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Examining How Saturation and Pore Solution Chemistry Impact Durability Test Methods, Specifications and Service Life Models (Keynote)

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Examining How Saturation and Pore Solution Chemistry Impact Durability Test Methods, Specifications and Service Life Models

Jason Weiss and Burkan Isgor
Oregon State University

August 27th, 2018

The Formation Factor as a Practical Tool for Concrete Specifications and Service Life Models

Jason Weiss and Burkan Isgor
Oregon State University

August 27th, 2018

Thank you to Our Hosts and RILEM



Concrete is not the Dinosaur



But its Specifications Often Are



Performance Specification Approach - AASHTO



Oregon State University
College of Engineering

TARGET: Improve
Long-Term Durability

- Freeze-Thaw
- Salt Damage
- Chloride Ingress
- ASR
- Shrinkage & Cracking



Today I would like to discuss the role of pores, pore solution, and pore solution chemistry as it can be crucial but its often not considered as explicitly as it should be

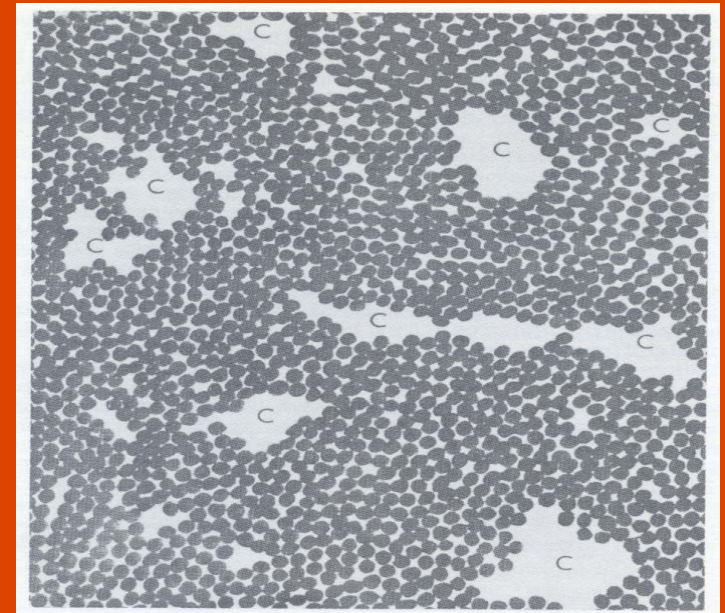
Weiss et al. 2015

Outline



Oregon State University
College of Engineering

- Porosity and Formation Factor
- F Factor, Resistivity and RCPT
- F Factor and Diffusion Coefficients
- F Factor and Absorption
- Pore Structure and Volume
- Degree of Saturation
- Solution & Immersion Testing
- Implementation in Practice

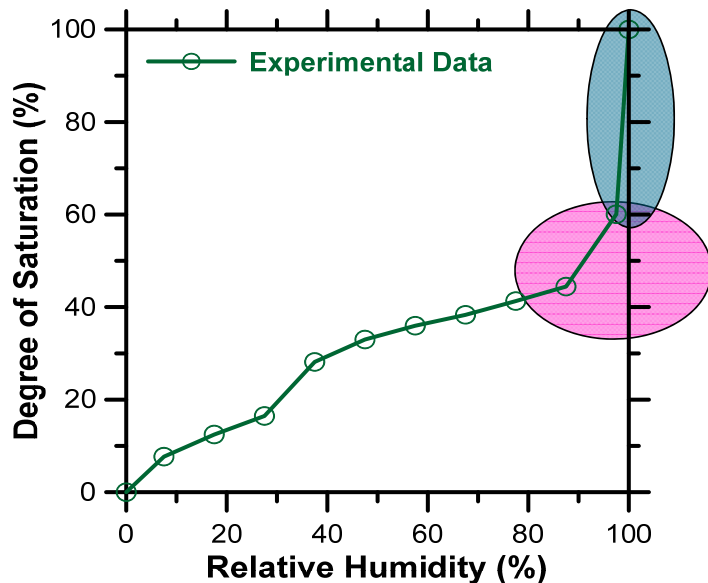


Pores are the Key

Entrained/Entrapped Air – Biggest pores that are important for freeze-thaw

Capillary Pores (5nm-10 mm) – Controlled by mix water, important for transport

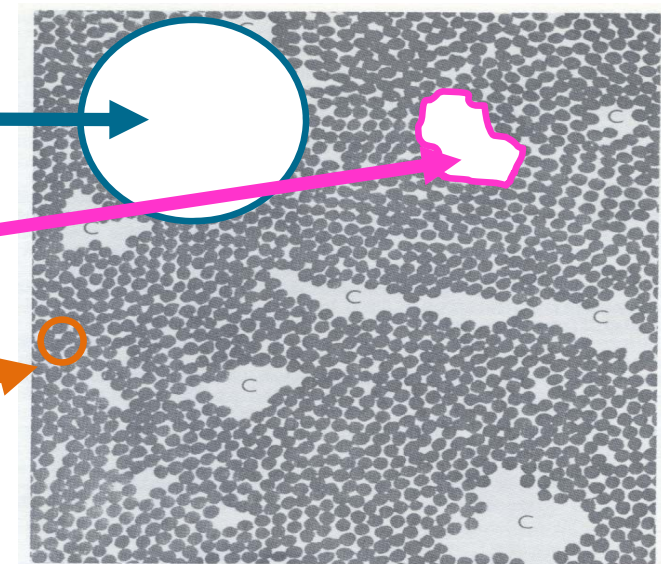
Gel Pores (2-5 nm) – Part of the structure, important for shrinkage



**Air controlled
by air**

**Capillary
controlled
by w/c**

**Gel is part
of the structure
(little we can do)**



Practical Descriptor



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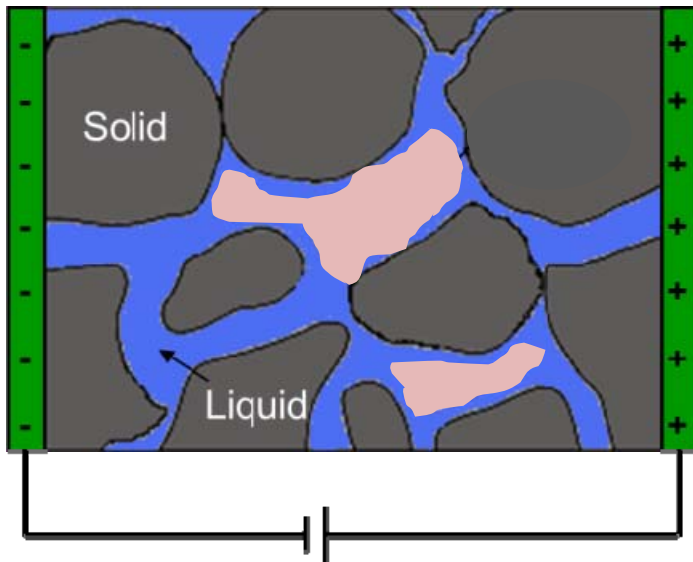


- Formation factor – Measure of pore volume and connectivity
- Gustavus (G.E.) Archie, worked for Shell Oil Company, Elk City OK
- Seminal work appeared in the 1940s
- Was quite useful for Oklahoma oil fields

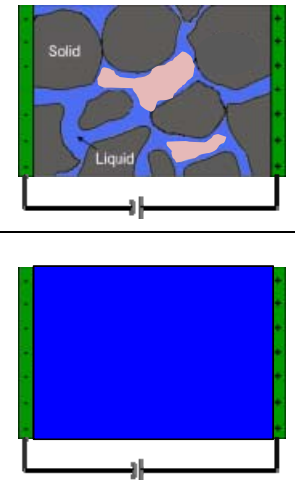


Formation Factor

- Related to pore volume (ϕ)
- Related to pore connectivity (β)



$$F = \frac{\rho}{\rho_0} = \frac{1}{\phi\beta}$$



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Resistivity and RCPT

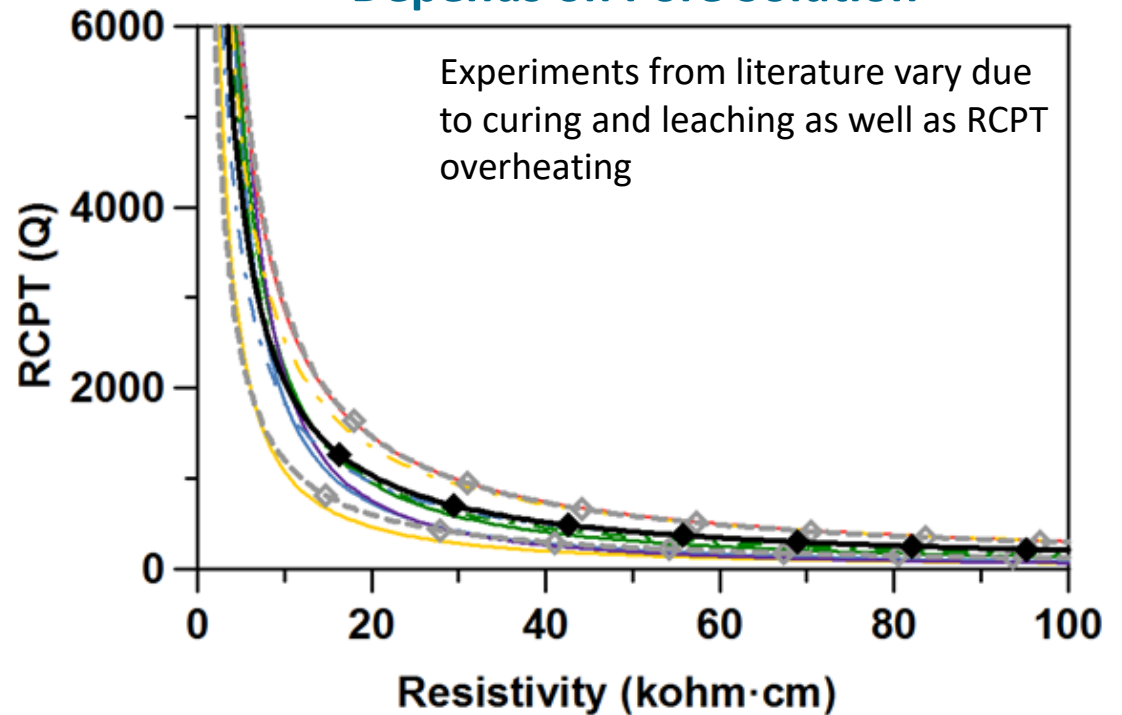


$$Q = \int_{0 \text{ hr}}^{6 \text{ hr}} I dt = \int_{0 \text{ hr}}^{6 \text{ hr}} \frac{V}{R} dt$$

$$Q = \int_{0 \text{ hr}}^{6 \text{ hr}} \frac{V A}{\rho L} dt$$

$$Q = V \frac{A}{L} t \frac{1}{\rho} = \frac{206,830 V m s}{\rho}$$

Depends on Pore Solution





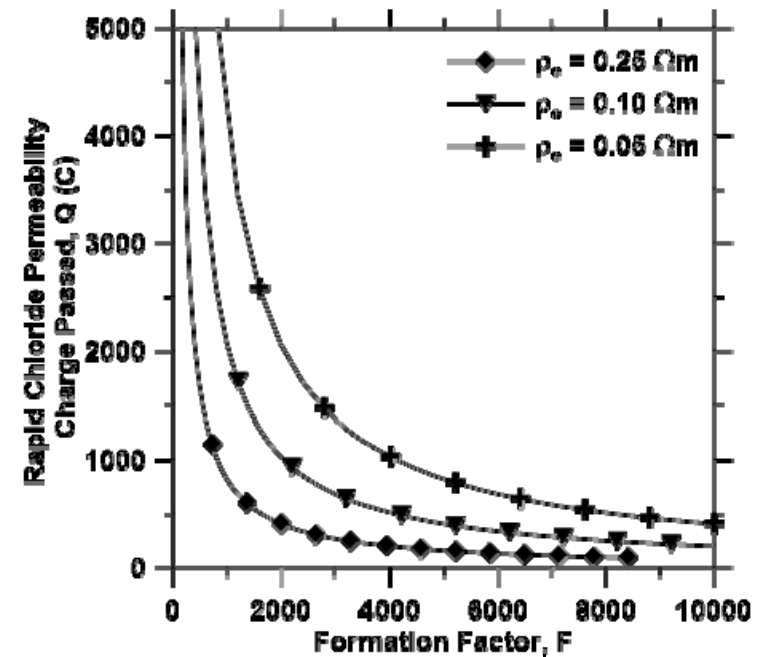
F Factor and RCPT

- This is written as F-Factor which shows errors in RCPT if ρ soln is not known

$$Q = V \frac{A}{L} t \frac{1}{\rho_0} \frac{1}{F}$$

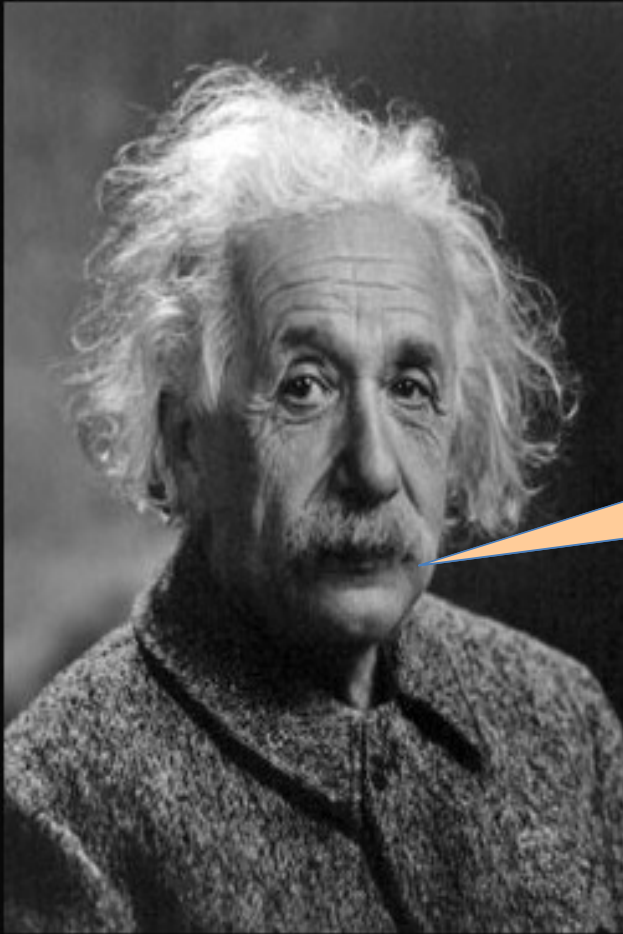
$$Q = 60V \frac{8107 \text{ mm}^2}{50.8 \text{ mm}} 21,600 \text{ s} \frac{1}{\rho_0} \frac{1}{F}$$

$$Q = \frac{206,830 \text{ V m s}}{\rho_0} \frac{1}{F}$$



Weiss et al. 2016

Theory of Everything



**Use the F Factor to
Obtain Other Transport
Properties**

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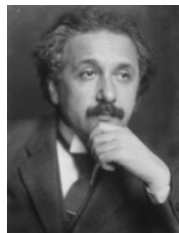
Service Life Models for Corrosion of Steel



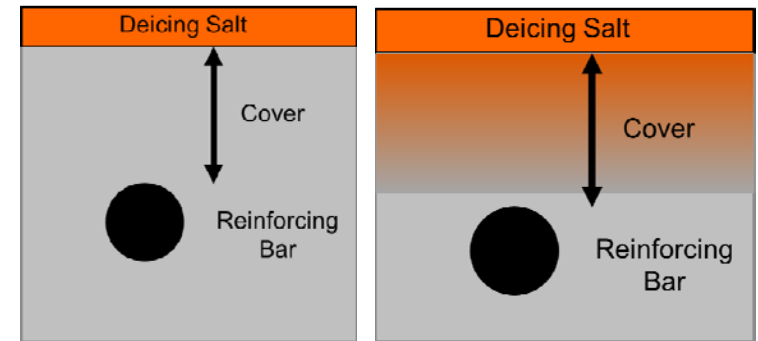
- Chloride ingress is important for concrete SLM's
- $D_{\text{Effective}}$ – No reaction/binding



$$\frac{1}{F} = \frac{D_i}{D_i^\mu} = \frac{\rho_{\text{Pore}}}{\rho_{\text{Bulk}}}$$



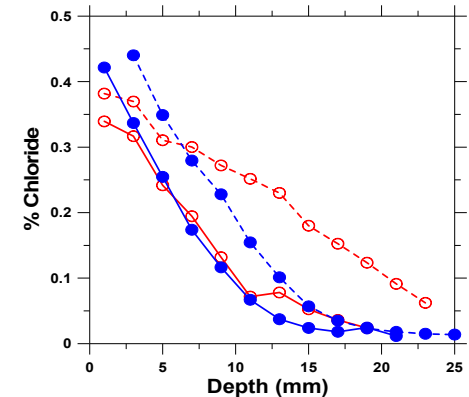
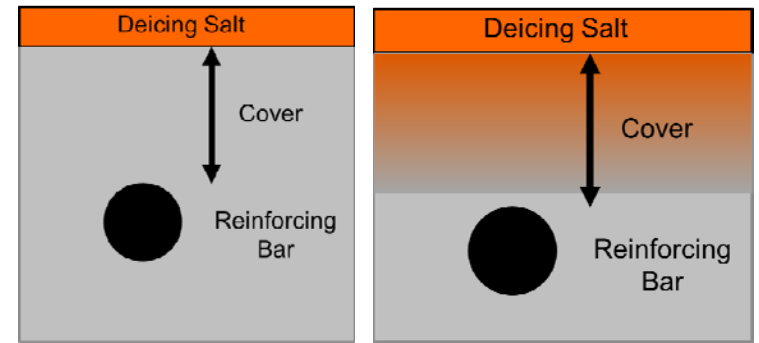
$$D_i = D_i^\mu \cdot \phi\beta = D_i^\mu \cdot \frac{1}{F}$$



Species	D_i^μ ($10^{-9} \text{ m}^2/\text{s}$)
OH^-	5.273
Na^+	1.334
K^+	1.957
SO_4^{2-}	1.065
Ca^{2+}	0.792
Cl^-	2.032
Mg^{2+}	0.706

Service Life Models for Corrosion of Steel

- Chloride ingress is important for concrete SLM's
- $D_{\text{Effective}}$ – No reaction/binding
- D_{Apparent} – Typically ponding tests





F Factor and D_{Apparent}

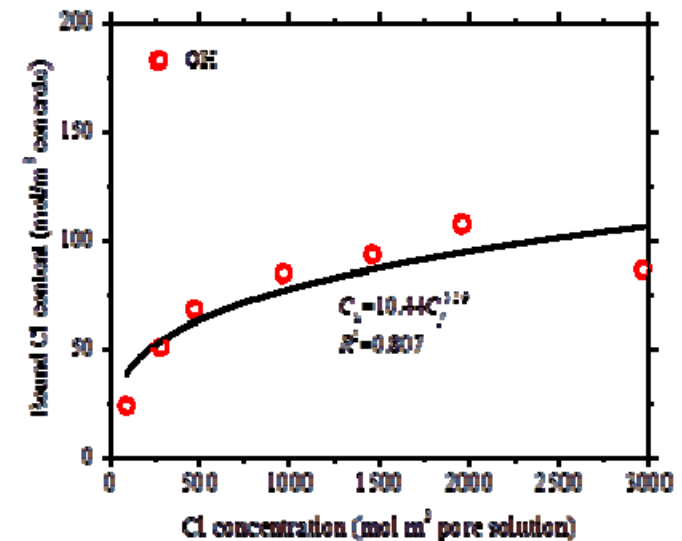
- Frequent criticism of F-Factor - it doesn't include binding
- While this is true (neither does any electrical measure), it can be shown that F Factor can easily be combined with a binding isotherm to predict performance.

- Nernst Plank equation:

$$\frac{\partial}{\partial t}(pC_i + C_i^b) = -div\left[-\frac{D_i^0}{F}\left(gradC_i + C_i grad\ln\gamma_i + \frac{z_i F}{RT} C_i grad\psi\right)\right]$$

- Freundlich binding: $c_{cl}^b = \alpha \cdot c_{cl}^\beta$

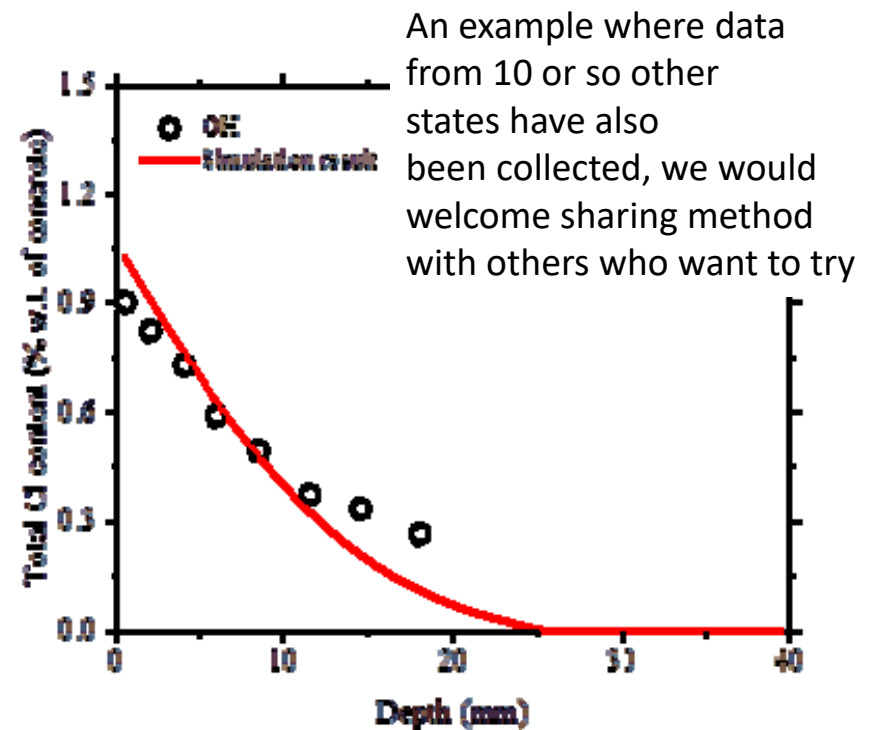
(Qiao et al. submitted)



Chloride Diffusion



- Here we see that combining the F -Factor and binding is very powerful .
- This does a good job at predicting chloride ingress.
- This is much faster than ASTM 1556.
- Further binding is a qualification test and F is a QC/QA test.



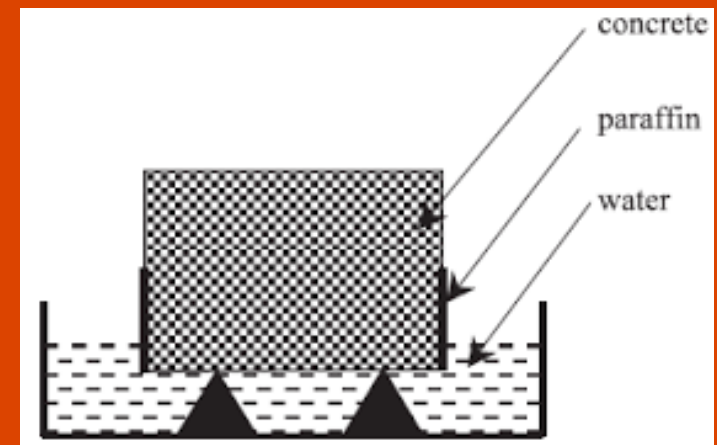
(Qiao et al. submitted)

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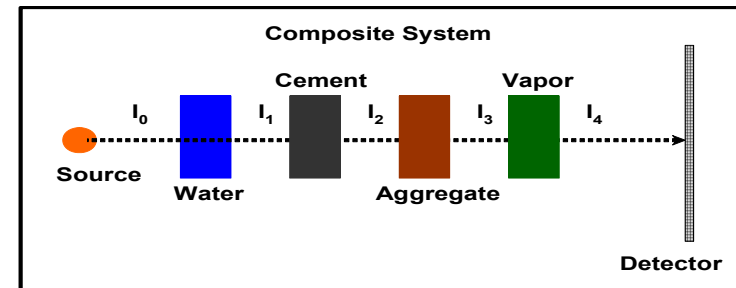
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F Factor and Absorption

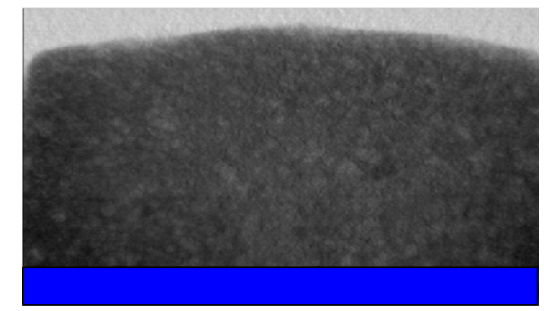
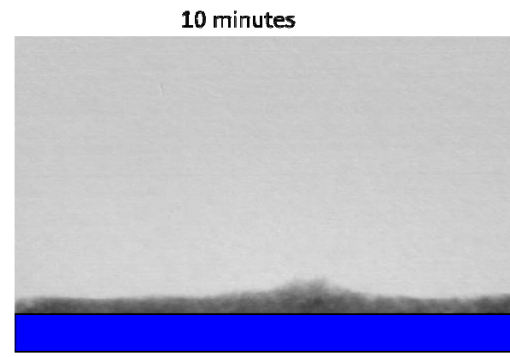


- Demonstrate that the mass of absorbed water (M) is related to ($F^{-0.5}$)
- Derived from first principles



$$I(t) = I_0 \exp \left[- \left(\sum_{i=1}^N (\mu_i V_i) \right) t \right]$$

$$M(t) = \frac{A\rho R_i}{2} \sqrt{\frac{\varepsilon P_{cap}}{\mu}} \sqrt{\frac{1}{F}} \sqrt{t}$$

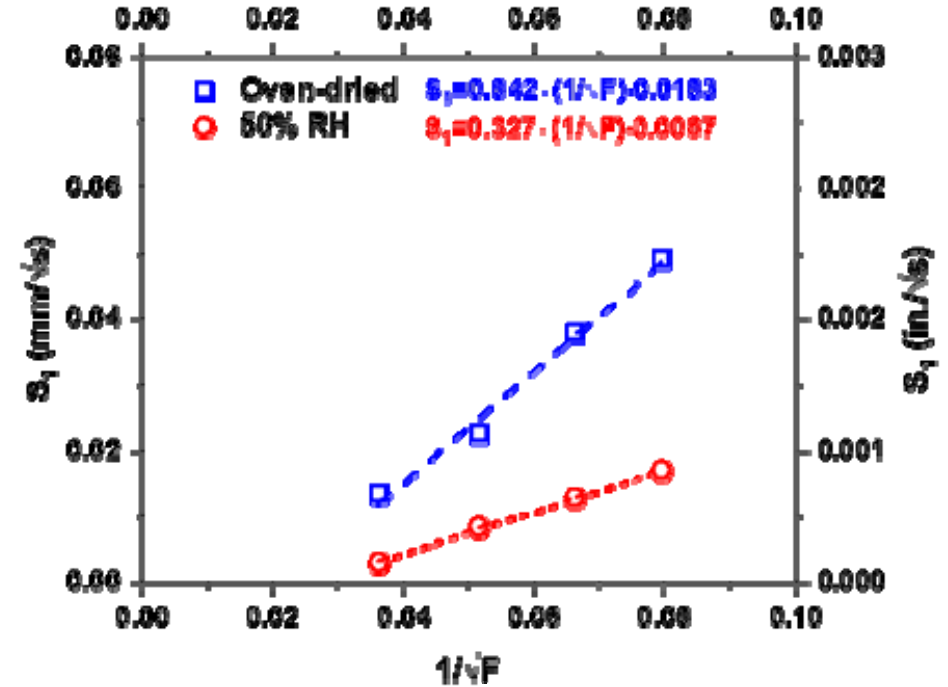
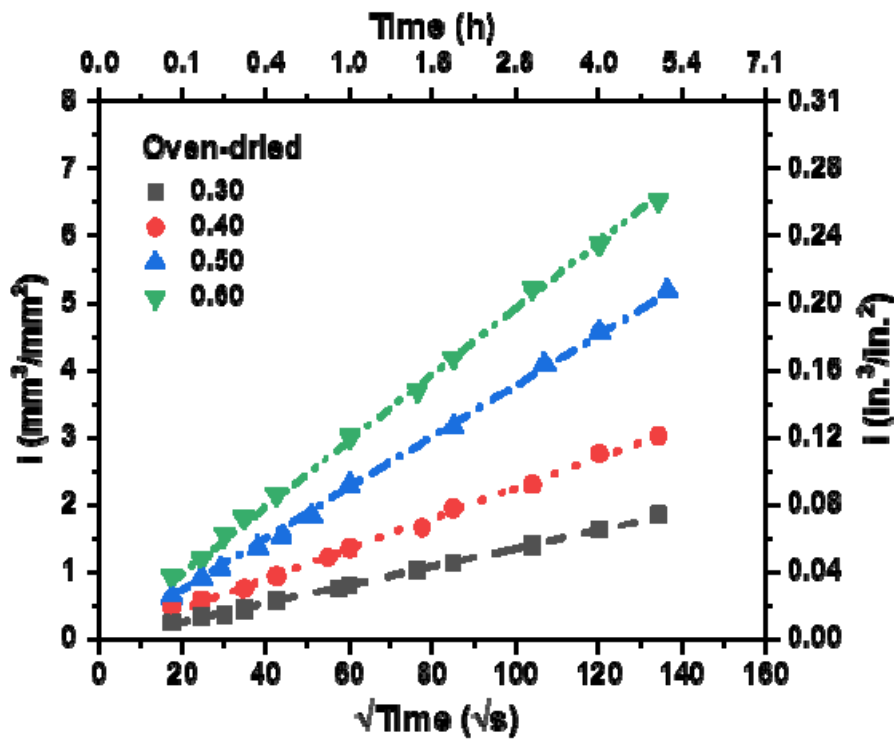


11 hours

Absorption



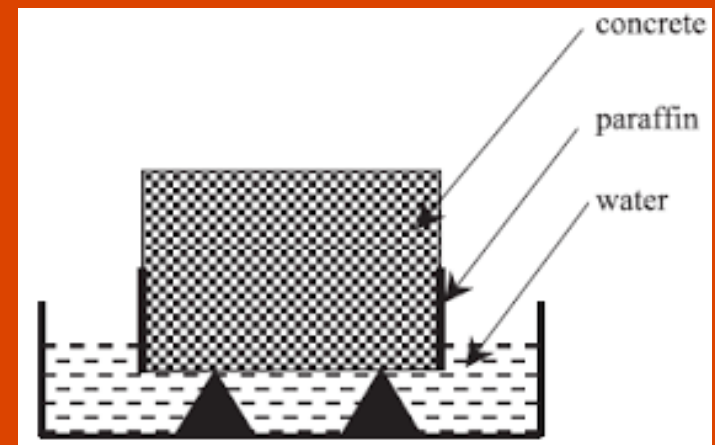
$$M(t) = \frac{A\rho R_i}{2} \sqrt{\frac{\varepsilon P_{cap}}{\mu}} \sqrt{\frac{1}{F}} \sqrt{t}$$



Outline



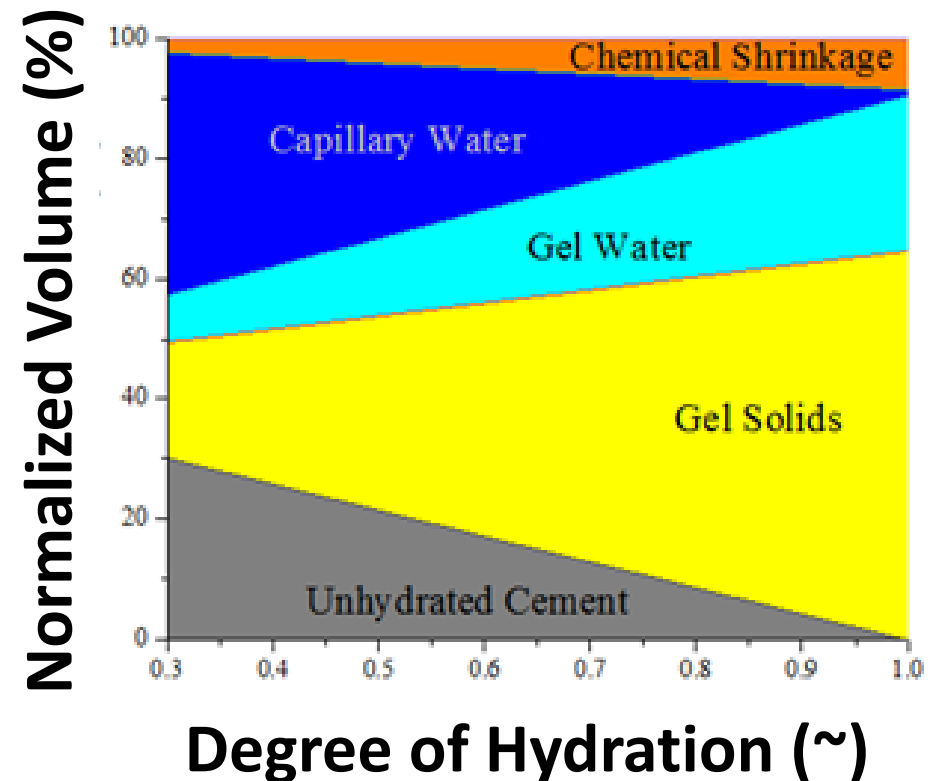
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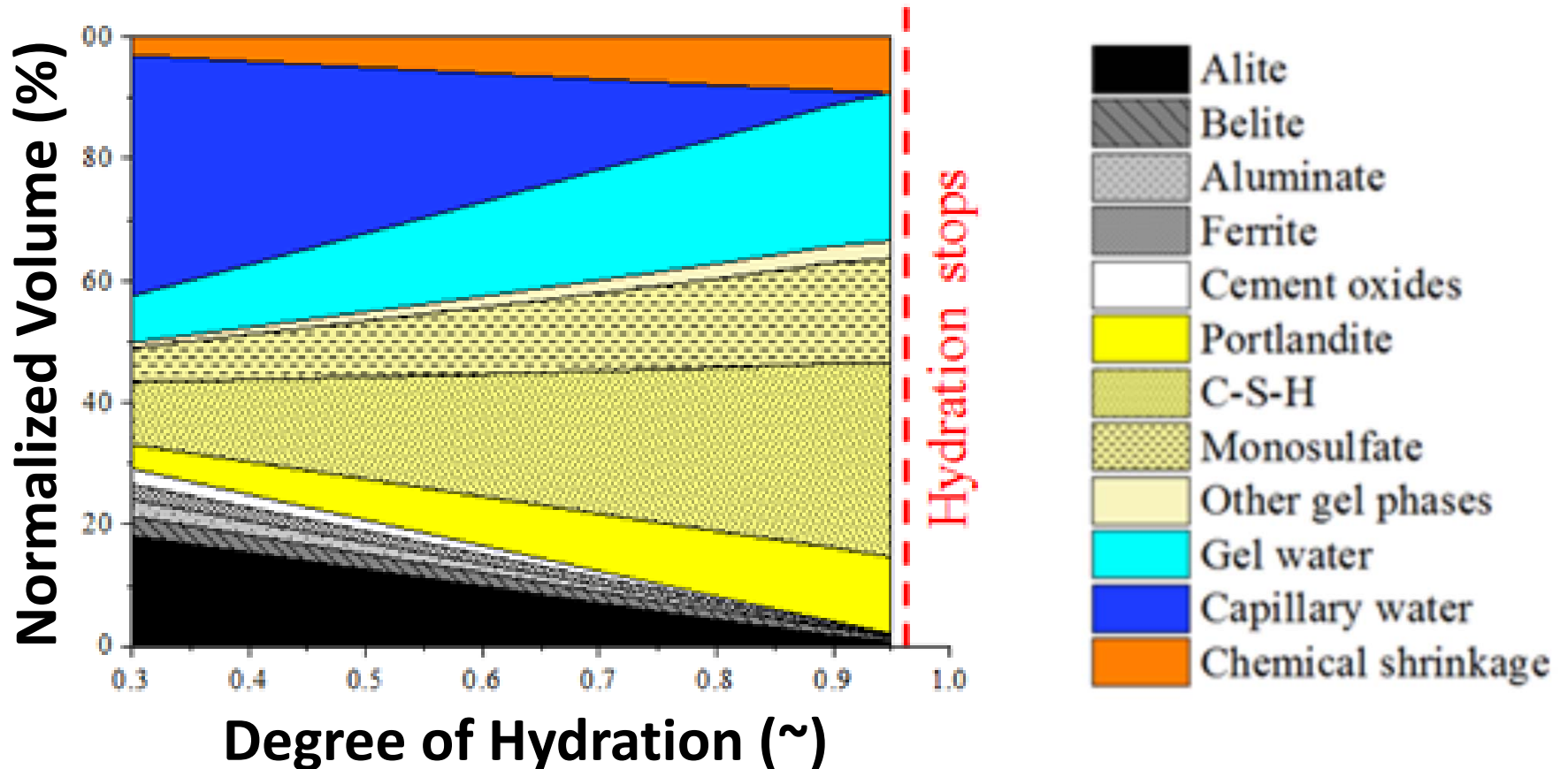
Pore Structure Model



- The Powers-Brownnyard model is used to determine pore volumes
- However it is limited to OPC systems, does not 'distinguish' between different solids, and does not provide pore solution composition



GEMS Simulations



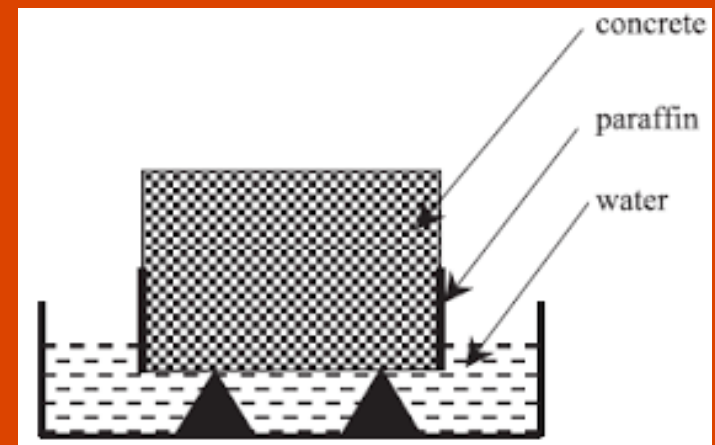
Glosser et al. to be submitted

Outline

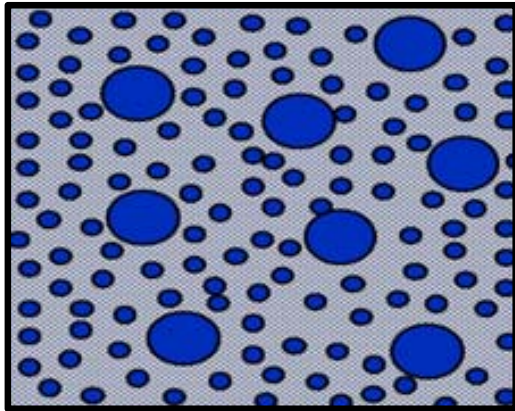


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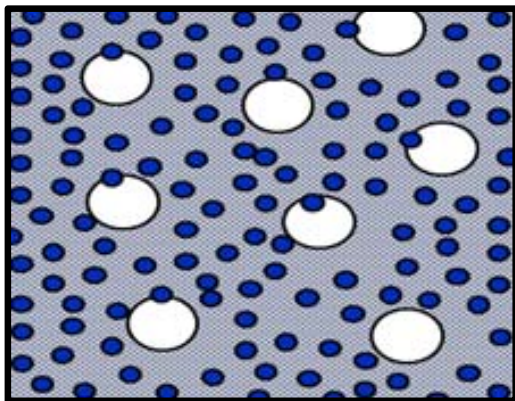
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Saturation: S vs S_{Matrix}

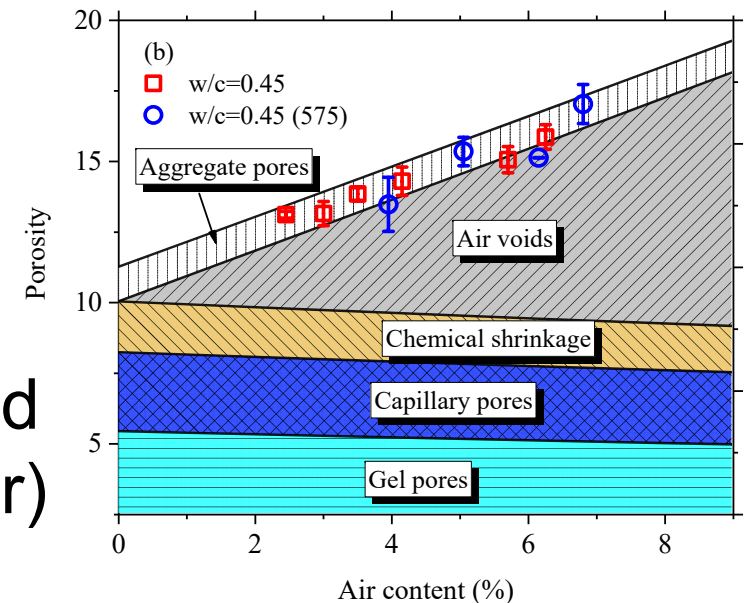


Saturated, S



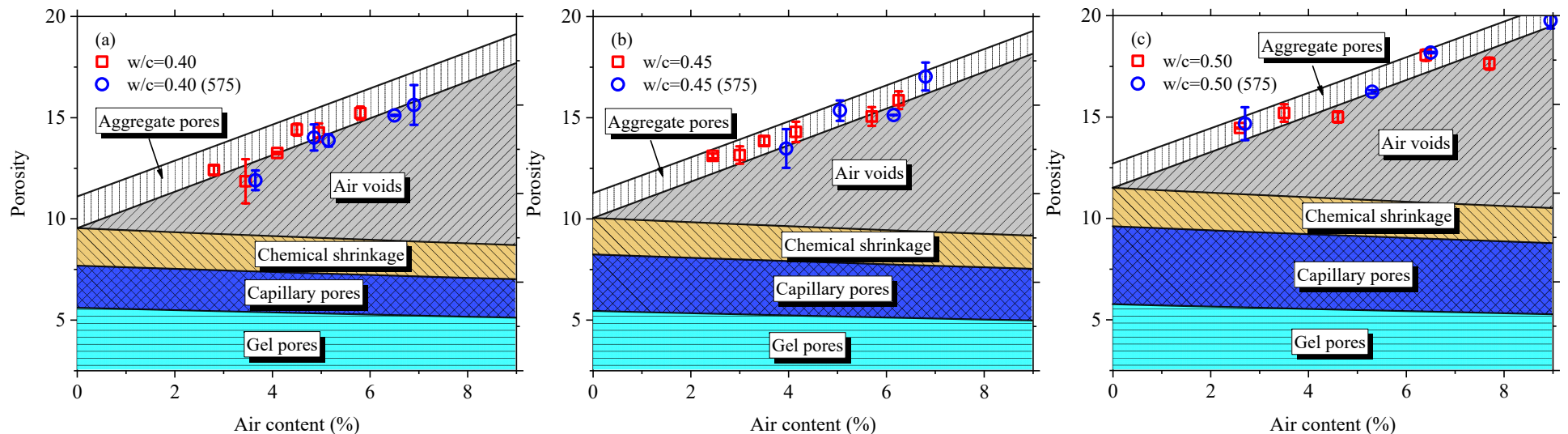
Saturated Matrix, S_M

- This will seem very academic and not very important however it is a crucial issue
- This applies both to testing and application
- Vacuum saturation fills all voids: gel, capillary, and air
- Submersion fills in matrix voids: gel and capillary (i.e., not air)

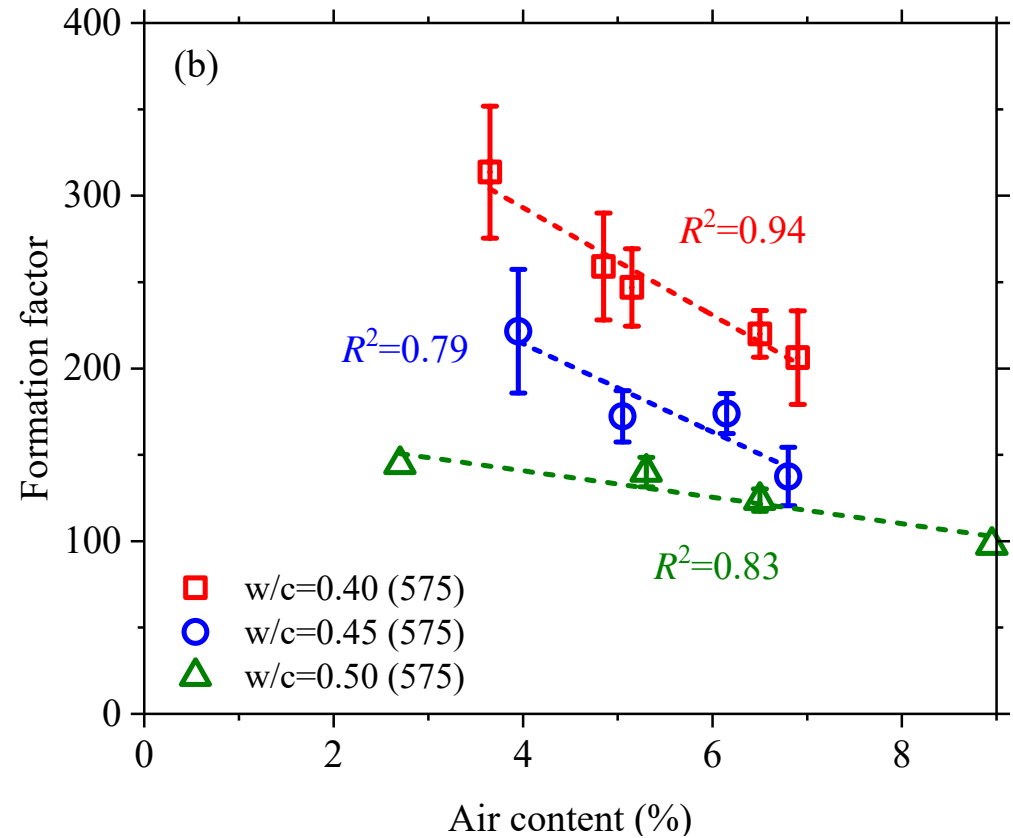
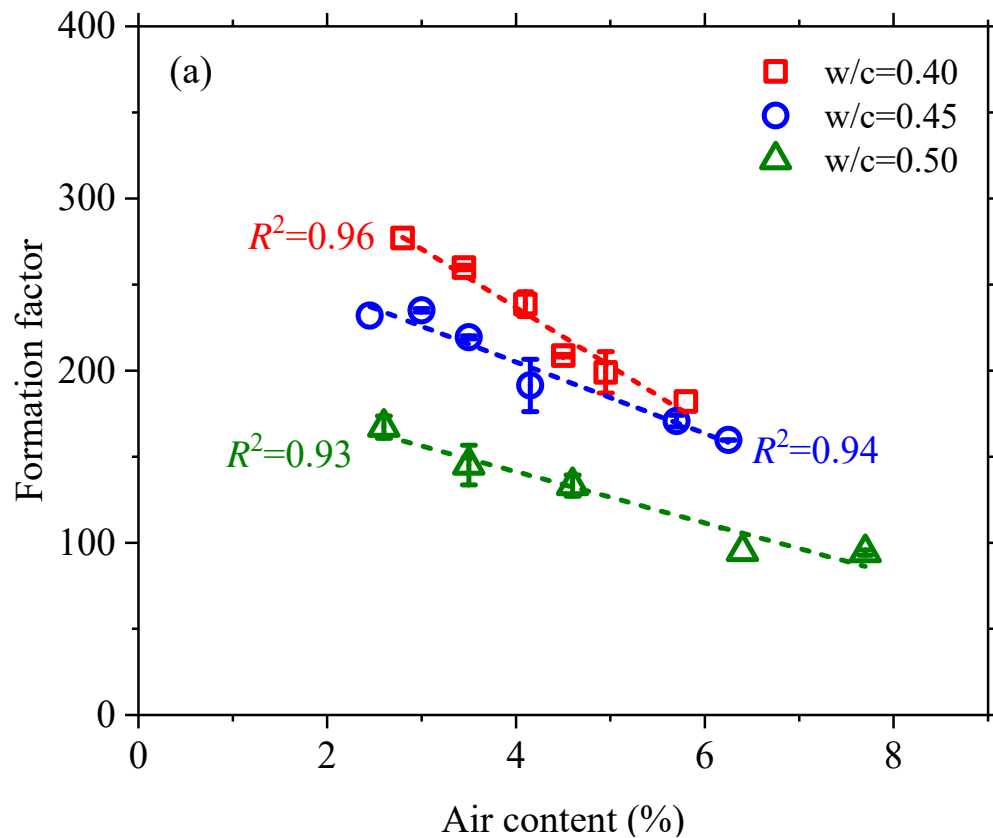


Porosity: Measured versus Theoretical

- We have compared numerous mixtures (32 shown here) but many more have been tested
- Theory matches practice well

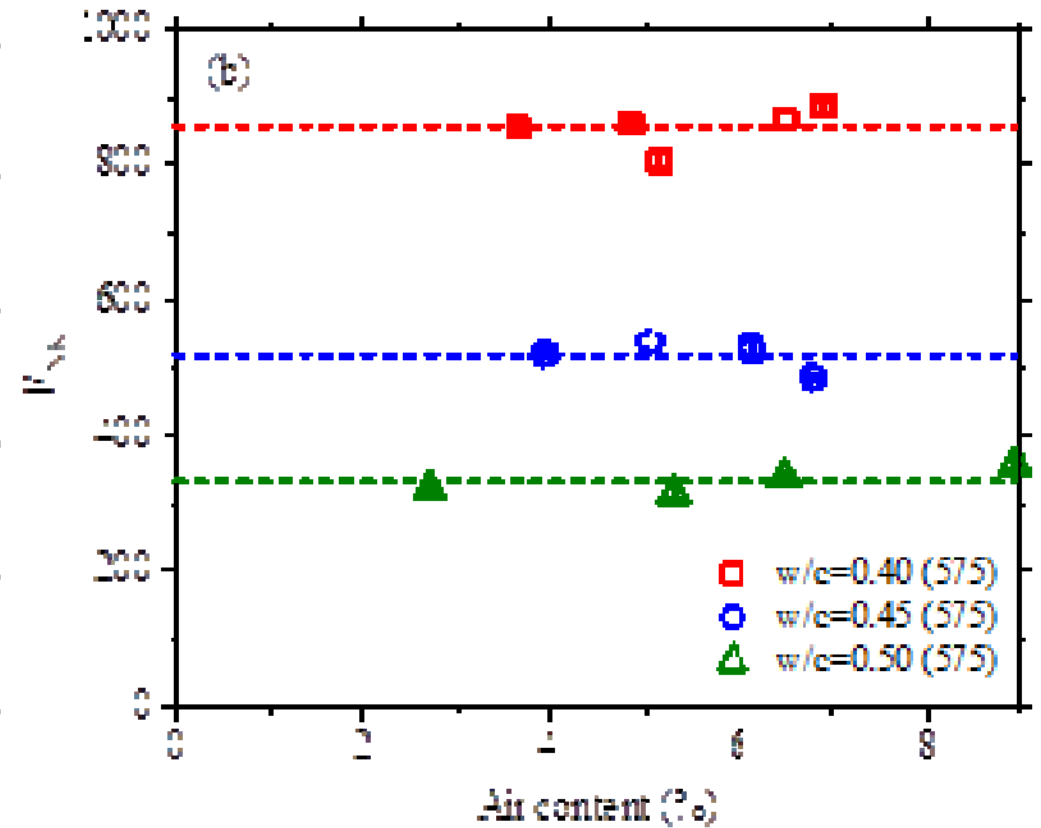
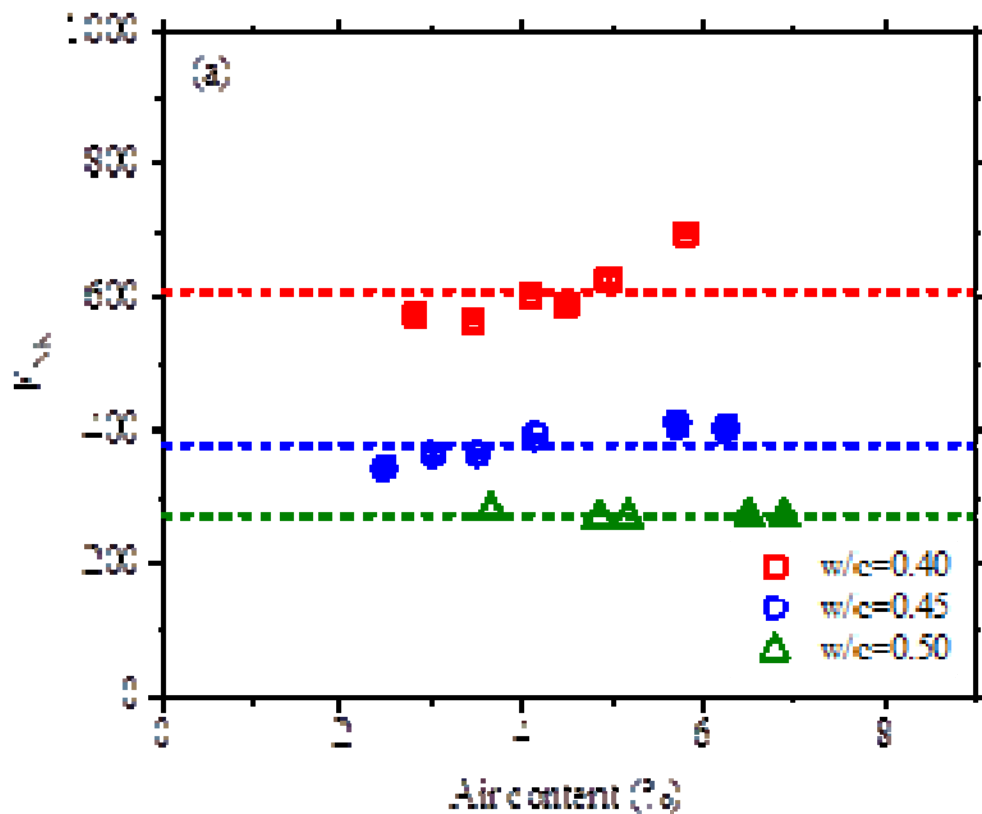


Saturated F Factor



Qiao et al. submitted

F Factor For Matrix Saturation

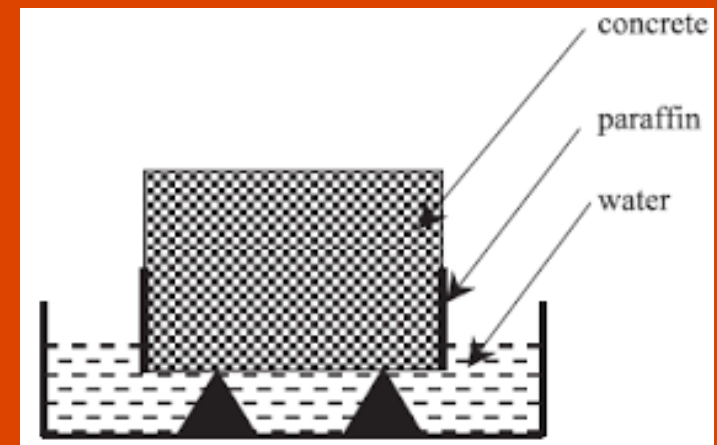


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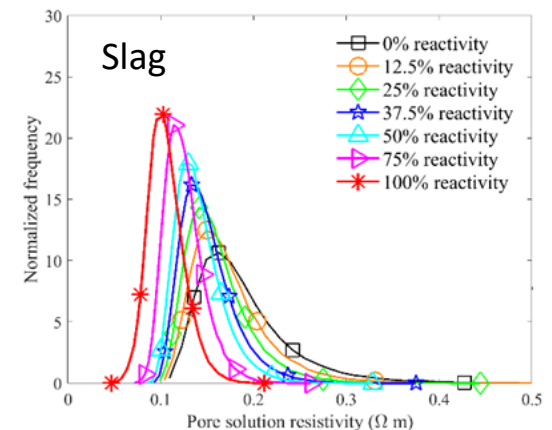


Computation of the Pore Solution



- Assume a value (0.04-0.12 Ω -m) (Spragg 2017)
- Estimate value from mill cert (NIST) (Bentz 2007)
- Estimate value from GEMS (Azad et al. 2018)
- Use of Sensors (Rajabiopour et al. 2007)
- Pore solution expression (Barneyback/Diamond 1981)
 - Direct measurements: resistance meter
 - Chemical analysis:
 - Inductively coupled plasma (ICP)
 - Atomic absorption (AA)
 - Ion chromatography (IC)
 - X-ray Fluorescence (XRF)

$$F = \frac{\rho_b}{\rho_o}$$



Are estimates perfect no, are the better than nothing (RCPT) yes

Ionic Composition to Resistivity



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$$\rho_{calc} = \frac{1}{\sum z_i c_i \lambda_i}$$

z_i = valence

c_i = ionic concentration

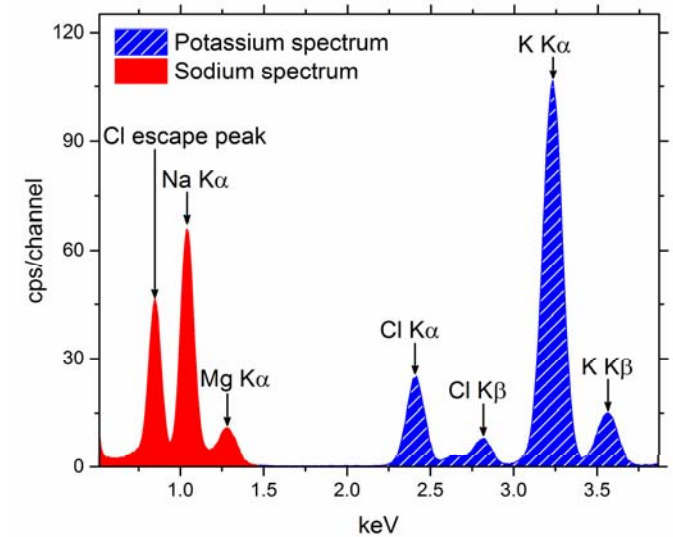
λ_i = individual equivalent conductivities

$$\lambda_i = \frac{\lambda_i^\circ}{1 + G_i I_M^{1/2}}$$

λ_i° = equivalent conductivity at infinite dilution

G_i = empirical conductivity coefficient

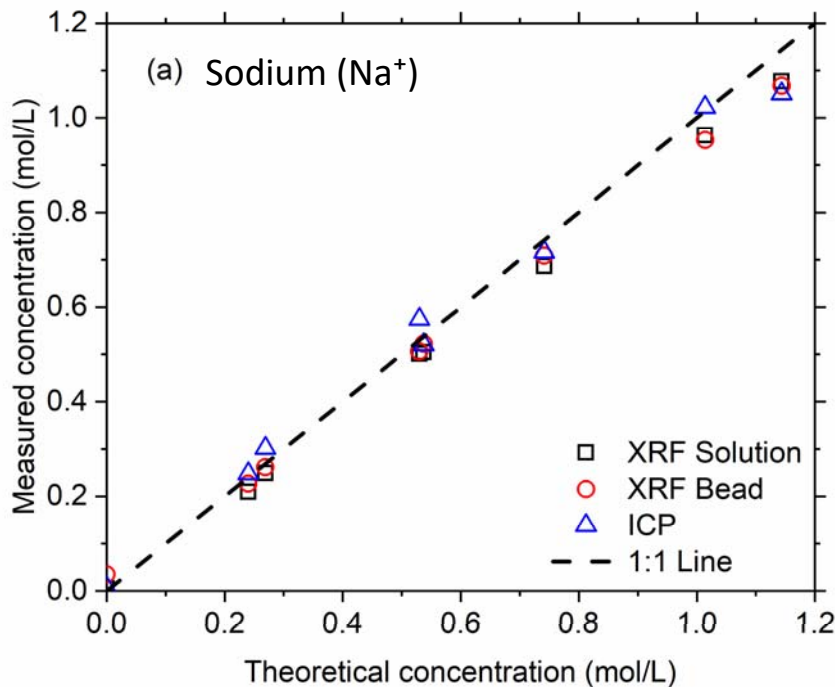
I_M = ionic strength



(Tsui-Chang et al. 2017)

Chemical Composition

For simulated pore solutions



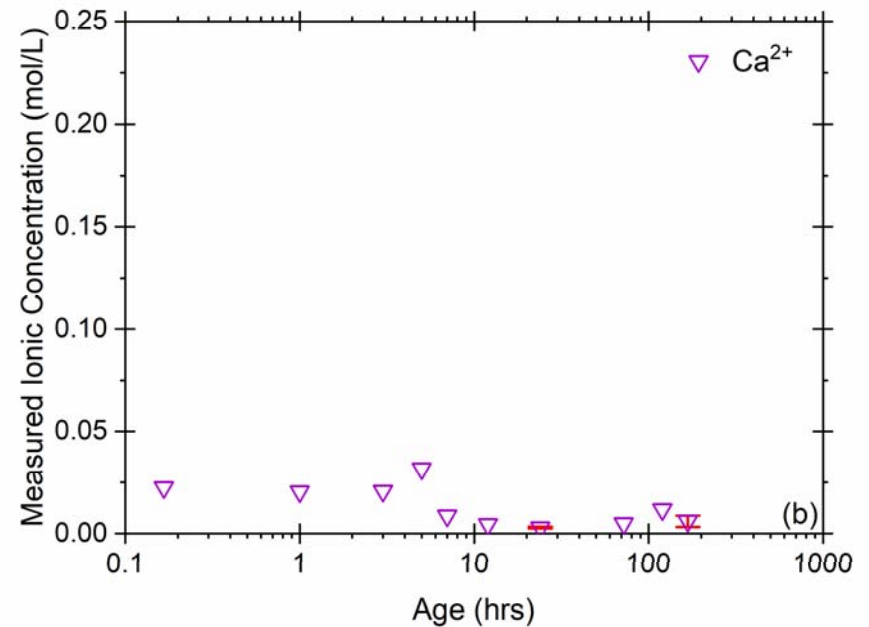
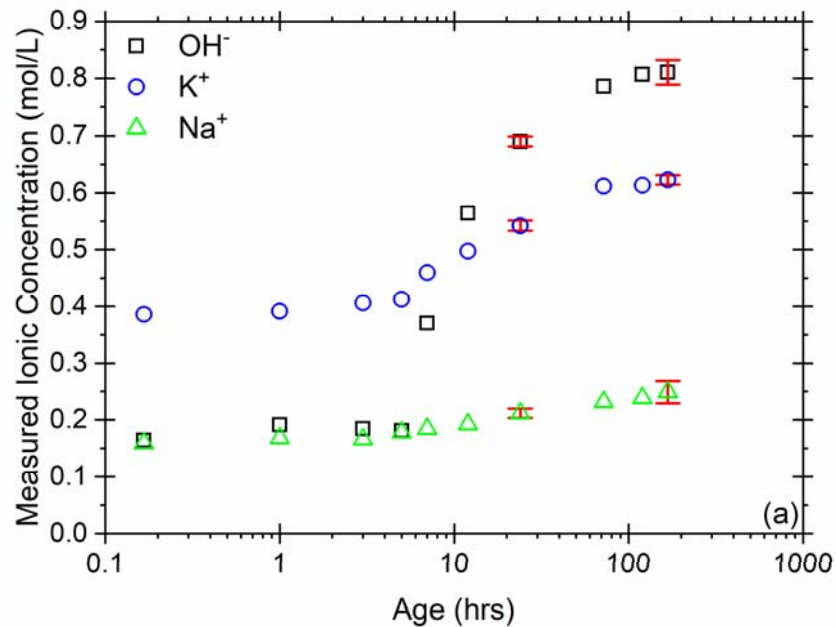
	XRF Solutions	XRF Bead	ICP
Linear Fit	$y=0.96x$	$y=0.96x$	$y=1.00x$
R^2	0.999	0.998	0.990
Average Percent Error (%)	-5.57	-3.05	3.10

x = theoretical concentration (mol/L)
 y = measured concentration (mol/L)

We can accurately measure the ionic concentrations using XRF

Chemical Composition

For expressed pore solutions



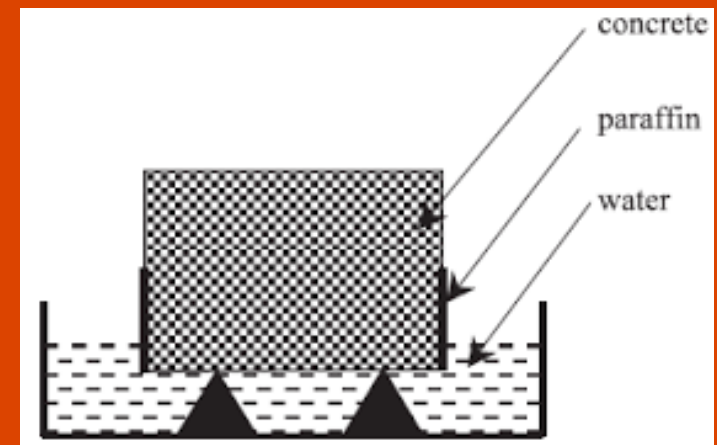
Similar trends and concentration ranges seen in literature

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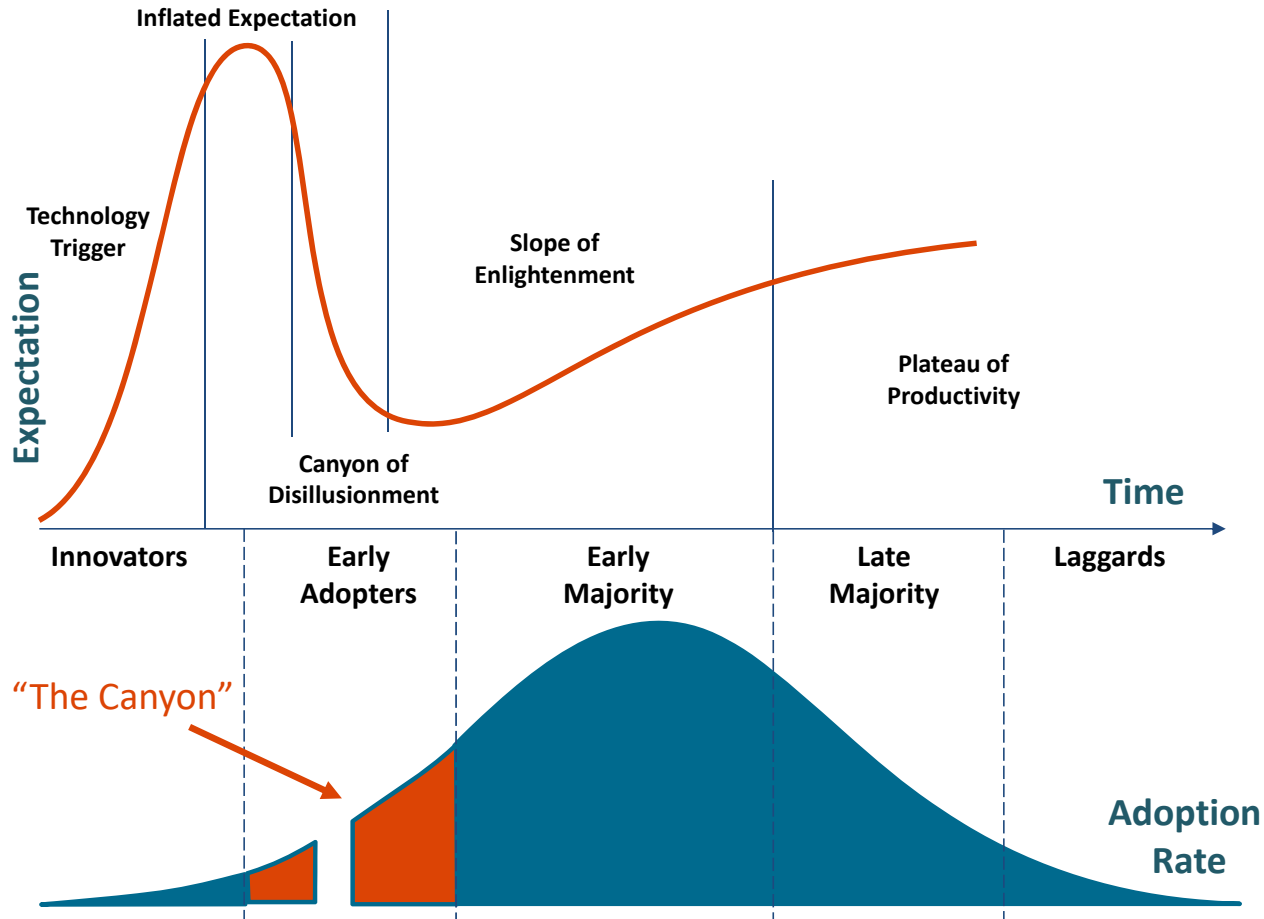




Research

Practice

Research and Adoption



- Hype Cycle
- Many times innovative research occurs but stalls at the 'canyon'
- How do we make it past this gap or minimize this gap



R2D2

© pixeltale



- 5 Gal Bucket
- Reliable
- Robust
- Simple
- Safe

R2S2

© pixeltale

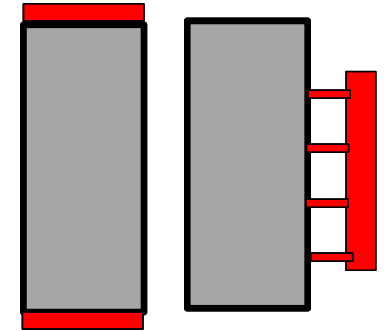
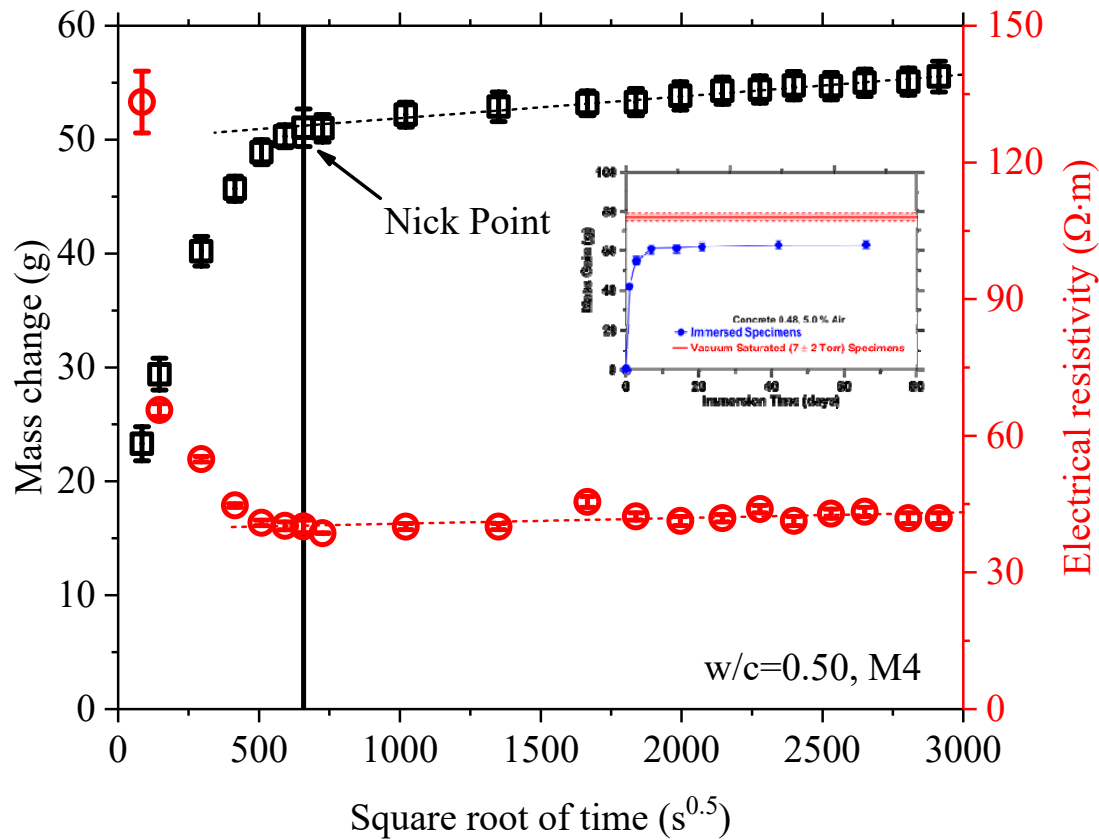
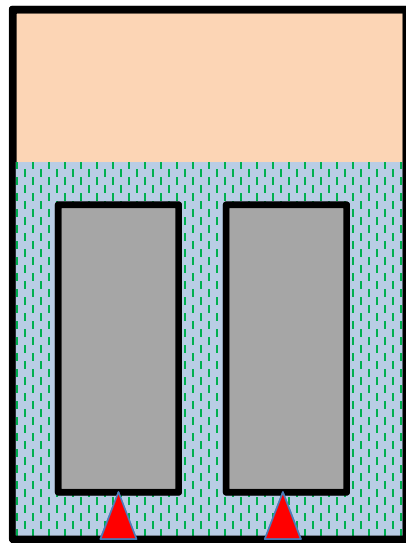
Current State

- There are 'three geometries' frequently used
- With proper geometry conditioning, all are similar



- There are two AASHTO test standards; while good a large issue in repeatability is related to conditioning
- Temperature, Moisture, Leaching, Degree of Saturation

"Bucket Test"



Conclusions



Oregon State University
College of Engineering

- Formation Factor – Measures Porosity & Connectivity
- F Factor – Directly Related to Resistivity and RCPT
- F Factor – Extended to Apparently Diffusion Coefficients
- F Factor – Extended and Fluid Absorption
- PB and GEMS Models for Pore Structure and Volume
- Importance of Computing Degree of Saturation
- Practical Impacts of Pore Solution & Immersion Testing
- Being Extended into Practice Through AASTHO PP-84

An aerial photograph of a university campus, likely Oregon State University, showing a dense collection of red brick buildings interspersed with green and yellow trees. In the background, a range of blue mountains is visible under a clear sky. The text "Thank You" is overlaid in the upper left quadrant.

Thank You

Conclusion



Recommended Tests



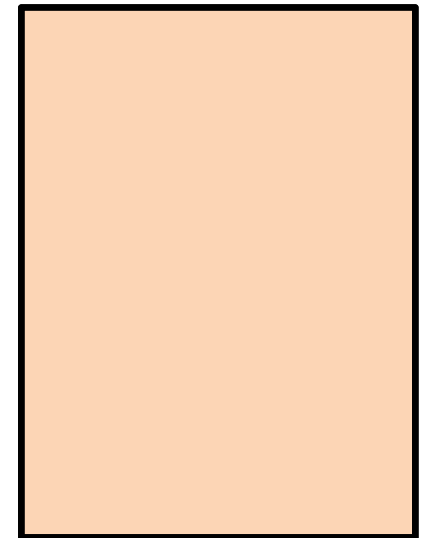
- Option 1 - “The Bucket Test”
 - Option 2 - Sealed Samples
 - Option 3 – Vacuum Saturation
 - ~~Option 4 – Moist Curing Room~~
-
- We will start by describing how to perform the tests using simplified procedures

Option 1 “Bucket Test”



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College of Engineering

- Begin with a 5 gallon bucket

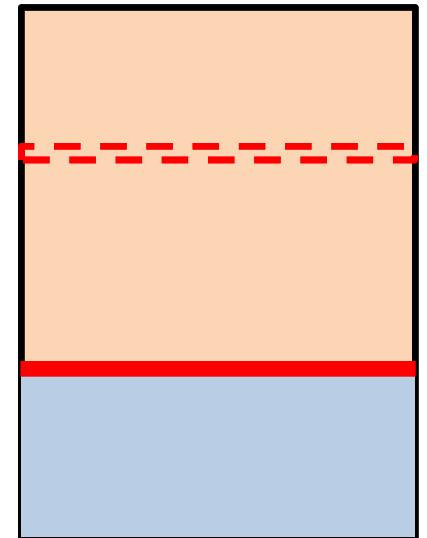


Option 1 “Bucket Test”



Oregon State University
College of Engineering

- Begin with a 5 gallon bucket
- Place a specific volume of fluid into the bucket (the solution to sample ratio is important, place a line on the bucket)

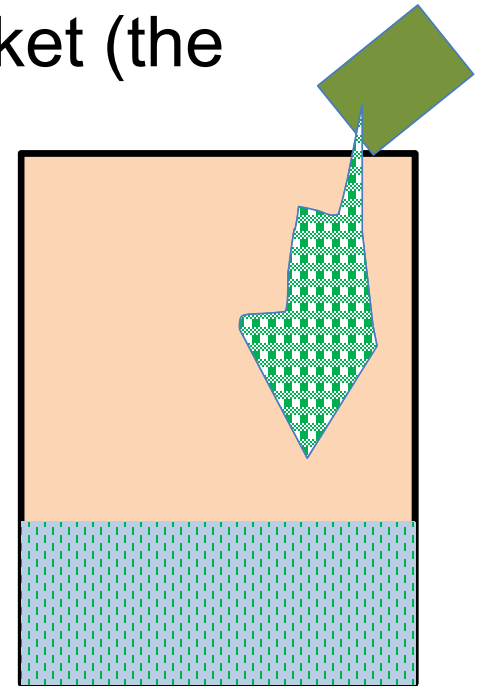


Option 1 “Bucket Test”



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College of Engineering

- Begin with a 5 gallon bucket
- Place a specific volume of fluid into the bucket (the solution to sample ratio is important, place a line on the bucket)
- Place a specified “CH-salt” into the solution (some adjust to the mixture, we suggest selecting a standard value)

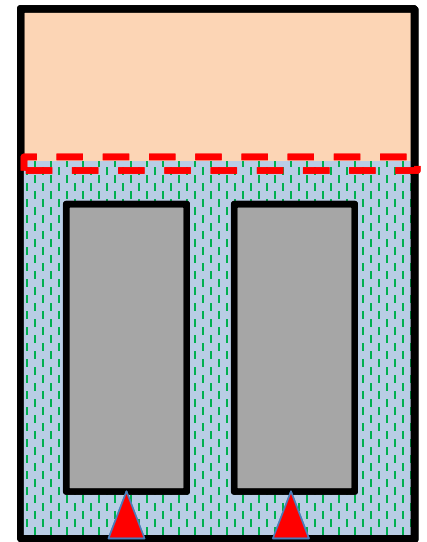


Option 1 “Bucket Test”



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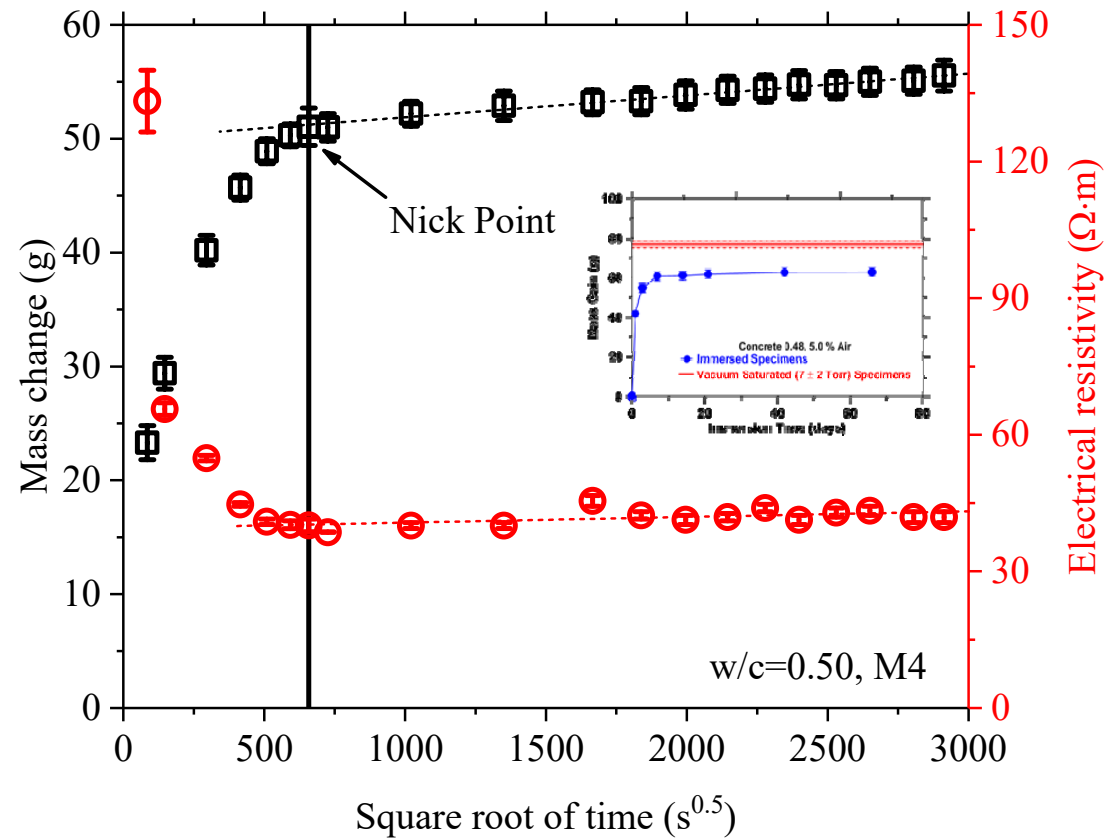
- Begin with a 5 gallon bucket
- Place a specific volume of fluid into the bucket (the solution to sample ratio is important, place a line on the bucket)
- Place a specified “CH-salt” into the solution (some adjust to the mixture, we suggest selecting a standard value)
- Place samples into the solution to allow the solution to reach the entire sample



Option 1 "Bucket Test"



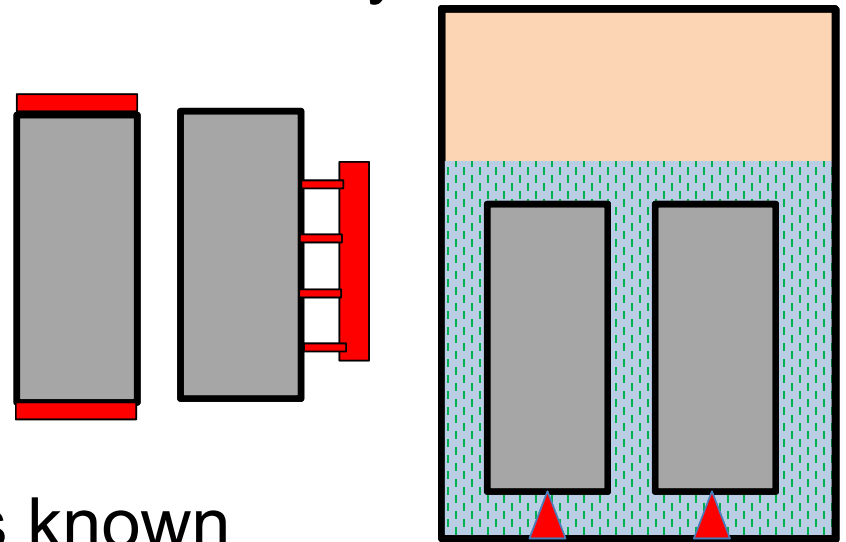
- Mass change and resistivity are shown
- Similar to absorption (like in ASTM C1585); however the time scale varies due to:
 - 1-sided vs immersion
 - sample geometry
- Approx. 5 vs. 1 days



Option 1 "Bucket Test"

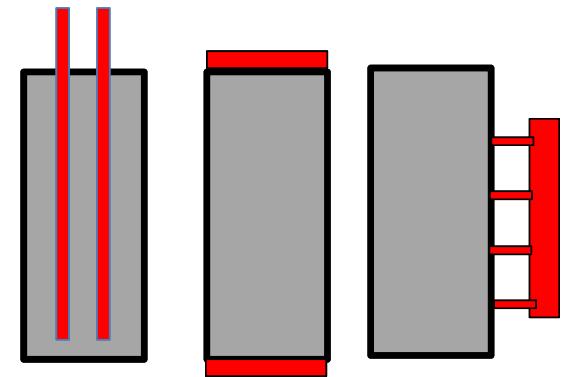


- At select ages remove sample from the bucket, towel/wash off the surface and perform either the surface resistivity test or the uniaxial bulk resistivity test
- After 5 days in solution the sample is assumed to be in matrix saturation
- This can provide a measure of ρ_{measured} or F_{matrix}
- Conditioning solution ρ_{solution} is known



Option 2 - Sealed

- Maintain a sealed sample (this can be done in a sealed sample or by placing the sample in double bags)
- The advantages of this approach:
 - Provides continual measure
 - Provides an “easy” test condition
- The disadvantages of this approach:
 - Will require a moisture correction
 - Requires some sort of ability to make it clear as to whether the sample has dried

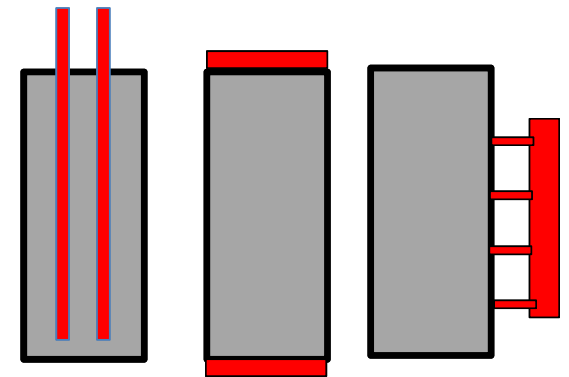


Option 3 – Vacuum Sat.

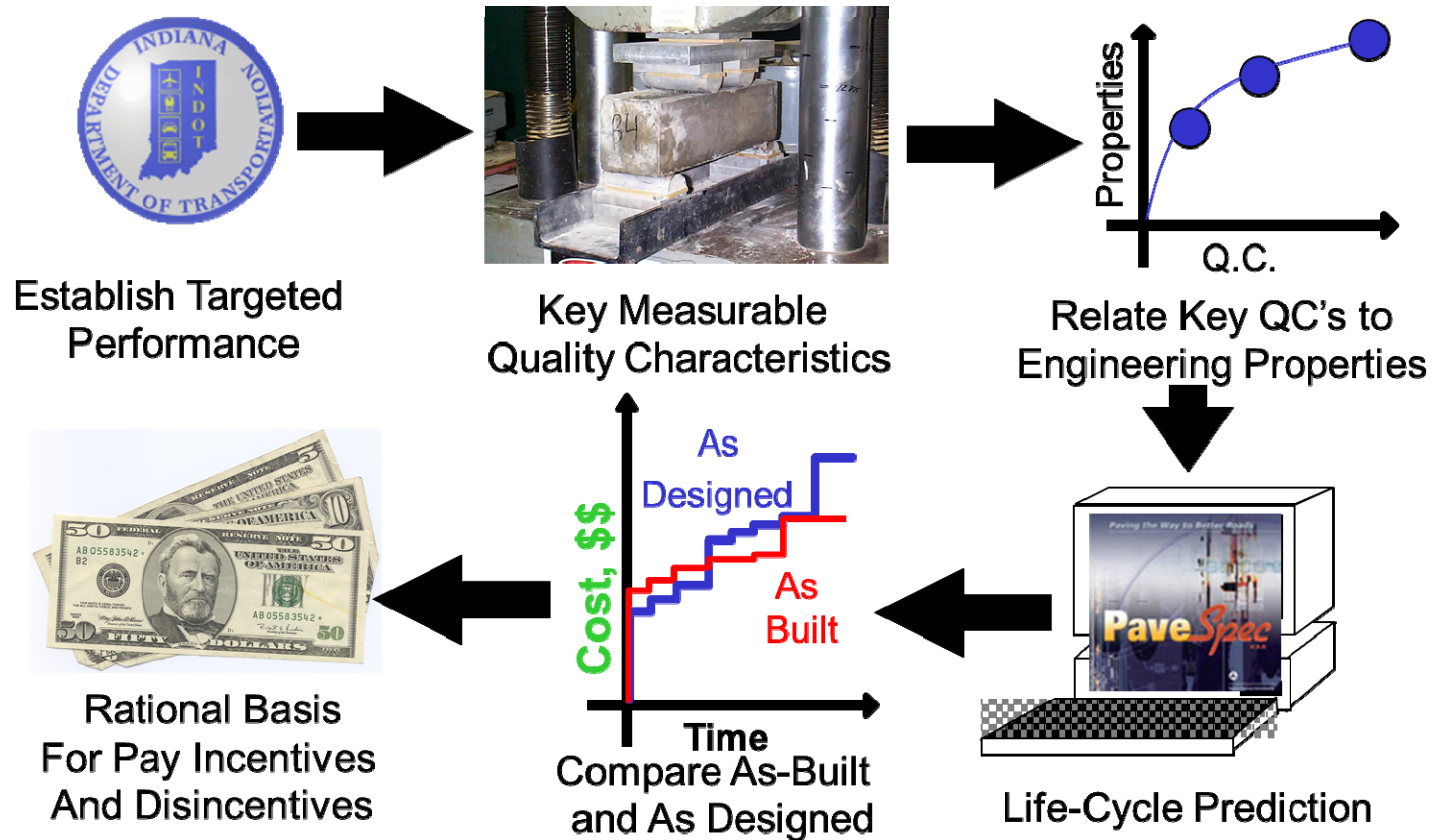


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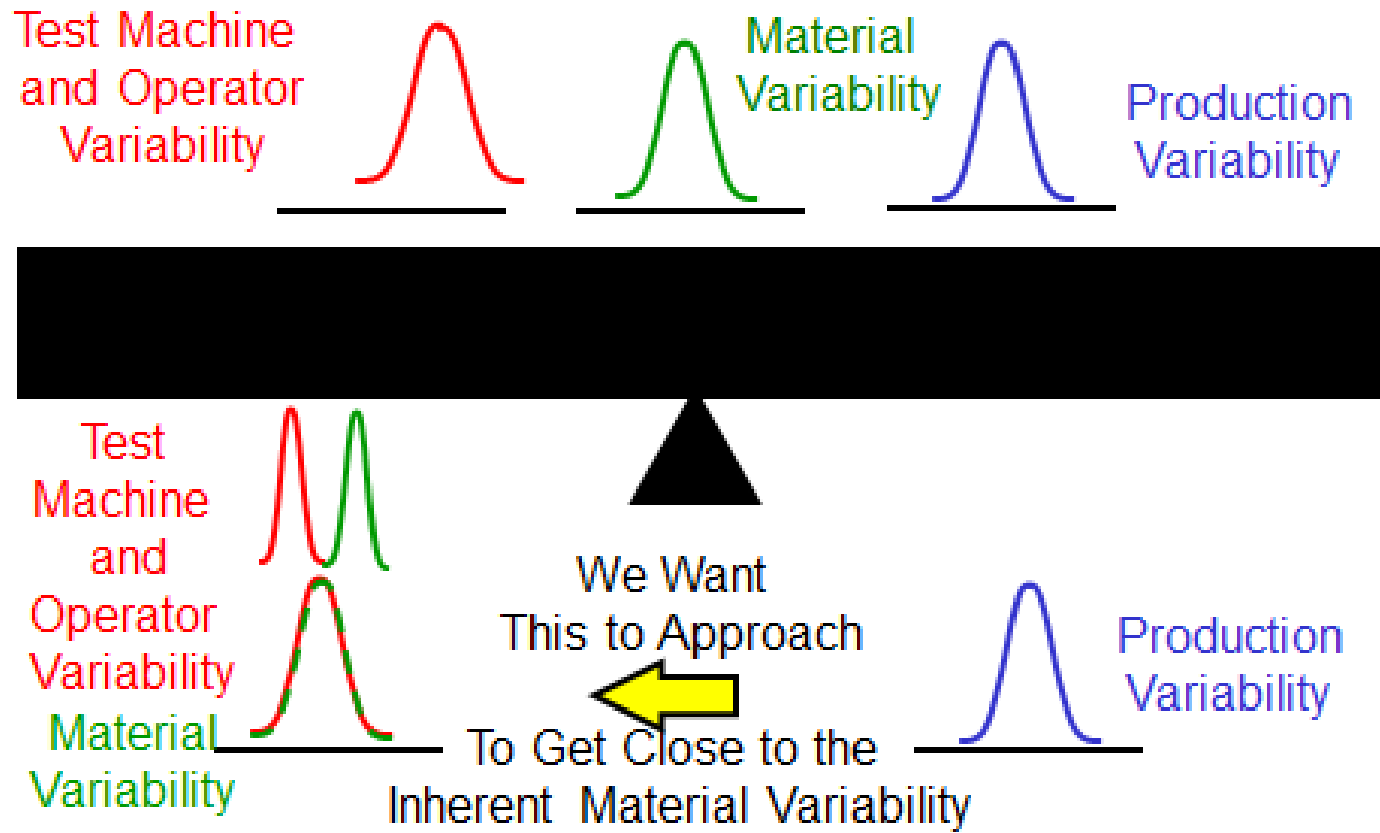
- This is not being recommended but its historical
- The advantages of this approach are:
 - This will match most closely to ASTM C1202
- The disadvantages of this approach are:
 - time consuming
 - difficult to do on 4x8 in samples



Performance Spec



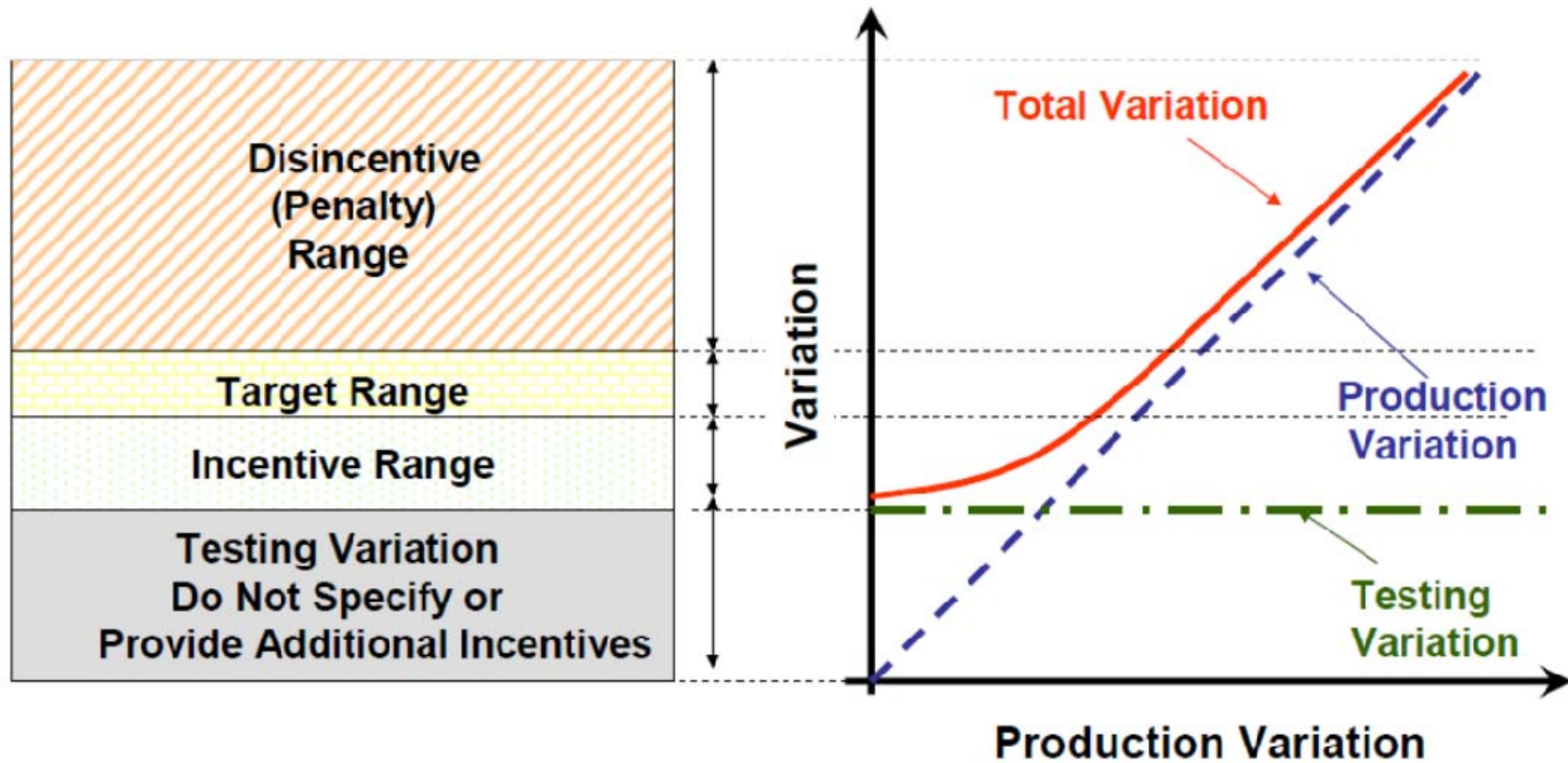
Sources of Variation



Setting Variability Limits



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Pellinen et al., 2005