxylate Super Ps

- The admixture system is now very complicated as we have ingredients that are trying to both build and destroy air bubbles
- Other admixtures may be used as well
- Behavior is cement dependant
- This interaction is very complicated and is still being studied



# Polycarboxylate Super Ps

- There have been a number of reports from the field about PCE concrete that loses air and slump simultaneously over time
- OSU has investigated this phenomenon with laboratory testing

# Laboratory Testing

- Concrete mixtures were made with five different PCEs, three different AEAs, and w/cm between 0.37 and 0.50

	CA	FA	Water	Cement	w/c
Mixture 1	1850	1203	275	611	0.45
Mixture 2	1815	1180	301.6	599.5	0.50
Mixture 3	1907	1240	231	630	0.37
Mixture 4	1900	1129	250.5	611	0.41

# Laboratory Testing

- These mixtures were designed to have a water slump between 0.5" and 2" and then PCE was used to bring the slump up to 8" to 10" while having an air content between 5 to 7%



# Laboratory Testing

- Mixtures were prepared and then discharged into a wheel barrow.
- Fresh property tests were run and samples were taken at different time intervals

ASTM	Description	Time After Mixing				
		0 min	30 min	60 min	90 min	120 min
C143	Slump	X	X	X	X	X
C138	Unit Weight	X	X	X	X	X
C231	Pressure Meter	X		X		X
N/A	Modified Pressure Meter					X
C666	Freeze Thaw	X		X		

Hardened air void samples were taken at 0, 60 and 120 min

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N/A	Modified Pressure Meter					X
C666	Freeze Thaw	X		X		

Hardened air void samples were taken at 0, 60 and 120 min

# What is the modified pressure meter?

- A thin garbage sack is placed on the inside of a unit weight bucket
- The unit weight bucket with the liner is filled and consolidated in the standard manner after discharge from the mixer (time = 0 minutes)
- The bucket is left to sit for 120 minutes and then tested with the pressure meter



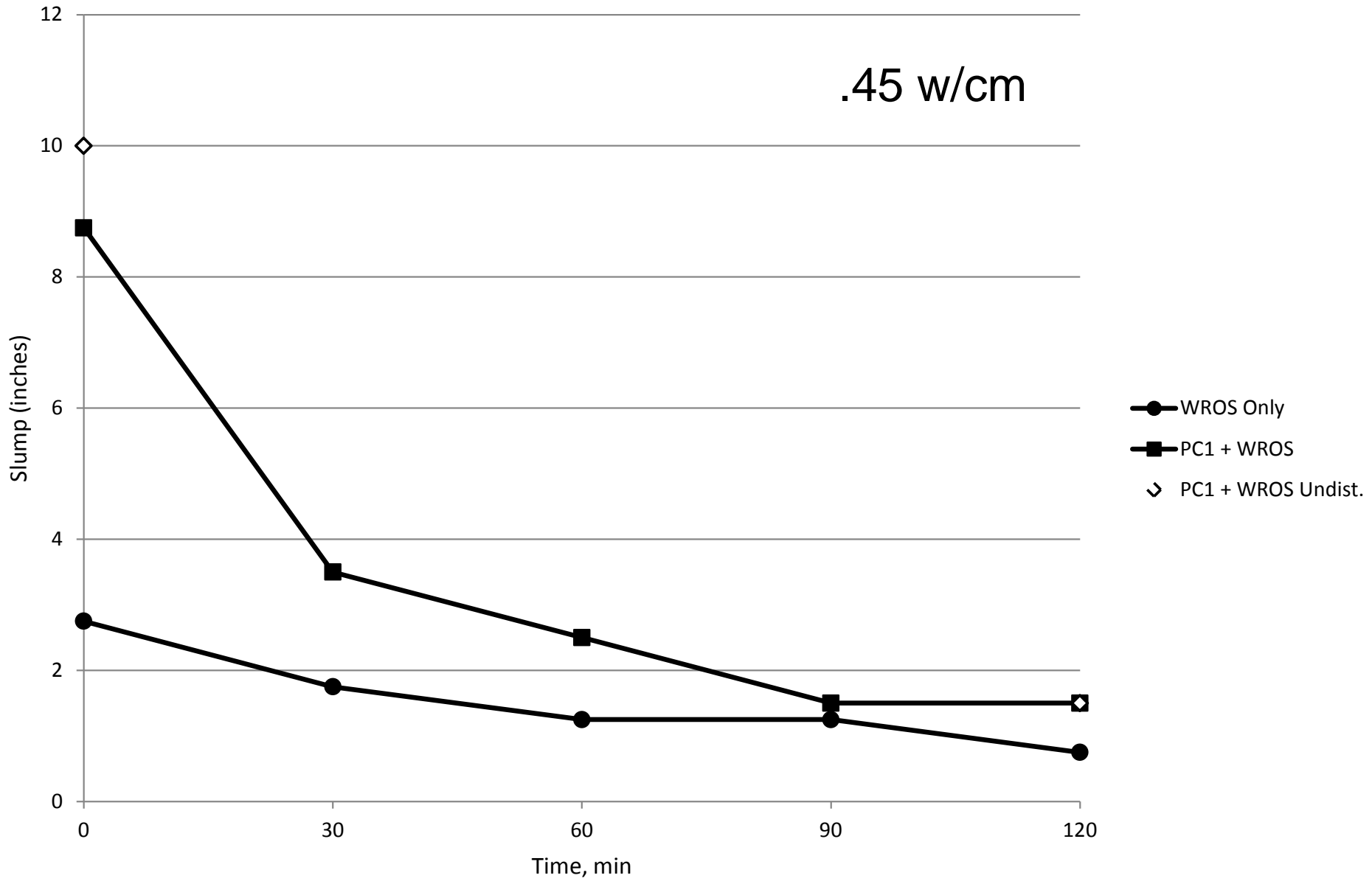
# Laboratory Testing

- All samples were taken without reconstituting the mixture
- Between sampling periods the materials were left in the wheelbarrow untouched
- A mixture was also made where the sample was never touched over the sampling period

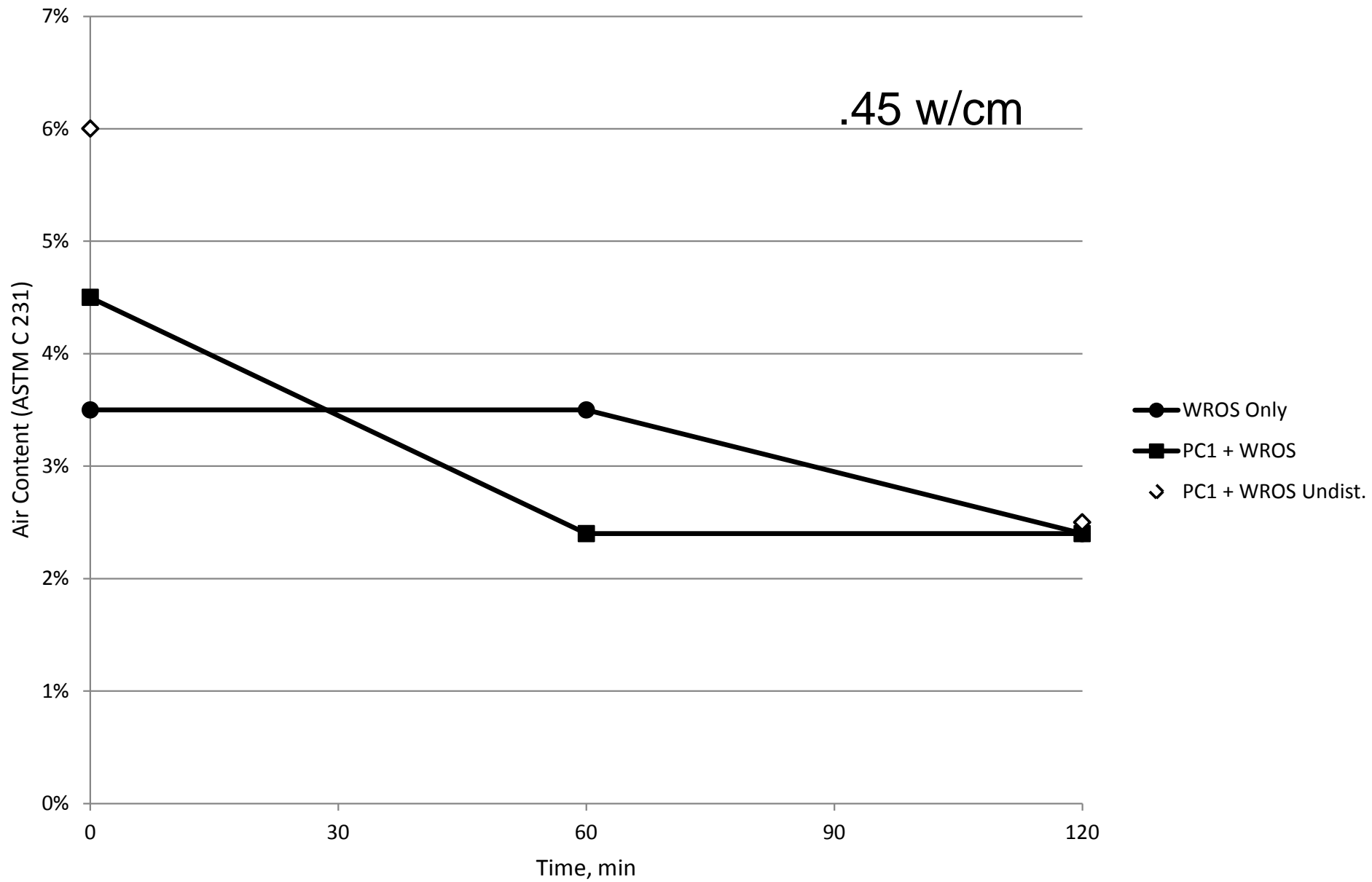
# Results

- Fresh property testing
- Hardened air-void analysis
- Freeze thaw performance

.45 w/cm



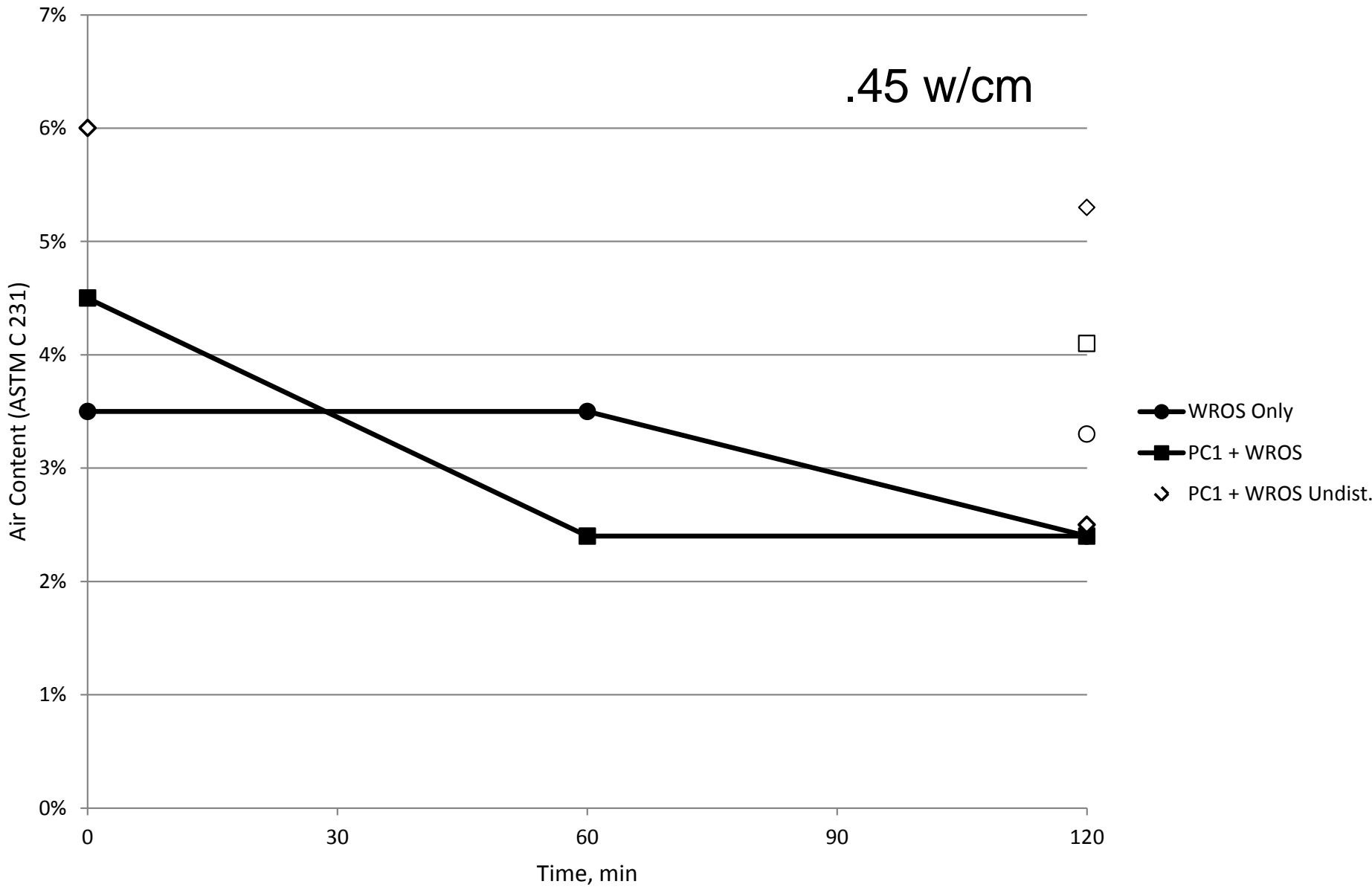




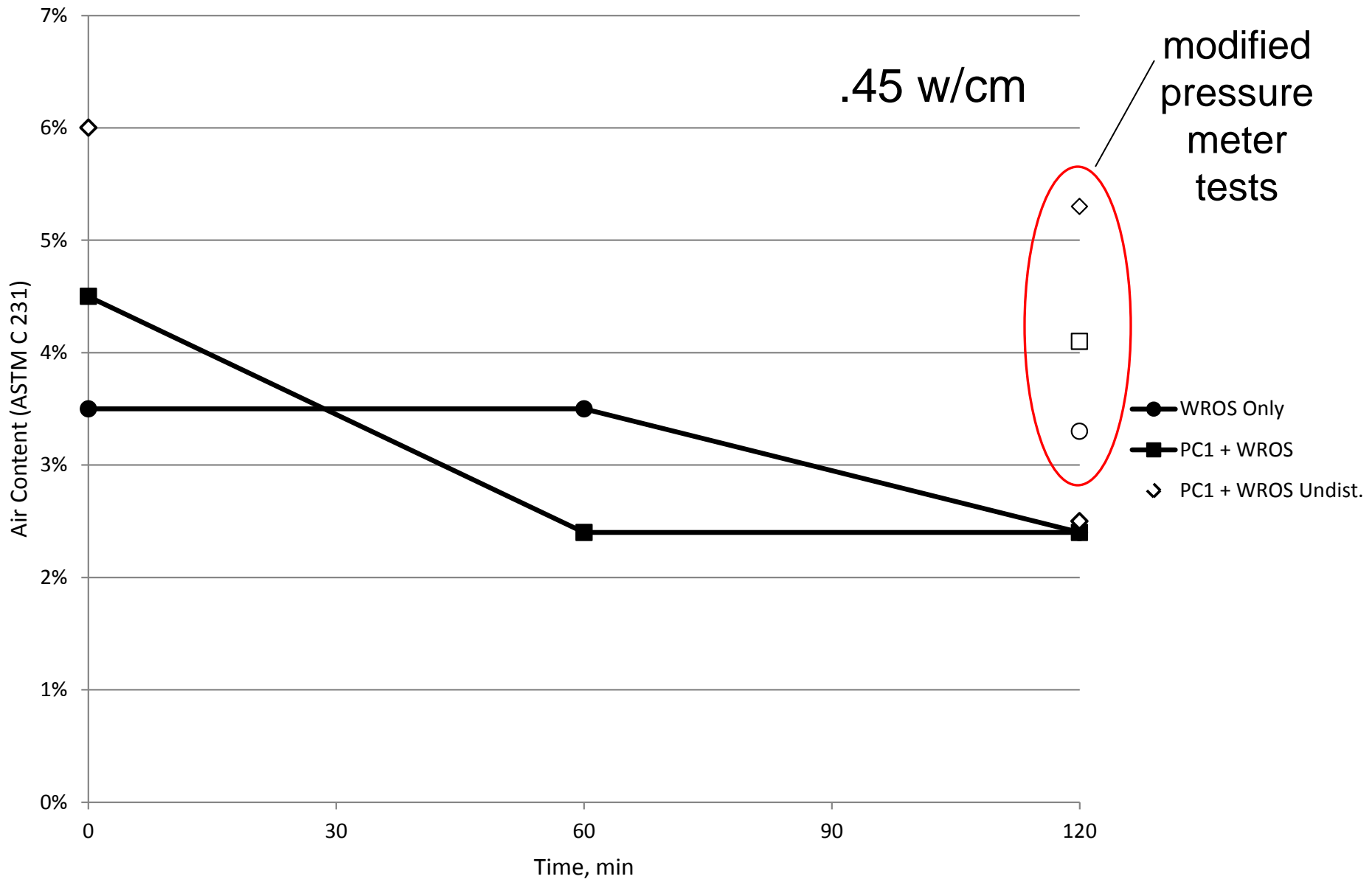
# Observations

- Both the PCE and non PCE mixture lose slump over time
- The PCE loses about 50% of the slump over the first 30 minutes
- The PCE mixture loses air more rapidly than the non PCE mixture
- Both mixtures eventually get to a similar air content

.45 w/cm

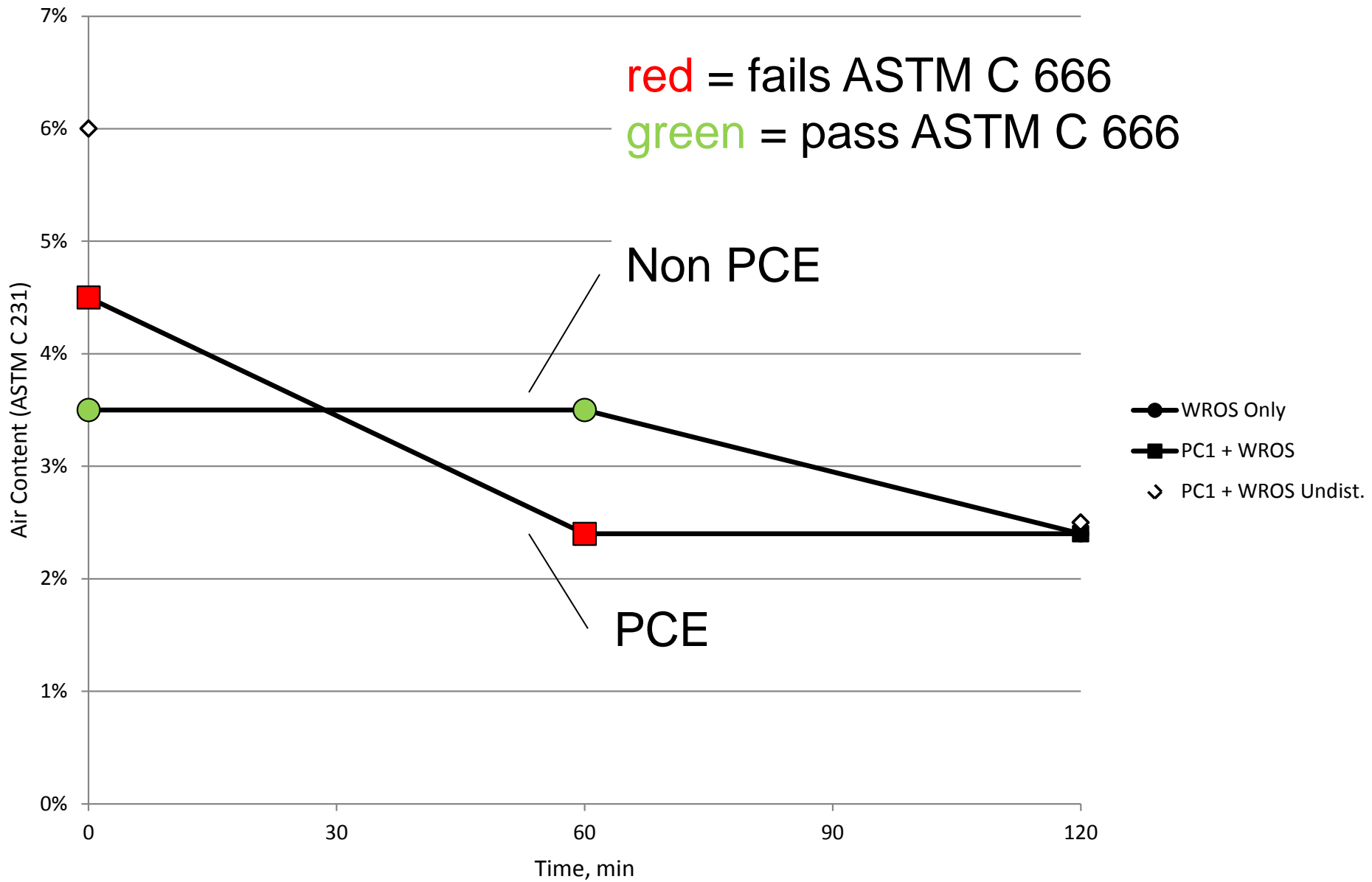




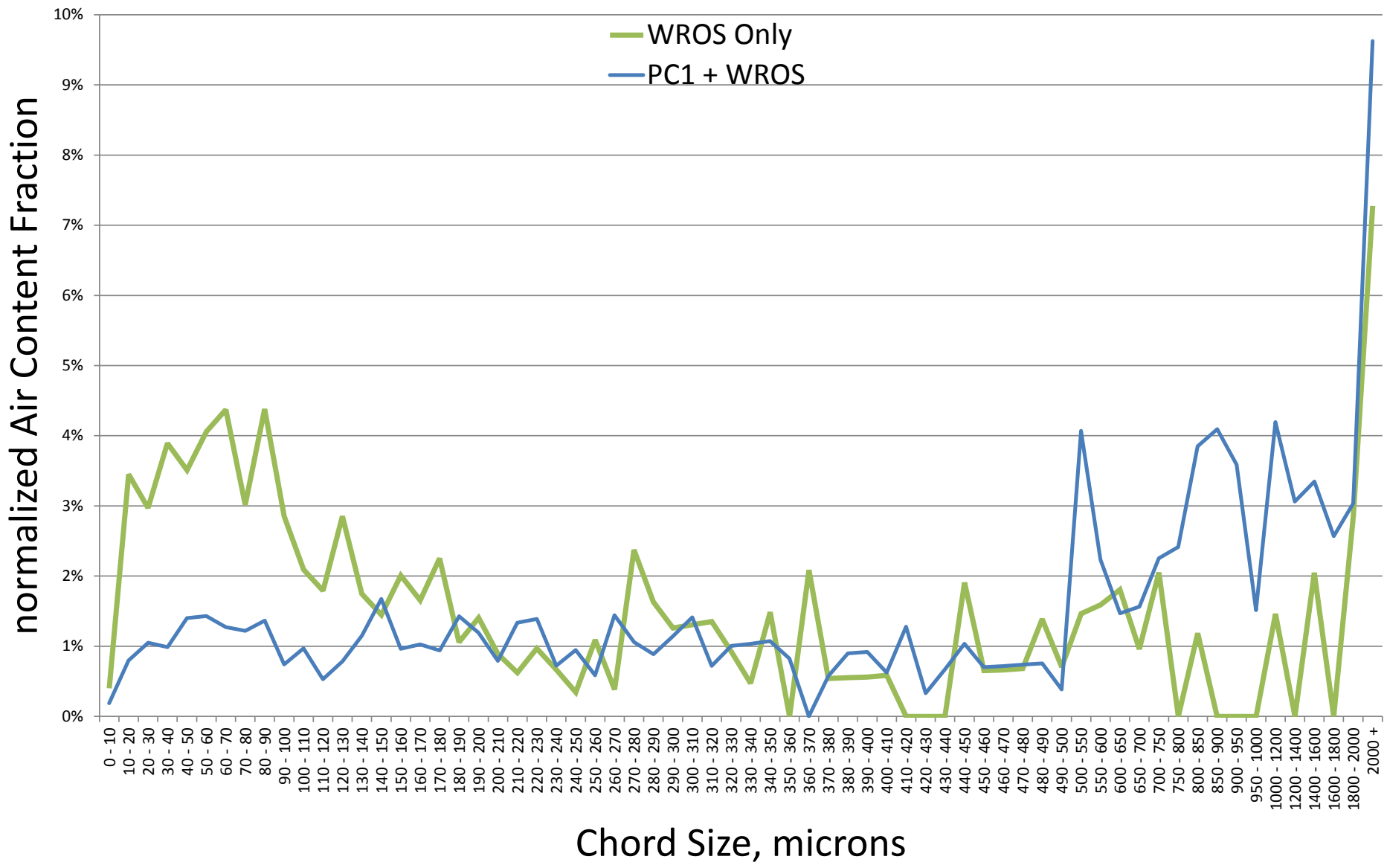


# Observations

- The air contents measured in the modified pressure meter test at 120 minutes are very close to the air contents at 0 minutes
- *It appears that the act of consolidating the concrete has locked the air void system in place!*





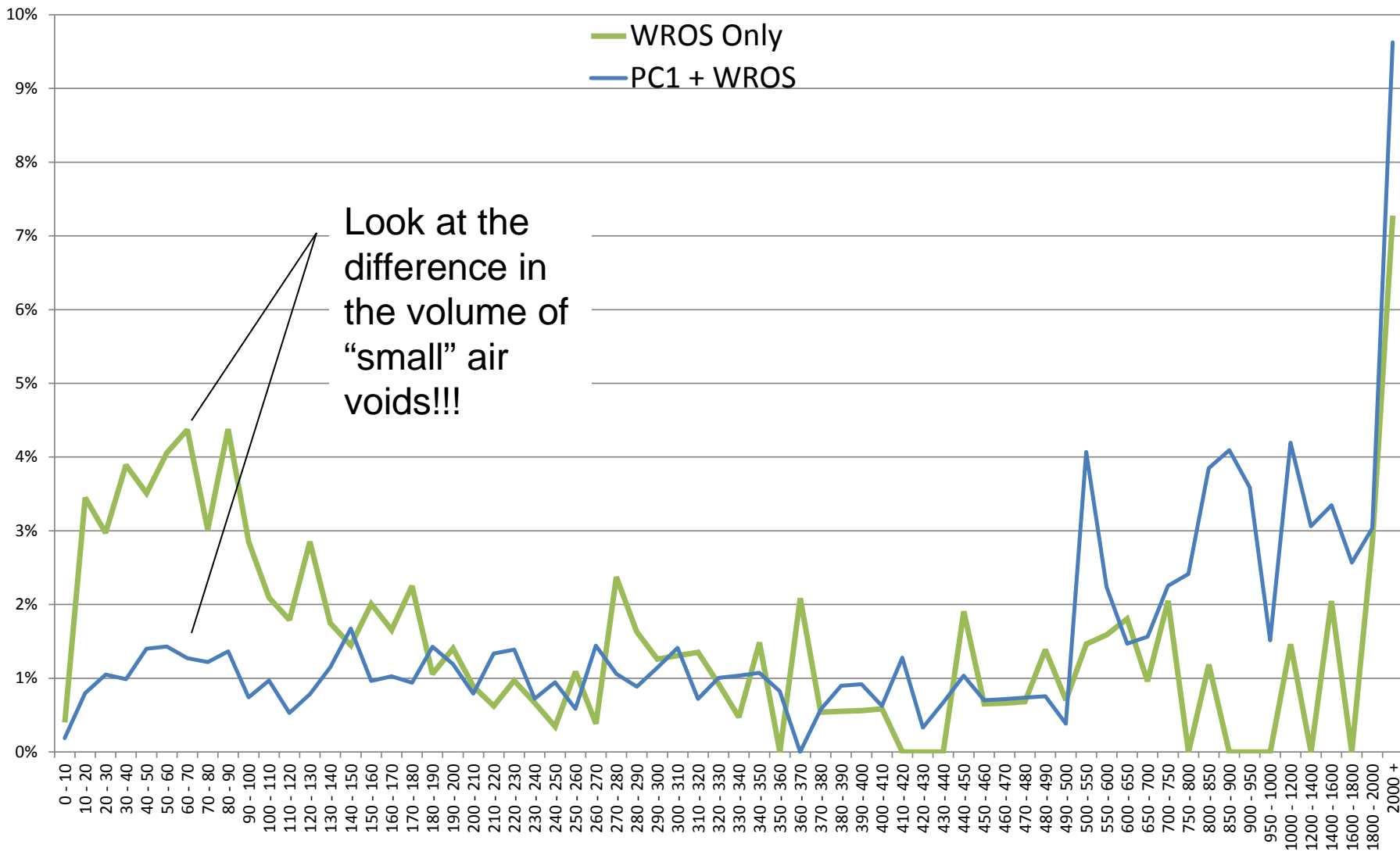


normalized Air Content Fraction

WROS Only

PC1 + WROS

Look at the difference in the volume of "small" air voids!!!

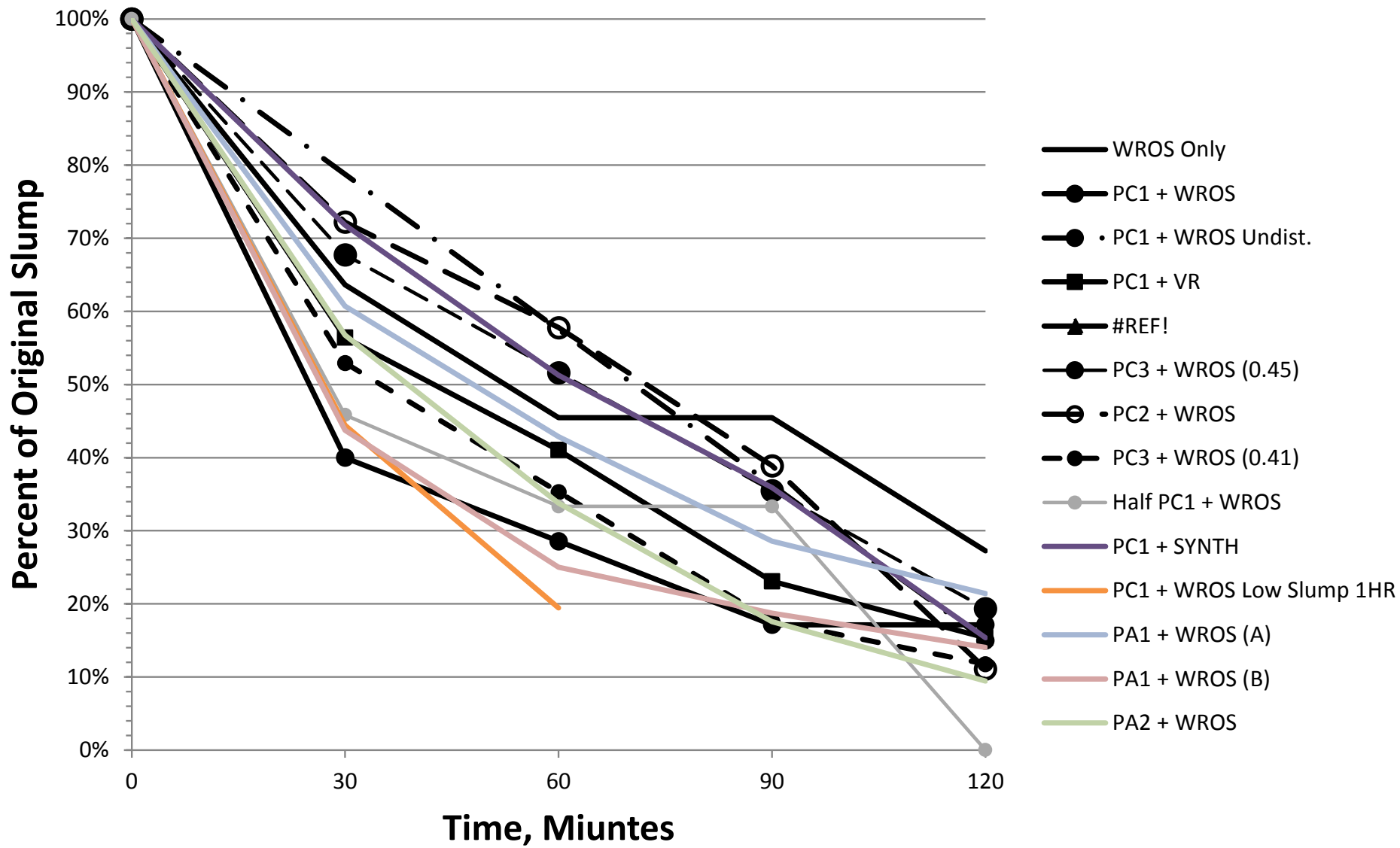


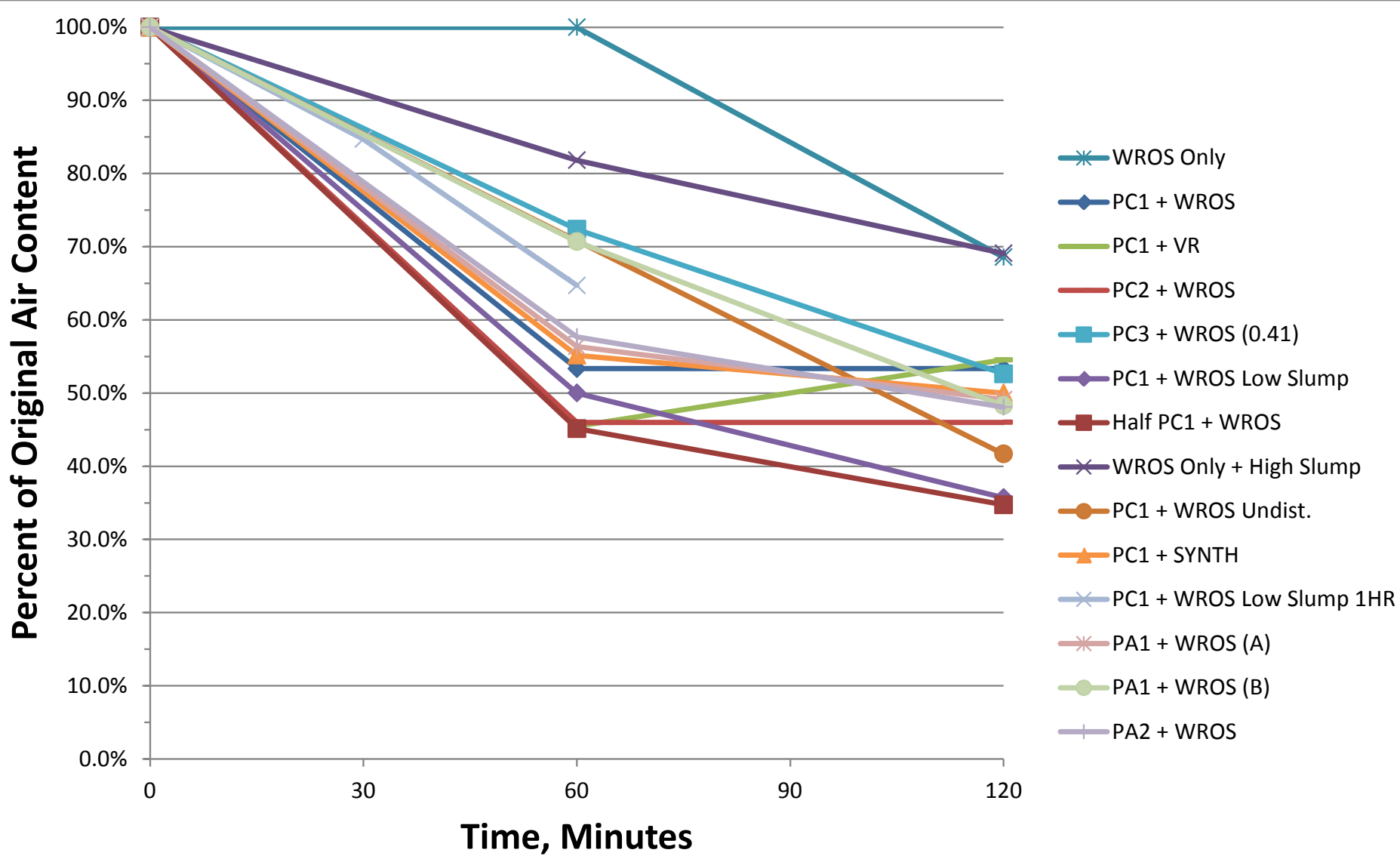
Chord Size, microns

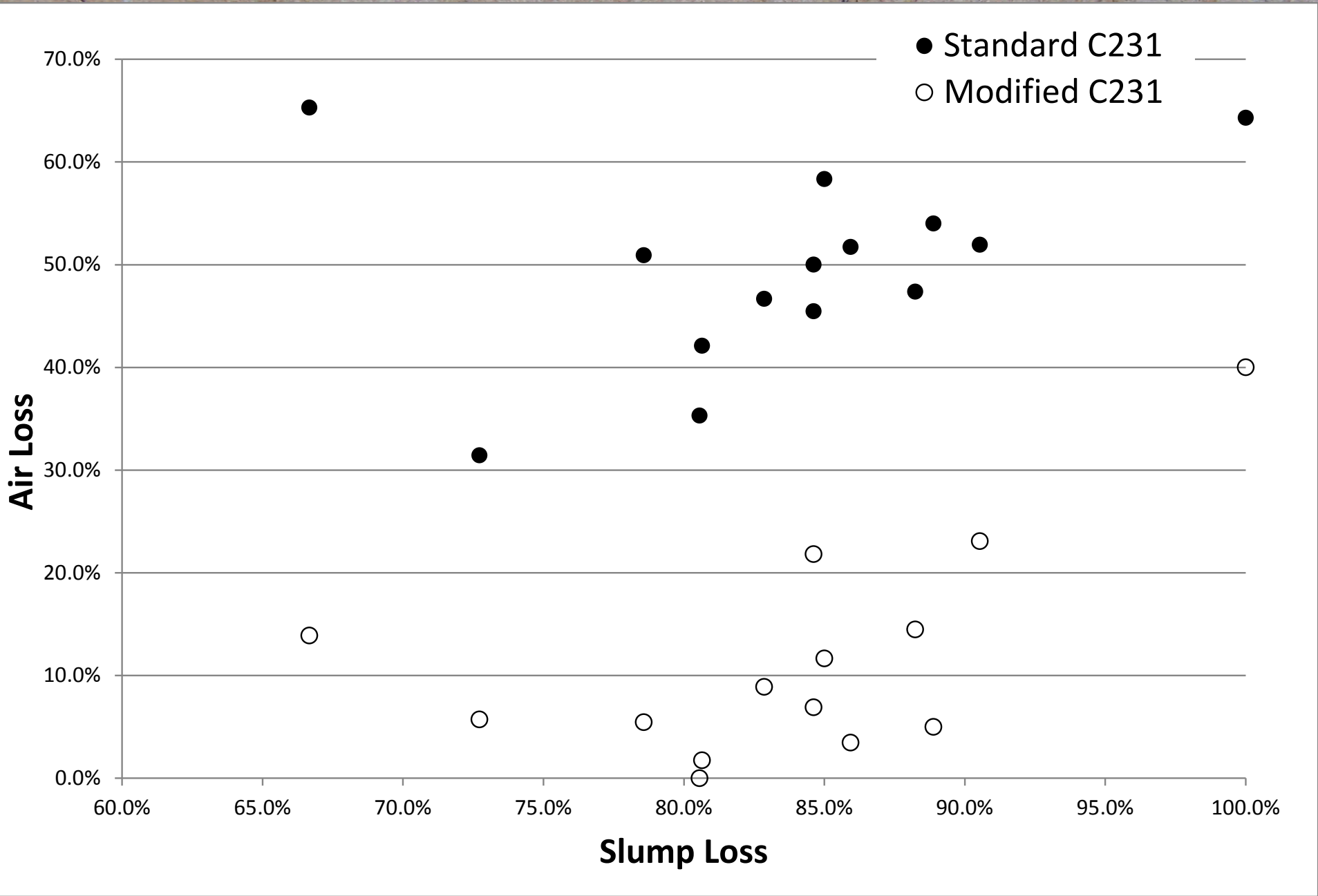
# Observations

- The concrete with out PCE passed the ASTM C666 test
- The concrete with the PCE failed the ASTM C666 test
- This happened despite the mixture with the PCE having the higher air content
- *The hardened air-void analysis suggests that the PCE has a higher percentage of large air-voids than the non PCE mixture*





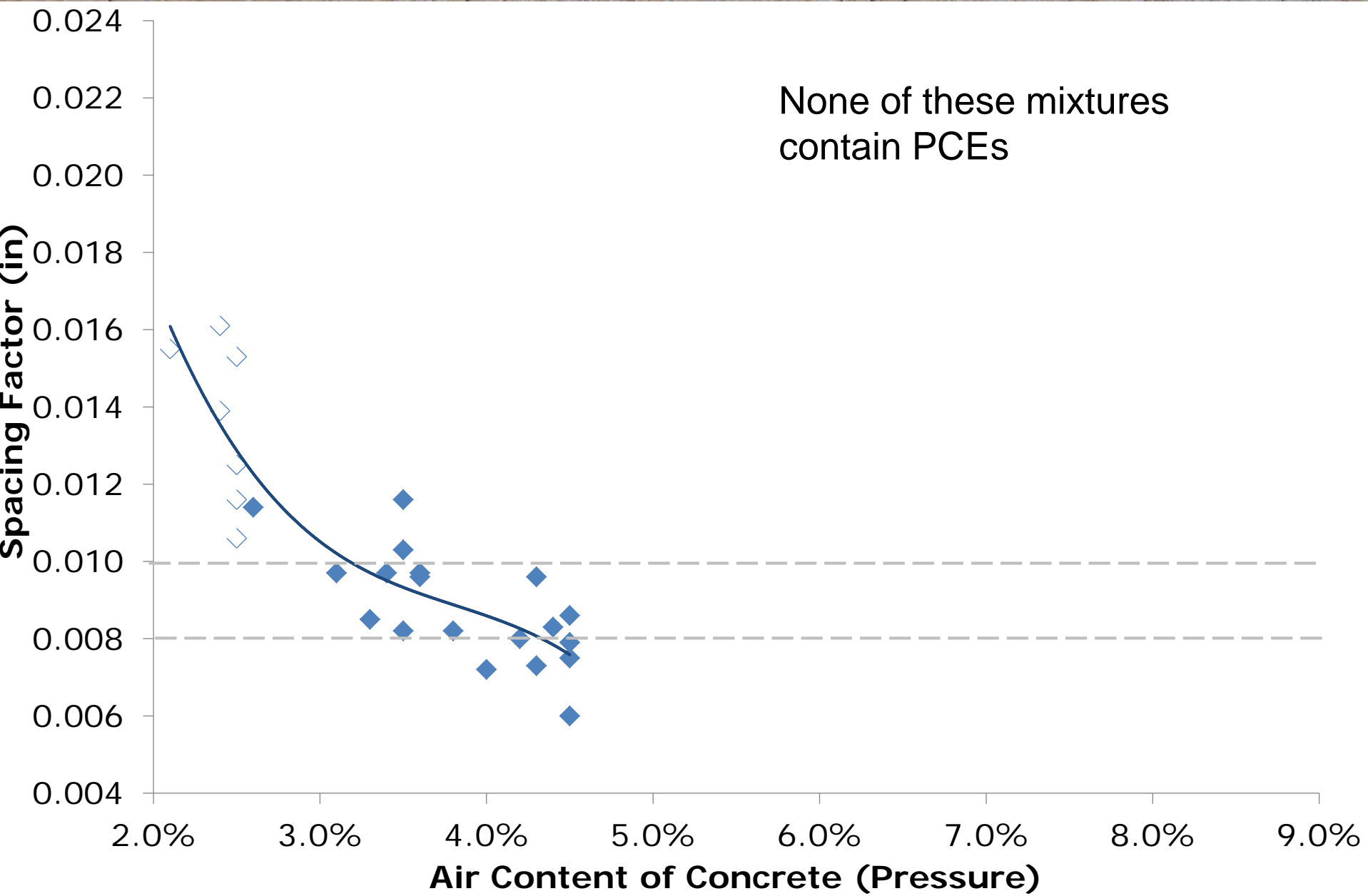


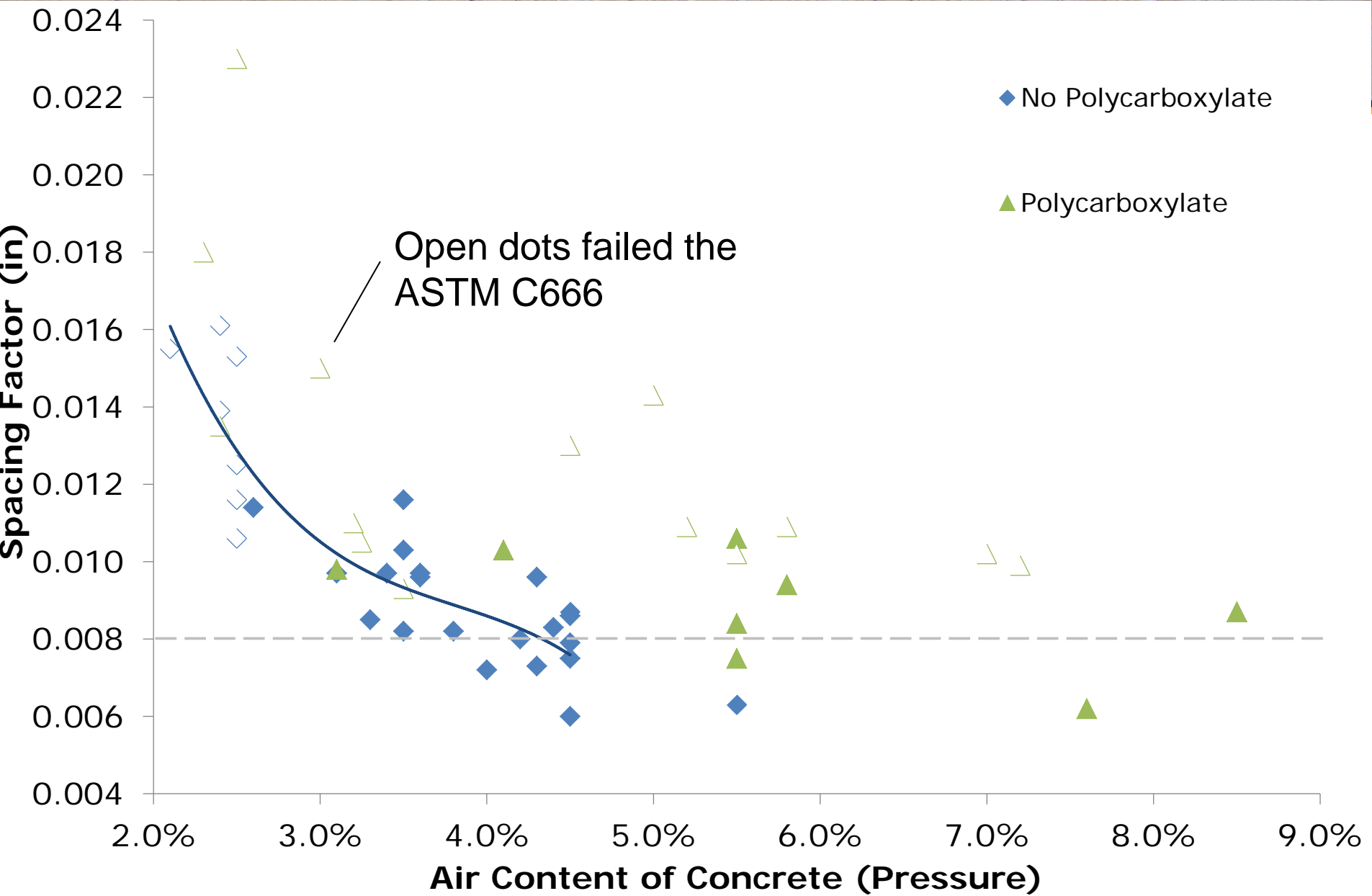




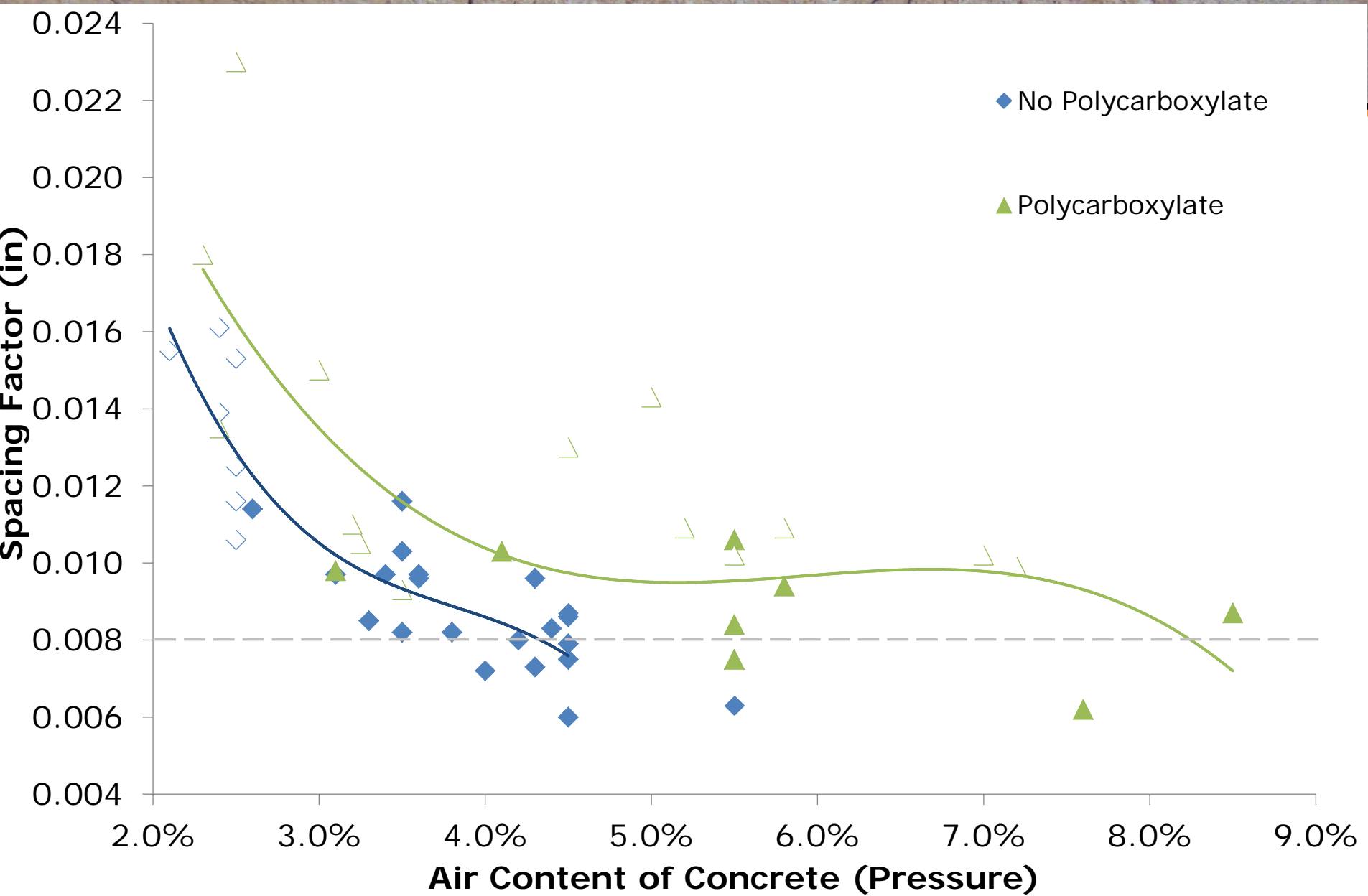
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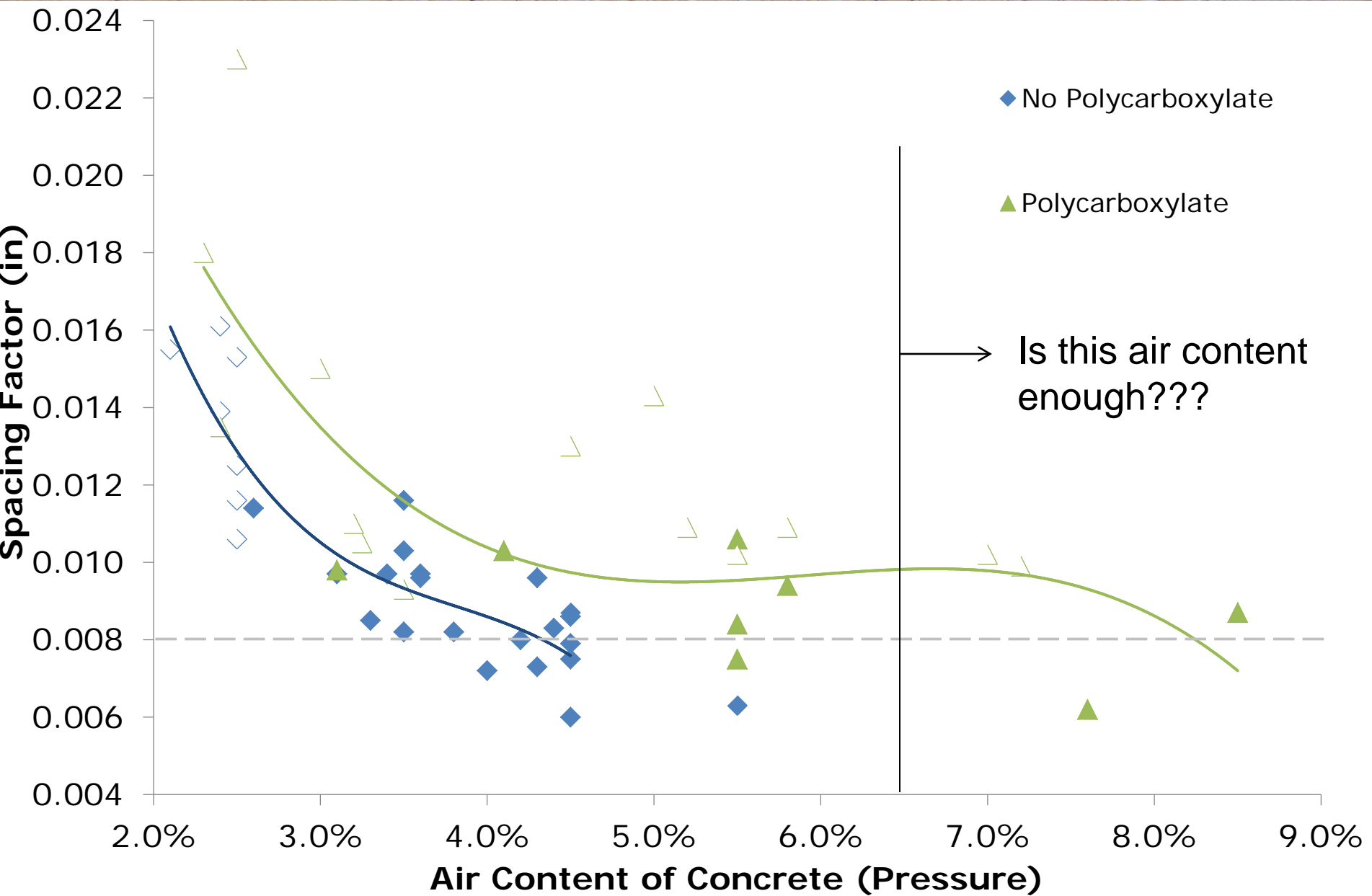
- Similar performance was observed for 5 different PCEs and with three different AEAs
- For all of the mixtures investigated there was a significant difference in the air content between the concrete consolidated at 0 and 120 minutes





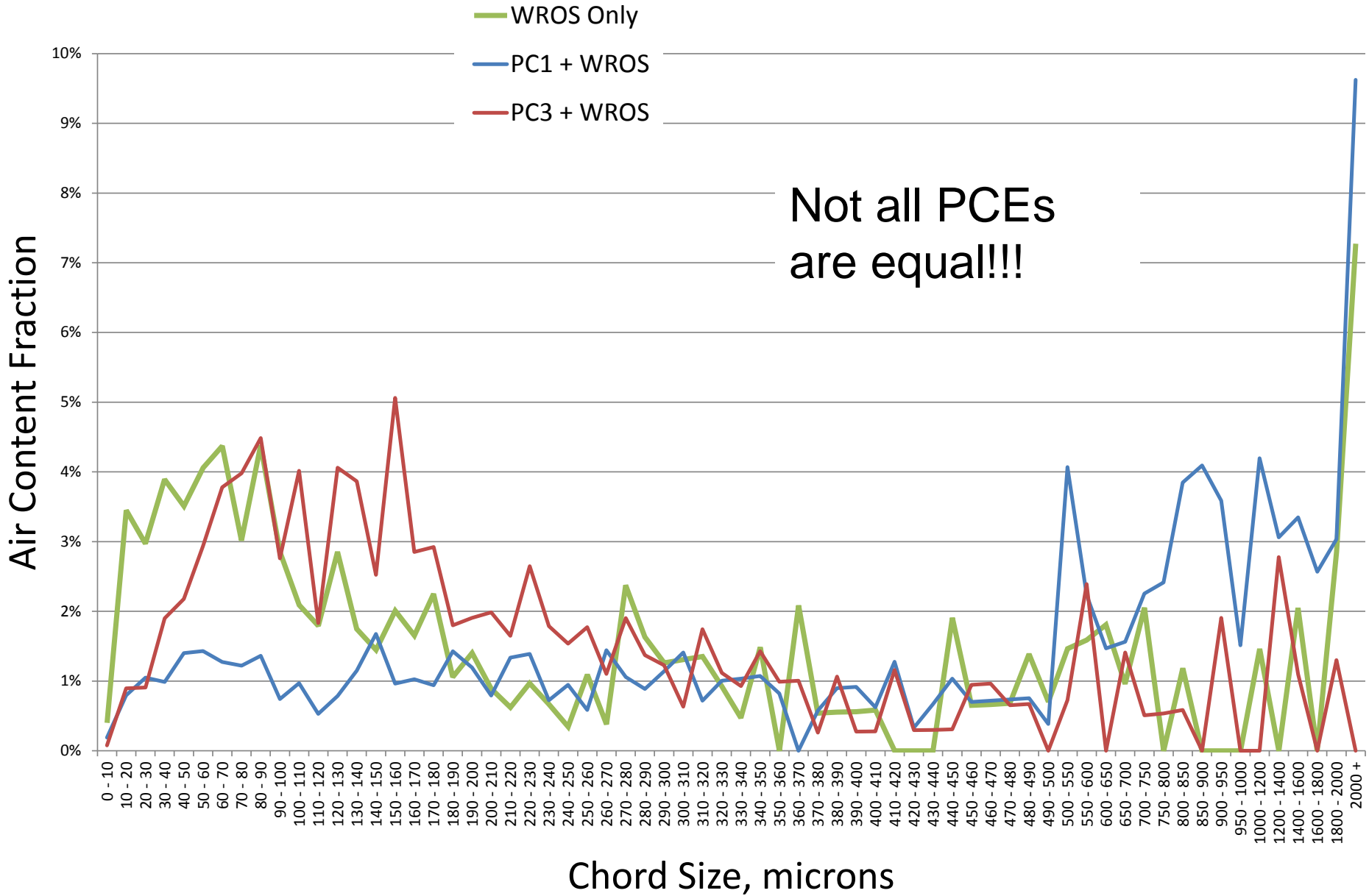












# Observations

- It appears that the air void system produced by PCEs are on average more coarse than mixtures with no PCEs
- Mixtures containing PCEs had similar air-void systems as non PCE with different freeze thaw performance.
- This suggests that the classic freeze thaw mechanisms may need to be modified

# Observations

- Higher volumes of air are likely required for mixtures containing PCEs to produce frost durable concrete
- Not all PCEs perform the same



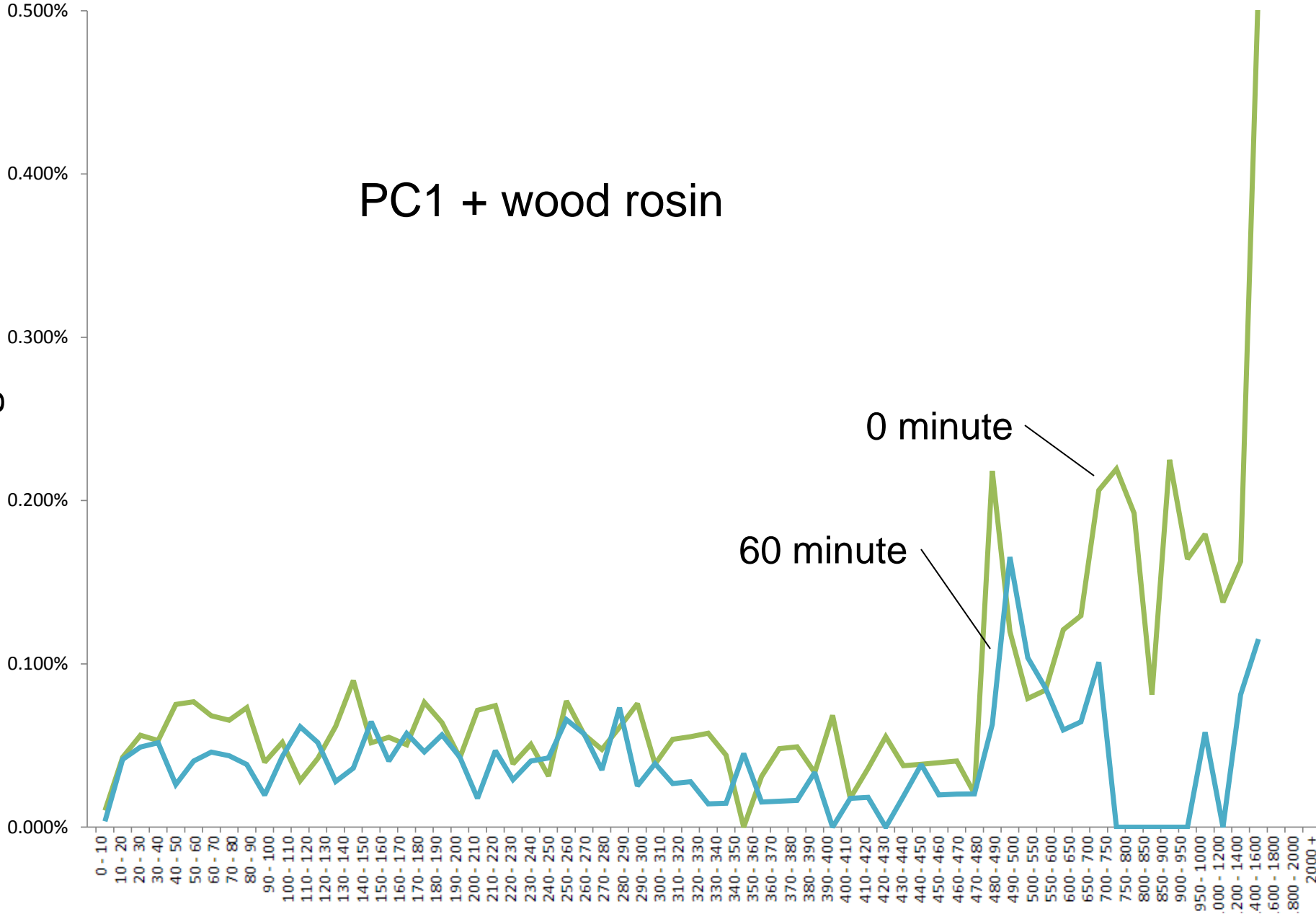
What is happening???

# PC1 + wood rosin

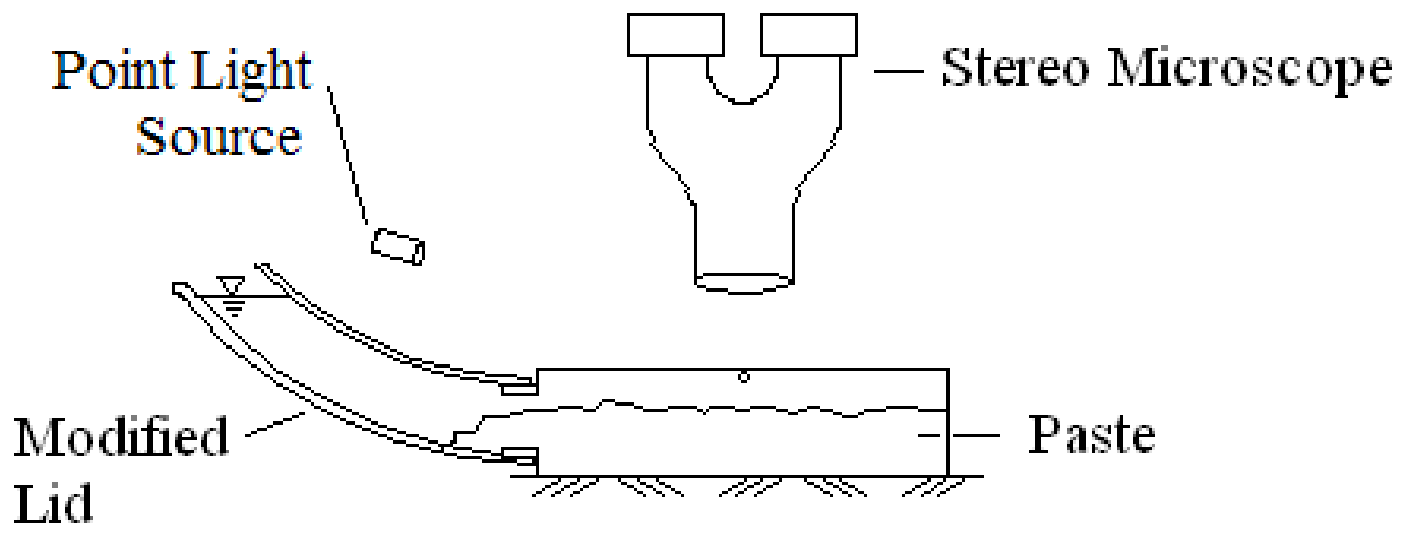
Percentage of air

0 minute

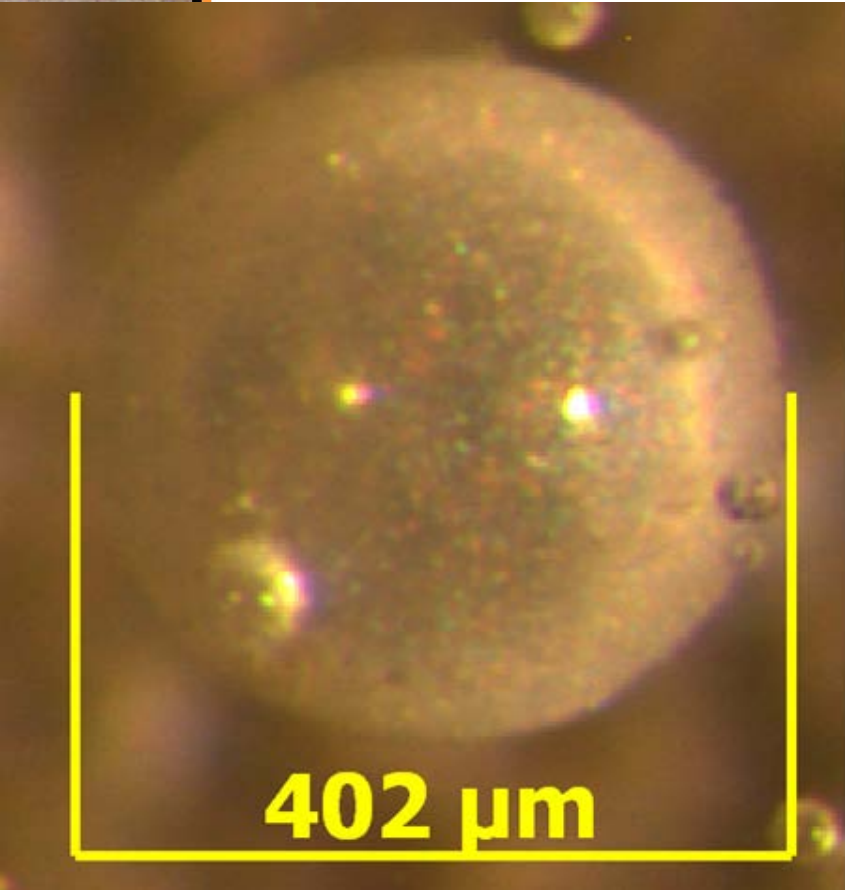
60 minute



Chord Size, microns

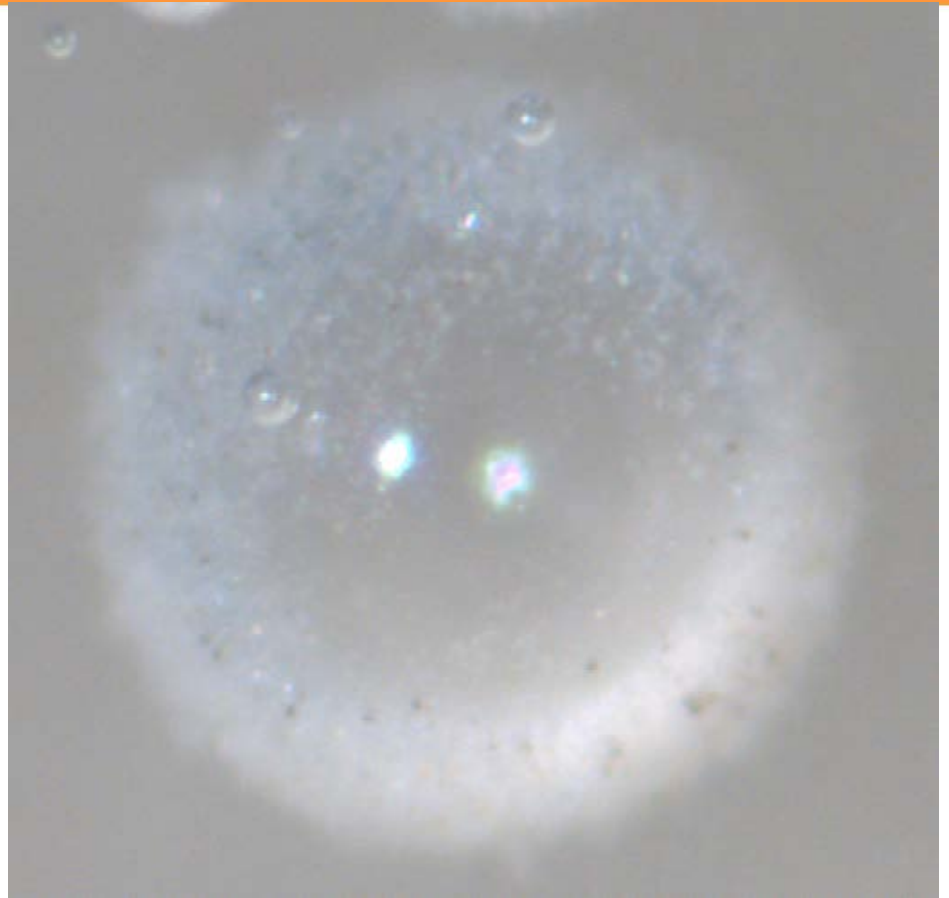




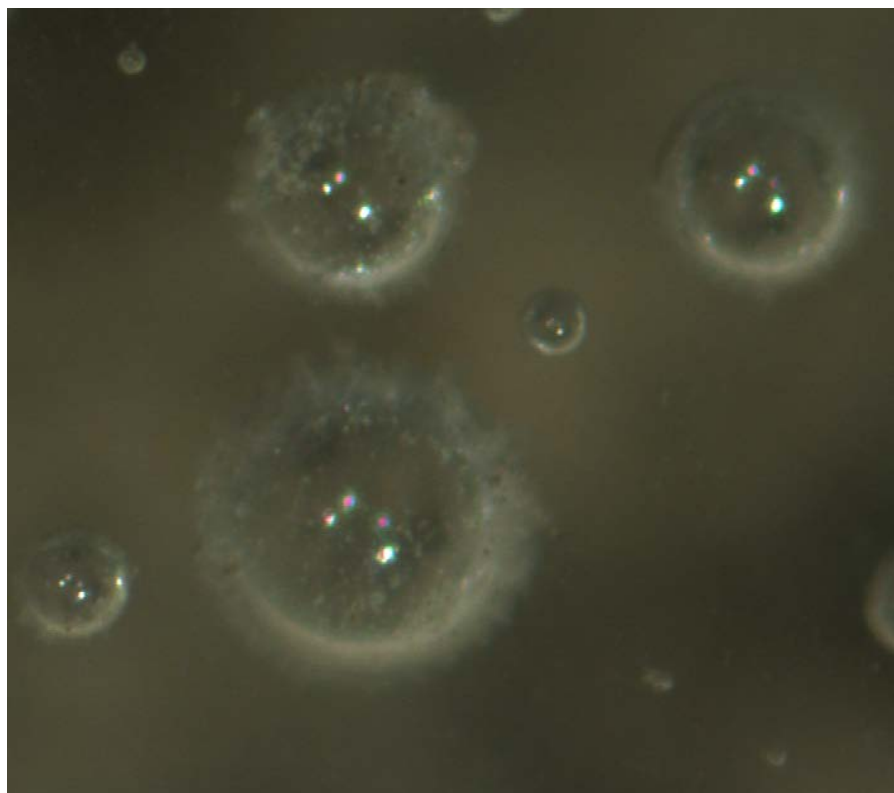
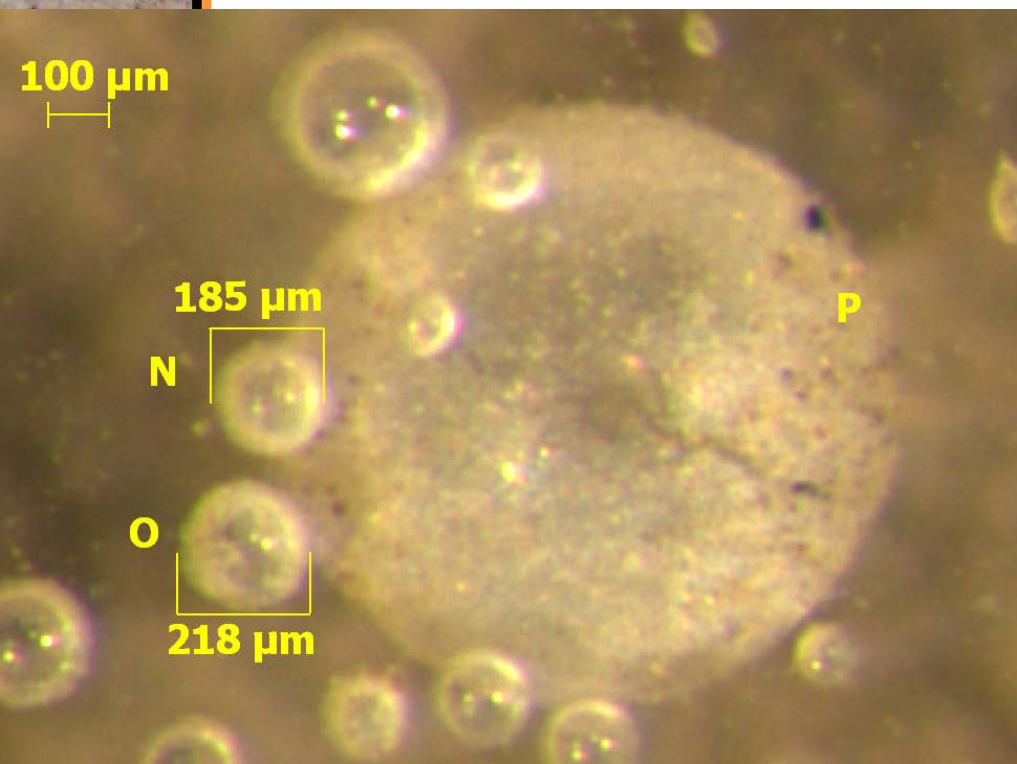


**402 μm**

Vinsol resin



PCE + Vinsol resin



Wood rosin

PCE + Wood rosin



# Observations

- Between 0 and 60 minutes it appears that the PCE concrete loses both small and large air bubbles over time
- There is a greater loss of the larger bubbles



# Observations

- The bubbles from the PCE + and AEA are not thoroughly coated with cement grains as with the AEA bubbles
- This may change their ability to form a hydration shell and their interaction with cement paste (Ley et al, 2007a and 2007b)

# Mechanisms

- Cement grains help hold bubbles in fresh concrete
- The PCE forces cement grains away from one another
- If these cement grains are not close together then the bubbles may have an easier time escaping from the fresh concrete

# Mechanisms

- When you consolidate the concrete (as was done with the modified pressure meter) you force cement grains close together and highly reduce the number of internal large voids
- This consolidation seems to lock the air void system in place



# What does this mean?

- PCEs are a valuable tool that provide great abilities to our concrete but users should be aware of their performance
- PCE concrete will lose slump and air over time on long hauls\*
- It is recommended to check the air content at the point of placement

\* *BASF has a new admixture that is supposed to reduce this*

# What does this mean?

- The air void system is typically coarser in PCE concretes and so higher volumes of air will likely be needed
- PCE concrete should be consolidated as soon as possible after mixing

# What does this mean?

- A spacing factor limit of .008” is recommended for concrete with PCE while a limit of .010” is satisfactory for non PCE mixtures
- PCEs are a valuable tool that provide great abilities to our concrete but users should be aware of the possible differences in performance



# What does this mean?

- We should not take a knee jerk reaction and not use PCEs because they modify the air void systems in the concrete. Instead we should re-evaluate how we specify and measure PCE concrete to ensure frost durability

# Freeze Thaw Research Needs

- While we have made recent progress with freeze thaw research much work is needed
- Current freeze thaw funding for this research has been expended
- I have been speaking with **Jason Weiss (Purdue)** and **Peter Taylor (Iowa State)** about proposing a pooled fund study

# Freeze Thaw Research Needs

A – Investigate ways to stabilize high quality air void systems in PCE concrete

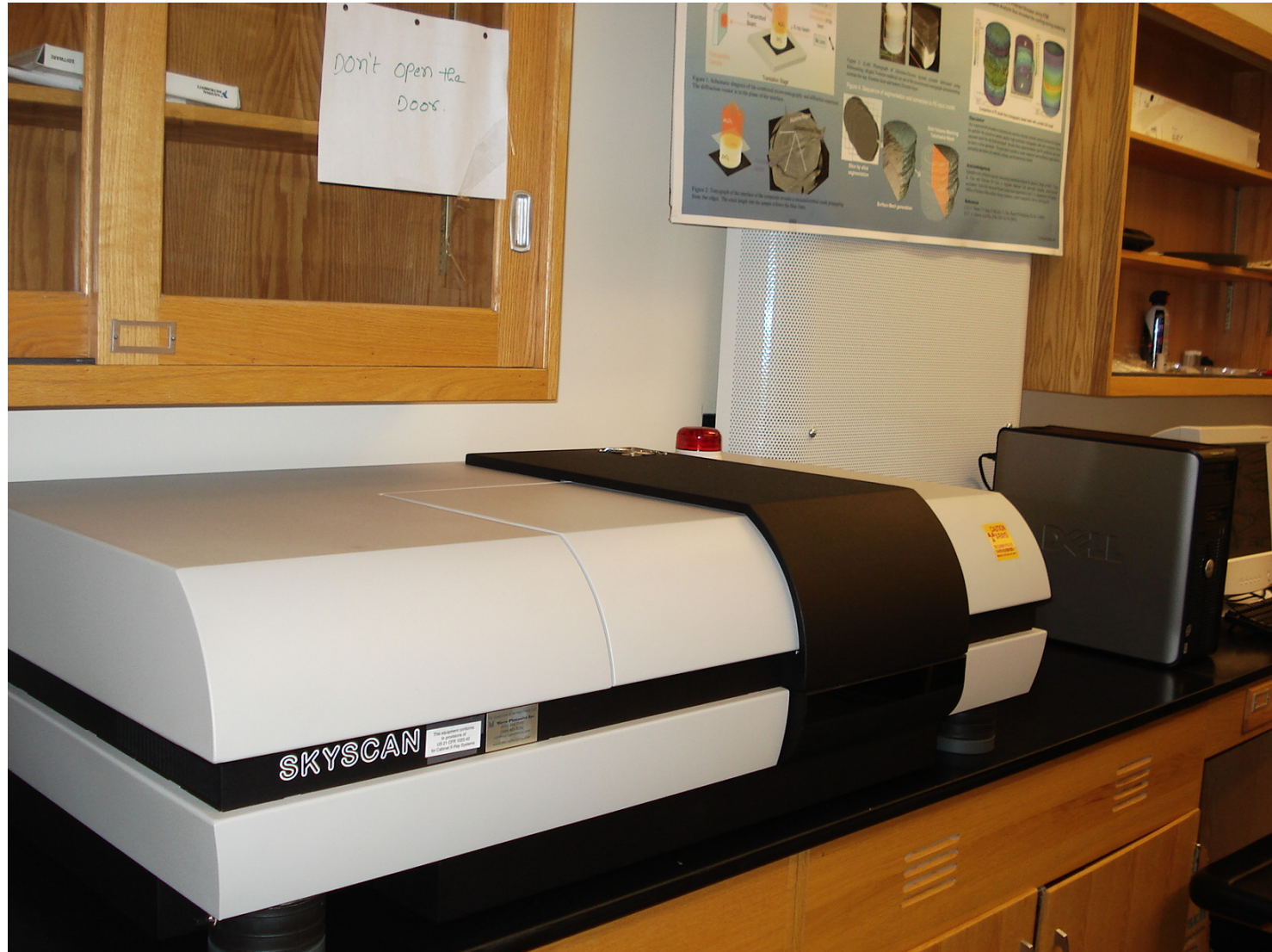
B – Evaluate the air void systems of field prepared PCE concrete

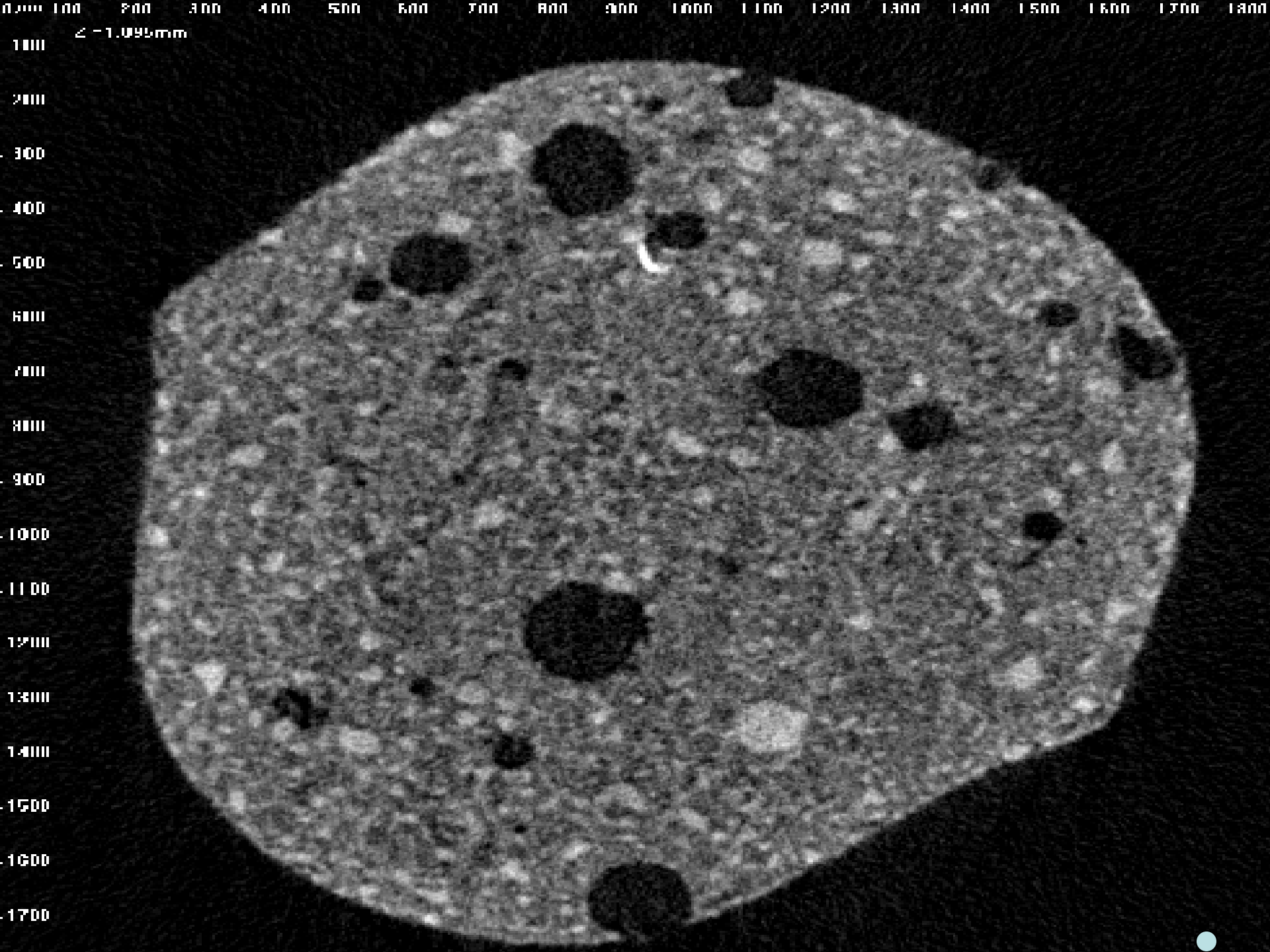
C – Establish a classification test method for PCE concrete

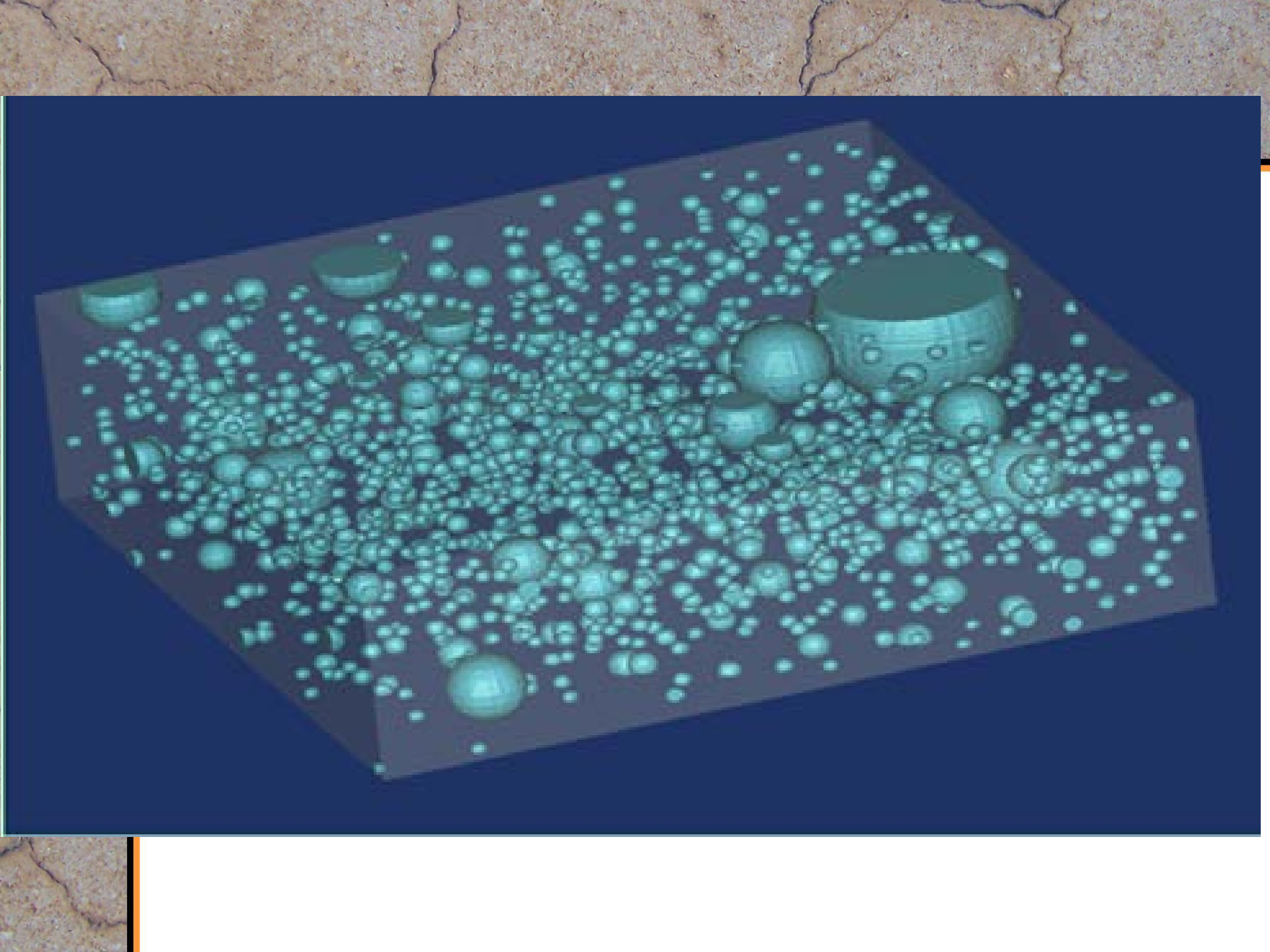
D – Reinvestigate the mechanisms of frost durability with modern investigation techniques



# $\mu$ CT Scanner





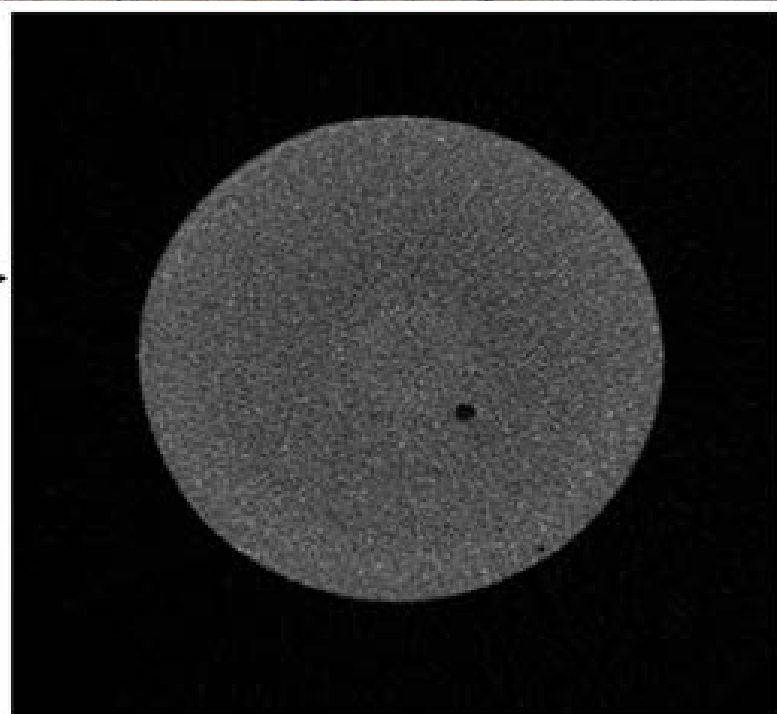




Non AEA paste

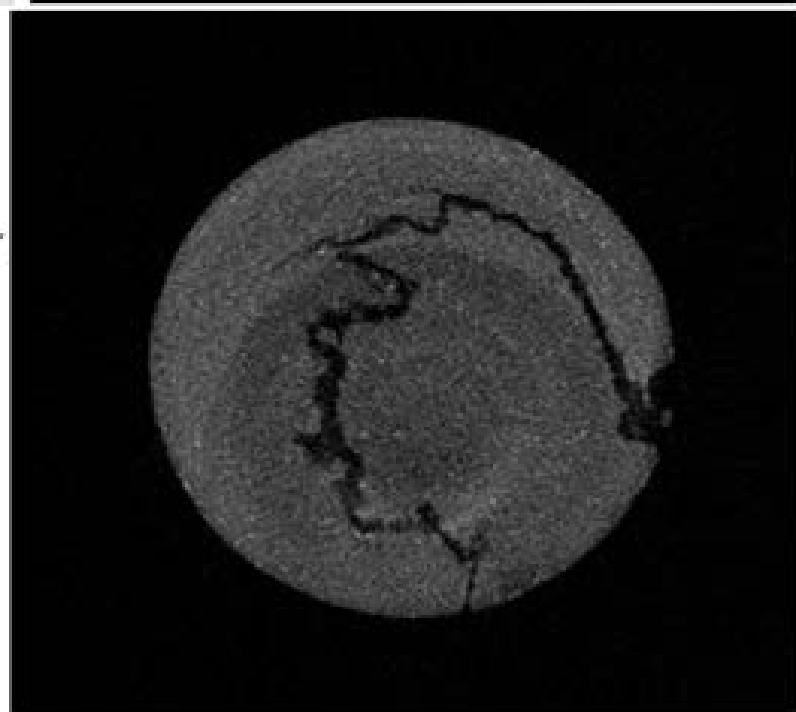
2.63 mm

0 cycles



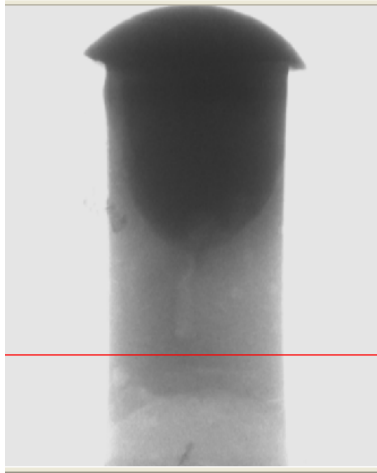
2.72 mm

15 cycles

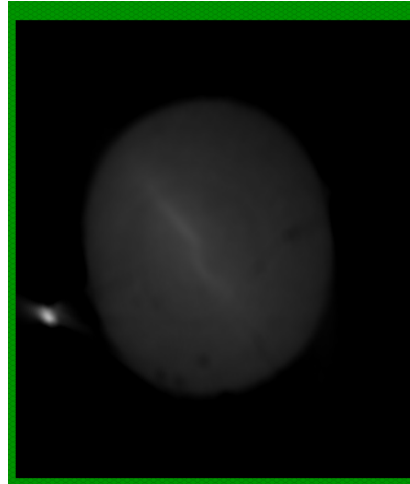


paste

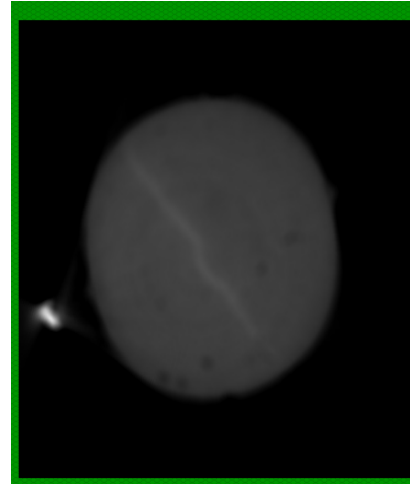
electron dense water



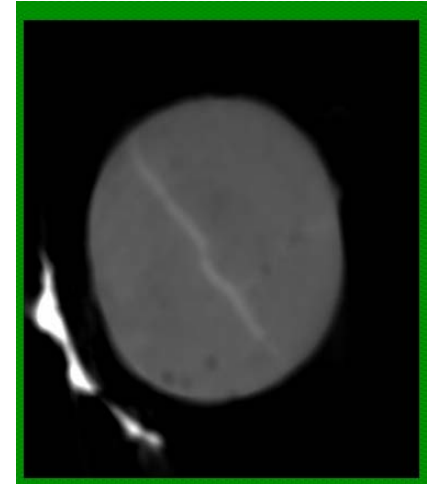
elevation view  
of the sample



0 min.



30 min.



90 min.

# Freeze Thaw Research Needs

E – Evaluate the field performance of frost susceptible concrete

F – Determine the air void requirements for concrete exposed to deicing salt solutions for bulk freeze thaw and scaling

G – Use these lessons to design a set of tests that better measure the frost durability of concrete



# Freeze Thaw Research Needs

We need a state to volunteer as the lead and then several others to volunteer to support these efforts

If there is support we can have a proposal together very quickly

# Questions???



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