Application of Microbial Induced Calcite Precipitation (MICP) to Stabilize Florida High-Organic Matter Soils for Roadway Construction

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Motivation and Objectives

- High organic content (OC) soil needs to be stabilized and treated to mitigate settlement

- Previous studies/attempts are either ineffective, prohibitively expensive, environmentally harmful, and/or not sustainable

- Goal: Determine Microbial Induced Calcite Precipitation (MICP) feasibility as an environmentally-friendly and sustainable method for treating Florida's OC soil for roadway construction.

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MICP Governing Chemical Reactions

- $CO(NH_2)_2 + 2H_2O \rightarrow 2NH_3 + H_2CO_3$ (Urea Lysis)
- $NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-$ (increasing pH)
- $H_2CO_3 + 2OH^- \leftrightarrow HCO_3^- + H_2O + OH^-$ (Carbonic acid to bicarbonate)
- $HCO_3^- + H_2O + OH^- \leftrightarrow CO_3^{2-} + 2H_2O$ (Bicarbonate to carbonate)
- $Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3$ (s) (Calcium carbonate precipitation)
- $Ca^{2+} + 2HCO_3^- \leftrightarrow CaCO_3\ (s) + CO_2\ (g) + H_2O$ (More calcium carbonate precipitation)

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Treatment Constituents

- Ureolytic bacteria – *Sporosarcina pasteurii*
- Calcium chloride solution
- Urea

Soil Mixing Versus Percolation

Soil Percolation Setup

Typical Percolation Results in 50/70 Ottawa Sand

Typical Mixing Result in 50/70 Ottawa Sand

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Percolation Results in Organics

50% OC, Post-Treatment, Wet

50% OC, Post-Treatment, Dry

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Treatment Procedure – Soil Mixing Method

1. Soil with an OC of approximately 50% was obtained from a natural soil deposit near SR-33 in Polk County, FL. This material was dried, homogenized, and sieved.

2. 50/70 Ottawa sand (quartz) was added to the 50% OC soil to yield three soil batches with OCs of 10%, 30%, and 50%.

3. The soil was pluviated into 2-inch by 4-inch cylinder molds until the molds were approximately 75% full.

Treatment Procedure – Soil Mixing Method (Continued)

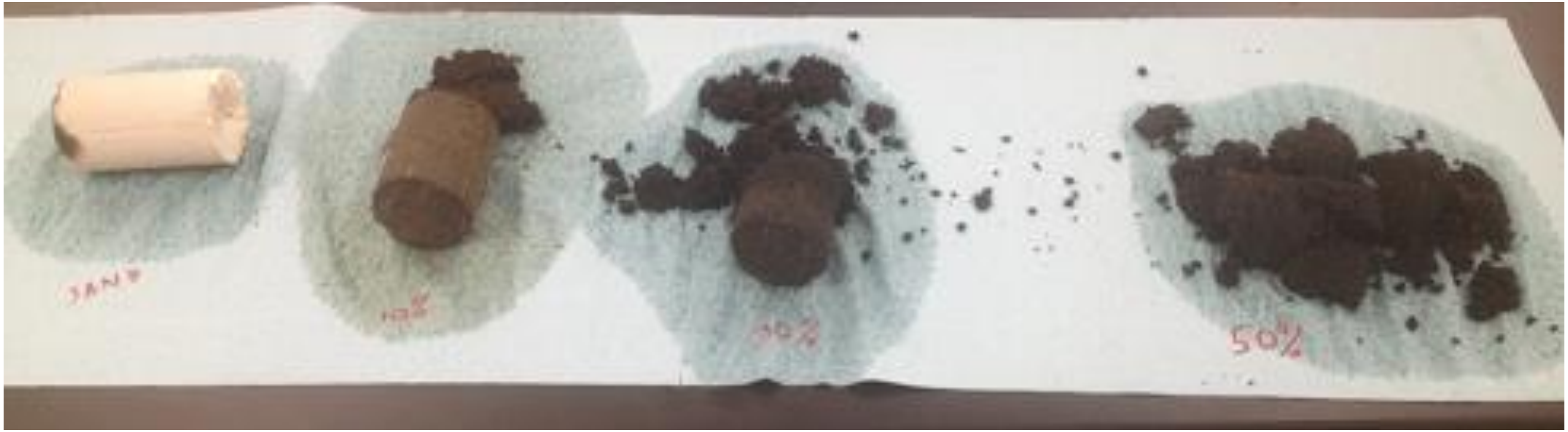
4. A bacterial solution of *Sporosarcina* cultured to an optical density (OD) greater than 2.0 was added to the soil and hand mixed using a spatula.

5. A 2.5M urea/2.5M calcium chloride solution was added to the soil/bacterial mixture. The urea/calcium chloride/bacteria/soil was hand-mixed using a spatula.

6. The specimens were allowed to cure for a minimum for 48 hours.

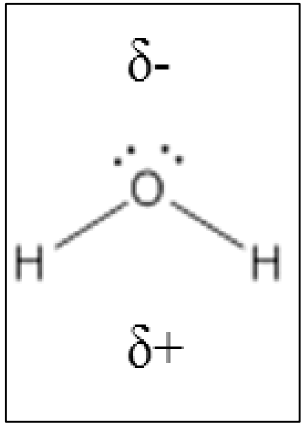
7. After curing, the molds were opened using a Dremel® tool and the specimens were extracted.

Initial Results – Soil Mixing Method

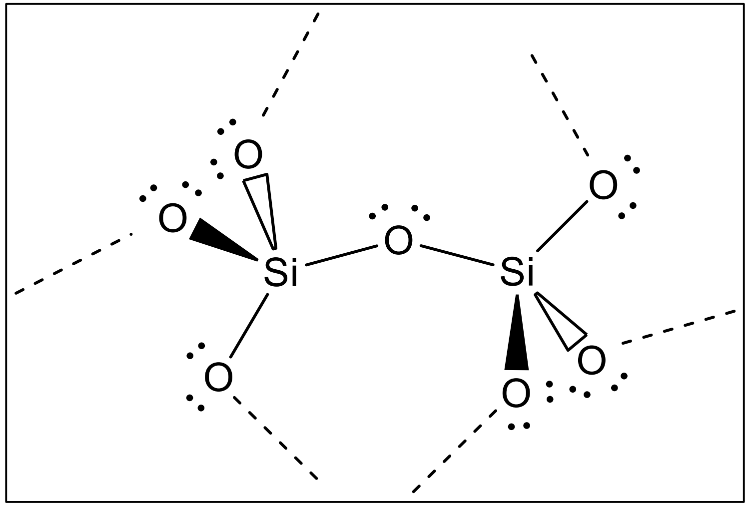


Ottawa Sand 10% OC Soil 30% OC Soil 50% OC Soil

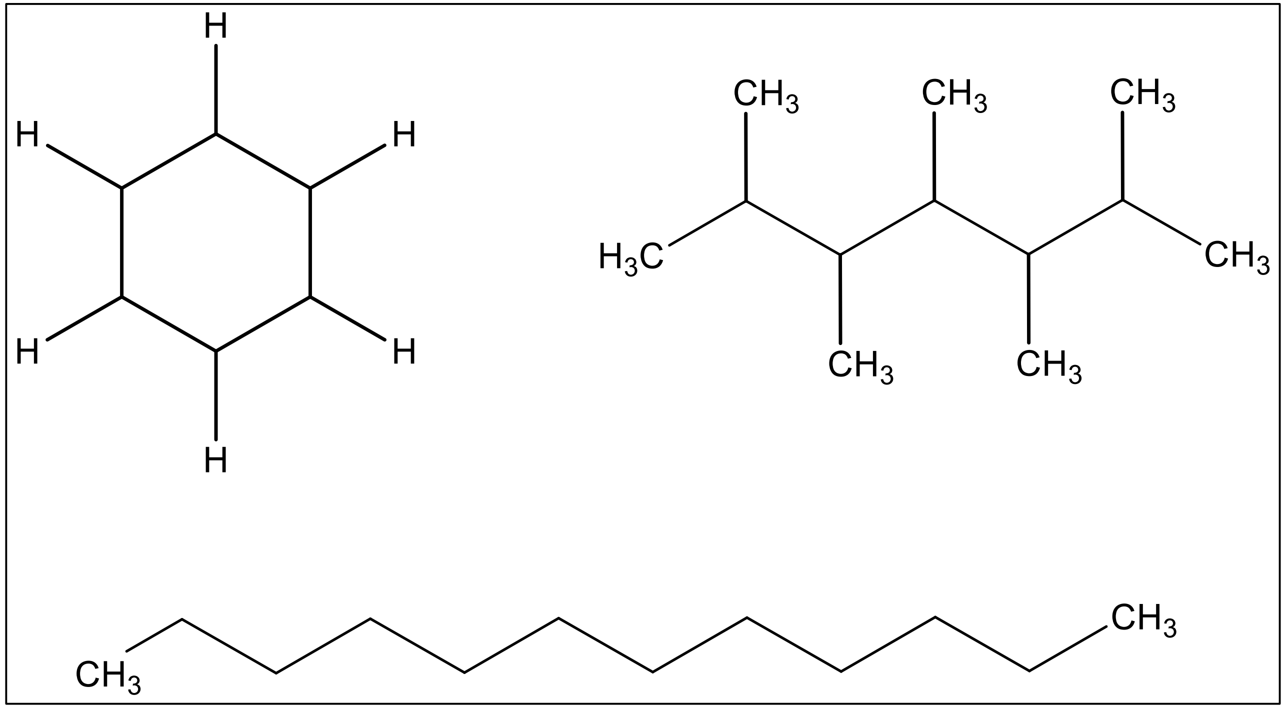
Some Geochemistry



Water Molecule



Silica Sand Molecule



Typical Organic Particle Molecules

Cementing High-OC Soils – Initial Thoughts

- OC soil is very hydrophobic
- Calcification difference between sands and OC soils may be an electrochemical charge issue; we know that $CaCO_3$ forms well on negatively charged surfaces such as metal and glass and poorly on neutral surfaces such as plastic
- Therefore – need to:
  - Wet the soil more efficiently
  - "Trick" microbes and/or calcium carbonate into "thinking" that OC soils are negatively charged
- **Use a surfactant – Sodium Dodecyl Sulfate (SDS)**

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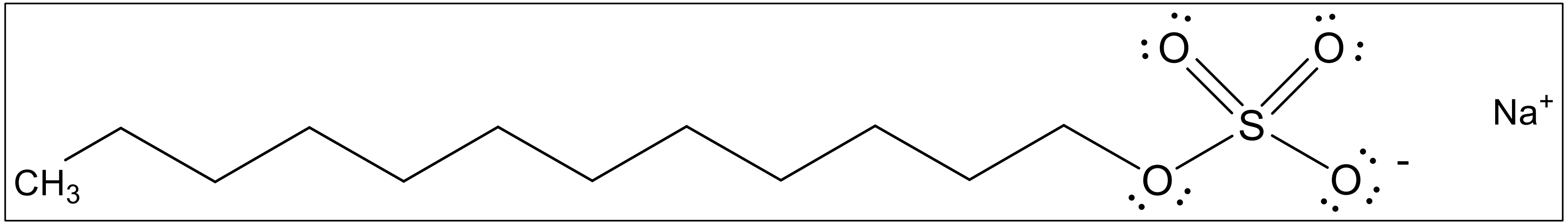
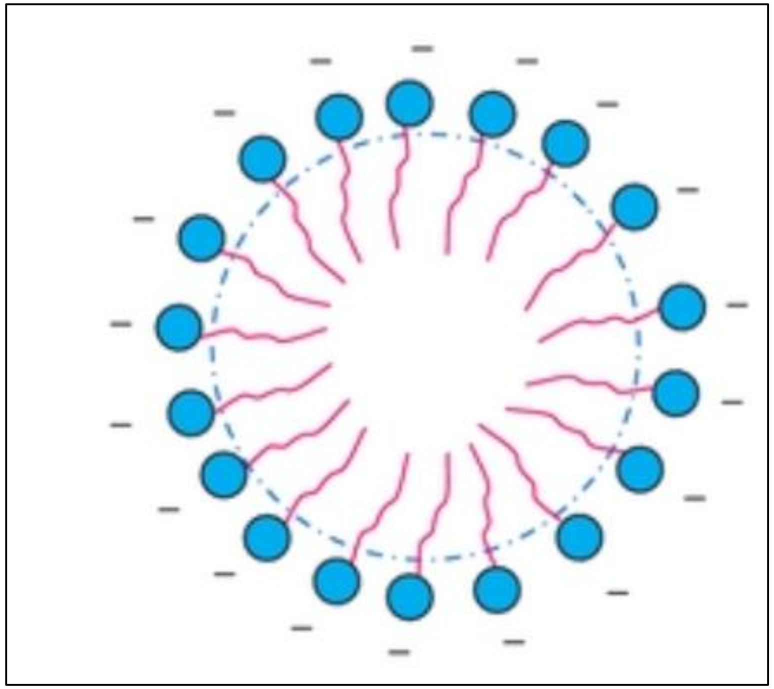
Sodium Dodecyl Sulfate



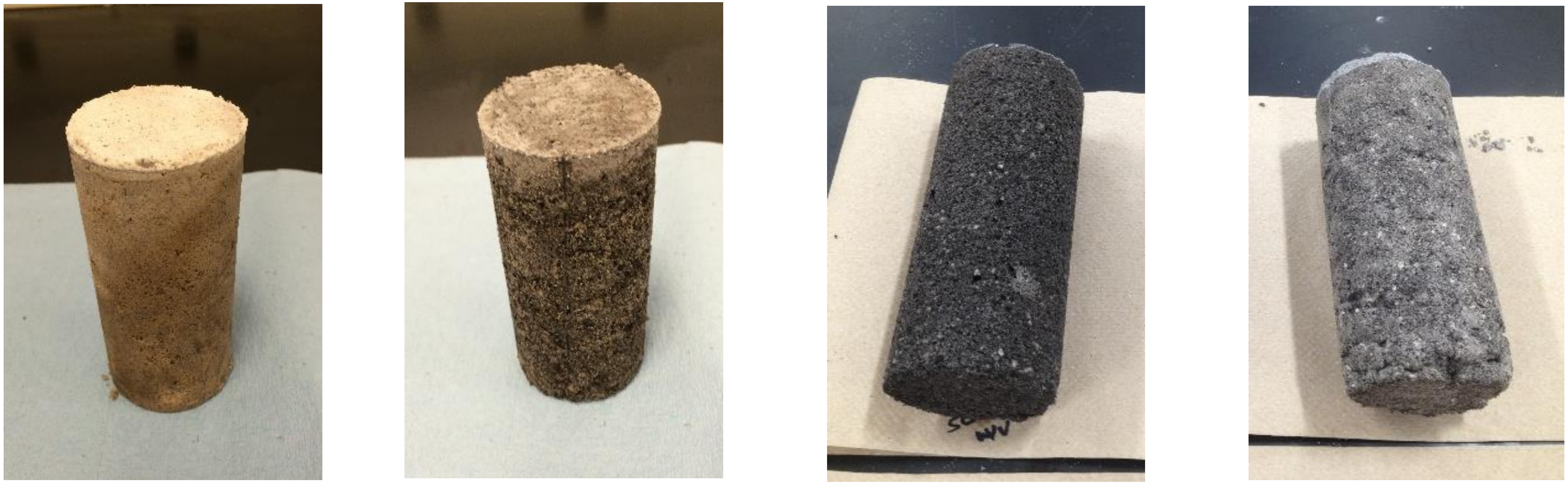


Top: SDS Chemical Structure

Bottom: Typical SDS Micelle; adapted from: Liu, R., Pu, W., Jia, H. Shang, X., Pan, Y., and Yan, Z. (2014). Rheological properties of hydrophobically associative copolymers prepared in a mixed micellar method based on methacryloxyethyl-dimethyl cetyl ammonium chloride as surfmer. *International Journal of Polymer Science,* 2014(17): 1-14.

New Treatment Procedure and Results

- Use same mixing procedure as before except add SDS in various percentages to soil before adding bacteria, urea, and $CaCl_2$



Fully Calcified OC Specimen Examples (post MICP treatment) Showing (a) 50/70 Ottawa Sand with 10% OC; (b) 50/70 Ottawa Sand with 30% OC; (c) 50% OC with 50% SDS; and (d) 50% OC with 80% SDS

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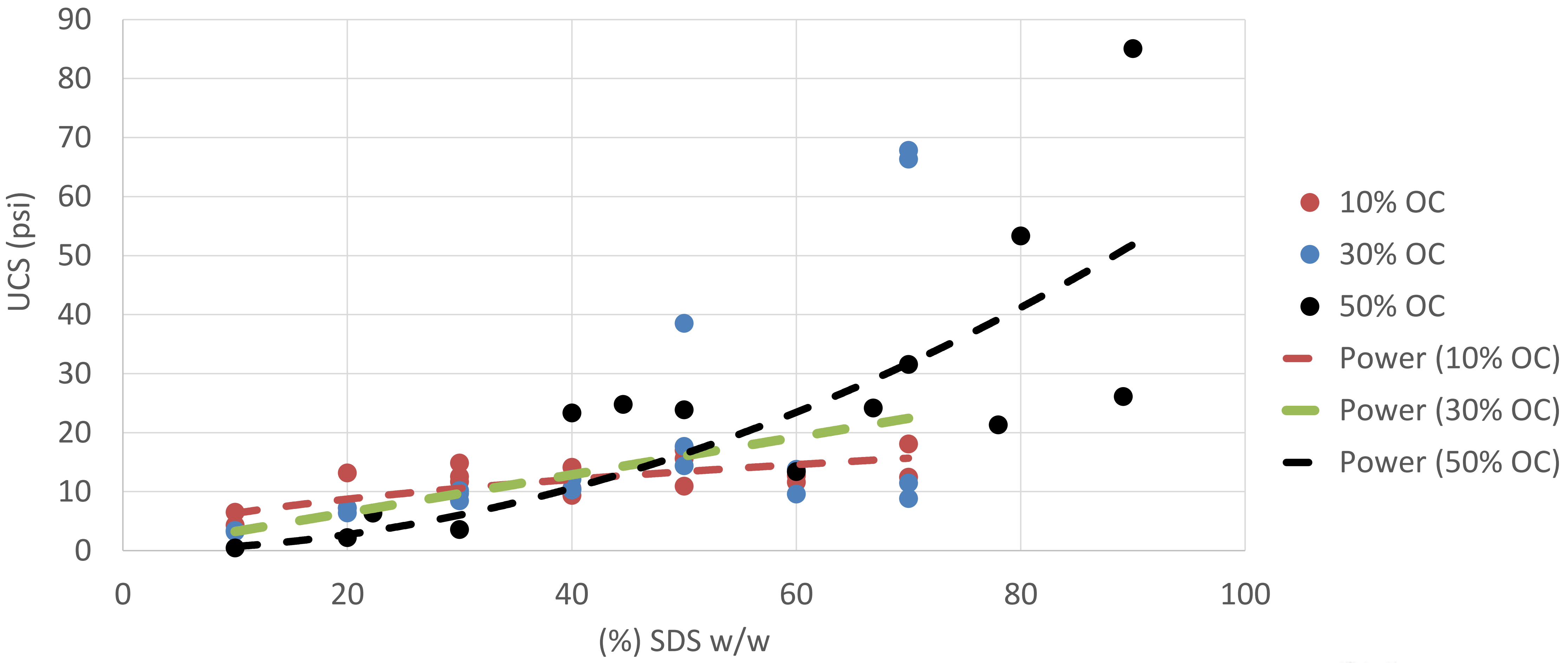
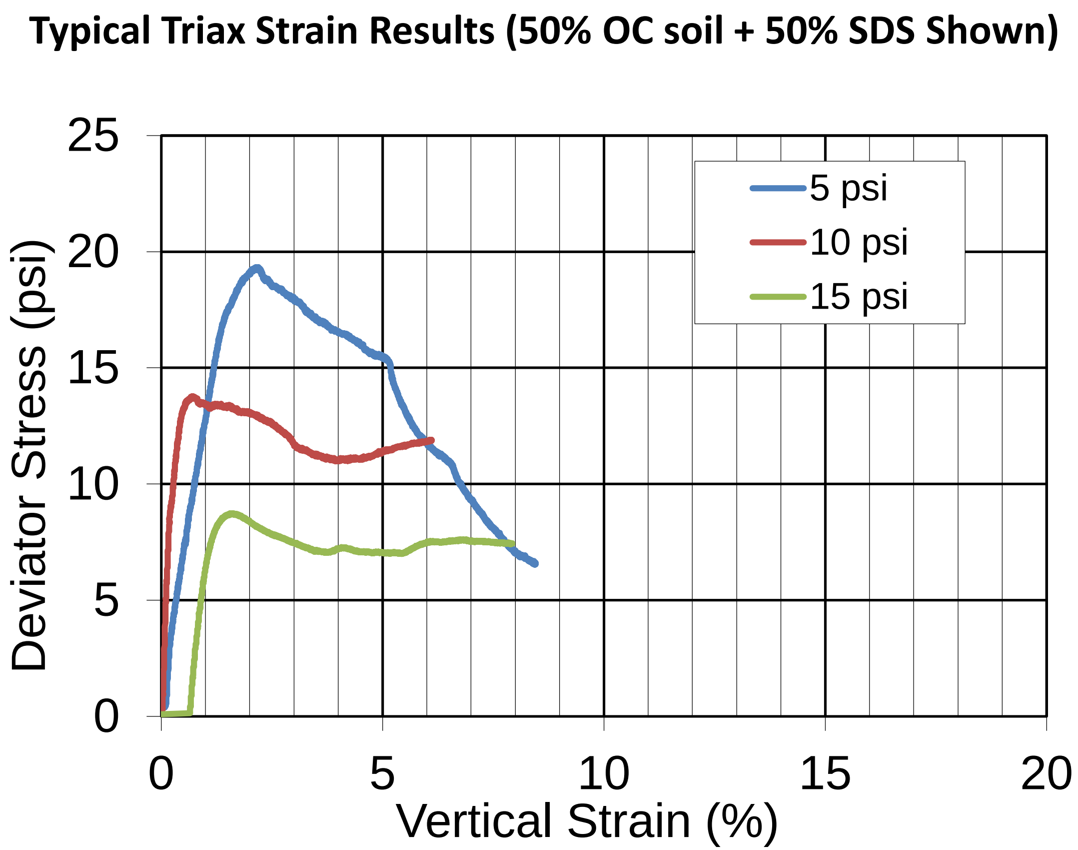
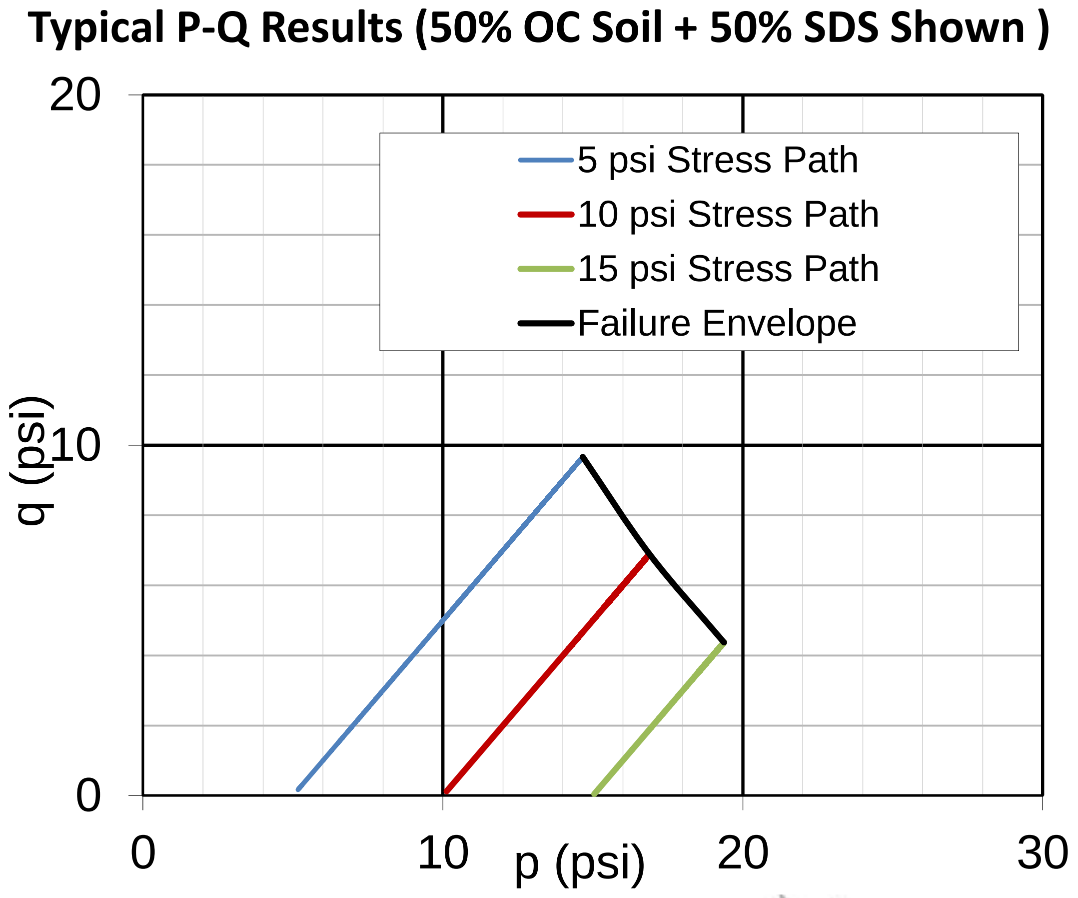
SDS + OC Soil UCS Results



Typical Traixial Test Results





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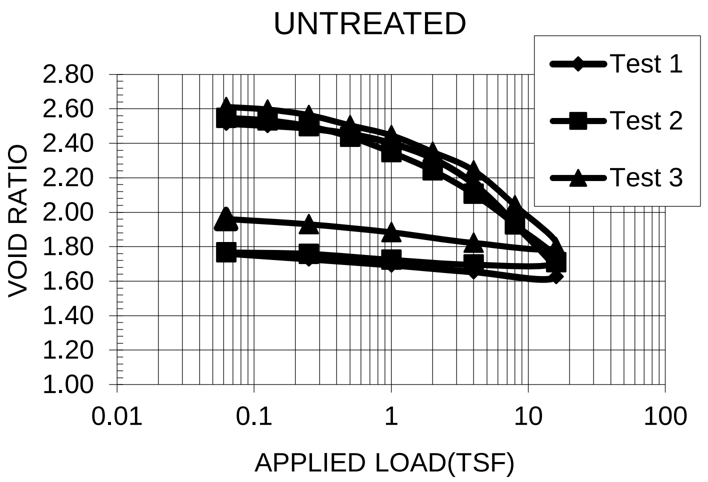
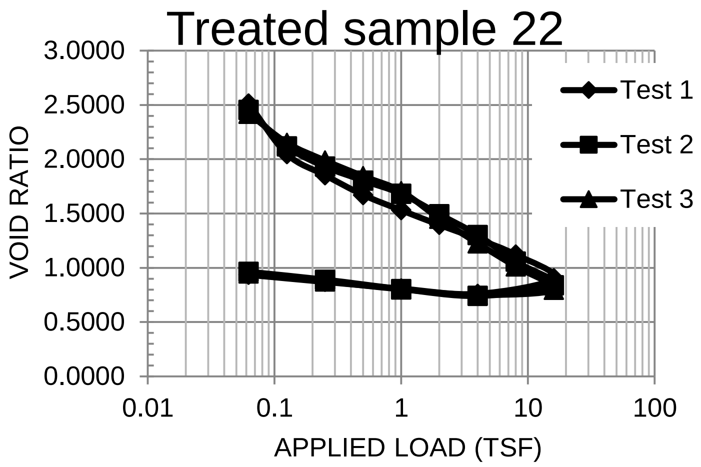
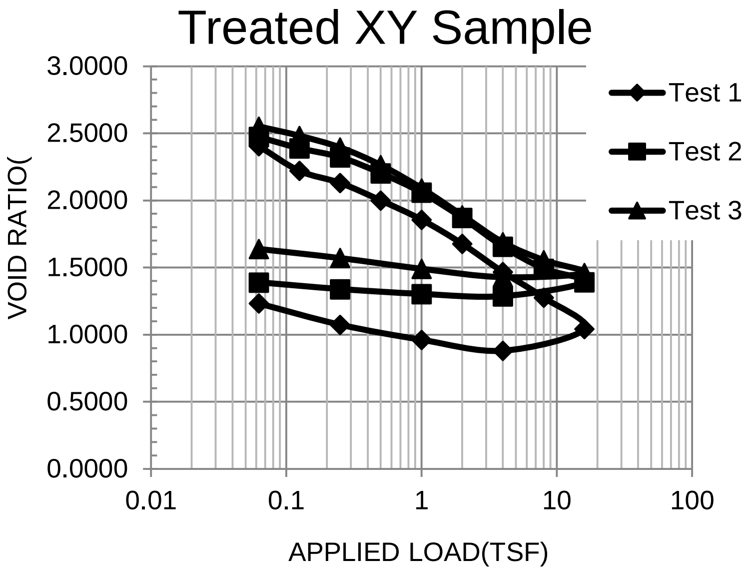
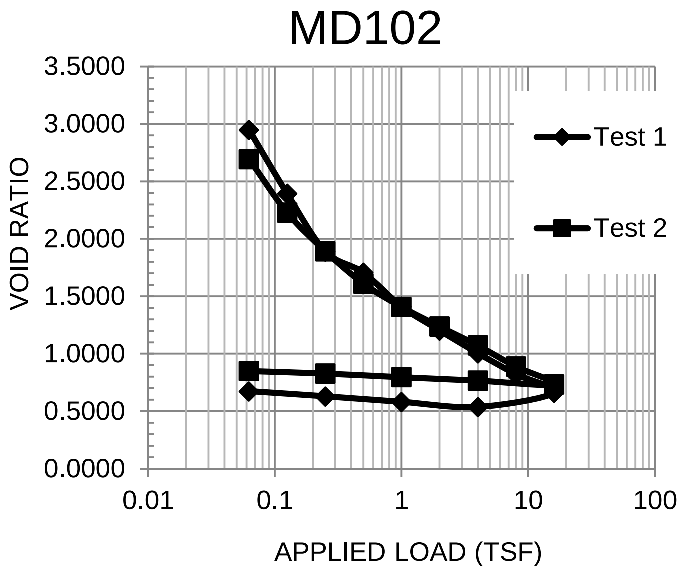
Typical Consolidation Test Results









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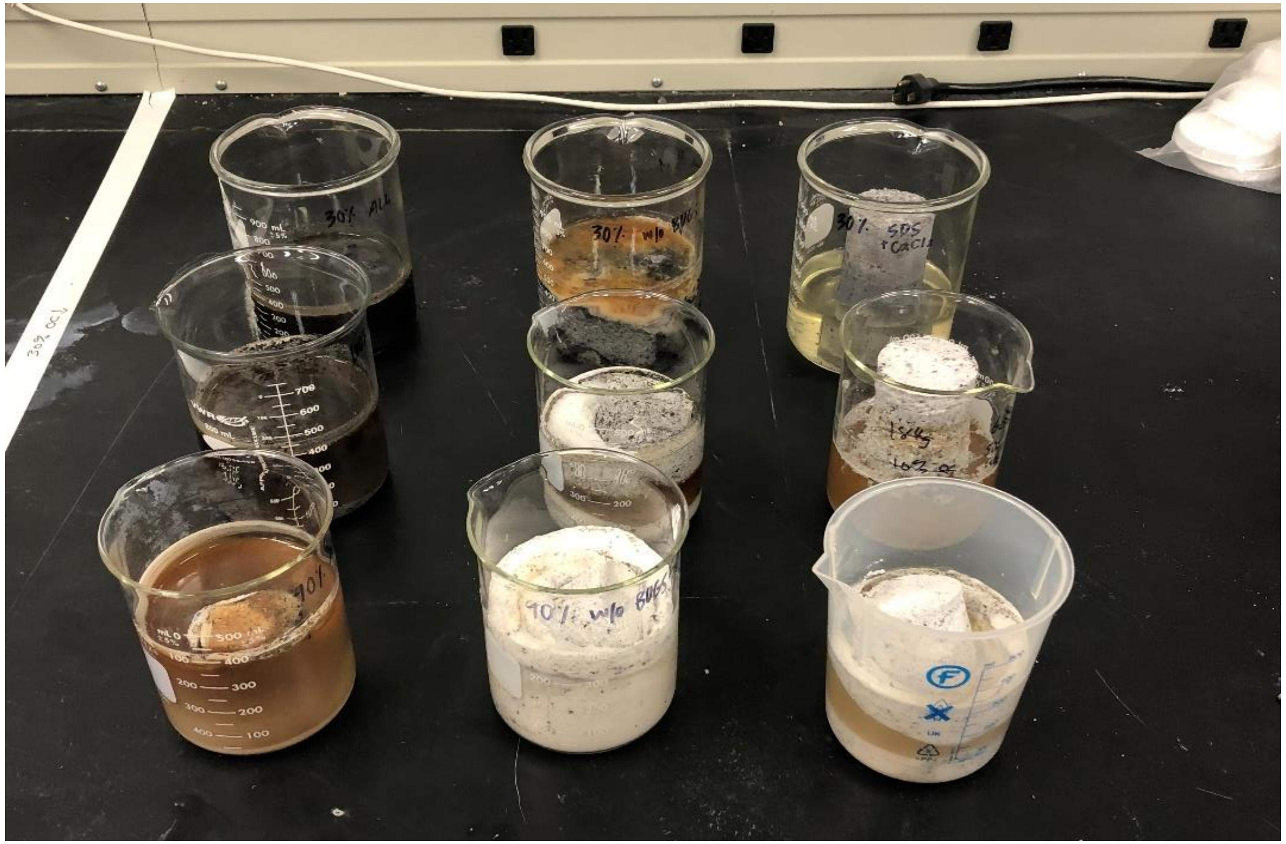
Dissolution Testing Results



Full Suite of Dissolution Tests. Far-left column are with microbes; middle column is without microbes; far-right column are without microbes and urea; top row is 30% SDS; middle row is 60% SDS; bottom row is 90% SDS

50% OC, 30% SDS, & 2.5M CaCl after 3 Weeks

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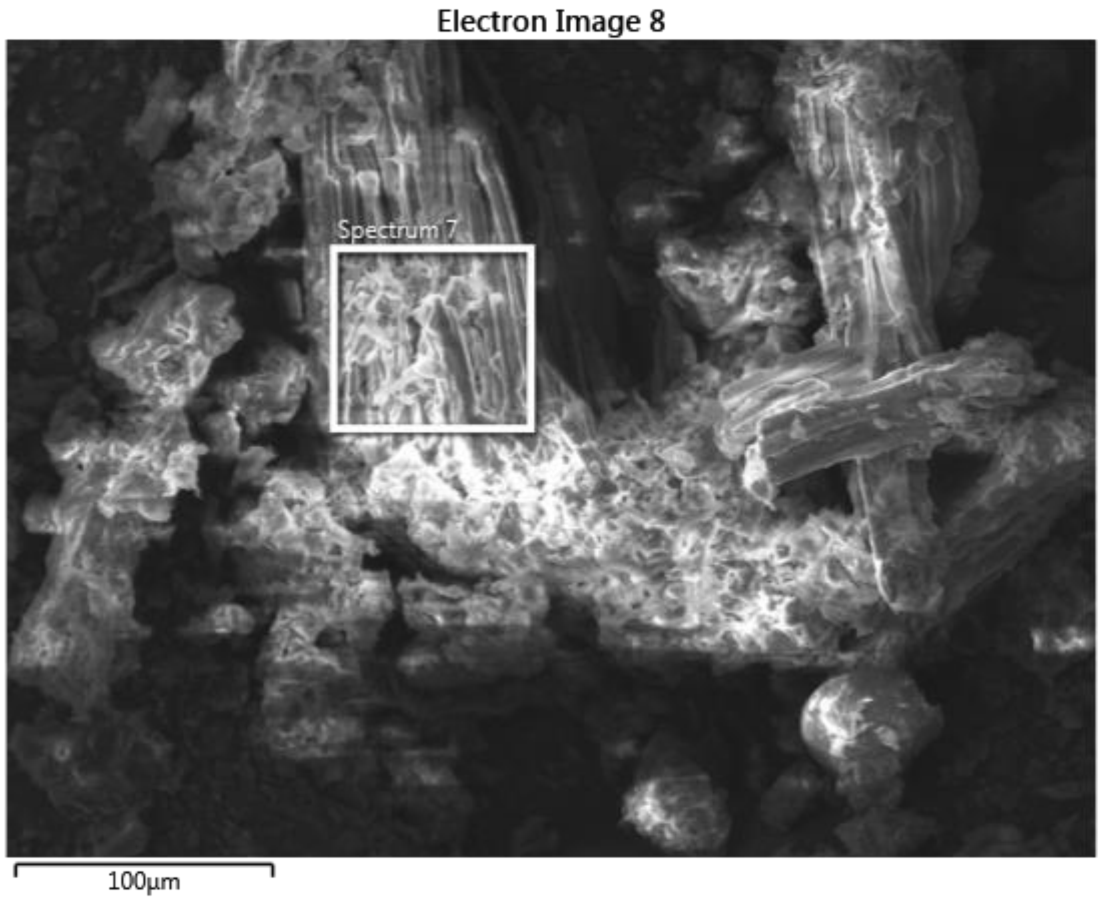
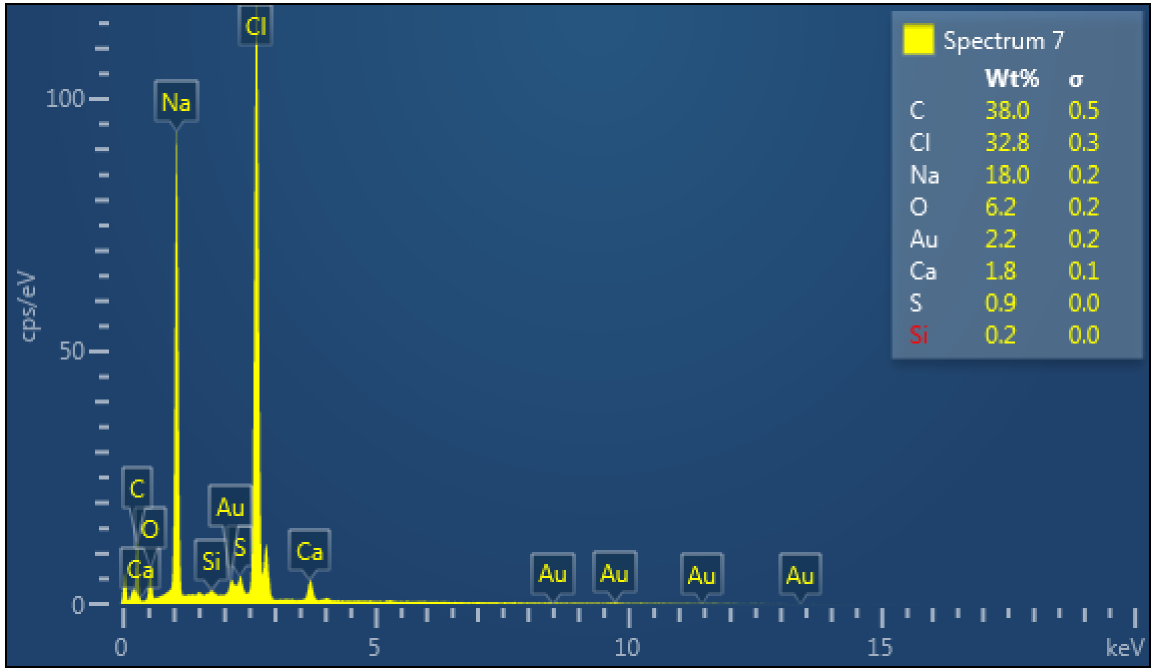
SEM/XRD Results

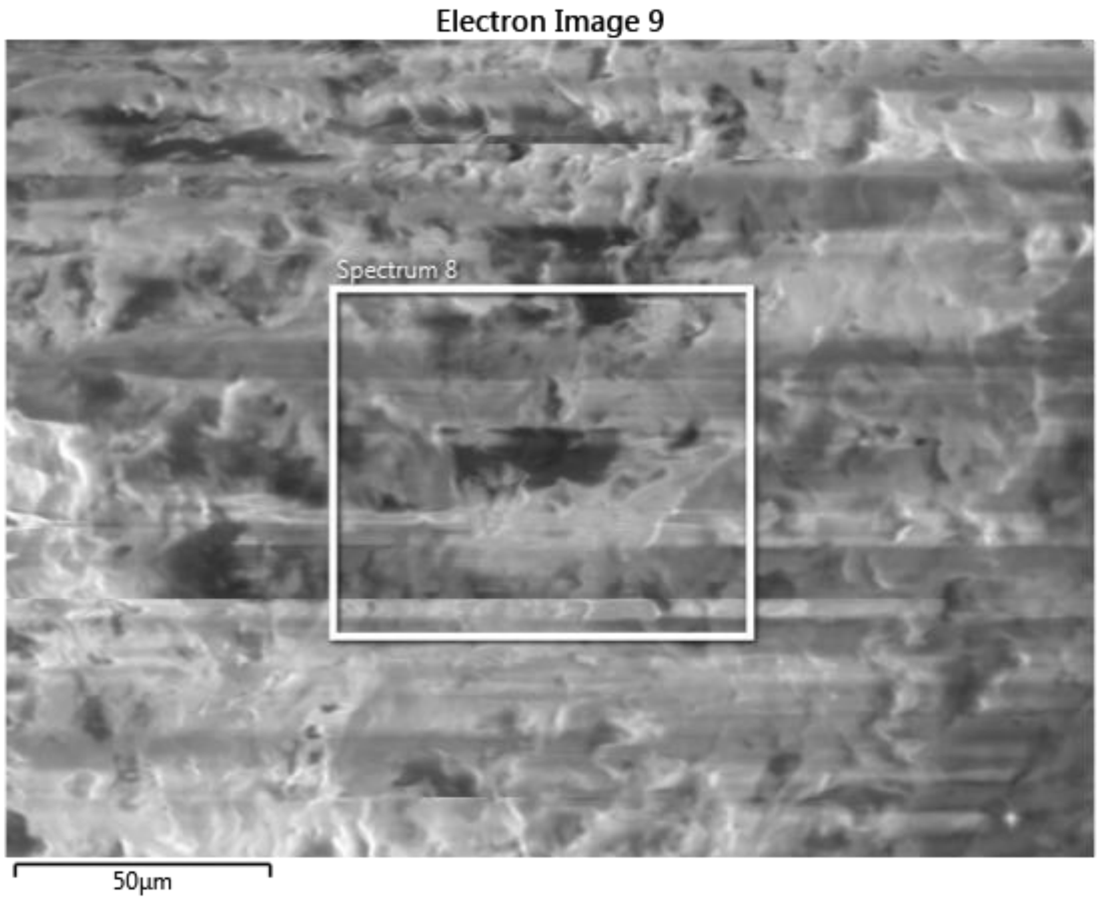


SEM Results from 30% SDS, no microbes, no urea

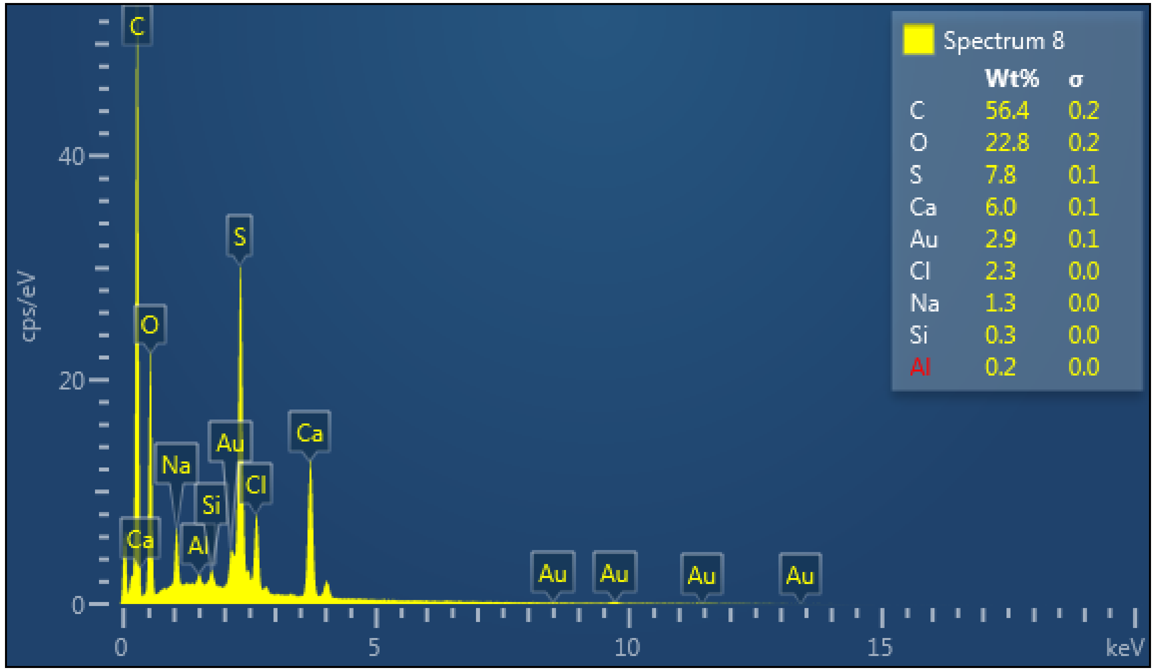


XRD Results from 30% SDS, no microbes, no urea

SEM/XRD Results



SEM Results from 30% SDS, no microbes, no urea



XRD Results from 30% SDS, no microbes, no urea

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New Thoughts on Geochemistry

- SDS = $NaC_{12}H_{25}SO_4$
- In water, $NaC_{12}H_{25}SO_4 \rightarrow Na^+ + (C_{12}H_{25}SO_4)^-$
- $Na^+ + Cl^- \rightarrow NaCl\ (s)$
- $Ca^{2+} + 2(C_{12}H_{25}SO_4)^- \rightarrow (C_{12}H_{25}SO_4) - Ca - (C_{12}H_{25}SO_4)$
- Calcium needed for $CaCO_3$ may be getting “locked” into “grime” matrix

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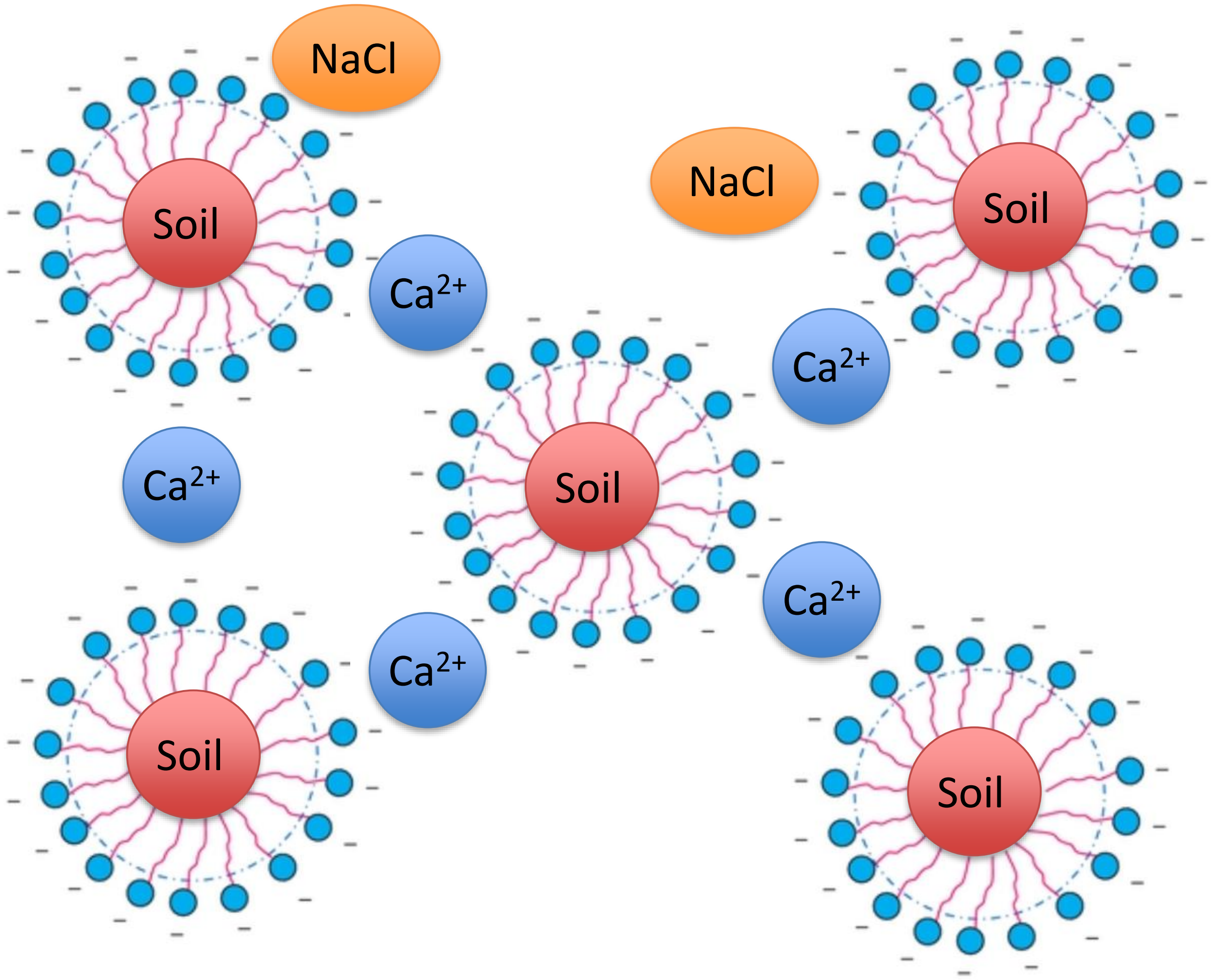
Results Explanation



What about MICP?

- 2.5M $CaCl_2$ is approximately the limit of dissolved $CaCl_2$ at typical Florida temperatures

- Therefore, flood system with $MgCl_2$

- $MgCl_2$ is more reactive than $CaCl_2$

- $MgCO_3$ is an order of magnitude more dissolvable than $CaCO_3$

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What about MICP?

- Data indicate that *s. pasteurii* does not act as a nucleation site for calcite precipitation.

- Rather, calcite may precipitate instead on exopolysaccharides (EPS).

- In failed MICP treatments, EPS was not observed; maybe in organics, EPS does not form for some TBD biological reason.

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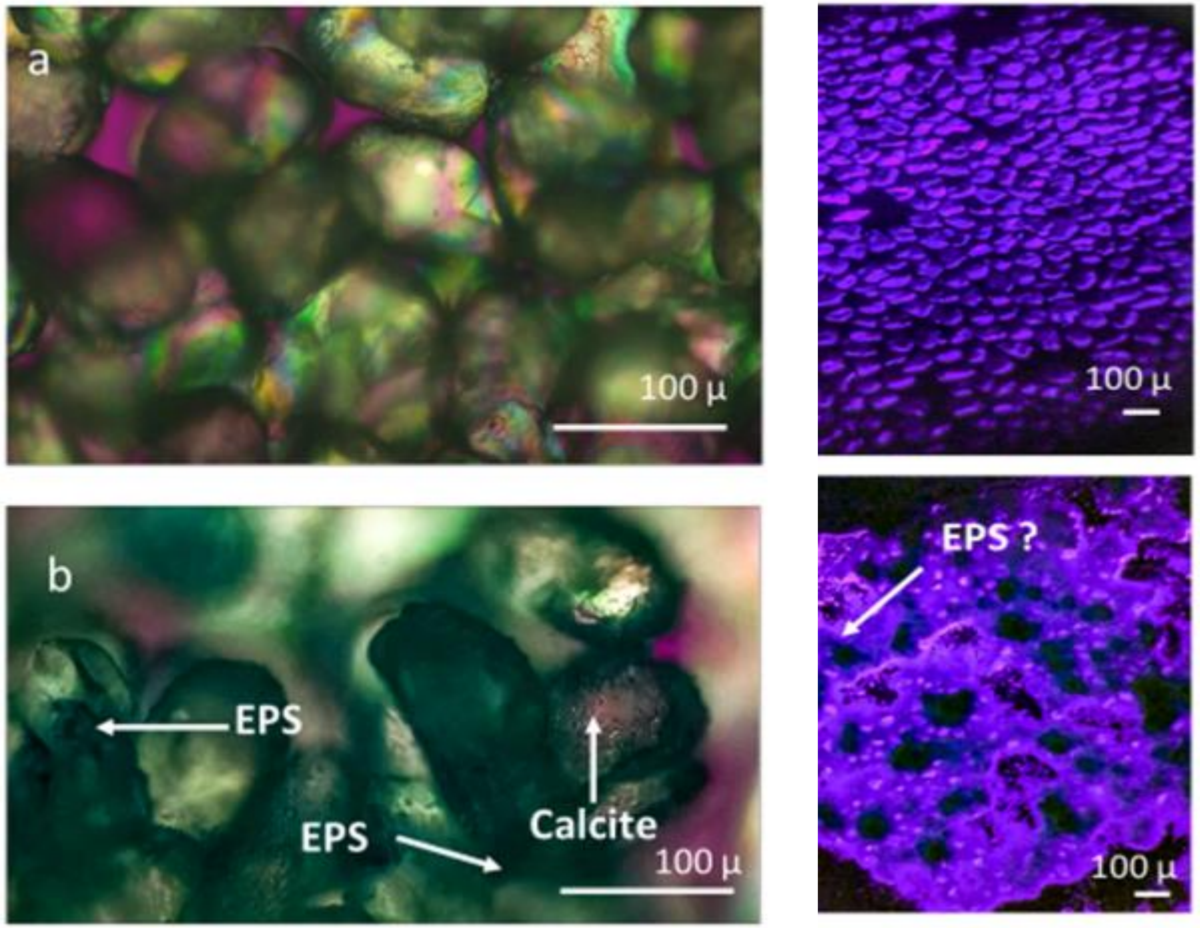
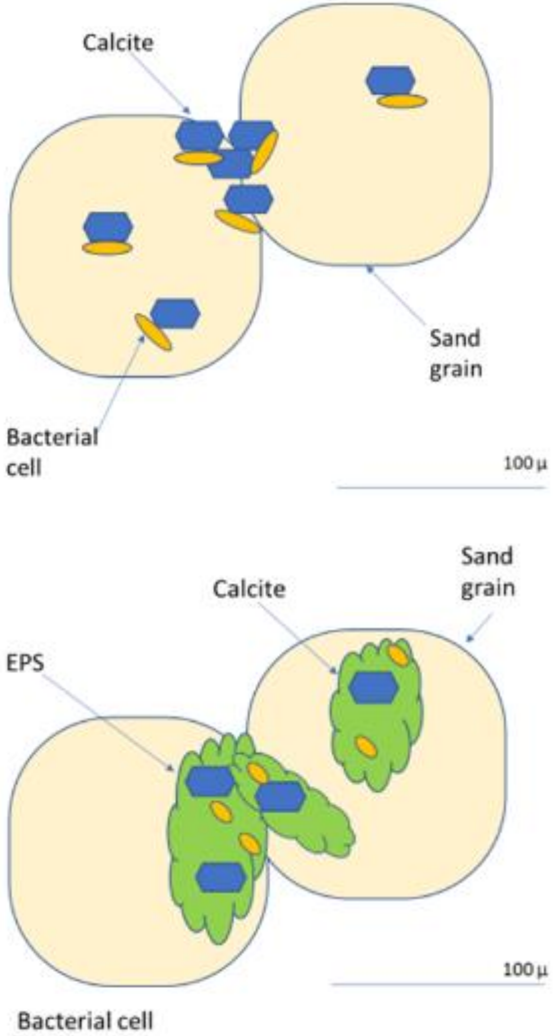
Exopolysaccharides (EPS)



Alcian blue stained MICP specimens showing untreated sand (top) and treated sand (bottom); left is standard lighting; right is under UV light; adapted from Ford 2018



Traditional MICP model (top) and MICP model with EPS (bottom); adapted from Ford 2018

Possible New MICP Governing Reactions

- $Mg^{2+} + 2(C_{12}H_{25}SO_4)^- \rightarrow (C_{12}H_{25}SO_4) - Mg - (C_{12}H_{25}SO_4)$

- $Ca^{2+} + 2(C_{12}H_{25}SO_4)^- \rightarrow (C_{12}H_{25}SO_4) - Ca - (C_{12}H_{25}SO_4)$

- $Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3\ (s)$

- $Ca^{2+} + 2HCO_3^- \leftrightarrow CaCO_3\ (s) + CO_2\ (g) + H_2O$

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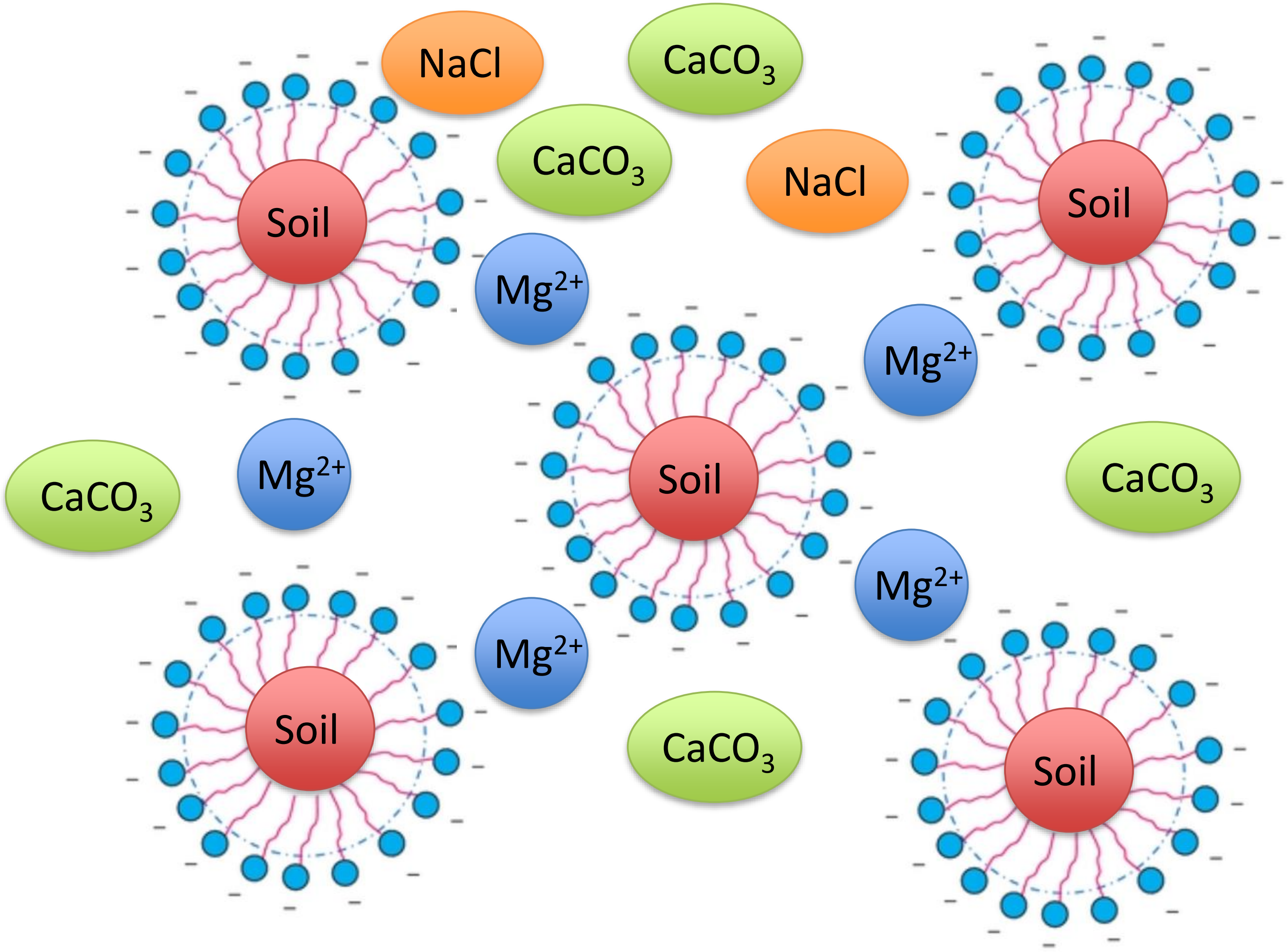
Hypothesized New Structure



Ongoing Testing

- 100% SDS + calcium chloride yielded stiff column that cured underwater
- Using magnesium chloride resulted in similar results
- Optimization ongoing when OC soil added to the mixture
- Testing whether or not MICP actually helps the “grime” matrix
- Native microbe testing – without (and maybe with) “grime”

Thank you!