ROLE OF SULFATES ON HIGHWAY HEAVING IN LAKE COUNTY, OHIO

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OUTLINE OF PRESENTATION

• Statement of Problem
• Introduction
• Objectives
  • Objective I. Characterizing Boring Samples
  • Objective II. Synthesizing Ettringite
  • Objective III. Swell Testing
• Conclusions/Recommendations
STATEMENT OF PROBLEM

• Summer 2009, existing pavement on Rt 2, Lake County, OH removed and replaced.
• Late winter 2010, longitudinal cracking discovered.
• Fall 2010, pavement heave of up to >4 inches observed.
• December 2010, subgrade borings at two stations yielded sulfate >10,000 ppm.

Could heave be due to high sulfate content of the soil?
SITE LOCATION
Lake County, OH
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Lake County, OH
GEOLOGIC SETTING

• Part of Galion Glaciated Low Plain Till Plain
• Soil is Conneaut-Painseville
  • Poorly drained soil formed in silty glacial till or loamy material over silty glacial till on lake plain during Wisconsinan glaciation.
• Underlying bedrock is Ohio Shale
  • Late-Devonian age brownish-black to black shale deposited in stagnant tropical sea.
SUSPECTED SOURCE OF HEAVE:

Expansive sulfate minerals such as ettringite
ETTRINGITE

- Ca-based stabilizers when mixed with water develop high pH conditions favorable for forming expansive minerals like **Ettringite**.

\[
\text{Ettringite:} \quad [\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26 \text{H}_2\text{O}]
\]

- Ca from lime/cement stabilizer
- Al from dissolution of phyllosilicates (eg, clays, micas)
- SO\(_4\) from gypsum dissolved in water

**Availability of sulfate is the most important consideration.**
RISK LEVELS ASSOCIATED WITH LIME STABILIZED SULFATE-BEARING CLAY SOILS

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<th>Soluble Sulfate Concentrations (ppm)</th>
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<tr>
<td>Moderate</td>
<td>Between 3,000 and 5,000</td>
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<td>Greater then 8,000</td>
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<tr>
<td>Unacceptable</td>
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</table>

From Texas Transportation Institute (after Little and Nair, 2009).
OBJECTIVES

I. Characterize soils from borings along Rt. 2 in Lake Co.
II. Synthesize and learn to identify ettringite
III. Replicate the soil swelling condition on Rt. 2 in Lake Co.
OBJECTIVE I

CHARACTERIZATION OF BORING SAMPLES
CHARACTERIZATION OF BORING SAMPLES

- Twenty eight samples collected from boring sites in the heaved road section were provided by ODOT.
- Most samples (22) were from subgrade soil with the remaining sampled at different depths.
- Samples were characterized by:
  - Soluble sulfate analysis (Tex-145-E colorimetric method)
    - Sulfate testing conducted to cross check methodology and results with ODOT
  - Mineralogical composition determined using XRD
SULFATE CONTENT OF SOIL SAMPLES

• Conducted sulfate tests using TxDOT procedure Tex-145-E colorimetric method.
  • Little and Nair (2009) recommend the TxDOT test method Tex-145-E over the AASHTO T-290 test.
  • The AASHTO T-290 test can under-report sulfate content.

• Sulfate testing procedures
  • Soil samples passed through a No. 40 (425µm) sieve
  • 10 g of soil / 200 ml deionized water
  • Samples shaken for 1 min / allowed to sit for 24 hrs
  • Filtered using Millipore filtering system / 0.45 µm filter paper
  • Two drops HCL / barium chloride packet
SULFATE CONTENT OF SOIL SAMPLES

- Used Thermo Scientific Orion AQUAfast AQ 3700 Colorimetry meter
  - Instrument reads concentrations of 5-100 mg; over-range solutions diluted
  - Solutions prepared in 1:20, 1:40, or 1:80 dilutions
  - Determined 5 minute optimum wait time before reading results
    - 90 ppm average difference between initial and 5 minute reading
    - 52 ppm average difference between 10 minute and 5 minute reading
SULFATE TESTING RESULTS

- Compared 23 sulfate results for boring samples between BGSU and ODOT.
  - 5 of 23 samples resulted in a difference of risk category
  - 3 of 5 significant; changing from low risk to moderate risk (above 3000 ppm)
  - 2 of 5 not significant; both results above 3000 ppm

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<tr>
<th>Sample</th>
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<th>ODOT PPM</th>
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<td>B4 Nat Sub</td>
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<td>B7 Top</td>
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<td>B49 Nat Sub</td>
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<td>4,002</td>
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XRD PROCEDURE

• Material crushed and placed in well of Al-planchet or, for smaller samples, crushed and placed on a glass slide using adhesive comprised of Duco cement and acetone.

• Scan performed using Rigaku Mini-flex II X-ray Diffractometer (Cu Kα source, 30 kV, 15 mA).

• Most scans were from 3° to 70° 2θ at scan rate of 1.5° per minute.

• Slower scans were run from 3° to 40° 2θ at a rate of 1° per minute to look for characteristic peaks of ettringite at 9.14° and 15.86° 2θ.
XRD RESULTS

- All samples were predominantly quartz with muscovite (sericite)
- Other common minerals were clays (clinochlore, kaolinite and illite)
- 57% contained measurable gypsum, anhydrite or combination of both
SUMMARY OBJECTIVE I

- TxDOT test method Tex-145-E for determining sulfate content resulted in consistent risk category levels between BGSU and ODOT labs.
- Found range in sulfates with some above moderate risk level (>3000 ppm).
- Found gypsum and/or anhydrite in over half of the samples, but highly heterogeneous distribution. Multiple aliquots from the same sample area yielded different mineralogical compositions in XRD.
- Differences in sulfate results for the same sample are due to the heterogeneity of the gypsum distribution in the soil and not the testing method.
OBJECTIVE II

SYNTHESIS AND CHARACTERIZATION OF ETTRINGITE
ETTRINGITE SYNTHESIS

- Synthesize pure ettringite crystals to use as standards for identification in soil samples.
- In X-ray diffraction procedures, pure sample is easier to identify peaks in than a more complex, mixed one.
- Having a pure sample provides a target chemical composition and crystal morphology to compare to SEM/EDAX analysis of soil samples.
- The synthesized material was characterized using SEM/EDAX and XRD.
SYNTHESIS:

- Procedure adapted from Cody et al. (2004).
- Saturated CaOH solution was prepared and dry N₂ gas was bubbled through the solution overnight to purge dissolved CO₂.
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- Procedure adapted from Cody et al. (2004).
- Saturated CaOH solution was prepared and dry N₂ gas was bubbled through the solution overnight to purge dissolved CO₂.
- Saturated AlSO₄ solution was then added to the CaOH solution and dry N₂ gas was bubbled through the mixed solution for remainder of procedure. (8-12 hours).
SYNTHESIS:

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• Fuzzy precipitate forms within 1 hr and reaction complete in 24 hrs.
SYNTHESIS:

- Procedure adapted from Cody et al. (2004).
- Saturated CaOH solution was prepared and dry N$_2$ gas was bubbled through the solution overnight to purge dissolved CO$_2$.
- Saturated AlSO$_4$ solution was then added to the CaOH solution and dry N$_2$ gas was bubbled through the mixed solution for remainder of procedure. (8-12 hours).
- Fuzzy precipitate forms within 1 hr and reaction complete in 24 hrs.
- Precipitate is filtered and placed in dessiccator to dry overnight.
- Note, if dissolved CO$_2$ is not purged from solutions calcite will precipitate instead of ettringite.
SEM/EDAX

- **SEM (Scanning Electron Microscope)**
  - Hitachi S-2700
  - Produces an image of a specimen by focusing a beam of electrons on the specimen.
  - When electrons strike the specimen there are a number of emanations. One of these is secondary electrons which produce the 3D image.

- **EDAX (Energy Dispersive Spectroscopy)**
  - X-ray energy analysis by the segregation of x-rays according to their energy.
  - The electron beam dislodges electrons from the inner shell. Outer shell electrons fill the vacancies. The difference in energy is emitted as an x-ray characteristic of a specific element.
SEM/EDAX

- **SEM Sample Preparation**
  - Soil samples air dried for at least three days versus oven dried to preserve the minerals present.
  - Small pieces broken off larger samples; soil is not ground or flattened to preserve structure.
  - Carbon adhesive or carbon planchet used to cover a 12.7 mm Al stub.
    - Prevents erroneously high Al EDAX results; true Al results for soil.
  - Sample affixed to the stub using liquid graphite (carbon).
  - Sample coated with C or Au/Pd for conductivity to prevent charging.
- **SEM Run Conditions**
  - Accelerating voltage = 20 kV; Working distance = 12 mm (optimum for EDAX).
SEM/EDAX

General Description of Minerals

- Clays: thin hexagonal plates
- Gypsum: tabular with perfect basal cleavage
- Ettringite: six-sided (hexagonal) needles
- Soil: platy structures
SEM/EDAX RESULTS FOR LAB ETTRINGITE

- SEM images showed a six-sided needle-like hexagonal morphology.
- EDAX confirmed the characteristic elemental abundances of O, Ca, S, and Al for ettringite.
- Using the empirical formula for ettringite the atomic weight percents are:
  - O 49.57%, Ca 31.04%, S 12.42%, and Al 6.97%

<table>
<thead>
<tr>
<th>ELEMENTAL WEIGHT PERCENT</th>
</tr>
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<tbody>
<tr>
<td>O</td>
</tr>
<tr>
<td>AVERAGE ABUNDANCE</td>
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XRD RESULTS

Initial runs without purging dissolved CO₂ with dry N₂ gas yielded calcite.

By purging dissolved CO₂ with dry N₂ gas the precipitate obtained was ettringite.
SUMMARY

- Lab synthesis of ettringite was verified with:
  - SEM images
  - EDAX composition
  - XRD scans
OBJECTIVE III

SWELL TESTS OF LAKE COUNTY ROUTE 2 SOILS
SWELL TESTING

- Replicated conditions that occurred along Lake County Route 2.
- Six buckets of soil provided by ODOT from Stations
  - 729+00
  - 730+00
  - 730+00
  - 730+80
- Samples of cement stabilized subgrade (CSS) and non-stabilized (NSS) soil directly below.
- Areas chosen because pavement heave was significant; full depth pavement repair required to fix pavement.
NON-STABILIZED SOIL

- Non-stabilized soil (NSS)
  - Brown and gray sandy silt
  - Gypsum crystals ranging from finely disseminated to 2 cm in size.
  - Black shale clasts

730+00 NSS

Gray clay layer
NON-STABILIZED SOIL

- SEM / EDAX
  - 21 samples / 7 per station
  - Quartz, clays, and iron oxides

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<th></th>
<th>O</th>
<th>Si</th>
<th>S</th>
<th>Ca</th>
<th>Fe</th>
<th>Al</th>
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<th>C</th>
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<td>0.97</td>
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<td>0.58</td>
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Non-stabilized Soil XRD

- Soil powdered prior to analysis.
NON-STABILIZED SOIL XRD

- Soil powdered prior to analysis.
- All samples primarily quartz/muscovite (sericite).
NON-STABILIZED SOIL XRD

- Soil powdered prior to analysis.
- All samples primarily quartz/muscovite (sericite).
- Highly heterogeneous distribution of gypsum.

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<td>Gypsum</td>
<td>3.46%</td>
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<td>Ferrihydrite</td>
<td>0%</td>
<td>0%</td>
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CEMENT STABILIZED SOIL

- Brown and gray sandy silt
- Gypsum crystals ranging from finely disseminated to 2 cm in size.
- Black shale clasts
- White precipitate

730+00 cement stabilized soil
CEMENT STABILIZED SOIL - XRD

- Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?

Lab-grown Sample
CEMENT STABILIZED SOIL - XRD

• Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?

• Used XRD to look for mineralogy of bucket samples
  • White precipitate was mostly calcite
CEMENT STABILIZED SOIL - XRD

- Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?
- Used XRD to look for mineralogy of bucket samples
  - White precipitate was mostly calcite
  - Found ettringite and thaumasite in stabilized soil
CEMENT STABILIZED SOIL - XRD

- Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?
- Used XRD to look for mineralogy of bucket samples
  - White precipitate was mostly calcite
  - Found ettringite and thaumasite in stabilized soil
CEMENT STABILIZED SOIL - XRD

- Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?
- Used XRD to look for mineralogy of bucket samples
  - White precipitate was mostly calcite
  - Found ettringite and thaumasite in stabilized soil
    - Easier to detect when sampling from areas of white precipitate
CEMENT STABILIZED SOIL - XRD

- Know what ettringite looks like (SEM/XRD). Can we find it in stabilized soil that experienced “swell”?
- Used XRD to look for mineralogy of bucket samples
  - White precipitate was mostly calcite
  - Found ettringite and thaumasite in stabilized soil
    - Easier to detect when sampling from areas of white precipitate
  - Major components

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CEMENT STABILIZED SOIL – SEM

- Looked at white precipitate areas with SEM.
- Ettringite needles were found in samples

730+00 CSS
CEMENT STABILIZED SOIL – SEM

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- Ettringite needles were found in samples

730+00 CSS
CEMENT STABILIZED SOIL – SEM

- Looked at white precipitate areas with SEM.
- Ettringite needles were found in samples
  - 730+00 CSS
  - 730+80 CSS
• Looked at white precipitate areas with SEM.
• Ettringite needles were found in samples
  730+00 CSS
  730+80 CSS
CEMENT STABILIZED SOIL – SEM

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- Ettringite needles were found in samples
  - 730+00 CSS
  - 730+80 CSS
CEMENT STABILIZED SOIL – SEM

- Looked at white precipitate areas with SEM.
- Ettringite needles were found in samples
  - 730+00 CSS
  - 730+80 CSS
CEMENT STABILIZED SOIL – SEM/EDAX

- Looked at white precipitate areas with SEM.
- Station 729+00 CSS: less ettringite; needles not as well developed.
CEMENT STABILIZED SOIL – SEM/EDAX

- Looked at white precipitate areas with SEM.
  - Station 729+00 CSS: less ettringite; needles not as well developed.
  - EDAX comparison: zooming in on ettringite needles the Si decreased from 26.15% to 14.13%, Ca increased 9.84% to 24.54%, S peak of 6.03%.
CEMENT STABILIZED SOIL – EDAX

- Typical cement stabilized EDAX spectrum.
- Average elemental abundance of 30 samples for cement stabilized soil.
- Average elemental abundance of 6 lab ettringite samples.

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<tr>
<td>AVERAGE ABUNDANCE</td>
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SULFATE CONTENT OF SOIL SAMPLES

• Conducted sulfate tests using TxDOT procedure Tex-145-E colorimetric method for NSS and CSS.
  • Determine the level of sulfate naturally in the soil.
  • 3 samples taken from stations 729+00, 730+00, and 730+80 for both NSS and CSS for a total of 18 samples.
  • 5 minute optimum wait before reading results.
### Sulfate Testing Results

#### Non-Stabilized Soil

<table>
<thead>
<tr>
<th>Date</th>
<th>Sample</th>
<th>pH</th>
<th>PPM</th>
<th>Risk</th>
<th>Settling Time</th>
<th>Date</th>
<th>Sample</th>
<th>pH</th>
<th>PPM</th>
<th>Risk</th>
</tr>
</thead>
<tbody>
<tr>
<td>3/5/13</td>
<td>729+00 NSS</td>
<td>3.400</td>
<td>moderate</td>
<td>Quick</td>
<td>3/5/13</td>
<td>729+00 SS</td>
<td>3.640</td>
<td>moderate</td>
<td>Quick</td>
<td></td>
</tr>
<tr>
<td>3/10/13</td>
<td>729+00b NSS</td>
<td>7.93</td>
<td>4,400</td>
<td>moderate</td>
<td>Moderate</td>
<td>3/10/13</td>
<td>729+00b SS</td>
<td>10.08</td>
<td>mod to high</td>
<td>Quick</td>
</tr>
<tr>
<td>3/10/13</td>
<td>729+00c NSS</td>
<td>7.68</td>
<td>5,120</td>
<td>mod to high</td>
<td>Moderate</td>
<td>3/10/13</td>
<td>729+00c SS</td>
<td>10.07</td>
<td>mod to high</td>
<td>Quick</td>
</tr>
<tr>
<td>Mean</td>
<td>7.81</td>
<td>4,307</td>
<td>moderate</td>
<td>Mean</td>
<td>10.08</td>
<td>5,720</td>
<td>mod to high</td>
<td>Mean</td>
<td>9.79</td>
<td>6,107</td>
</tr>
<tr>
<td>Stan Dev</td>
<td>0.18</td>
<td>864</td>
<td>Stan Dev</td>
<td>0.01</td>
<td>1,805</td>
<td>Range</td>
<td>0.11</td>
<td>1,499</td>
<td>Range</td>
<td>0.04</td>
</tr>
<tr>
<td>Range</td>
<td>0.25</td>
<td>1,720</td>
<td>Range</td>
<td>0.01</td>
<td>3,240</td>
<td>Range</td>
<td>0.09</td>
<td>2,520</td>
<td>Range</td>
<td>0.07</td>
</tr>
</tbody>
</table>

#### Stabilized Soil

<table>
<thead>
<tr>
<th>Date</th>
<th>Sample</th>
<th>pH</th>
<th>PPM</th>
<th>Risk</th>
<th>Settling Time</th>
<th>Date</th>
<th>Sample</th>
<th>pH</th>
<th>PPM</th>
<th>Risk</th>
</tr>
</thead>
<tbody>
<tr>
<td>3/5/13</td>
<td>730+00 NSS</td>
<td>3,640</td>
<td>moderate</td>
<td>Quick</td>
<td>3/5/13</td>
<td>730+00 SS</td>
<td>4,960</td>
<td>moderate</td>
<td>Quick</td>
<td></td>
</tr>
<tr>
<td>3/10/13</td>
<td>730+00b NSS</td>
<td>7.46</td>
<td>6,160</td>
<td>mod to high</td>
<td>Moderate</td>
<td>3/10/13</td>
<td>730+00b SS</td>
<td>9.82</td>
<td>mod to high</td>
<td>Quick</td>
</tr>
<tr>
<td>3/10/13</td>
<td>730+00c NSS</td>
<td>7.37</td>
<td>6,080</td>
<td>mod to high</td>
<td>Moderate</td>
<td>3/10/13</td>
<td>730+00c SS</td>
<td>9.75</td>
<td>mod to high</td>
<td>Quick</td>
</tr>
<tr>
<td>Mean</td>
<td>7.42</td>
<td>5,293</td>
<td>mod to high</td>
<td>Mean</td>
<td>9.79</td>
<td>6,107</td>
<td>mod to high</td>
<td>Mean</td>
<td>9.75</td>
<td>4,880</td>
</tr>
<tr>
<td>Stan Dev</td>
<td>0.06</td>
<td>1,432</td>
<td>Stan Dev</td>
<td>0.05</td>
<td>994</td>
<td>Range</td>
<td>0.09</td>
<td>1,760</td>
<td>Range</td>
<td>0.07</td>
</tr>
<tr>
<td>Range</td>
<td>0.09</td>
<td>2,520</td>
<td>Range</td>
<td>0.07</td>
<td>1,760</td>
<td>Range</td>
<td>0.09</td>
<td>2,520</td>
<td>Range</td>
<td>0.07</td>
</tr>
</tbody>
</table>
SULFATE TESTING RESULTS

• Given:
  • The threshold level for swell damage is 2,000 to 3,000 ppm (Little and Nair, 2009).
  • NSS sulfates ranged from 3,400 to 6,880 ppm; above the threshold level for damage.
  • Average pH of CSS samples ranged from 9.75 to 10.08.

• Determined:
  • Additional sulfate in the form of gypsum was not needed for swell testing.
SWELL SAMPLES

- Modeled swell experiment after a Texas study by Harris et al. (2004).
- Modifications made to the Texas study to better represent Ohio conditions.
  - The Texas soil was sulfate deficient; Ohio soil from Lake County Route 2 tested high in sulfates; additional gypsum was not added to the soil.
  - All Texas samples were air dried for 3 days to simulate Texas conditions of very dry soil that is suddenly saturated with water by a thunderstorm. Lake County Route 2 soil is wetter due to the availability of water. Half the samples were air dried for 3 days and half were kept moist.
SWELL SAMPLES

• Replicate conditions that occurred at Lake County Route 2
  • NSS passed through a #4 sieve
  • 6% Saint Mary’s cement
  • Wet-side of optimum using distilled water; target 16% moisture; actual 15-22%
  • Samples compacted using standard proctor machine
    • Used 2.5 kg (5.5 lb) rammer; 305 mm (12 in) drop
    • 4 inch x 4.5 inch mold
    • 3 soil layers which received 25 drops per layer
SWELL SAMPLES

- 4 samples per station: 729+00, 730+00, and 730+80; 12 total
- All samples left in plastic bag overnight to cure.
- Dry samples air dried for 3 days then placed in latex membranes.
- Wet samples placed in latex membranes.
- Samples placed on porous stones.
- 6 measurements per sample
  - Height 3 place 120° apart
  - Circumference: top, middle, bottom; averages used to calculate volume change
- Measurements taken everyday for 56 days

<table>
<thead>
<tr>
<th>Wet samples</th>
<th>Dry samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>(6)</td>
<td>(6)</td>
</tr>
<tr>
<td>Distilled water</td>
<td>Distilled water</td>
</tr>
<tr>
<td>7000 ppm sodium sulfate solution</td>
<td>7000 ppm sodium sulfate solution</td>
</tr>
</tbody>
</table>
RESULTS OF SWELL TESTING

Initial Sulfate levels in Soil

<table>
<thead>
<tr>
<th>Layer</th>
<th>Initial Sulfate</th>
<th>ppm</th>
<th>Swell Range</th>
<th>Weight Gain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-4</td>
<td>730+80</td>
<td>6,027</td>
<td>0.5% to 1.5% swell</td>
<td>39.0 g</td>
</tr>
<tr>
<td>5-8</td>
<td>730+00</td>
<td>5,329</td>
<td>6.0% to 8.0% swell</td>
<td>87.0 g</td>
</tr>
<tr>
<td>9-12</td>
<td>729+00</td>
<td>4,307</td>
<td>2.0% to 4.0% swell</td>
<td>44.8 g</td>
</tr>
</tbody>
</table>
RESULTS OF SWELL TESTING

- Circumference verses height

**Wet Samples**

- 1-4 730+80 0.5% to 1.5% swell
- 5-8 730+00 6.0% to 8.0% swell
- 9-12 729+00 2.0% to 4.0% swell

**Dry Samples**
SWELL TESTING OBSERVATIONS

- By day 4, solution had begun rising up the sides of the samples.
- Initially, bottom circumference measurements were the largest followed by middle, and then top. This trend was not consistent over time.
- In general, dry samples swelled slightly more than wet samples.
- Samples with additional sulfate in the form of sodium sulfate swelled slightly more than samples in distilled water.
After 56 days latex membranes removed and samples allowed to air dry.

Many samples contained cracks.

Samples were cut in half with a diamond-tipped saw.

Sodium sulfate samples 3, 8, & 11 had a white precipitate ring around edge of sample. Water samples 6 & 9 also had the ring. All these samples were “dry” samples.
Initially focused on white precipitate areas looking for ettringite and gypsum. Found calcite.
SWELL SAMPLES - XRD

- Initially focused on white precipitate areas looking for ettringite and gypsum. Found calcite.
- Ettringite found in soil areas near white precipitate or soil from the center section of the samples.
SWELL SAMPLES - SEM

- Ettringite needles found in soil near white precipitate and in soil from the bottom, middle, and top sections.
- Presence of ettringite needles found in all swell samples. Including samples 1-4 from station 730+80 that only swelled 0.5% to 1.5%.
SWELL SAMPLES – SEM

- Sample 1; 730+80; DW; Dry
- Sample 2; 730+80; DW; Wet
- Sample 6; 730+00; DW; Dry
- Sample 9; 729+00; DW; Dry
- Sample 10; 729+00; DW; Wet
- Sample 12; 729+00; SS; Wet

<table>
<thead>
<tr>
<th>ELEMENTAL WEIGHT PERCENT</th>
<th>O</th>
<th>Ca</th>
<th>Si</th>
<th>Al</th>
<th>S</th>
<th>Fe</th>
<th>C</th>
<th>K</th>
<th>Cl</th>
<th>Ti</th>
<th>Mg</th>
<th>Na</th>
</tr>
</thead>
<tbody>
<tr>
<td>AVERAGE ABUNDANCE</td>
<td>35.42</td>
<td>26.18</td>
<td>15.82</td>
<td>6.79</td>
<td>5.34</td>
<td>5.78</td>
<td>5.28</td>
<td>2.53</td>
<td>0.86</td>
<td>0.77</td>
<td>0.61</td>
<td>0.31</td>
</tr>
</tbody>
</table>
SUMMARY OBJECTIVE III

• Replicated soil swelling using material from Lake County Route 2
  • Soil swell does not follow sulfate concentration of soil likely due to the heterogeneous distribution of sulfates in the soil.
  • However, soils that produced swell were above 3000 ppm. Therefore, 3000 ppm is good sulfate limit.
• Established ettringite was linked to soil swelling, and was present in both NSS and lab soil specimens.
CONCLUSIONS

- Tex-145-E Colorimetry method provides relatively rapid and reliable sulfate analysis.
- Dissolved CO$_2$ in CaO-AlSO$_4$ solutions promotes precipitation of calcite over ettringite.
- Laboratory experiment using soils from Lake County Route 2 show significant swelling and formation of ettringite.
- However, amount of swelling apparently is not related to total amount of sulfate present.
IMPLICATIONS/RECOMMENDATIONS

• Following Little and Nair (2009):
  • Sulfate contents should be determined and heterogeneity of distribution considered.
  • Sulfate risk levels should be considered when using Ca-based stabilization.

• Following Harris et al. (2004)
  • Mellowing should be considered
    • Primary vs secondary ettringite formation may explain high sulfate but low amount of swell in our sample 730+80.
ACKNOWLEDGMENTS

- We would like to thank:
  - Jeff Wigdahl, ODOT Subject Matter Expert for all of his help and input.
  - Marilyn Cayer, BGSU Center for Microscopy and Microanalysis for help with the SEM/EDAX analyses.
  - ODOT Student Studies Program for funding this research project (State Job #134694).
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• Cody, AM, Lee, H, Cody, RD, and Spry, PG (2004) The effects of chemical environment on the nucleation, growth, and stability of ettringite \([\text{Ca}_3\text{Al(OH)}_6\text{]_2(SO}_4\text{)}_3\text{26H}_2\text{O}\). Cement and Concrete Research, 34, 869-881.


• Little, DN and Nair, S (2009) NCHRP Web-only document 145: Recommended practice for stabilization of sulfate rich subgrade soils. Texas Transportation Institute, Texas A & M University, College Station, Texas, 66 pg.