Techniques to Fingerprint Construction Materials--R06B
XRF and FTIR Spectroscopy

Committee on Materials and Pavements
Cincinnati, OH
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Techniques to Fingerprint Construction Materials (R06B)

Challenge
• Verify materials without sampling delays

Solution
• Technologies to verify specific construction materials in real time
• Technologies that can be used at the project site to determine specification compliance
Potential Benefits

- Rapid testing on site or in the lab
- Reduce testing time and cost
- Minimize noncompliance risk
X-Ray Fluorescence Spectroscopy (XRF)

- Rapid elemental analysis of materials
- Specific application developed in R06B—testing traffic paints for Ti content
XRF Advantages and Limitations

**Advantages**
- Pre-calibrated for wide range of elements
- Automatic reading—no analysis
- 1-3-minute testing time
- Little or no sample prep required
- No maintenance required
- Numerous applications

**Limitations**
- Requires user certification
- Upper and lower limits—different calibrations needed for trace metals vs. ores
Fourier Transform Infrared (FTIR)

- Identifies compounds
- Simple testing process
- Analysis more difficult than XRF
R06B—Maine

- **XRF**
  - Chlorides in bridge deck cores
  - Titanium in traffic paint
  - REOB in PG Binder
  - SS Rebar
  - Galvanized coating thickness
  - Glass Beads – lead, arsenic
  - Presence of RAS in HMA?

- **FTIR**
  - Polymer in PG Binder
  - Library of all Binders
  - Lime in HMA
R06B—Maine

Quantitative

Qualitative
Chloride Content – Bridge Deck Cores

• Current method: AASHTO T 260 (Gran Plot Method)
  – Requires nitric acid and silver nitrate
  – Numerous steps
  – 10 tests/day

• XRF method
  – No chemicals
  – 25+ tests/day
  – Less training required
Chloride Content – XRF method
- Split-sample comparison
  - Evaluated numerous binding agents for pelletized samples, XRF settings, direct measurement of concrete
  - Selected the settings that provided the best correlation on a limited amount of measurements vs. titration values
  - Expanded population of comparisons
  - In-progress statistical validation

<table>
<thead>
<tr>
<th>Item</th>
<th>Levels</th>
<th>Details</th>
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</thead>
<tbody>
<tr>
<td>Analysis Mode</td>
<td>3</td>
<td>AllGeo and Two Mining Modes</td>
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<tr>
<td>Time Breakdown</td>
<td>2</td>
<td>5/5/5/45 &amp; 15/15/15/15</td>
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<tr>
<td>Binding Agent</td>
<td>6</td>
<td>None and 5 recommended agents</td>
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<tr>
<td>Binding %</td>
<td>2</td>
<td>5% &amp; 10%</td>
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<tr>
<td>Replicates</td>
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<td>Three measurements on each pellet</td>
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</table>
Surface Testing of Core Slices

Top, bottom, edge of slice
Average of all readings v. Titration
$R^2 \approx 0.91$

- Exposed aggregate a problem
- Higher variability
Pulverized specimens

N = 282 samples

\[ y = 0.9672x \]

\[ R^2 = 0.9659 \]
Conclusions from study
- Pellets of pulverized material superior to surface readings of slices
- No binding agent required
- In process of:
  - testing lab-prepared reference samples
  - validating correlation with independent split-sample comparisons
Stainless steel rebar

<table>
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<tr>
<th>El</th>
<th>%</th>
<th>+/- 2σ</th>
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<tr>
<td>V</td>
<td>0.110</td>
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<tr>
<td>Cr</td>
<td>23.490</td>
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<tr>
<td>Mn</td>
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<td>Fe</td>
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<td>Ni</td>
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<td>Cu</td>
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<td>Zr</td>
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<td>0.001</td>
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<td>Nb</td>
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<td>0.001</td>
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<tr>
<td>Mo</td>
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<td>0.004</td>
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<tr>
<td>W</td>
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<td>0.005</td>
</tr>
<tr>
<td>Pb</td>
<td>0.007</td>
<td>0.002</td>
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</table>
• XRF
  – Silica and Calcium Carbonate in Limestone
  – Titanium in Thermoplastic
  – Glass Beads – lead, arsenic
  – REOB in Binder?
  – Galvanized coating thickness?

• FTIR
  – Polymer in PG Binder
  – Library of all Binders
  – PPA in Binder
Heavy Metals in Glass Beads

• Current Practice:
  – Tennessee requires every lot to be tested with EPA tests 3052, 6010B, or 6010C.

• Future Method:
  – Perform XRF testing on every lot. Allow manufacturer to Certify lots to Federal Aid Standard.
Silica in Limestone

- Currently tested by standard-less program on WDXRF in Lab.

- Handheld can perform same testing but still requires a lot of sample prep to be accurate.
Titanium in Thermoplastic

• Current Practice:
  – Tennessee currently accepts thermoplastic on certification.

• Future Practice:
  – The handheld can perform verification testing in the field/lab on Thermoplastic.
  – There may be some issues with some fillers in the Thermoplastic.
Future for this Product in TN

- Looking into other materials
  - Following Maine and using XRF as a rapid test for Chloride Content of Bridge Decks.
  - Using the XRF and FTIR to detect REOB’s and PPA’s in our Binders.
  - Using the FTIR to verify Qualified Products List materials, such as Texture Coating and Additives for Asphalt and Concrete.
What’s Next for R06B

The Future

- Webinar - August 22, 2018
- Peer Exchange - September 26-27, 2018
  - https://fs6.forms site.com/Mrussell/form204/index.html
- Regional User Producer Group Meetings?
- Others?
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Additional Resources:
GoSHRP2 Website:  
fhwa.dot.gov/GoSHRP2

AASHTO SHRP2 Website:  
http://shrp2.transportation.org

R06B Product Page  
Coming soon