Proposed Testing and Research Approach for Pyrrhotite-Induced Concrete Deterioration

Mr. Christopher M. Moore, Mr. Cody M. Strack, Dr. Robert D. Moser, Dr. Gordon W. McMahon, Engineer Research and Development Center (ERDC) US Army Corps of Engineers (USACE)

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This briefing presents recommendations for test methods and approaches for addressing pyrrhotite-induced expansion in concrete. There is no standard procedure to apply or guidance available. Almost all of the recommendations require R&D ranging from months to years to develop a solution.
Phased approach

• Approach focused on addressing near-term regulatory needs and developing approaches to identify, prioritize, and manage structures susceptible to concrete damage from pyrrhotite-induced expansion.
• Quarry oversight / putting a “clamp” on deleterious aggregates
  • 1st phase conservative guidance based on chemistry
  • 2nd phase to consider both mineralogy and chemistry – needs short term R&D
• Forensics on existing homes
  • Near-term recommendations for improving petrographic analysis
  • Med-term improved analysis methods – needs med term R&D
• Projection of future damage – long term (5+ years) R&D needed
• Develop mitigation options – long term (1-3 years) R&D needed
• Best practices for concrete replacement - med term (1 year) R&D needed
Aggregate Acceptance
Based on Chemistry

Develop Fe-S
Mineral Analysis
Method(s)

Aggregate Acceptance
Based on Chemistry
and Mineralogy

Use Petrography Methods and Standardize
Reporting of Field Conditions and Analysis Results

Develop New Petrography / Forensic Analysis
Methods: Chemistry, Mineralogy, Magnetic

Revised Approach for
Field Assessment and
Forensic Analysis

R&D on Service Life Modeling Approaches

R&D on Mitigation Options: Environmental, Repair/Retrofit

R&D / Tradespace on Approaches for Replacement

Inform Approaches for
Managing Existing Homes,
and Prioritizing
Mitigation, Repair, and
Replacement Activities

Immediate

Near-Term

Mid-Term

Long-Term

Year 1-2

Year 3-4

Year 5-7

Year 8+

IMPLEMENT

IMPLEMENT

IMPLEMENT
Quarry oversight

- Regulations on quarries for minimum testing and acceptance requirements for supply of aggregate materials to concrete production industry
- Lean on testing standards from ASTM, AASHTO currently used for aggregates, cements, etc.
- Reference acceptance / rejection requirements (i.e., limits on test results) from Canadian and European Standards
- Frequency of testing based on quantity of material that adjusts based on observed variation in results
Quarry Oversight

**Phase 1 - Immediate**
- Combustion IR
- ASTM C33
- Overly restrictive limits

**Phase 2 – Long term**
- ASTM C295
- X-ray diffraction (ASTM C1365)
- Development of pyrrhotite specific standards
  - Accurate accept/reject limits

- X-ray Fluorescence
- Testing regimen for aggregate sources
Quarry oversight – basic requirements

• Aggregate must meet minimum requirements of ASTM C33 Standard Specification for Concrete Aggregates
  - Gradations
  - Deleterious Materials
• This requirement should already be in place for materials to be used in certified ready-mix concrete plant
• Does not cover chemical and mineralogical concerns with pyrrhotite in aggregate
Quarry oversight – basic requirements

- No general standards that cover specifics of chemical and mineralogical analysis of aggregate
- Specific test method to identify pyrrhotite at relevant resolution (e.g., 0.2-0.3%) needs to be developed and vetted.
- Suggest immediate conservative focus that assumes pyrrhotite is present and uses standard test methods for chemical analysis to qualify and aggregate for use in concrete.
- 2nd phase would include mineral ID rather than conservative focus on pyrrhotite.
- Recommend using specific standards / test methods for chemistry and mineralogy.
Quarry Oversight – 1st phase sulfur content by XRF or IR combustion

- Leco infrared combustion sulfur analysis
  - Obtain elemental sulfur (S) content
- X-ray fluorescence (XRF) measurements on aggregate to identify bulk chemical composition
  - Performed on fused glass samples following procedures in AASHTO M85 for cement
    - Pulverize aggregate to produce fused glass sample
      - Pulverize sample to 90% by mass passing #325 (45 μm) sieve
  - Obtain bulk chemistry using XRF
  - Quantify elemental sulfur (S) content

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<td>C₄AF</td>
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Quarry Oversight – 1st phase accept / reject limits for aggregate

- Assume that pyrrhotite is present in aggregate
  - Accept aggregate if sulfur (S) content <0.1%
  - Otherwise, reject aggregate for use in concrete
- Suggest that CT State Geologist has input on this and if it is possible to just apply this analysis regionally. However, pyrrhotite geological maps are for non-trace compositions. Relevant amounts of pyrrhotite likely extend far beyond mapped regions.
Quarry Oversight – 1st phase qualification of laboratories

- Certification programs for laboratories to conduct this testing are recommended:
  - AASHTO Accredited
  - ISO 17025
  - Participate in Cement and Concrete Reference Laboratory (CCRL) testing proficiency sample program.

- Sulfur (S) content measurements are less than typically present in cement. New low S content calibrations will need to be performed by laboratories to calibrate / verify instruments.
Quarry Oversight – 1st phase frequency of testing

- Recommend testing for every 25k tons or 3 months, whichever is most frequent
  - Based on making testing less than 1% of operating cost
  - Assumed testing cost $5k and rock price $25/ton
- Perform this testing four times
  - If variability in results is less than +/-10% of mean of four test results, switch to conducting once per year
  - If higher variability observed, continue at testing frequency specified above
- NOTE: CT State Geologist should have input on this. The frequency / weight for each interval of testing should be based on the strata / heterogeneity in the formation.
Quarry oversight – 2nd phase mineralogy + sulfur content

- A 2nd phase of implementation to also consider mineralogy to understand Fe-S mineral present
- Fe-S mineral identification would guide selection of acceptable limits on sulfur content
- Recommend optionally using either thin section petrography by ASTM C295 or X-ray diffraction (or other undiscovered) to qualitatively identify Fe-S minerals
- Recommend using specific standards / test methods for chemistry and mineralogy.
- Recommend a quality materials consultant / laboratory services laboratory perform short-term research to develop method rather than university.
Quarry Oversight – 2nd phase X-ray diffraction for mineral ID

- X-ray diffraction (XRD) measurements on aggregate to identify deleterious minerals
- No specific procedures to apply. Can lean on ASTM C1365 and D934
- Example recommended operating procedures
  - Pulverize sample to 90% by mass passing #325 (45 μm) sieve
  - Random powder pack sample preparation
  - Scan resolution of <0.1° 2θ
  - Scan speed to achieve >10k counts on 100% peak
    - Modern XRDs can do this with a 2hr scan / 0.67 °/min
  - Scan between 20-100° 2θ
    - Applicable to Cu and Co K-α sources
    - Scan correlated to d-space between approx. 1-5 Å
  - Identify if pyrrhotite and/or pyrite are present
    - Using ICDD verified powder diffraction reference patterns
    - If no pyrrhotite or pyrite is detected, no additional testing (i.e., XRF) is required
- Recommend a short study by quality analysis laboratory to run through method and “tweak” any parameters
Quarry Oversight – 2\textsuperscript{nd} phase X-ray diffraction for mineral ID
Quarry Oversight – 2\textsuperscript{nd} phase sulfur content by XRF or IR combustion

- Leco infrared combustion sulfur analysis
  - Obtain elemental sulfur (S) content
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- Performed on fused glass samples following procedures in AASHTO M85 for cement
  - Pulverize aggregate to produce fused glass sample
    - Pulverize sample to 90\% by mass passing #325 (45 \( \mu \text{m} \)) sieve
- Obtain bulk chemistry using XRF
- Quantify elemental sulfur (S) content
Quarry Oversight – 2nd phase Accept / reject limits for aggregate

- If pyrrhotite is observed by XRD and/or ASTM C295
  - Accept aggregate if sulfur (S) content <0.1%
  - Otherwise, reject aggregate for use in concrete
- If pyrrhotite is not observed but other Fe-S minerals are (e.g., pyrite) by XRD and/or ASTM C295
  - Accept aggregate if XRF sulfur (S) content <1%
  - Otherwise, reject aggregate for use in concrete
- If no Fe-S minerals (i.e., pyrrhotite, pyrite) are observed by XRD and/or ASTM C295, no objection to acceptance based on chemistry and mineralogy

NOTE: Other methods for mineralogy and chemistry may be available such as examination of magnetic properties.
Quarry Oversight – 2nd phase qualification of laboratories for testing

- Certification programs for laboratories to conduct this testing are recommended:
  - AASHTO Accredited
  - ISO 17025
  - Participate in Cement and Concrete Reference Laboratory (CCRL) testing proficiency sample program.
- Sulfur (S) content measurements are less than typically present in cement. New low S content calibrations will need to be performed by laboratories to calibrate / verify instruments.
Quarry Oversight – 2nd phase frequency of testing

- Recommend testing for every 25k tons or 3 months, whichever is most frequent
  - Based on making testing less than 1% of operating cost
  - Assumed testing cost $5k and local rock price $25/ton
- Perform this testing four times
  - If variability in results (max vs. min) <10%, switch to conducting once per year
  - If higher variability observed, continue at testing frequency specified above
- NOTE: CT State Geologist should have input on this. The frequency / weight for each interval of testing should be based on the strata

Stony Creek Quarry – Branford, CT
Forensics – Using current procedures ASTM C856

- Currently ASTM C856 Standard Practice for Petrographic Examination of Hardened Concrete
- Need to standardize sample collection
  - Sample from consistent location (i.e., basement wall with maximum soil elevation)
  - Report site conditions: water table, waterproofing systems, sump present, dehumidifier, HVAC in basement
- Report environmental conditions: internal temperature and relative humidity in basement
- Report damage: standard “classes” of damage with visual rating guidance, note efflorescence, etc.
Forensics – Using current procedures ASTM C856 (cont’d)

• Need to standardize reporting:
  • Note presence of Fe-S minerals: pyrrhotite, pyrite, or others
  • Note if present in coarse and/or fine aggregate
  • Note any other relevant features: SCMs used, rough estimate of water-to-cement ratio, large voids and air entrainment observations
  • Provide semi-quantitative estimate of composition. Below is an example potential binning:
    ▶ <0.1% / minor
    ▶ 0.1%-1% moderate
    ▶ 1%-10% high
    ▶ >10% very high
    ▶ Can base off of visual estimators applied to thin sections
    ▶ The resolution in these bins is difficult to obtain w/ current analytical methods

• Would require some short term trials with concrete from affected structures to refine method.
Forensics – New method development, med term R&D needed

- Full concrete petrography by ASTM C856 is expensive and low resolution
- Need better method with high resolution to quantify Fe-S content in existing concrete
- Ideally coring would not be required
- Many potential options:
  - On-site forensic analysis using handheld instruments
  - Collection of powders for laboratory-based analysis
  - Improved petrographic analysis procedures to apply to cores to improve resolution and speed of analysis
- Would recommend medium term R&D conducted by quality materials consultant with expertise in concrete materials, petrography, and unique test methods
Service Life Prediction for Infrastructure

Current State of Distress → Future State of Distress → Influence on the Structure

Risk-informed decisions for prioritizing funding for future maintenance and repair activities.
Projecting future concrete deterioration, long term R&D needed

- To project how concrete damage will occur in future
- Map space of material, construction, exposure:
  - Different pyrrhotite contents, coarse and/or fine aggregate
  - Different construction / basement types
  - Different environments: water table, RH, etc.
- Laboratory testing:
  - Simulate different variables with accelerated laboratory-based expansion testing
- Goal would be to correlate observations from forensic analysis and “bins” structure types to project future damage
  - Minimal expansion capacity, moderate, high, very-high, etc.
Develop potential mitigation options, long term R&D needed

- Focus on R&D to understand if there’s anything that can be done to help mitigate expansion
  - French drains, upstream waterproofing, dehumidifiers, or other (more outside-the-box) ways
  - Results from the service life modeling research would provide information to guide potential mitigation options
- Would be tied to “bins”
  - 1: Minimal damage anticipated, no mitigation needed
  - 2: Mitigation can actually help to extend life of concrete
  - 3: Nothing you can do – full replacement needed
Best practices for full concrete replacement, med term R&D needed

• Need to generate some best practices for remediation / replacement of basements
  • Engineered solutions for typical basements
  • Jack up house vs. wall-by-wall replacement
  • Other innovative construction approaches
• In future, this guidance would roll into the high risk “bin” for structures where full replacement is needed
Aggregate Acceptance
Based on Chemistry

Develop Fe-Substitution Method(s)

Aggregate Acceptance Based on Chemistry and Mineralogy

Use Petrography Methods and Standardize Reporting of Field Conditions and Analysis Results

Develop New Petrography / Forensic Analysis Methods: Chemistry, Mineralogy, Magnetic

Revised Approach for Field Assessment and Forensic Analysis

R&D on Service Life Modeling Approaches

Inform Approaches for Managing Existing Homes, and Prioritizing Mitigation, Repair, and Replacement Activities

R&D on Mitigation Options: Environmental, Repair/Retrofit

R&D / Tradespace on Approaches for Replacement

Year 1-2

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