AN INVESTIGATION OF NANO SILICA (SiO$_2$) IN THE CEMENT HYDRATION PROCESS

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Overview

• Introduction
• A Brief History of Concrete
• What Makes This Project Unique?
  – Comparing new vs old materials
  – What has been investigated
• Materials and Experiments
  – What worked
  – What didn’t work
• The Cement Hydration Process
  – 5 Stages of the Heat of Hydration
• Results and Analysis
  – What Happened
• Summary
• Questions
• References
A Brief History of Concrete

• Concrete Technology through time (*Finding Solutions in the Face of Adversity*)
  – Appian, Rome: Circa 125 BC
  – First Concrete Roadway, Ohio: 1893
  – Confederation Bridge, Canada: 1993

• Problems in Need of Solutions
  – Concrete/Cement Expensive
  – ASR, Min. Fly Ash Req.
    • GAO – 85 – 10R

• Engineering Responsibility
  – Preserving the Environment
  – Economic Vitality
  – Durable/Sustainable Roadways
What Makes this Project Unique?

• Comparing old vs new materials
  – Silica, a pozzolanic material
    • Introduced in the 1970s
  – Nano Silica, a pozzolanic material
    • Introduced in the new millennium

• What has been investigated
  – A nano silica changing a microscopic crystal structure which has macroscopic affects.
Materials and Experiments

- **Constituents**
  - Type I/II Portland Cement
  - Polycarboxylate Comb Polymer (HRWR)
  - Silica

- **Mix Design**
  - No Aggregate
  - w/c of 0.32

- **Preliminary Trials**
  - Contamination
  - Modifying the Standards

<table>
<thead>
<tr>
<th>Mix</th>
<th>kg/m³ (lb/yd³)</th>
<th>L/m³</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Water</td>
<td>Cement</td>
</tr>
<tr>
<td>ST CMT</td>
<td>133 (224)</td>
<td>425 (716)</td>
</tr>
<tr>
<td>SF</td>
<td>133 (224)</td>
<td>425 (716)</td>
</tr>
<tr>
<td>8 (nano)</td>
<td>133 (224)</td>
<td>425 (716)</td>
</tr>
<tr>
<td>50 (nano)</td>
<td>133 (224)</td>
<td>425 (716)</td>
</tr>
<tr>
<td>508 (nano)</td>
<td>133 (224)</td>
<td>425 (716)</td>
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</tbody>
</table>
Materials – Silica Fume (SF)

- “This byproduct is the result of the reduction of high-purity quartz and coal in an electric arc furnace in the manufacture of silicon or ferrosilicon alloy.” (PCA 60).
- Commercialization ~ 1970’s
- Surface area of SF ~ 20,000 m²/kg
  - Cigarette smoke ~ 10,000 m²/kg
- Size ~ Average 1.0 µm
  - ST DEV of 0.5 µm
- Variability
Materials – Nano Silica

• Less variability
• Size distribution
  – Mix 8, Size ~ Average 35 nm
    • Range of sizes: 2 to 100 nm
  – Mix 50, Size ~ Average 5.5 nm
    • Range of sizes: 4 to 6 nm
  – Mix 508: Combination of 8 and 50
• Manufacturer’s Specification
  – Diameter > 40 nm, Filler
  – Diameter < 10 nm, Chemical

Micrograph of Nano Particles
From Brian H. Green at the Army Corp of Engineers
Experiments

With a Brief Discussion on Standards

- **Calorimetry**
  - Grace Adiacal, Semi-Adiabatic Calorimetric Chamber
  - Specimens: 76.2 mm (diam.) by 152.4 mm (length)
  - Standard: ASTM C 31-09

- **X-Ray Diffraction**
  - Siemmin’s E500, X-Ray Diffractor
  - Specimens: 25 mm by 50 mm by 10 mm
  - Standard: N/A

- **Scanning Electron Microscopy**
  - Jeoul 5800 LV, Scanning Electron Microscope
  - Specimens: 25 mm by 50 mm by 10 mm
  - Standard: N/A

- **Compressive Tests**
  - Forney Pilot, Closed Loop Hydraulic Press
  - Specimen: 50 mm by 50 mm by 50 mm
  - Standard: ASTM C 109-08
Cement Hydration Process

- **Stage 1** → \( C_3A + 6H \rightarrow C_3AH_6 \)
- **Stage 2, 3, & 4** → \( 2(C_3S) + 6(H_2O) \rightarrow C_3S_2H_3 + 3(Ca(OH)_2) \)
- **Stage 4, 5** → \( 2(C_2S) + 4(H_2O) \rightarrow C_3S_2H_3 + Ca(OH)_2 \)
- **Pozzolanic Reaction** → \( S + Ca(OH)_2 \rightarrow C_3S_2H_3 + Ca(OH)_2 \)
Results – Calorimetry

SF MIX, POZZ REACTION AT 120 Hr
Results – XRD

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>Calcium Hydroxide (%)</th>
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<tbody>
<tr>
<td>8</td>
<td>22.7%</td>
</tr>
<tr>
<td>SF</td>
<td>23.9%</td>
</tr>
<tr>
<td>508</td>
<td>24.9%</td>
</tr>
<tr>
<td>ST CMT</td>
<td>27.4%</td>
</tr>
<tr>
<td>50</td>
<td>27.5%</td>
</tr>
</tbody>
</table>
Results - SEM

Mix ST CM

Mix SF
Results – SEM, Cont’d

Mix 8

Mix 50

Mix 508 →
Results – SEM, Cont’d

Mix 508

Mix 8
Results – Compressive Strength

- ST DEV for all mixes conformed to the ASTM C 109-08
  - Did not exceed 3.5 MPa (500PSI) between samples
Summary

• Thesis Goals
  – An Investigation of Nano Silica on The Cement Hydration Process

• As the Size of the Nano Particle Decreases and the Distribution of Sizes Increases
  – Increase in Temperature
  – Reduction in CH
    • Possibility of a Change in CSH
  – Increase in Compressive Strength
  – SF Mix, An Outlier
QUESTIONS
References

- Balaguru, P., and Chong, K. Nanotechnology and Concrete: Research Opportunities. ACI SP-2008.
- Christensen P. Structural and Ingredient Analysis of Concrete – Methods, Results, and Experience. Nordisk Betong. 1984.
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