Update on Deicer Distress

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Michigan Tech Material Science & Engineering


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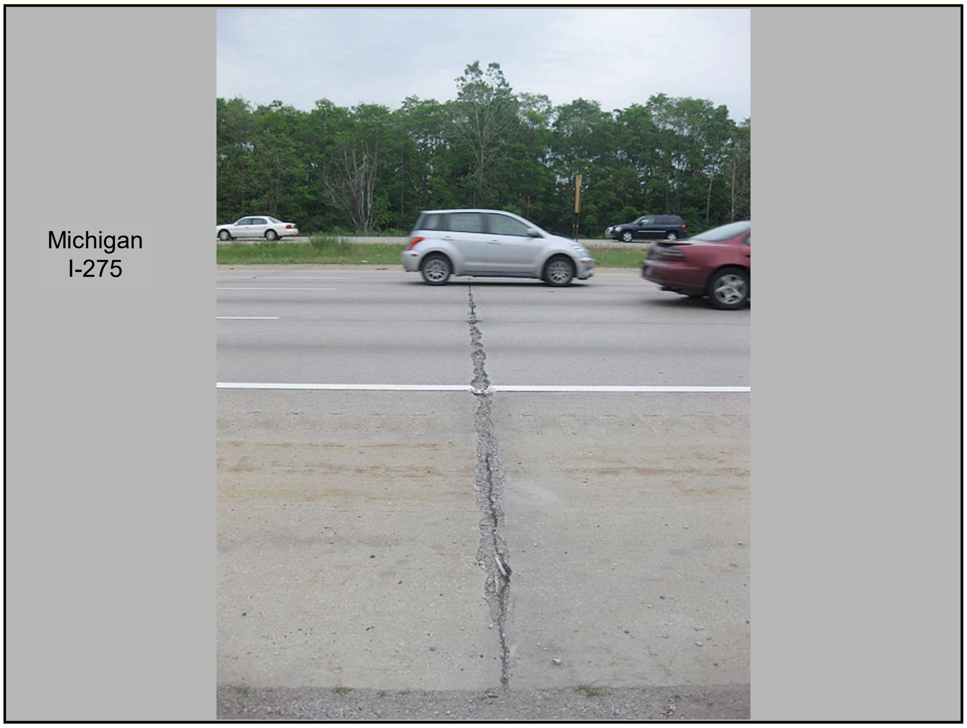
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Many Suspects

- Air entraining agents
- Early entry sawing
- Curing
- Deicing practices

Many Suspects

- Air entraining agents
- Early entry sawing
- Curing
- **Deicing practices** ←

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What Do We Know?

- The causes
  - Physical Attack
    - Saturated paste localized at joints
    - Freeze-Thaw deterioration located at joints
  - Chemical Attack from deicers
    - Oxychloride formation – paste deterioration

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What Do We Know?

- The causes
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Physical Attack

- Conventional paste freeze-thaw (F-T) action but exacerbated by deicers
- The F-T action of water is one of the most destructive forces that concrete faces
  - Places concrete ”under tension“
- The hydrated cement paste is generally considered protected against F-T damage by
  - Acceptable air-void system
  - Reduced permeability

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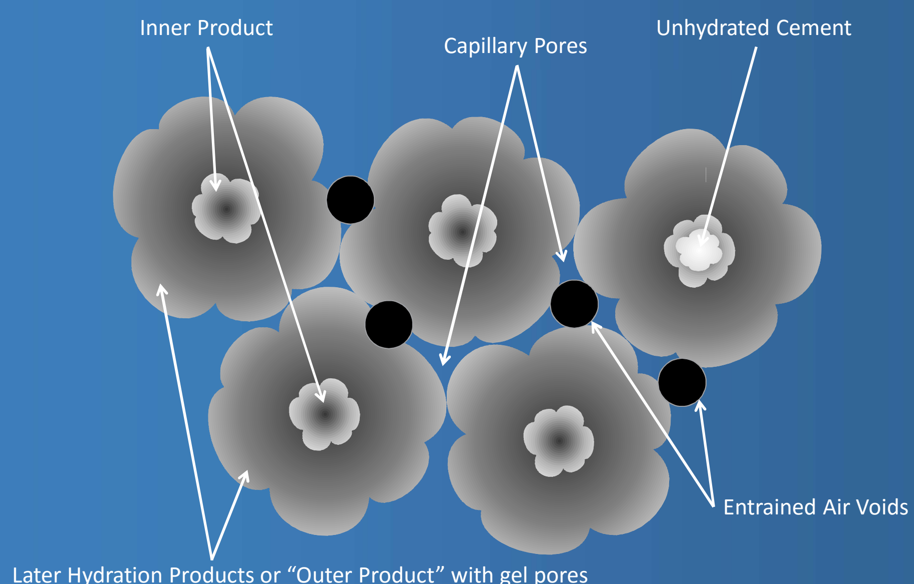
Physical Attack

- **Concrete is a porous material – water goes in, out and is absorbed**
- <u>Durability is achieved through reduced permeability</u>
- Porosity (permeability) is a result of:
  - The cement hydration process
  - Other materials used (aggregates)

Hydration Reactions Example: Portland Cement



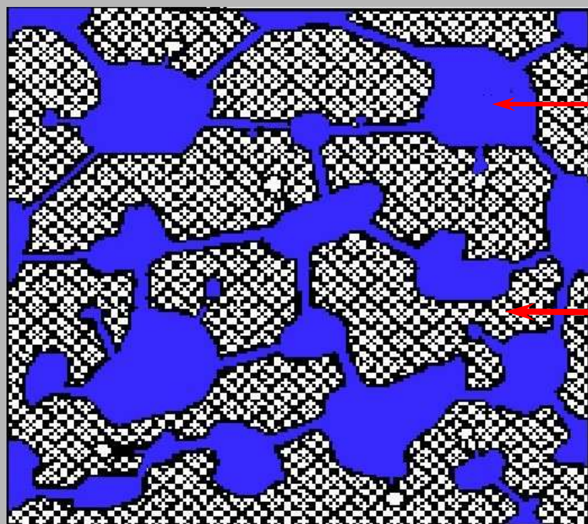
Pore Structure

- Gel Pores
  - Space between layers in calcium silicate hydrate (C-S-H
  - Sizes range between 0.5 and 2.5 nm
  - Can contribute 28% of C-S-H porosity
  - Little impact on strength and permeability
  - Can influence shrinkage and creep

Pore Structure

- Capillary Pores
  - Depend on initial separation of cement particles, which is **controlled by the *w/cm***
  - Space not taken up by cement or hydration products is capillary porosity
  - On the order of 10 to 50 nm, although larger for higher *w/cm* (3 to 5 um)
  - Larger voids affect strength and permeability, smaller voids impact shrinkage

High Permeability (Capillary Pores Interconnected)

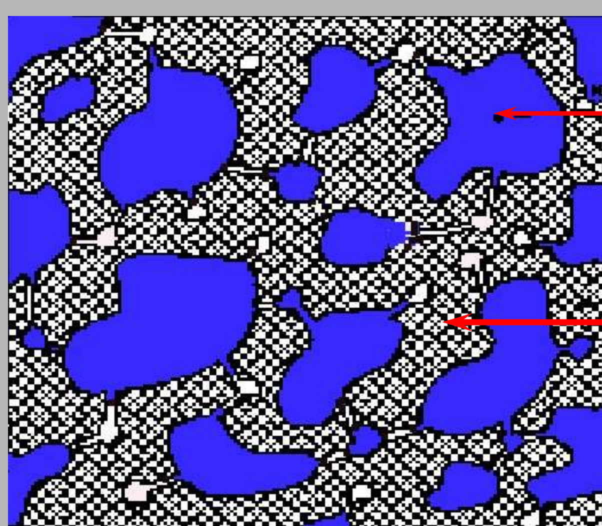


Capillary Pores

CSH Framework

Neville

Low-Permeability Capillary Pores Segmented and Only Partially Connected



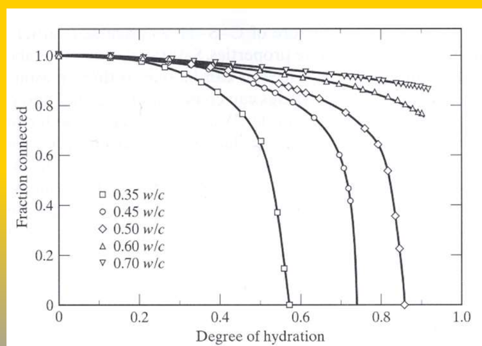
Capillary Pores

CSH Framework

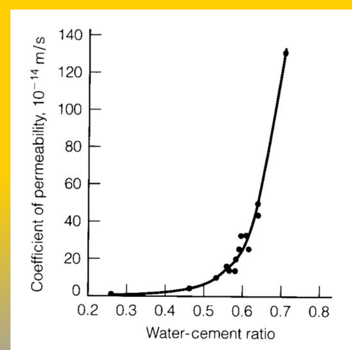
Neville

Porosity & Permeability

Connectivity



Permeability



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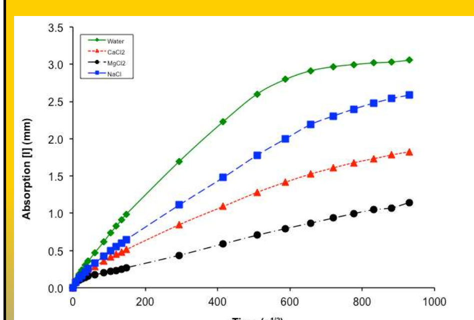
w/c Effects on Permeability

0.55 *w/c* OPC Sorptivity



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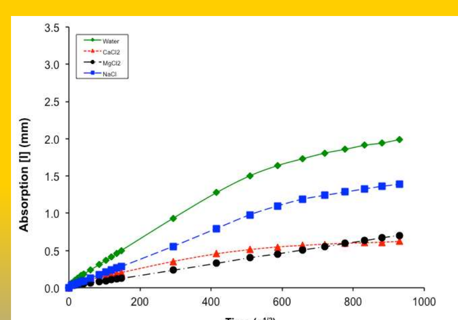

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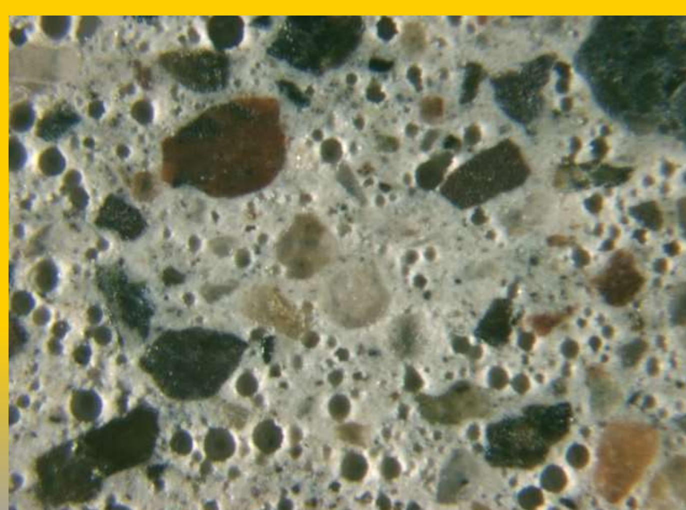
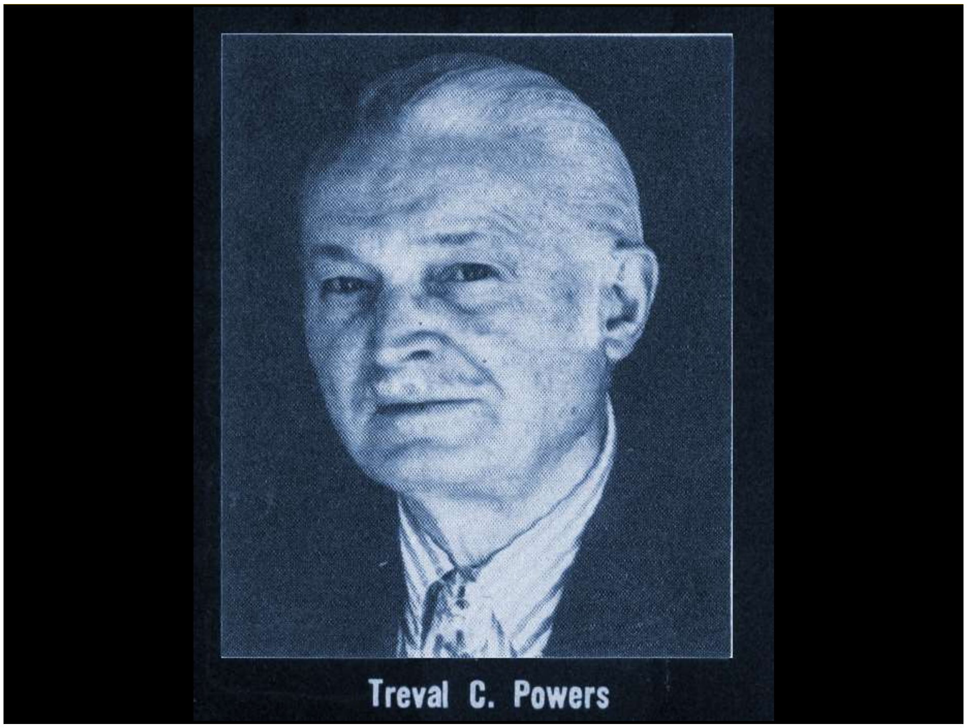
Pore Structure

- Air Voids
  - Intentionally entrained and unintentionally entrapped
  - Entrained air voids are spherical and randomly distributed throughout the cement paste.
  - Size of entrained air voids ranges from ~10 μm to over 1 mm
    - Often voids larger than 1 mm, and/or irregular in shape, are labeled as entrapped
  - Entrapped air voids can range in size from microscopic to over 3 mm

Air-Void System



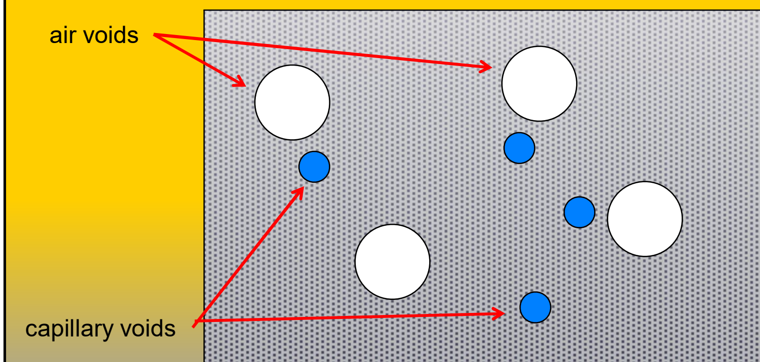


Hydraulic Pressure Theory – Powers 1945

- Attributed damage to **excessive hydraulic pressures resulting from the expansion of ice**

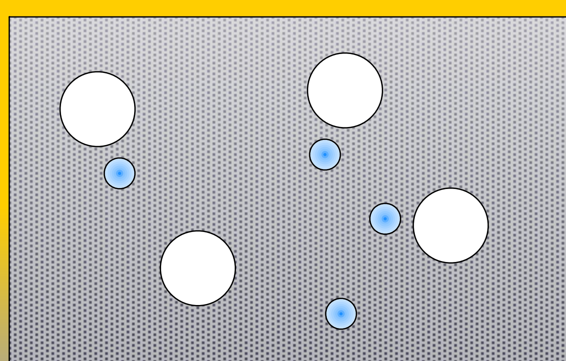
Hydraulic Pressure Theory – Powers 1945

- Water fills capillary pore space



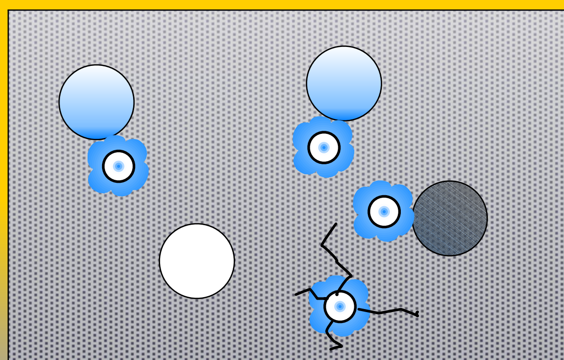
Hydraulic Pressure Theory – Powers 1945

- **Ice forms** in a saturated capillary pore system



Hydraulic Pressure Theory – Powers 1945

- Volume expansion causes the **unfrozen water to be expelled away from the freezing sites**



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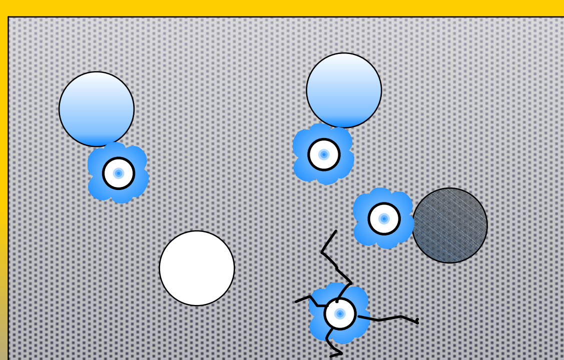
Hydraulic Pressure Theory – Powers 1945

- Depending on the nature of the pore system, **excessive internal stresses result from resistance to this flow**



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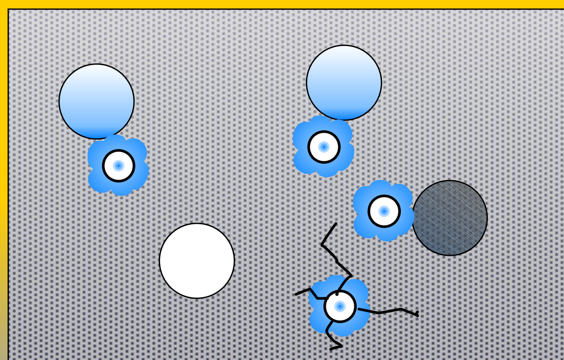
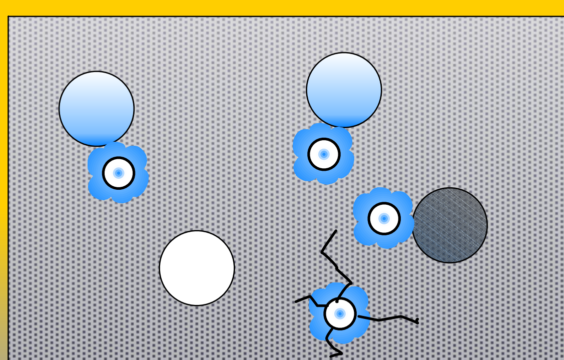
Hydraulic Pressure Theory – Powers 1945

- The **pressurized water** moving away from the freezing sites **finds relief at the air voids**, where it freezes without causing damage



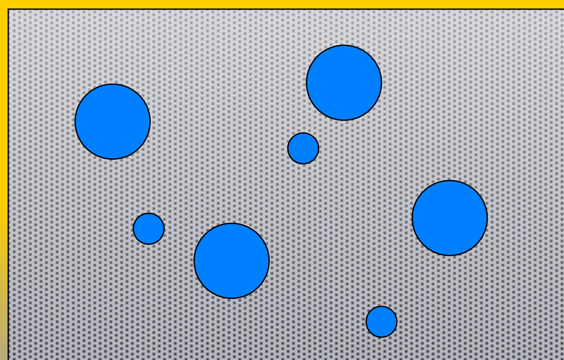
Hydraulic Pressure Theory – Powers 1945

- Powers recognized **the spacing between voids**, rather than total volume of air, was **the better measure of resistance to F-T damage**



Hydraulic Pressure Theory – Powers 1945

- If the total void system is saturated…



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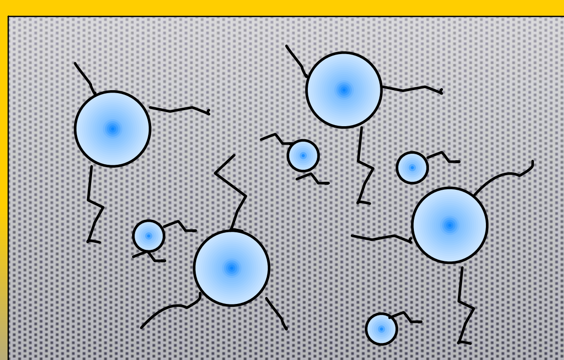
Hydraulic Pressure Theory – Powers 1945

- **Volume expansion leads to tensile forces and cracking**



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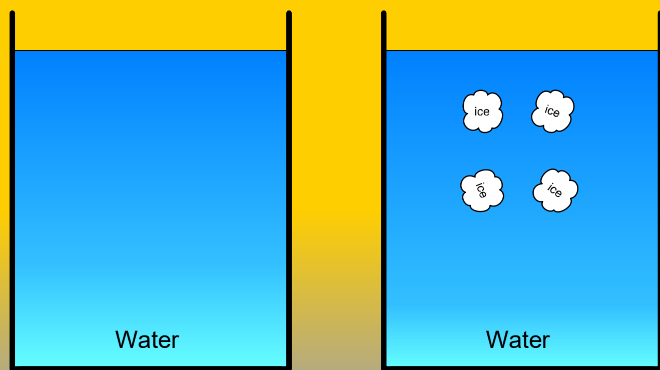
Osmotic Pressure Theory – Powers 1975

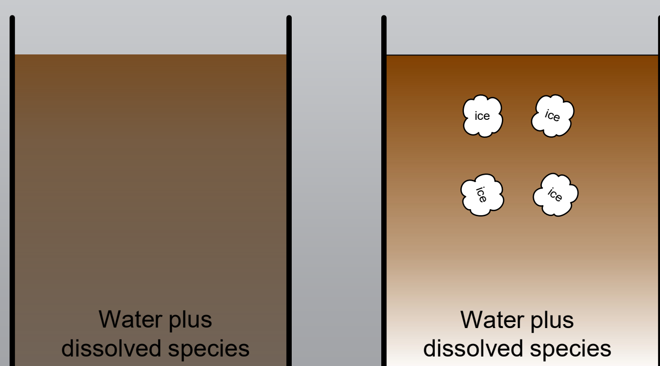
- Less obvious... Need to know a couple concepts
- **Ice is solid water (water is liquid ice)**



Decrease temperature below freezing →

Osmotic Pressure Theory – Powers 1975

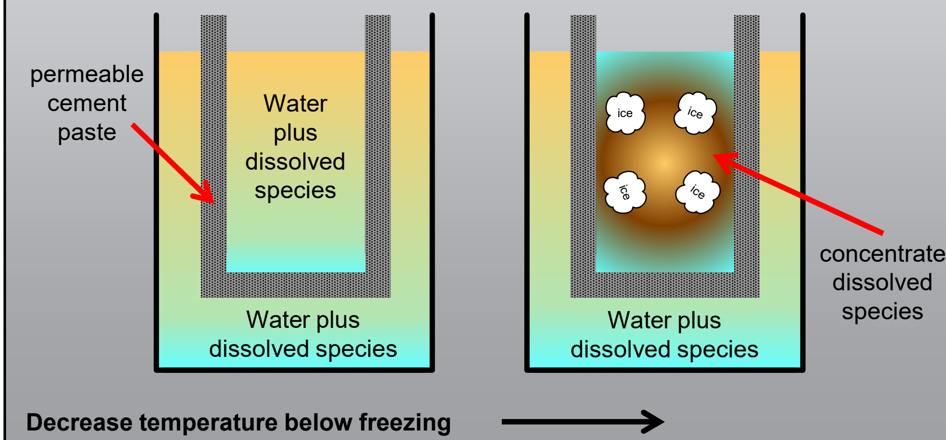
- When the liquid is <u>**water plus dissolved material**</u>, ice forms (water) and makes the liquid more concentrated with the dissolved material



Decrease temperature below freezing →

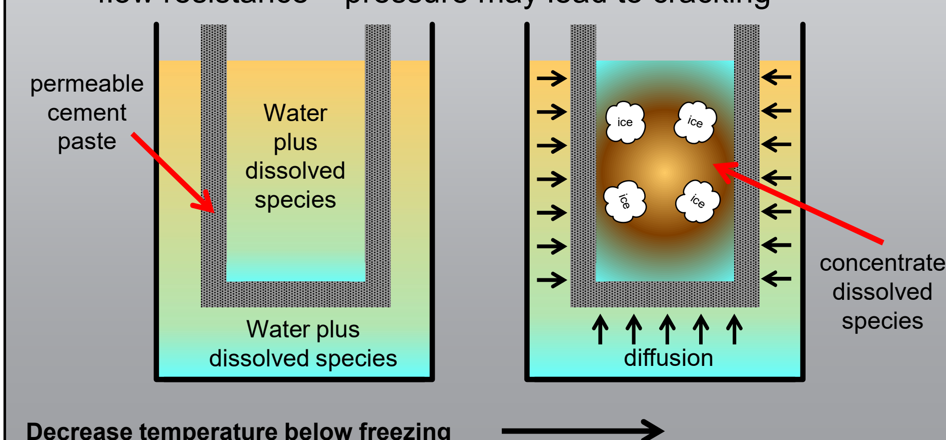
Osmotic Pressure Theory – Powers 1975

- As ice forms the remaining solution becomes more concentrated with dissolved ions



Osmotic Pressure Theory – Powers 1975

- More dilute solution diffuses through paste to equalize concentrations – develops “osmotic” pressure due to flow resistance – pressure may lead to cracking



Osmotic Pressure Theory – Powers 1975

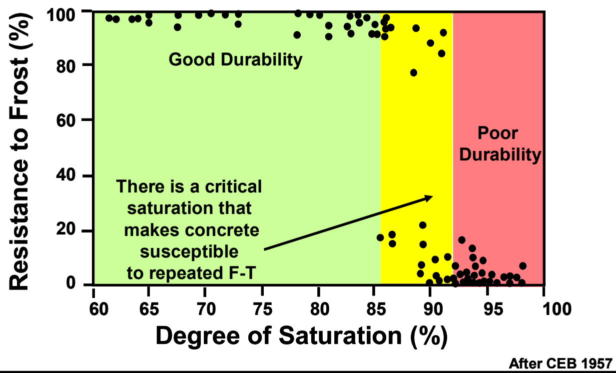
- Because of their relatively large size, air voids are initial freezing sites
- Liquid at the freezing sites becomes a more concentrated alkaline solution
- Alkaline solution in the surrounding paste is **drawn to the freezing** – driven by the alkali concentration gradient
- Resistance to flow through the cement paste creates tension – drawing action creates compresiion

Osmotic Pressure Theory – Powers 1975

- If protected – all freezable water will reach an air void and freeze – no damage
- If not protected –
  - Osmotic pressures will increase due to the remaining differences in alkali concentrations
  - Freezable water will remain in the capillary pores
  - Cracking may occur

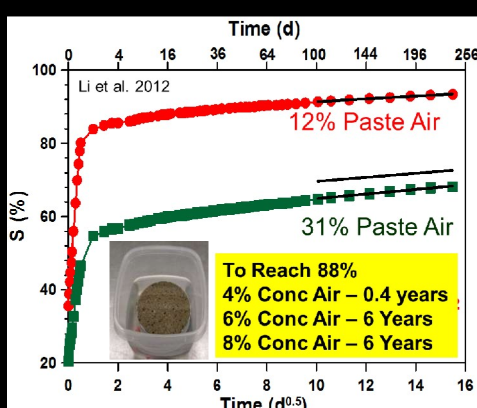
So what does this mean for joint distress?

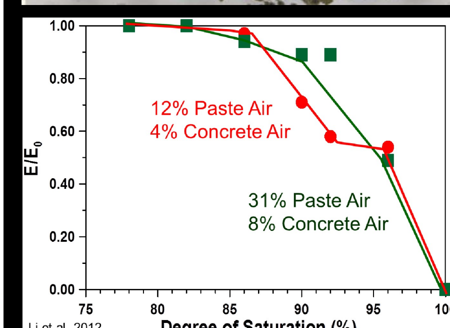
- Mechanism
  - Results when the paste becomes “critically saturated” and concrete under goes F-T cycles
  - The expansion of ice causes tensile forces that crack concrete



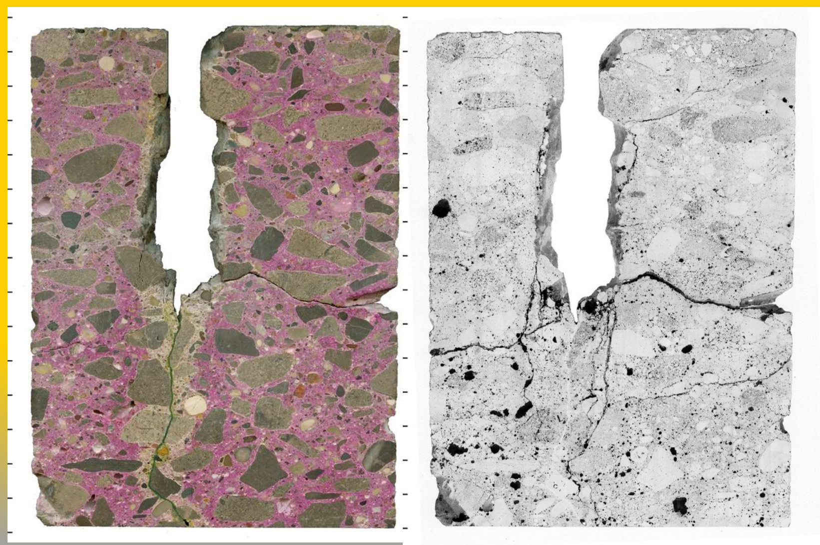
Freeze-Thaw Damage and the Degree of Saturation

- Rate to reach DOS
- FT at different DOS



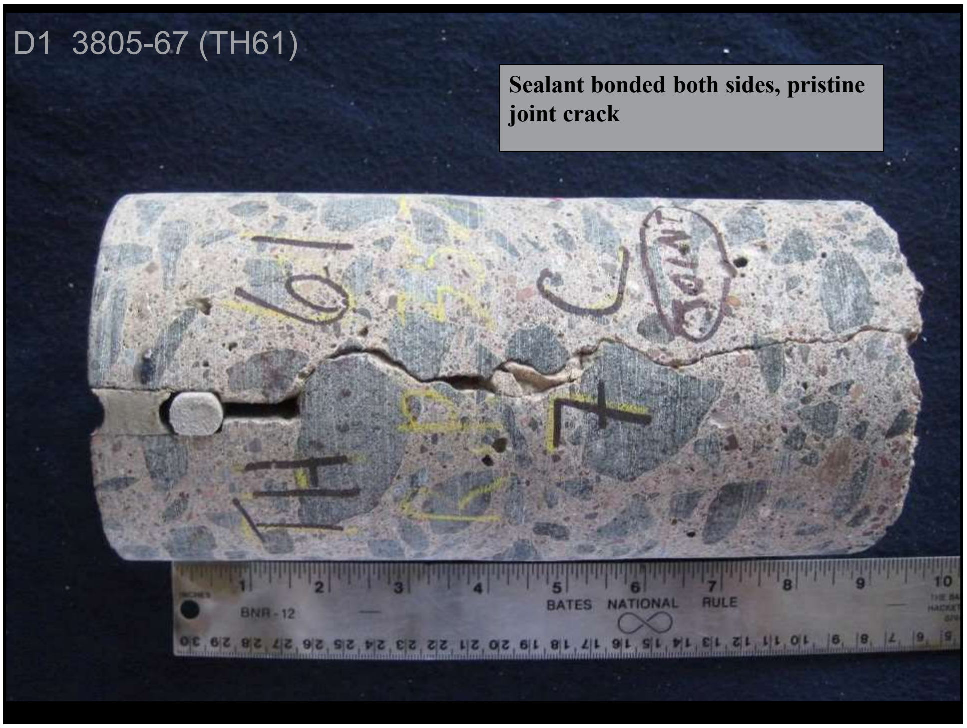


Joint Detail & Seals Important




D1 0980-127
Silicone sealant completely de-bonded.
Severe joint distress.
3B
BATES NATIONAL RULE
1992


D1 3805-67 (TH61)
Sealant Bonded
1997


D1 3805-67 (TH61)
Sealant bonded both sides, pristine joint crack


D3 7380-199
Well bonded
1999

Take Aways - Paste F-T

- Concrete porosity cannot be avoided
- Concrete porosity (permeability) is directly linked to *w/cm* and curing (connectivity)
- Air is entrained to protect the paste – but it is not a bullet-proof solution
  - Critically saturated paste will crack when frozen
- Need proper air content and air-void system (spacing factor, specific surface)

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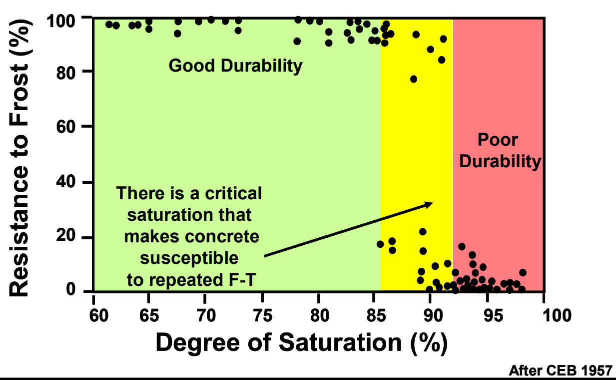
Take Aways - Paste F-T

- Do deicers play a role?

- YES

Remember Critical Saturation?

- Mechanism
  - Results when the paste becomes “critically saturated” and concrete under goes F-T cycles
  - The expansion of ice causes tensile forces that crack concrete



Take Aways - Paste F-T

- Salts suck up water & hold it
- Helps concrete reach saturation sooner

Photos courtesy Peter Taylor, Iowa State University

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A Couple Words on Deicer Scaling…

Deicer Scaling

Scaling

- Typically appears within 1 to 5 years after construction
- Deicer chemicals play a role
  - Will amplify scaling deterioration **(physical attack)**
  - Deicers can chemically react with hydrated paste constituents and degrade strength AND cause expansion **(chemical attack)**
- Scaling prevented through proper air entrainment (hardened concrete) and a relatively low water-to-cement ratio
- Minimize finishing, which can reduce air content at surface

Scaling

- Mechanism
  - Not well understood
  - Predominately a **physical attack**
  - **Chemical attack** (deicers) is also a contributor

Mechanism

- **Physical Attack**
  - Current research indicates scaling is due to tensile forces developed in the surface layer of concrete due to expansion of the ice layer (glue spalling)†
  - The expansive forces of the ice are at a maximum when the solution freezing on the surface contains ~3% dissolved salt, and **the type of salt is not a factor† (for physical attack)**

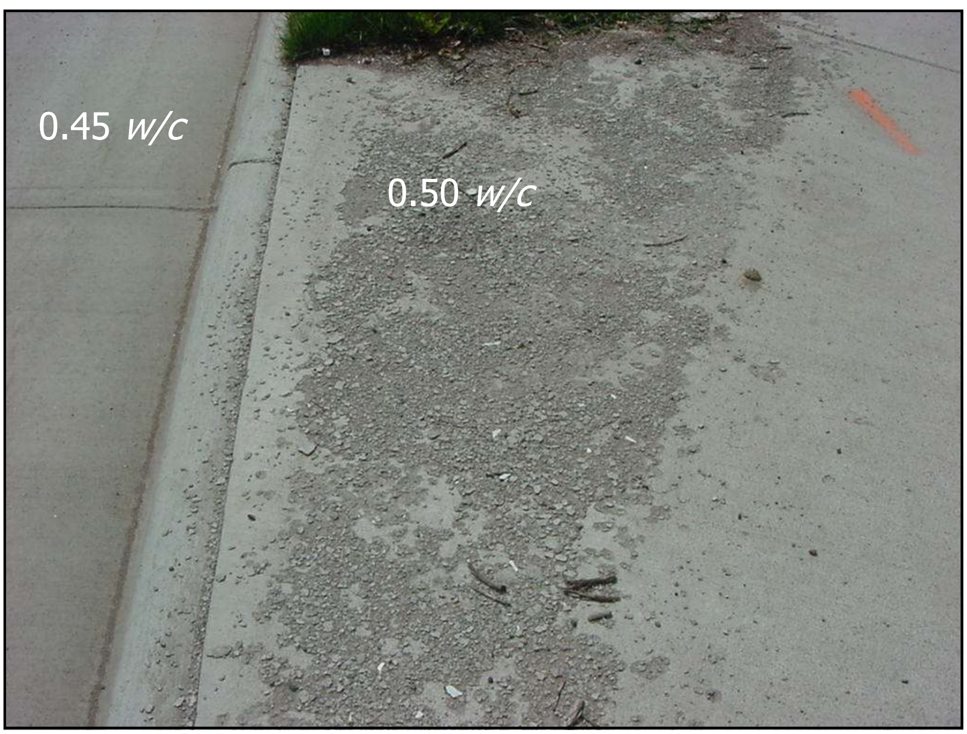
† Valenza, J. J., and G. W. Scherer. 2007. A Review of Salt Scaling: II. Mechanisms, *Cement and Concrete Research*. 37(7), 1022-1034.

Mechanism

- **Physical Attack**
  - The mechanical properties of the ice layer are affected by brine pockets in the ice layer – expansive forces maximized with dilute brine solutions present†
  - The strength of the concrete surface (resistance to expansion forces) is key to resisting the expansion
  - **High *w/c* = lower strength (blessing the concrete)**

† Valenza, J. J., and G. W. Scherer. 2007. A Review of Salt Scaling: II. Mechanisms, *Cement and Concrete Research*. 37(7), 1022-1034.


0.45 w/c
0.50 w/c


0.45 w/c
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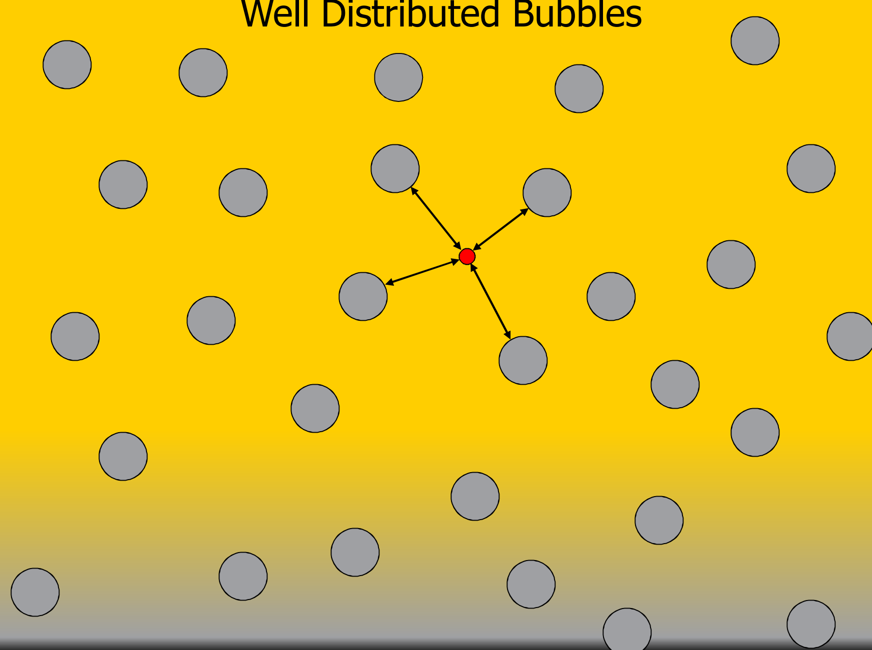
Role of Entrained Air

- Air entrainment reduces bleeding
  - Bleeding will increase the *w/c* in the surface layer
- Ice formation in air voids sucks water from the surrounding capillary pores
  - Creates a compression force that resists the tensile forces of the ice layer
  - **DON'T WORK THE AIR OUT OF THE SURFACE**

Well Distributed Bubbles



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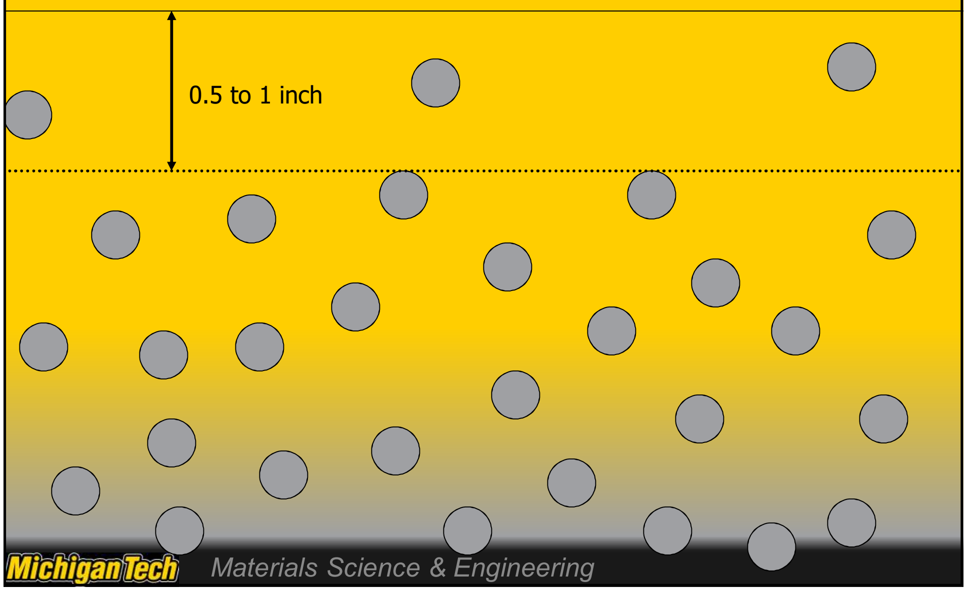
Impact of Poor Finishing



Scaling

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Back to Joint Distress

- The causes
  - Physical Attack
    - Saturated paste localized at joints
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  - Chemical Attack from deicers
    - Oxychloride formation – paste deterioration

Cylinders exposed to $MgCl_2$ solution after 84 days of constant low temperature test. From left to right: 0.40, 0.50, and 0.60 *w/c*

Cylinders exposed to $CaCl_2$ solution after 84 days of constant low temperature test. From left to right: 0.40, 0.50, and 0.60 *w/c*

Cylinders exposed to NaCl solution after 84 days of constant low temperature test. From left to right: 0.40, 0.50, and 0.60 *w/c*

Cylinders exposed to $Ca(OH)_2$ solution after 84 days of constant low temperature test. From left to right: 0.40, 0.50, and 0.60 *w/c*

Hi conc. $CaCl_2$ – 500 days – 40 ºF

Hi conc. $MgCl_2$ – 500 days – 40 ºF

Cement Hydration Reaction

Hydraulic Cement + Water ➔ C-S-H + C-A-H + CH

CSH – Calcium silicate hydrate

CH – Calcium hydroxide

Calcium hydroxide dissolution is at the center of chemical deicer attack

Chemical Mechanisms of Deicer

- $CaCl_2$ *(Monosi & Collepardi, 1990)*

  $3Ca(OH)_2 + CaCl_2 + 12H_2O \rightarrow 3CaO \cdot CaCl_2 \cdot 15H_2O$

  Calcium Oxychloride

- NaCl *(Marchand 1994)*

  $2NaCl + Ca(OH)_2 \rightarrow CaCl_2 + 2NaOH$

- $MgCl_2$ *(Mindess, Young and Darwin, 2002)*

  $Ca(OH)_2 + MgCl_2 \rightarrow CaCl_2 + Mg(OH)_2$

  $C\text{-}S\text{-}H + MgCl_2 \rightarrow CaCl_2 + M\text{-}S\text{-}H$

Chemical Mechanisms of Deicer

- How **Calcium Chloride** ($CaCl_2$) Attacks

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Arrow means shaken, not stirred

Chemical Mechanisms of Deicer

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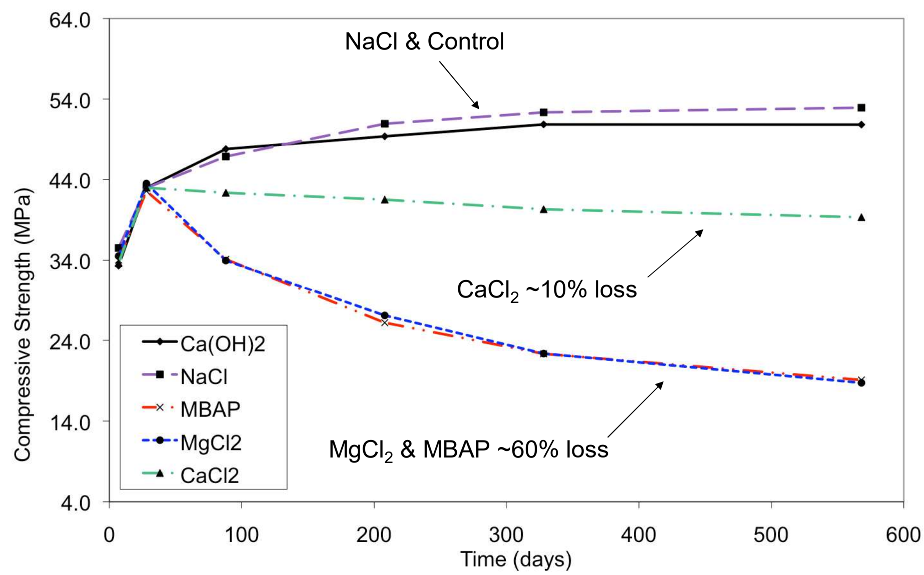
Compressive strength evolution with time of mortar cubes



So what's new on chemical attack?

- Work from Jason Weiss et al. (Purdue, Oregon State) has confirmed the formation and destructive nature of oxychloride

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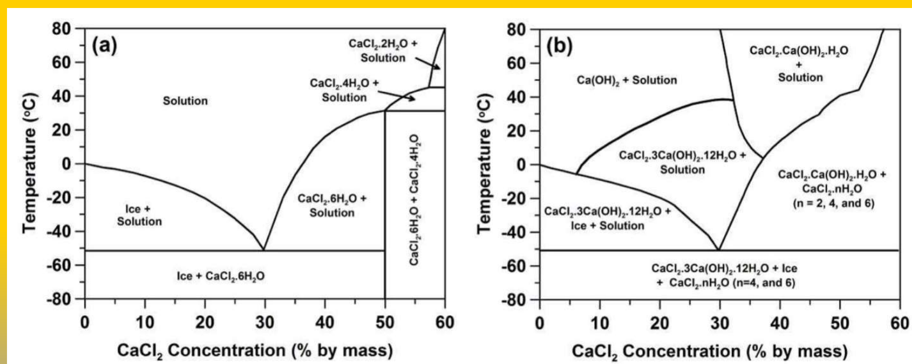
The Theory

- The solutions that freeze are not a mixture of salt and water (a) – there are alkalis and other species in the solution (b)



0 ºF = -17.8 ºC
-58 ºF = -50 ºC

Farnam Y., S Dick, A Wiese, J Davis, D Bentz, J Weiss. "The Influence of Calcium Chloride Deicing Salt on Phase Changes and Damage Development in Cementitious Materials." Cement and Concrete Composites, Volume 64, November 2015, Pages 1-15.

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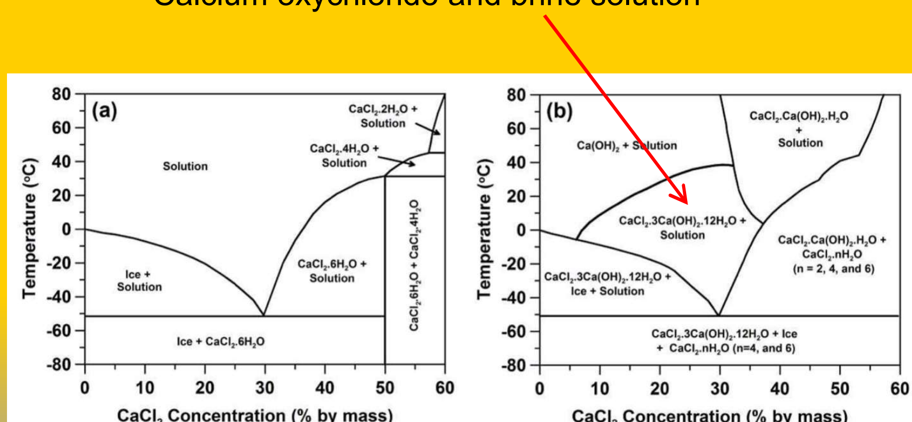
The Theory

Calcium oxychloride and brine solution



0 ºF = -17.8 ºC
-58 ºF = -50 ºC

Farnam Y., S Dick, A Wiese, J Davis, D Bentz, J Weiss. "The Influence of Calcium Chloride Deicing Salt on Phase Changes and Damage Development in Cementitious Materials." Cement and Concrete Composites, Volume 64, November 2015, Pages 1-15.

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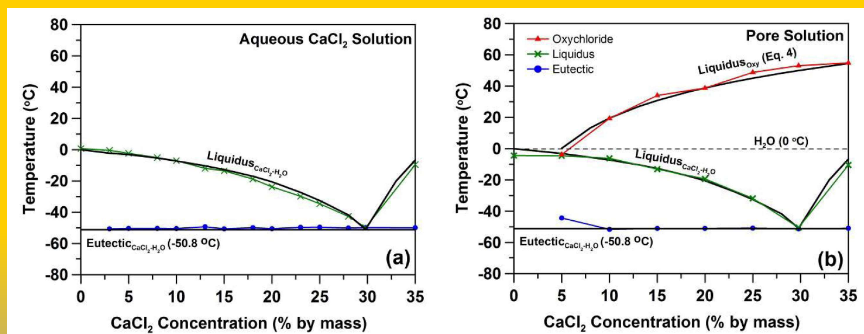
Purdue Research

- Results show clear evidence of oxychloride forming above freezing temperatures



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Oxychloride Formation

- Work at Michigan Tech and now confirmed at Purdue shows oxychloride forms at temperatures above freezing
- **Implications** – residual salt in concrete pore structure will form oxychloride – chemical attack year round ????
- To what extent is this happening ????

Take Aways – Chemical Attack

- Brines of magnesium and calcium chloride have been demonstrated to react deleteriously with hydrated cement paste
  - Expansive calcium oxychloride forms
  - Reaction is slower than physical attack mechanisms
  - Reaction can occur year round

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What do we do?

- Reduce permeability
- Use SCMs
- Drainage

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Reduce Permeability

- Keep the water out and reduce the salt brine ingress
  - Lower *w/cm* (0.40 or less)
  - Penetrating sealers
  - Permeability reducing admixtures
  - Use SCMs

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A word on sealers...

- Silane and siloxane are effective at reducing the ingress of fluid into concrete

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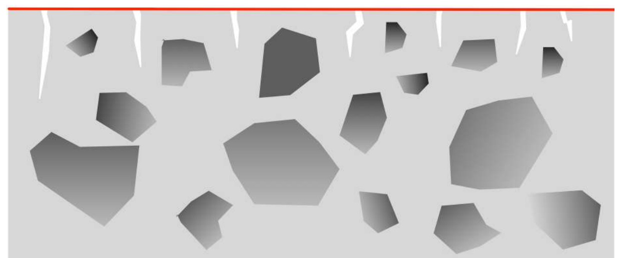
Film Formers

- Form complete surface barrier with little penetration
- Do not react with concrete
- Susceptible to wear and abrasion
- In some cases susceptible to UV
- Can be removed
- Capable of penetrating and sealing large cracks
- May be applied with aggregate top coat to improve friction
- May be thinned (pore blocker)

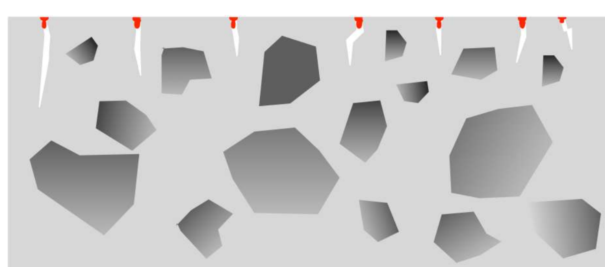
Ex. epoxies, polyurethanes, methacrylates, waxes, acrylics



Chemically Reactive – Pore Blocker

- React with cement paste (CH) forming gels that "block" pores
- Can not be removed
- In some cases susceptible to UV
- Increase surface hardness
- Decrease permeability
- Block moisture transport both in *AND* out of concrete

Ex. Silicates of lithium or sodium, linseed oil

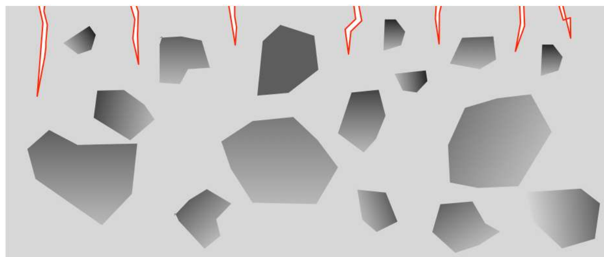


Chemically Reactive – Water Repellent

- React with cement paste by bonding with Si
- Can not be removed
- In some cases susceptible to UV
- Not a “sealant”

- Water enters pore but the water repellent coating prevents ingress into the cement paste
- Allow moisture transport out of concrete

Ex. silanes, siloxanes



A word on sealers...

- Silane and siloxane are effective at reducing the ingress of fluid into concrete
- Questions?
  - How long do they last?
  - F-T durability of the sealer?
  - Cost effectiveness compared to other options?
  - Can you get it where you need it (in the joint)?

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Affect of FT on Sealer Performance

AASHTO T 260

Cl conc. @ depth of 1 inch

Ratio of Cl measured on sealed & unsealed samples

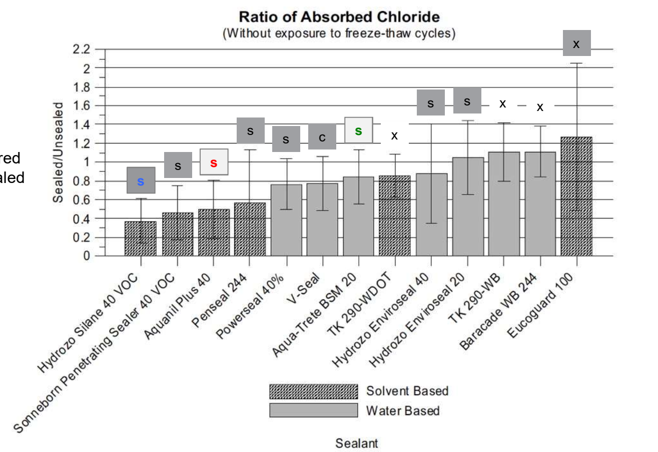

Figure 6.2.7 Ratio of absorbed chloride in specimens without exposure to freeze-thaw cycles

s - silane
x - siloxane
c - siliconate

J.A. Pincheira, M. Dorhorst. "Evaluation Of Concrete Deck And Crack Sealers." Wisconsin Highway Research Program 06-09. Madison, WI. December 2005.

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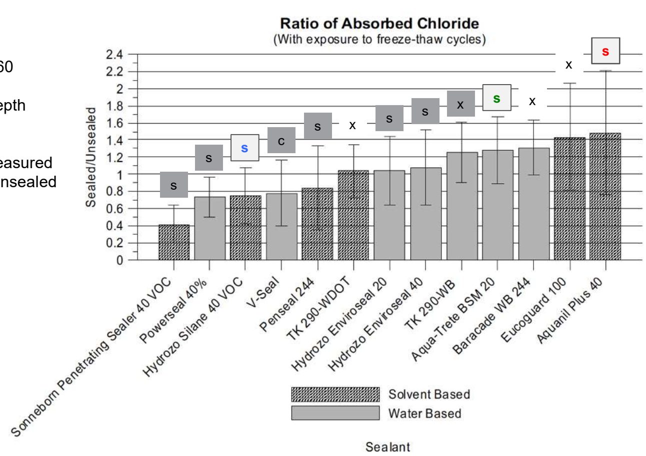

Figure 6.2.8 Ratio of absorbed chloride in specimens exposed to freeze-thaw cycles

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Depth of Penetration

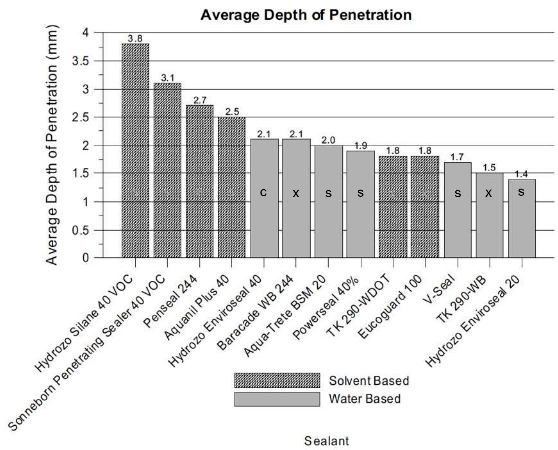

Figure 6.2.11 Average depth of penetration of each sealant arranged in order of decreasing value.

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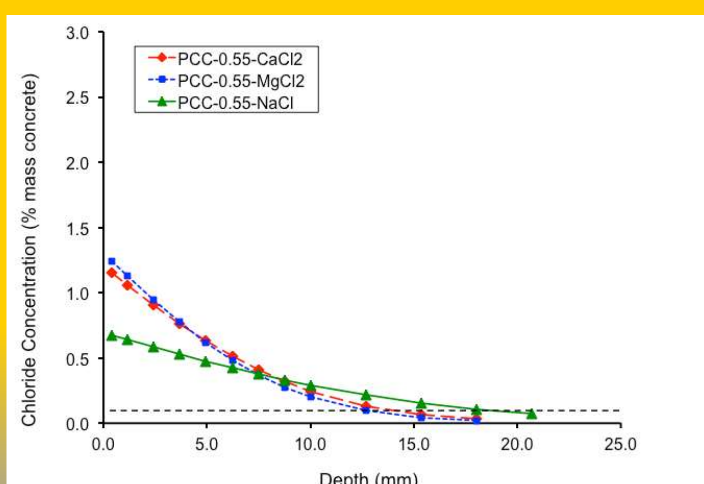
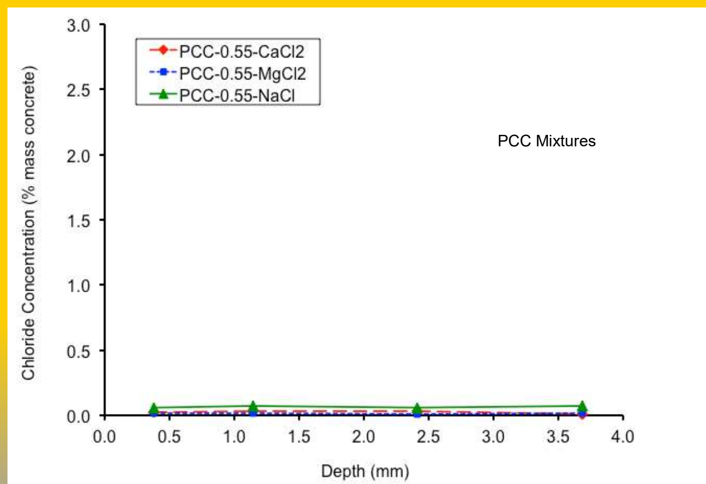
J.A. Pincheira, M. Dorhorst. "Evaluation Of Concrete Deck And Crack Sealers." Wisconsin Highway Research Program 06-09. Madison, WI. December 2005.

Salt Ingress for OPC mixture

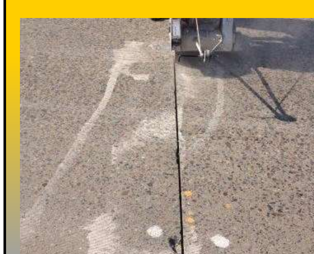


Tri-siloxane 12% (aliphatic hydrocarbon)

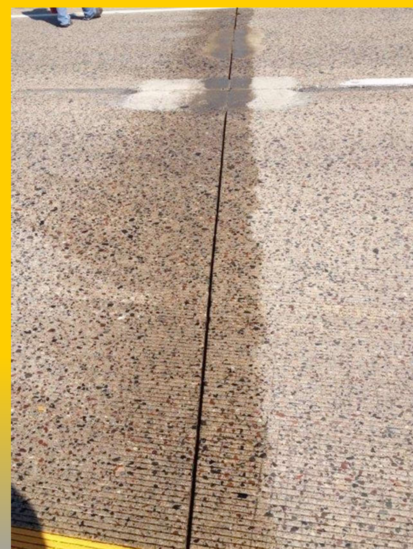


Preparation



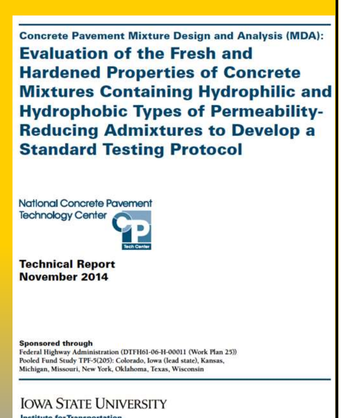



Application



Permeability Reducing Admixtures

- Different types – different mechanisms
  - Water repellents
  - Crystal formers
  - Colloidal silica/silicates
- Need more research
- Similar questions to sealers
- MiDOT research underway
  - Durability testing included



Concrete Pavement Mixture Design and Analysis (MDA):
Evaluation of the Fresh and Hardened Properties of Concrete Mixtures Containing Hydrophilic and Hydrophobic Types of Permeability-Reducing Admixtures to Develop a Standard Testing Protocol

National Concrete Pavement Technology Center

Technical Report
November 2014

Sponsored through
Federal Highway Administration (DTFH61-06-H-00011 (Work Plan 25))
Pooled Fund Study TPF-5(205): Colorado, Iowa (lead state), Kansas, Michigan, Missouri, New York, Oklahoma, Texas, Wisconsin

IOWA STATE UNIVERSITY
Institute for Transportation

SCMs

- Oxychloride requires calcium hydroxide (CH) to form
- Reduce the CH – reduce oxychloride formation

Portland Cement Reaction

Cement + Water -> C-S-H + CH

Pozzolanic Reaction (example: Class F Fly Ash)

CH + Pozzolan + Water -> C-S-H

- Forming C-S-H reduces permeability
- Consuming CH reduces oxychloride formation

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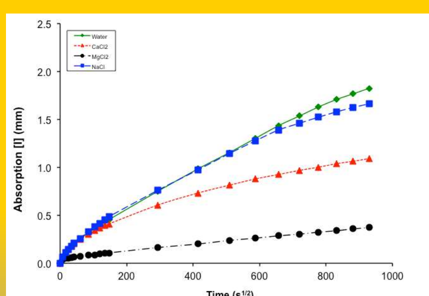
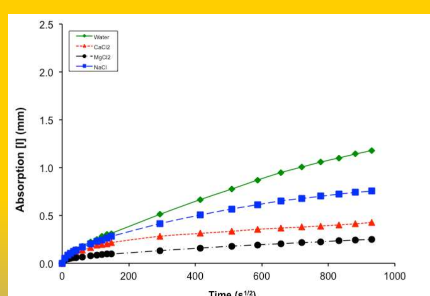
SCM Effects on Permeability

0.45 *w/c* OPC



0.45 *w/c* OPC + 15% Class F



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New Developments

- Based on research reported by Purdue, a new test is being developed to determine the necessary pozzolan addition to minimize oxychloride formation
- Test based on Low Temperature Differential Scanning Calorimetry (LT-DSC)

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Take Aways – Actions

- Sealants can be effective – we still do not have a good way to install
- PRAs offer opportunity – more research is needed
- SCMs are the only sure bet right now

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Take Aways – Actions

- Use of SCMs reduces susceptibility of concrete for chemical attack
  - Reduces CH available to react
  - Permeability of concrete not compromised by CH leaching
  - Improves concrete strength (affects physical and chemical attack)

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Summary

- Keep fluids out of concrete and all materials-related distress is minimized
- Materials selection is very important
  - Low *w/c*
  - Low paste content
  - Use SCMs
- Curing is essential
  - Keep the mixture water in and allow the materials time to form dense, impermeable, hydration products

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Summary

- Deicing chemicals are a serious concern
  - Contribute to physical attack
  - Contribute to chemical attack
- Sealants may be required to offset deicing fluids
  - Keep brines out
  - Slow ingress of salt solutions

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Questions?

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