Effect of Laboratory Aging on Asphalt Binders and Intermediate-Temperature Cracking of Asphalt Mixtures

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- Advanced Asphalt Mixture Characterization Group
  - LTRC

Outline

- Background
- Objective
- Scope
- Experimental Design
- Results
- Summary

Background -- Aging

- Aging significantly affects stiffness and ductility properties of asphalt materials
  - Contributing factors
    - Oxidation
    - Polymerization
    - Volatilization
    - Thixotropy
    - Syneresis
    - Separation
  - Oxidative aging is major reaction for asphalt hardening
    - Stiffer/brittle asphalt mixture:
      - Increased cracking

Asphalt Mixture Design

- Volumetrics
  - Voids in the Total Mix, VTM
  - Voids in the Mineral Aggregate, VMA
  - Voids Filled with Asphalt, VFA
- Densification
  - Stages during lab compaction process

Fatigue Cracking  Block Cracking

VOLUME MASS

Total Volume  Total Mass

Air  Asphalt  Aggregate
Concerns

- Optimum asphalt binder content
  - Quantity
  - NOT QUALITY
  - Aged Binders
    » Replace virgin binder
    » RAP and/or RAS

Balance Asphalt Mixture Design (BMD)

- Evaluate
  - Quality of a mixture design relative to anticipated performance using a rational approach
- Address
  - Performance issues
    » Cracking
      - Increased aged asphalt binder
      - Use of recycled materials
      - High RAP and/or RAS
- Allow
  - Innovation in designing mixtures for performance
  - Sustainable development

Balance Asphalt Mixture Design (BMD)

- Volumetric and Performance Mixture Testing
  - Rutting (AASHTO T 324): LWT test (50°C, Wet)
  - Cracking (ASTM 8044): SCB test (25°C)

2016 Louisiana DOTD Specifications for Roads and Bridges

<table>
<thead>
<tr>
<th>Table 502-7: Asphalt Concrete General Criteria</th>
<th>SCB, min. Jc, kJ/m² @ 25°C, Aged</th>
</tr>
</thead>
<tbody>
<tr>
<td>All mix design level 1 must meet minimum 0.5 Jc</td>
<td>All mix design level 2 must meet minimum 0.6 Jc</td>
</tr>
</tbody>
</table>

Research projects to create new specification parameters


Transportation Pool Fund TPF 5(294)

Develop Mix Design and Analysis Procedures for Asphalt Mixtures Containing High-RAP Contents
**Objective**

- Evaluate fatigue/fracture tests that can be conducted on plant mixtures (lab or field compacted) from participating states
  - ranking quality of RAP and or RAP/RAS mixtures as compared to virgin mixtures.
- Develop score card

**Mixture Experiment**

- **Specimen Types**
  - Plant produced laboratory compacted (PL)
  - Plant Produced Field compacted (PF, Cores)
- **Challenging**
  - Triplicates
- **Fracture/fatigue testing**
  - Semi-circular bend test, SCB
  - Overlay Jetter test, OT
  - Energy Ratio Test
  - Beam Fatigue Test
  - Direct Tension Cyclic Fatigue
  - S-VECD
- **Per mixture and Specimen type**
  - 5 tests x 3 = 15 mixes

**Development of Ranking Score Card**

- Each test are ranked
  - Specimen preparation
  - Instrumentation
  - Standard test method
  - Testing oversight
  - Testing time
  - Training
  - Interpretation
  - Sensitivity to mix composition parameters
  - Routine Application
  - Correlation to field performance
  - Data Analysis
  - Repeatability & variability
  - Equipment cost
  - Required technical ability

**Summary of Score Card – Max. 56**

<table>
<thead>
<tr>
<th>Score</th>
<th>SCB</th>
<th>Texas Overlay</th>
<th>UF</th>
<th>IDT</th>
<th>Beam Fatigue</th>
<th>S-VECD</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Background -- Aging**

- **Asphalt mixture cracking testing**
  - Design, production, Installation
  - Long Term Aging
  - AASHTO R 30
  - Compacted samples
    - Five days, 85°C
  - NCHRP Report 871
  - 16 years field aging in South Louisiana requires
    - loose mixture at 95°C
      - 27 days (6mm)
      - 12 days (20 mm)

**Background -- Aging**

- Laboratory aging of asphalt binders
- Laboratory aging of asphalt mixtures
- Relationships to link
  - Design
  - Production
  - Construction
- Performance-based
  - QC/QA system
Objective

- Evaluate effect of laboratory aging on
  - Chemical properties of asphalt binders
  - Rheological properties of asphalt binders;
  - Intermediate-temperature cracking of asphalt mixtures

Scope:

- Materials
  - Four plant-produced mixtures
  - Two gradations: 12.5 and 19-mm NMAS;
  - Asphalt binder contents: optimum and optimum + 0.2%
  - Asphalt binder type: Louisiana PG76-22m;
  - RAP

<table>
<thead>
<tr>
<th>Mixture Characteristics</th>
<th>Mix 1</th>
<th>Mix 2</th>
<th>Mix 3</th>
<th>Mix 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC Grade</td>
<td>76-22</td>
<td>76-22</td>
<td>76-22</td>
<td>76-22</td>
</tr>
<tr>
<td>Total % AC</td>
<td>5.0</td>
<td>5.2</td>
<td>4.8</td>
<td>5.0</td>
</tr>
<tr>
<td>RAP RBR, %</td>
<td>~18</td>
<td>~18</td>
<td>~25</td>
<td>~25</td>
</tr>
<tr>
<td>Film Thickness (µm)</td>
<td>9.9</td>
<td>10.3</td>
<td>11.1</td>
<td>11.6</td>
</tr>
</tbody>
</table>

Scope:

- Asphalt Binder Experiment
  - Chemical characterization
    - Gel permeation chromatography (GPC)
  - Rheological characterization
    - Continuous high performance grading
    - Frequency sweep test at multiple temperatures
- Intermediate temperature cracking
  - Semi Circular Bend (SCB) test
  - ASTM D 8044
- Aging treatment
  - Compacted samples
  - 85°C
  - 0-, 2-, 5-, and 7-days.

Experimental Design:

- Specimen fabrication and conditioning
  - Specimens Compaction: 150 × 57 mm, 7±0.5% air voids,
    - Superpave Gyratory Compactor (SGC);
  - Cutting cylindrical specimen into two halves;
  - Placed at smooth, flat plates separately in a forced oven at 85°C.

<table>
<thead>
<tr>
<th>Aging Duration, days</th>
<th>Abbreviation</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>PLM</td>
<td>Plant-produced loose mixture</td>
</tr>
<tr>
<td>2</td>
<td>LTGA 2D</td>
<td>Long-term oven aging</td>
</tr>
<tr>
<td>5</td>
<td>LTGA 5D</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>LTGA 7D</td>
<td></td>
</tr>
</tbody>
</table>

Experimental Design:

- Extraction of asphalt binder
  - ASTM D8159
  - Trichloroethylene
  - Automated Asphalt Analyzer

<table>
<thead>
<tr>
<th>Process of Extraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
</tr>
<tr>
<td>(2)</td>
</tr>
<tr>
<td>(3)</td>
</tr>
<tr>
<td>(4)</td>
</tr>
<tr>
<td>(5)</td>
</tr>
</tbody>
</table>

Experimental Design:

- Recovery of extracted asphalt binder
  - AASHTO R 59: Abson method
  - Remove TCE
  - Distillation within temperature of 160° to 166°C
  - Protective gas environment of carbon dioxide (CO2)
**Chemical characterization: Gel Permeation Chromatography (GPC)**

- A chromatographic method of separating molecules based on their molecular size, a method analogous to aggregate sieving.
- Inject 0.25% Tetrahydrofuran (THF) sample into porous columns:
  - Molecules pass the columns:
  - 0.35 ml/min THF flow rate, at 40 °C;
- Large molecules pass first, followed by the smaller ones;
- The concentration of size-separated molecules are detected using Differential Refractive Index detector (DRI):
  - Recorded Refractive index (RI) and elution time.

**Asphalt Binder**

**Gel Permeation Chromatography (GPC)**

- **Test principle** – chromatographic method of separating asphalt molecules based on their sizes, a method analogous to aggregate sieving

**Experimental Design:**

- Chemical characterization: Gel Permeation Chromatography (GPC)
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**Asphalt Binder**

**Gel Permeation Chromatography (GPC)**

- **Test principle** – chromatographic method of separating asphalt molecules based on their sizes, a method analogous to aggregate sieving

**Asphalt Binder**

**Rheological characterization**

- Continuous high PG grade
  - Dynamic shear rheometer (DSR)
  - AASHTO R 29

**Asphalt Binder**

**Rheological characterization**

- Frequency sweep test at multiple temperatures
  - 16 frequency: 0.1 -100 rad/s, with even distribution in log scale.
  - Multiple temperatures: 45°C, 35°C, and 15°C
  - Specimen: 8 × 2 mm
  - Record dynamic shear modulus (G*) and phase angle (°)
  - Results: three isotherms → Master curve

**Asphalt Binder**

**Rheological characterization**

- Frequency sweep test at multiple temperatures
  - 16 frequency: 0.1 -100 rad/s, with even distribution in log scale.
  - Multiple temperatures: 45°C, 35°C, and 15°C
  - Specimen: 8 × 2 mm
  - Record dynamic shear modulus (G*) and phase angle (°)
  - Results: three isotherms → Master curve
**Chemical characterization: GPC Test Results**

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Aging Level</th>
<th>HMW 300K-45K, %</th>
<th>Associated Asphaltenes, 45-19K, %</th>
<th>Asphaltenes 19-3K, %</th>
<th>Maltenes&lt;3K, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 1</td>
<td>0</td>
<td>2.93</td>
<td>3.05</td>
<td>23.9</td>
<td>71.13</td>
</tr>
<tr>
<td></td>
<td>2D</td>
<td>2.91</td>
<td>3.01</td>
<td>23.9</td>
<td>71.13</td>
</tr>
<tr>
<td></td>
<td>5D</td>
<td>2.82</td>
<td>3.22</td>
<td>23.3</td>
<td>70.84</td>
</tr>
<tr>
<td></td>
<td>7D</td>
<td>2.85</td>
<td>3.49</td>
<td>23.1</td>
<td>70.65</td>
</tr>
<tr>
<td>Mix 2</td>
<td>0</td>
<td>2.97</td>
<td>3.01</td>
<td>23.6</td>
<td>70.84</td>
</tr>
<tr>
<td></td>
<td>2D</td>
<td>2.91</td>
<td>3.07</td>
<td>23.1</td>
<td>70.65</td>
</tr>
<tr>
<td></td>
<td>5D</td>
<td>2.75</td>
<td>3.36</td>
<td>23.1</td>
<td>70.84</td>
</tr>
<tr>
<td></td>
<td>7D</td>
<td>2.89</td>
<td>3.59</td>
<td>23.1</td>
<td>70.84</td>
</tr>
</tbody>
</table>

**Discussion Results**

- Continuous high PG grading results
- For each mixture type, the chart shows the continuous high PG grade and the corresponding film thickness for two different mixes.

- **Mix 1 - Optimum**
- **Mix 2 - Optimum+0.2%**

**12.5 mm NMAS Mixes**

- **Mix 3**
- **Mix 4**

**19 mm NMAS Mixes**

- **Mix 1**
- **Mix 2**
- **Mix 3**
- **Mix 4**

- **Chemical characterization: GPC Test Results**

- For each mixture, the table shows the aging level, HMW 300K-45K, %, Associated Asphaltenes, 45-19K, %, Asphaltenes 19-3K, %, and Maltenes<3K, %.

- **Mixture NMAS AC Content Film Thickness (µm)**

- **Mix 1**
- **Mix 2**

- **Discussion of Results**

- **Continuous high PG grading results**

- For each mixture type, the chart shows the continuous high PG grade and the corresponding film thickness for two different mixes.

- **Mix 1 - Optimum**
- **Mix 2 - Optimum+0.2%**

- **12.5 mm NMAS Mixes**

- **19 mm NMAS Mixes**
Discussion of Results

- Continuous high PG grading results
  - 19 mm NMAS Mixes

Discussion of Results

- Frequency sweep test at multiple temperatures
  - Master Curves
  - G-R Parameter
  - Measurement of ductility
    - $|G'|$ and $\delta$ values (15°C and 0.005 rad/s)

Discussion of Results

- G-R Parameter Results
  - 12.5 mm NMAS Mixes
### Discussion of Results

#### G-R Parameter Results

<table>
<thead>
<tr>
<th>Mixture Type</th>
<th>NMAS</th>
<th>AC Content</th>
<th>Film Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 1</td>
<td>12.5</td>
<td>5.0</td>
<td>9.9</td>
</tr>
<tr>
<td>Mix 2</td>
<td>12.5</td>
<td>5.2</td>
<td>10.3</td>
</tr>
<tr>
<td>Mix 3</td>
<td>19</td>
<td>4.8</td>
<td>11.1</td>
</tr>
<tr>
<td>Mix 4</td>
<td>19</td>
<td>5.0</td>
<td>11.6</td>
</tr>
</tbody>
</table>

#### Semi-Circular Bend Test Results, 25°C

<table>
<thead>
<tr>
<th>Mixture Type</th>
<th>Aging Duration, days</th>
<th>Jc, kJ/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 1-Optimum</td>
<td>0 - days</td>
<td>0.78</td>
</tr>
<tr>
<td>Mix 2-Optimum+0.2%</td>
<td>2 - days 85C</td>
<td>0.55</td>
</tr>
<tr>
<td>Mix 3-Optimum</td>
<td>5 - days 85C</td>
<td>0.51</td>
</tr>
<tr>
<td>Mix 4-Optimum+0.2%</td>
<td>7 - days 85C</td>
<td>0.50</td>
</tr>
</tbody>
</table>

### Discussion of Results

<table>
<thead>
<tr>
<th>Mixture NMAS AC Content Film Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 1 12.5 5.0 9.9</td>
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<tr>
<td>Mix 3 19 4.8 11.1</td>
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<tr>
<td>Mix 4 19 5.0 11.6</td>
</tr>
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</table>

### Discussion of Results

**Correlation between SCB Jc and G-R Parameter**

\[
y = -0.0045x + 1.3175 \\
R^2 = 0.89 \\
y = -0.0073x + 1.2076 \\
R^2 = 0.86
\]
**Aging Factor**

- **Superpave asphalt PG**
  - Rutting factor: $G' \sin \delta$;
  - Original binder: 1.0 kPa;
  - RTFO binder (STA): 2.2 kPa;
- **Aging factor = 2.2**

**Summary**

- **GPC results**
  - Increased asphaltenes and reduced maltenes fractions in asphalt binder composition with an increase in aging treatment
- **G-R parameter showed increased with aging treatment**
  - SCB $J_c$ values decreased with aging.
  - Addition of 0.2% AC increased SCB $J_c$ values
  - Improve cracking resistance
- **Strong correlation B/W G-R parameter and SCB $J_c$ at the adding levels considered**
  - G-R has potential to be used as an aging factor