Freeze Thaw Durability of Modern Concrete Mixtures

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Acknowledgements

- Oklahoma Transportation Center
- CP Tech Center
• 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
What is...

Air-entrained concrete
Air-Entrained Concrete

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

Hydrophilic “Head”

Hydrophobic “Tail”
Hydrophilic "Head"

Hydrophobic "Tail"

Air

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.
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?

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

Case B has a lower spacing factor and a higher specific surface.
Current Measuring Techniques
Current Measuring Techniques

PCA photo
ASTM C 231

PCA photo
ASTM C 173

ASTM C 138
Current Measuring Techniques

These only measure volume!!!
How Much Air Do We Need?
If $f'c > 5,000$ psi then these recommendations can be reduced by 1%.

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

<table>
<thead>
<tr>
<th>Nominal maximum aggregate size, in.*</th>
<th>Air content, percent</th>
<th>Exposure Class F1</th>
<th>Exposure Classes F2 and F3</th>
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<tbody>
<tr>
<td>3/8</td>
<td>6</td>
<td>7.5</td>
<td></td>
</tr>
<tr>
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<td>7</td>
<td></td>
</tr>
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<td>6</td>
<td></td>
</tr>
<tr>
<td>1</td>
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<td>6</td>
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<tr>
<td>1-1/2</td>
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<td>5.5</td>
<td></td>
</tr>
<tr>
<td>2†</td>
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<tr>
<td>3†</td>
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*See ASTM C33 for tolerance on oversize for various nominal maximum size designations.
†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.
How Much Air Do We Need?

- Current air volume specifications are based on work by Klieger at PCA (1952 and 1954)
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As max nom. Aggregate size ↓ paste ↑
• There was no standard freeze-thaw testing method at the time
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• To investigate the freeze-thaw durability specimens were manually wheeled in and out of freezers for 300 cycles!!!
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• 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
Expansion During 300 Cycles of Freezing and Thawing - %

Air Content of Concrete - % (Pressure)

Max. Size Aggregate
No. 4

5 1/2 sks. per cu. yd.
2 to 3-in. slump

Klieger 1952
Mixtures have increasing paste contents

Klieger 1952
If $f'_c > 5,000$ psi then these recommendations can be reduced by 1%.

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Klieger varied the water content of the mixtures to get a constant slump.
Expansion During 300 Cycles of Freezing and Thawing - percent

3/4" Maximum Nominal Aggregate Size

Slump: 2-3 inches

Variable W/C Ratio

Paul Klieger (1952)

Recommended Concrete Air Content

5/4 Sack Mix

0.49

0.48

0.47

0.46

0.45

0.42

Air Content of Concrete (Pressure)
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
<table>
<thead>
<tr>
<th>w/c ratio</th>
<th>Cement $\text{lb/yd}^3$</th>
<th>Coarse $\text{lb/yd}^3$</th>
<th>Fine $\text{lb/yd}^3$</th>
<th>Water $\text{lb/yd}^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.38</td>
<td>611</td>
<td>1950</td>
<td>1203</td>
<td>232</td>
</tr>
<tr>
<td>0.41</td>
<td>611</td>
<td>1900</td>
<td>1129</td>
<td>250.5</td>
</tr>
<tr>
<td>0.45</td>
<td>611</td>
<td>1850</td>
<td>1203</td>
<td>275</td>
</tr>
</tbody>
</table>
Laboratory work at OSU

• The following were measured:
  – Slump
  – Unit weight
  – Pressure meter air
  – Strength
  – Hardened air
    • spacing factor
    • specific surface
    • total air
W/C = 0.45

- failed ASTM C666

Spacing Factor (in)

C231 Air Content (%)
Spacing Factor (in) vs. C231 Air Content (%)

- Vinsol Resin
- Wood Rosin
- Synthetic

W/C = 0.41
Spacing Factor (in)

C231 Air Content (%)
Spacing Factor (in)

C231 Air Content (%)

- 0.41+ Wood Rosin
- 0.41+ Synthetic
- 0.41+ Vinsol Resin
- 0.45+ Wood Rosin
- 0.45+ Synthetic
- 0.45+ Vinsol Resin

Passes
ASTM C666
Passes
ASTM C666

- 0.41+ Wood Rosin
- 0.41+ Wood Rosin+ 3.7 oz/cwt Midrange
- 0.41+ Wood Rosin+ 10.2 oz/cwt Midrange
- 0.38+ Wood Rosin+ 10.2 oz/cwt Midrange

Spacing Factor (in)

C231 Air Content (%)

2.0% 2.5% 3.0% 3.5% 4.0% 4.5% 5.0%
The graph shows the relationship between Spacing Factor (in) and Air Content of Concrete (Pressure). The Spacing Factor decreases as the Air Content of Concrete increases. The data points are plotted with a trend line that illustrates this relationship.
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!!!!
• 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.
Positive and negative charges

PCE molecule

Negative charges

Positive and negative charges
No PCE

cement grain
with PCE
• 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

<table>
<thead>
<tr>
<th></th>
<th>CA</th>
<th>FA</th>
<th>Water</th>
<th>Cement</th>
<th>w/c</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixture 1</td>
<td>1850</td>
<td>1203</td>
<td>275</td>
<td>611</td>
<td>0.45</td>
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<tr>
<td>Mixture 2</td>
<td>1815</td>
<td>1180</td>
<td>301.6</td>
<td>599.5</td>
<td>0.50</td>
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<tr>
<td>Mixture 3</td>
<td>1907</td>
<td>1240</td>
<td>231</td>
<td>630</td>
<td>0.37</td>
</tr>
<tr>
<td>Mixture 4</td>
<td>1900</td>
<td>1129</td>
<td>250.5</td>
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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

<table>
<thead>
<tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td>0 min</td>
</tr>
<tr>
<td>C143</td>
<td>Slump</td>
<td>X</td>
</tr>
<tr>
<td>C138</td>
<td>Unit Weight</td>
<td>X</td>
</tr>
<tr>
<td>C231</td>
<td>Pressure Meter</td>
<td>X</td>
</tr>
<tr>
<td>N/A</td>
<td>Modified Pressure Meter</td>
<td></td>
</tr>
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<td>C666</td>
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Hardened air void samples were taken at 0, 60 and 120 min
Laboratory Testing

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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

Slump (inches) vs. Time, min

- WROS Only
- PC1 + WROS
- PC1 + WROS Undist.
Air Content (ASTM C 231)

- WROS Only
- PC1 + WROS
- PC1 + WROS Undist.

Time, min

0% 1% 2% 3% 4% 5% 6% 7%

0 30 60 90 120

.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.
Air Content (ASTM C 231)

- WROS Only
- PC1 + WROS
- PC1 + WROS Undist.

0.45 w/cm

Time, min

0  30  60  90  120
Air Content (ASTM C 231) vs. Time, min

- WROS Only
- PC1 + WROS
- PC1 + WROS Undist.

Modified pressure meter tests

.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!*
red = fails ASTM C 666
green = pass ASTM C 666

Non PCE

PCE
Look at the difference in the volume of “small” air voids!!!
Observations

• The concrete **without** 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*
Observations

• 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.
None of these mixtures contain PCEs
Open dots failed the ASTM C666
Is this air content enough???
Same air void system and different freeze thaw performance!!!
Not all PCEs are equal!!!
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
PC1 + wood rosin

Percentage of air

Chord Size, microns

0 minute
60 minute
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
µCT Scanner
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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