Accelerated Test Method to Identify Freeze-Thaw Durability of Aggregates

Belayneh Desta, Nancy Whiting, Mark Snyder, Jan Olek, and Tommy Nantung
General Outline

Part I: Study Findings & Background

Part II: Natural & Recycled Concrete Aggregates (RCA)

Part III: Highlights of the Ongoing Tests
Part I
Study Findings & Background

• Study Findings
  o Natural Aggregates
  o RCA

• Background
  o Freeze-Thaw Related Damages in Concrete
  o The Certified Aggregate Producers (CAP) Program
  o ASTM C666 (ITM 210)
  o History and Protocol of the HFT
  o INDOT HFT Equipment (History, Design, Features Calibration)
Study Findings

• The Hydraulic Fracture Test (HFT) equipment and refined procedures developed at INDOT accurately predicted the ITM 210 90-day freeze-thaw test results of nearly all aggregates in 8 days.

• The RCA testing showed:
  – All 5 aggregate sources made from older INDOT concrete pavement passed the ITM 210 FT test
  – RCA is responsive to testing in the HFT
  – Testing of additional RCA sources is expected to lead to a reliable model to predict FT durability of RCA in 8 days
Aggregate-Related Freeze-Thaw Damages

D-Cracking

Pop-Outs
Identification of Freeze-Thaw Durability of Aggregates

Rapid Freezing and Thawing - ASTM C666/ AASHTO T161

- INDOT's ITM 210
  - Commonly used test
  - Evaluates concrete beams exposed to freeze-thaw cycles
  - Determines % dilation and DF of beams
  - Requires expensive equipment

- Can take months to complete

Freeze-Thaw Chamber
Certified Aggregate Producer (CAP) Program

The Indiana Department of Transportation (INDOT):

- Tests (ITM 210) all their concrete aggregate sources every 1-3 years

- Frequency of test depends on source variability and historical test results
Why Accelerated Test Method?

• The natural variability within aggregate sources may require frequent testing

• Confirm quality for sources that frequently go in and out of spec

• A quick approval or confirmation of approval of certified sources just before construction
Objective

To develop a **reliable, quick test method** for determining the freeze-thaw resistance of carbonate aggregates quarried in Indiana using the **Hydraulic Fracture Test (HFT)** equipment.
The HFT

- The Hydraulic Fracture Test (HFT) was developed under SHRP Program in the early 1990’s

- AASHTO developed a provisional standard TP12-93 modified and reapproved as TP12-96 “Method for Determining the Hydraulic Fracture of Coarse Aggregate”

- The equipment available and method for interpreting the results at that time had marginally acceptable accuracy
HFT Test Protocol

- Oven-dry aggregates are placed in chamber (~28lb)
- Chamber is filled with water

- Apply pressure (1300 psi) using nitrogen to force water into the pores of the aggregate
- Pressure is then released rapidly causing compressed air trapped within the aggregate pores to expand, expel water from pores creating internal stresses
Test Protocol

- If the pore structure of the aggregate cannot allow the water to exit rapidly and the resulting hydraulic pressure exceeds the strength of the aggregate then it will fracture aggregate particles.

Schematic diagram of INDOT HFT equipment

Fractured aggregate after subjected to 50 HFT cycles (example)
Evolution of INDOT HFT Equipment
The HFT Chamber Calibration

• The amount of fracturing caused by HFT is directly related to the pressure release rate

• The goal of calibration was to establish the combination of actuator pressure and the chamber pressures that consistently produced release rate curves similar to the “standard” that was adopted in previous studies
The HFT Chamber Calibration

The INDOT HFT equipment was calibrated by running a series of pressurizing and depressurizing cycles at different pressures combination.

Pressure release profiles for chamber pressure of 1300psi and different actuator pressure.

A Chamber pressure of 1300 psi, & actuator pressure of 175psi selected for further testing.
Part II

Quarried and Recycled Concrete Aggregates

• Quarried Aggregate
  – Selection
  – Testing
  – Analysis
  – Model development
  – Conclusions
  – Implementation

• Recycled Concrete Aggregate (RCA)
  – Testing
  – Analysis
  – Model development
  – Conclusions
  – Implementation
<table>
<thead>
<tr>
<th>Group A</th>
<th>Group B</th>
<th>Group C</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>B1</td>
<td>C1</td>
</tr>
<tr>
<td>A2</td>
<td>B2</td>
<td>C3</td>
</tr>
<tr>
<td>A3</td>
<td>B3</td>
<td>C4</td>
</tr>
<tr>
<td>A4</td>
<td>B4</td>
<td>C5</td>
</tr>
<tr>
<td>A5</td>
<td>B5</td>
<td>C6</td>
</tr>
<tr>
<td>A6</td>
<td>B6</td>
<td>C7</td>
</tr>
</tbody>
</table>

**Group A:** Freeze-thaw durable sources  
**Group B:** Freeze-thaw nondurable sources  
**Group C:** Variable or unknown freeze-thaw performance
Aggregate Sample Preparation

Representative samples from each aggregate source were separated by sieving into three size ranges:

- 3/4 to 1 in. (19.0 to 25 mm)
- 5/8 to 3/4 in. (16.0 to 19.0 mm)
- 1/2 to 5/8 in. (12.5 to 16.0 mm)
Aggregate Sample Preparation

Individual fractions were recombined into an HFT test sample (about 28lb) using the gradation shown in the table.

<table>
<thead>
<tr>
<th>Sieve Size</th>
<th>% Passing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 in (25 mm)</td>
<td>100</td>
</tr>
<tr>
<td>¾ in (19 mm)</td>
<td>89</td>
</tr>
<tr>
<td>5/8 in (16 mm)</td>
<td>40</td>
</tr>
<tr>
<td>1/2 in (12.5 mm)</td>
<td>0</td>
</tr>
</tbody>
</table>
Aggregate Sample Preparation

Aggregate samples:

- Washed and oven dried
- Submerged in a water-based silane solution for 60 seconds
- Drained and oven dried

Double boiler used to soak aggregate sample
Aggregate Sample Preparation

Aggregate samples:
- Tumbled in a rock tumbler
- Sieved over the ¾”, 5/8” and ½” sieves, any piece passing the ½” sieve discarded
HFT Sample Sieving

After each 10 HFT cycles, the samples removed from the chamber, oven-dried, tumbled and sieved.
### HFT Result Example (B3)

<table>
<thead>
<tr>
<th>Sieve size</th>
<th>0 Cycle</th>
<th>10 Cycles</th>
<th>20 Cycles</th>
<th>30 Cycles</th>
<th>40 Cycles</th>
<th>50 Cycles</th>
<th>PCMR*</th>
</tr>
</thead>
<tbody>
<tr>
<td>3/4&quot;</td>
<td>1462.5</td>
<td>1462</td>
<td>1412.8</td>
<td>1363.6</td>
<td>1375.9</td>
<td>1263.8</td>
<td>-13.59</td>
</tr>
<tr>
<td>5/8&quot;</td>
<td>6344.0</td>
<td>6338.4</td>
<td>6328.8</td>
<td>6270.2</td>
<td>6099.8</td>
<td>6266.7</td>
<td>-1.22</td>
</tr>
<tr>
<td>1/2&quot;</td>
<td>5187.0</td>
<td>5180.5</td>
<td>5193.7</td>
<td>5281.2</td>
<td>5355.7</td>
<td>5243.8</td>
<td>1.10</td>
</tr>
<tr>
<td>3/8&quot;</td>
<td>0.0</td>
<td>0.0</td>
<td>13.7</td>
<td>9.4</td>
<td>44.7</td>
<td>78.9</td>
<td>0.61</td>
</tr>
<tr>
<td>5/16&quot;</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>1.3</td>
<td>4.0</td>
<td>0.0</td>
<td>0.03</td>
</tr>
<tr>
<td>1/4&quot;</td>
<td>0.0</td>
<td>0.0</td>
<td>0.5</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.00</td>
</tr>
<tr>
<td>#4</td>
<td>0.0</td>
<td>0.0</td>
<td>0.6</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.00</td>
</tr>
<tr>
<td>Pan</td>
<td>0.0</td>
<td>12.6</td>
<td>24.3</td>
<td>26.6</td>
<td>25.3</td>
<td>22.2</td>
<td>0.85</td>
</tr>
<tr>
<td>Total Mass</td>
<td>12993.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>12875.4</td>
</tr>
</tbody>
</table>

*Percent Change in Mass Retained (after 50 cycles)
Beams were fabricated from concrete mixtures that were produced using each aggregate source

- \( w/c = 0.43 \)
- 6.5 (±1.5) % air
- Beams (3 in. X 4 in. X15 in.)
- % dilation & durability factor (DF) measured

Expansion < 0.060% after 350 cycles of F/T
Analysis

\[ y = -0.0041x + 0.4121 \]

\[ R^2 = 0.9144 \]

0.000 0.050 0.100 0.150 0.200 0.250 0.300

0 20 40 60 80 100

% Dilation

\[ 0.060\% \text{ INDOT acceptance criteria} \]

\[ DF = 86 \]

Source A

Source B

Source C
Analysis

0.060% INDOT acceptance criteria

Aggregate source

Dilation, %
Model Development

A linear regression model was developed to predict the average % dilation of freeze-thaw test beams using parameters obtained from HFT results.

% Dilation = $8.25 \times 10^{-2} + 6.33 \times 10^{-3}P_{34} + 9.64 \times 10^{-2}P_{38} - 3.12P_{14} + 4.3P_{4}$

Model statistics:

$R^2 = 0.892,$

$R^2 \ (adj.) = 0.853,$

$SEE = 0.029, \ n=16,$

Model P-value $< 0.0001$
Measured vs Predicted Dilations

\[ y = 0.892x + 0.0087 \]

\[ R^2 = 0.8922 \]

Predicted dilation, %

Measured dilation, %

Ideal 1:1 relationship of x and y axes

Data Trend line excluding A3 and B2
The developed dilation model correctly predicted the freeze-thaw durability for all sources tested except for the four sources shown in the table (14 of the 18 sources).

<table>
<thead>
<tr>
<th>Source (Group)</th>
<th>Measured Dilation, %</th>
<th>Predicted Dilation, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>A3</td>
<td>0.0071</td>
<td>0.1068</td>
</tr>
<tr>
<td>B3</td>
<td>0.0849</td>
<td>0.0551</td>
</tr>
<tr>
<td>B6</td>
<td>0.0835</td>
<td>0.0597</td>
</tr>
<tr>
<td>C7</td>
<td>0.0809</td>
<td>0.0569</td>
</tr>
</tbody>
</table>
Measured vs Predicted Dilations

#### Graph Details

- **Axes:**
  - **Y-axis:** % Dilation
  - **X-axis:** Source

- **Bars:**
  - **A1 to C7**

- **Bars Colors:**
  - Blue: Measured
  - Red: Predicted
  - Dashed Red Line: INDOT 0.060% FT Criterion
  - Solid Purple Line: Proposed 0.050% HFT Criterion

- **Legend:**
  - Measured
  - Predicted
  - INDOT 0.060% FT Criterion
  - Proposed 0.050% HFT Criterion

#### Source Details

- **A1 to A7**
- **B1 to B7**
- **C1 to C7**

**Note:**
- The graph visually compares measured and predicted dilations for various sources, showing deviation from the criteria.
Conclusions

• The refined INDOT HFT equipment, procedures and analysis appear to provide a quick method to evaluate the freeze-thaw resistance of carbonate aggregates quarried in Indiana predicting the 90-day FT test results in 8-days.

• Testing of additional aggregate sources, and possible refinement of the regression model, the reliability of this test is expected to increase.
Significance

• ITM 210 - INDOT plans to continue testing and certifying their aggregate sources using ITM 210 every 1 to 3 years as part of their Certified Aggregate Producers (CAP) program.

• The HFT will be used as a quick approval, or confirmation of approval of certified sources. This is especially useful for quarry sources that have ledges with questionable or variable performance.

• HFT results will be a good check that INDOT receives for construction similar quality material that was tested during the certification process.
Implementation

At this time, it is recommended that the developed regression model be used as a screening test with the following criteria:

<table>
<thead>
<tr>
<th>Predicted Dilation</th>
<th>Expected Durability</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;0.050%</td>
<td>Durable</td>
</tr>
<tr>
<td>Between 0.050% and 0.060%</td>
<td>ITM210 is required to determine the aggregate durability</td>
</tr>
<tr>
<td>&gt; 0.060%</td>
<td>likely nondurable</td>
</tr>
</tbody>
</table>
Recycled Concrete Aggregate (RCA)
## RCA Sources

<table>
<thead>
<tr>
<th>Source No.</th>
<th>Structure</th>
<th>Original Aggregate Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1</td>
<td>SR26 INDOT pavement</td>
<td>Gravel</td>
</tr>
<tr>
<td>R2</td>
<td>US 52 INDOT pavement</td>
<td>Gravel</td>
</tr>
<tr>
<td>R3</td>
<td>I-65 INDOT pavement</td>
<td>Gravel</td>
</tr>
<tr>
<td>R4</td>
<td>SR19 INDOT pavement</td>
<td>Gravel</td>
</tr>
<tr>
<td>R5</td>
<td>SR912 INDOT pavement</td>
<td>Crushed carbonate rock</td>
</tr>
<tr>
<td>R6</td>
<td>Misc. structures from Indianapolis Dept. of Public Works</td>
<td>Primarily gravel, some crushed carbonate rock</td>
</tr>
</tbody>
</table>
# Analysis

## Summary of HFT and freeze-thaw test results

<table>
<thead>
<tr>
<th>RCA Source</th>
<th>Response Variables</th>
<th>Predictor Variables</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>FT test result</td>
<td>HFT test result (PCMR Values)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Dilation</td>
<td>DF</td>
<td>P34</td>
</tr>
<tr>
<td>R1</td>
<td>0.019</td>
<td>98.4</td>
<td>-14.39</td>
</tr>
<tr>
<td>R2</td>
<td>0.006</td>
<td>99.6</td>
<td>-27.46</td>
</tr>
<tr>
<td>R3</td>
<td>0.040</td>
<td>94.2</td>
<td>-10.41</td>
</tr>
<tr>
<td>R4</td>
<td>0.006</td>
<td>88.4</td>
<td>-19.24</td>
</tr>
<tr>
<td>R5</td>
<td>-0.002</td>
<td>96.0</td>
<td>-17.60</td>
</tr>
<tr>
<td>R6</td>
<td>0.640</td>
<td>30.0</td>
<td>-21.56</td>
</tr>
</tbody>
</table>
Analysis

0.060% INDOT acceptance criteria
Previous Models

The model developed for predicting freeze-thaw durability of the carbonated quarried aggregates cannot be used to predict the freeze-thaw durability of RCA

<table>
<thead>
<tr>
<th>Source</th>
<th>Dilation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measured</td>
</tr>
<tr>
<td>R1</td>
<td>0.019</td>
</tr>
<tr>
<td>R2</td>
<td>0.006</td>
</tr>
<tr>
<td>R3</td>
<td>0.04</td>
</tr>
<tr>
<td>R4</td>
<td>0.006</td>
</tr>
<tr>
<td>R5</td>
<td>-0.002</td>
</tr>
<tr>
<td>R6</td>
<td>0.64</td>
</tr>
</tbody>
</table>
A linear regression model was developed to predict the average % dilation of freeze-thaw test beams using parameters obtained from HFT results.

\[
\text{% Dilation} = -1.447 + 4.65 \times 10^{-2} P34 + 1.21 \times 10^{-1} P58 + 3.29 \times 10^{-1} P12 + 9.24 \times 10^{-1} P38
\]

**Model statistics:**

- \( R^2 = 0.999 \),
- \( R^2 \text{ (adj.)} = 0.997 \),
- \( \text{RMSE} = 0.012, n=6 \),
- \( \text{Model } P\text{-value} < 0.0316 \)
Measured vs Predicted

<table>
<thead>
<tr>
<th>Dilation</th>
<th>Measured</th>
<th>Predicted</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1</td>
<td>0.019</td>
<td>0.010</td>
</tr>
<tr>
<td>R2</td>
<td>0.006</td>
<td>0.004</td>
</tr>
<tr>
<td>R3</td>
<td>0.04</td>
<td>0.041</td>
</tr>
<tr>
<td>R4</td>
<td>0.006</td>
<td>0.008</td>
</tr>
<tr>
<td>R5</td>
<td>-0.002</td>
<td>0.006</td>
</tr>
<tr>
<td>R6</td>
<td>0.64</td>
<td>0.640</td>
</tr>
</tbody>
</table>
Conclusions

- RCA is responsive to testing in the HFT
- The models developed for predicting freeze-thaw durability of the carbonated quarried aggregates cannot be used to predict the freeze-thaw durability of RCA
It is recommended that the dilation model developed be used for research and preliminary screening purpose only.

Further HFT and FT testing of additional RCA sources that represent a greater spectrum of FT performance is needed to improve these models prior to using as part of the acceptance criteria for RCA as coarse aggregate in INDOT concrete paving projects.
Part III

Highlights of the Ongoing Tests

• Highlight of the ongoing tests
  – Objective
  – Testing
  – Analysis
  – Preliminary Conclusions/Observations

• Acknowledgements
Objectives

• To investigate the role of aggregate’s mineralogy, microstructure on the HFT result & freeze-thaw performance
• To further refine the HFT test procedure, data analysis and models developed
• To explain why some sources did not fit in the model
Acid Insoluble Residue (IR)- ASTM D3042

- IR is % by weight of silt & clay size particles when the aggregate is dissolved in 6N HCl

- No correlation between IR and dilation or DF observed

- Source B1 had the highest IR, and percent fracture in HFT

IR, %

Dilation, %
Thermal Analysis (TGA)

- B1 had the highest amount of fracture in HFT
- A3 did not fit in the model

Mass Loss:
Min. 30%, B1
Max. 53%, A3
Avg. 43%
Comparison of A3 with a particular source, A4
<table>
<thead>
<tr>
<th>Author</th>
<th>Date</th>
<th>Critical Pore Size Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shakoor 1982</td>
<td>10nm-10μm</td>
<td></td>
</tr>
<tr>
<td>Salcedo 1984</td>
<td>45nm-10μm</td>
<td></td>
</tr>
<tr>
<td>Kaneuji, Winslow and Dolch 1980</td>
<td>4.5nm-5μm</td>
<td></td>
</tr>
<tr>
<td>Dubberke and Marks 1985</td>
<td>40nm-0.2μm</td>
<td></td>
</tr>
</tbody>
</table>

Pittenger and West, 1995
Sorption Analysis

- Three sources were considered
- Dynamic Vapor Sorption (DVS) analysis conducted on about 50mg, 0.8mm thick specimen
- Mass loss was recorded by changing the RH from 97% all the way to 0%

<table>
<thead>
<tr>
<th>Source</th>
<th>Dilation</th>
<th>DF</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>0.0035</td>
<td>96.6</td>
</tr>
<tr>
<td>B1</td>
<td>0.1631</td>
<td>69.3</td>
</tr>
<tr>
<td>B2</td>
<td>0.27</td>
<td>36.2</td>
</tr>
</tbody>
</table>
Kelvin –Laplace Equation

The Kelvin–Laplace equation used to approximate the pore radius that corresponds to a specific relative humidity

\[ RH = \exp \left( \frac{-2\gamma \cos(\theta)}{r} \cdot \frac{V_m}{RT} \right) \]

where

- \( R \): universal gas constant (8.314 J/mol·K),
- \( T \): temperature (K),
- \( RH \): internal RH, and
- \( V_m \): molar volume of pore solution (≈18 \times 10^{-6} \text{ m}^3/\text{mol})

\( r_p \): pore radius
\( r_k \): Kelvin radius
\( t \): adsorbed layer thickness
Desorption Isotherm

![Graph showing desorption isotherm with points labeled A1, B1, and B2](image)
Cumulative Porosity

Cumulative Porosity
(gram of water/gram of oven dry sample), %

Kelvin radius, nm

A1
B1
B2
Preliminary Conclusions

• No correlation observed between IR and dilation or DF observed
• Incorrectly predicted source had the highest mass loss in TGA
• Cumulative porosity (pore size<80nm) determined by DVS identified durable from nondurable sources
Acknowledgements

Joint Transportation Research Program Indiana Department of Transportation
Purdue University

Indiana Aggregate Producers
Indiana Mineral Aggregates Association
American Concrete Pavement Association-ACPA Indiana chapter

Ron Walker
Steve Dick
Robert Rees
Dr. Donald Janssen
Michael Byers
Prof. Terry West
Contact

- Belayneh Desta, desta@purdue.edu
  Purdue University
  Lyles School of Civil Engineering
- Nancy Whiting, whiting@ecn.purdue.edu
  Purdue University
  Applied Concrete Research Initiative
- Jan Olek, olek@ecn.purdue.edu
  Purdue University
  Lyles School of Civil Engineering
- Mark Snyder, mbsnyder2@yahoo.com
- Tommy Nantung, tnantung@indot.in.gov
  Indiana Department of Transportation
  Division of Research and Development

Full report on Part I and II is available online:

http://docs.lib.purdue.edu/jtrp/1565/