Recycled Engine Oil Bottoms as Asphalt Binder Additive

Terry Arnold & Nelson Gibson

North Central Asphalt User Producer Group Feb 3 2015
Acknowledgments

• State DOTs and FHWA Federal Lands

• Crystal Clean / Heritage Research Group

• Safety-Kleen

• SES Group / Turner Fairbank Highway Research Center
  – Anant Shastry
  – Susan Needham
  – Scott Parobeck
  – Frank Davis
  – Adrian Andriescu
  – Xinjun Li
  – Lakesha Perry
AASHTO Task Force

Christopher Abadie  Louisiana DOT
Bill Ahearn  Vermont DOT (leader)
Terry Arnold  FHWA
Richard Bradbury  Maine DOT
Matthew Corrigan  FHWA
Nelson Gibson  FHWA
John Grieco  Massachusetts DOT
Matt Mueller  Illinois DOT
Christopher Peoples  North Carolina DOT
Michael San Angelo  Alaska DOT
Eileen Sheehy  New Jersey DOT
AASHTO Task Force Goals and Objectives

1. Develop a Common Understanding of the published information about REOB – mid January

2. Finalize a State by State summary of REOB specification/use status IBNLT known or unknown – end January

3. Define data gaps in knowledge and timeframes for resolution – mid February

4. Develop consensus on risk and recommended action – end February

5. Finalize response to SCOH – early March
Recycled Engine Oil Bottoms are Liquids at Room Temperature
Their Brookfield Viscosities Differ Widely Between Producers

- Producer A: 257.3 cps @ 135°C
- Producer B: 28.2 cps @ 135°C
Lubricating Oil additives
XRF-Spectrometer
Variation and Complications 
*between and within REOB Suppliers*

- Phosphorous: 1.5 - 1.9%
- Sulfur: 1.5 - 1.9%
- Calcium: 7,204 - 10,901 ppm
- Iron: 372 - 1,838 ppm
- Copper: 704 - 1,563 ppm
- Zinc: 4,554 - 7,213 ppm
- Molybdenum: 288 - 669 ppm
Variation and Complications – Asphalt

• XRF Phosphorous and Sulfur Peaks Overlap
• Sulfur 3.05 - 11.49%
• Iron 8 - 115 ppm
• Molybdenum 0 - 15.7 ppm
• May contain Zinc H₂S Scavengers
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<th>GTR</th>
<th>Asphalt</th>
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XRF Analysis of REOB Modified Binder

- **Calcium**
  
  \[ y = 72.616x - 159.01 \]
  
  \[ R^2 = 0.9996 \]

- **Zinc**
  
  \[ y = 47.826x - 5.9077 \]
  
  \[ R^2 = 0.9998 \]

- **Molybdenum**
  
  \[ y = 5.4177x + 7.369 \]
  
  \[ R^2 = 1 \]

- **Copper**
  
  \[ y = 7.463x - 19.525 \]
  
  \[ R^2 = 1 \]
XRF Analysis of GTR Modified Binder

\[ y = 156.87x + 13.386 \]

\[ R^2 = 0.9911 \]
## REOBO Content of Binders

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Conclusions on XRF Analysis

• You can readily detect REOB presence

• You cannot tell exactly how much is there
Field study

- FHWA/ARC/WRI and FHWA/ARC/NCAT Validation sites in the US and Canada -> MN site
- Only difference between the sites is the binder used??
Field Study - Rochester, MN Comparative Test sites

- 2012 Distress Data (500 feet test sites)

**Transverse Cracking**
- Low Severity: MN1-2, MN1-3
- Moderate Severity: MN1-4, MN1-5
- 8% REOB

**Low Severity Fatigue Cracking**
- 8% REOB

**Longitudinal Cracking**
- Non-Wheel Path
- 8% REOB

**Rutting**
- Average Rut Depth (mm)
- 8% REOB
Low Severity Transverse Cracking

MN1-2

MN1-2

MN1-4
8% REOB

MN1-5

*NOT ACTUAL CRACK MAPS* - GRAPHICAL REPRESENTATION OF DATA TO SCALE
Low Severity Non-Wheelpath Longitudinal Cracking

MN1-2

MN1-2

MN1-4

8% REOB

MN1-5

NOT ACTUAL CRACK MAPS - GRAPHICAL REPRESENTATION OF DATA TO SCALE
Low Severity Fatigue Cracking

MN1-2

MN1-2

MN1-4
8% REOB

MN1-5

NOT ACTUAL CRACK MAPS - GRAPHICAL REPRESENTATION OF DATA TO SCALE
Western Research Institute WRI
Atomic Force Microscopy AFM

Average Diameter of Human Hair

100 μm

Binder Microstructure study: AFM

- Neat AAG-1 topography

Typical neat AAG surface Nearly flat and featureless
Binder Microstructure study: AFM

- AAG-1 + REOB topography

Topographic image indicates a relatively smooth flat surface with a number of small “holes”
Binder Microstructure study: AFM

- AAG-1 + REOB topography + Aging

Topographic image indicates a relatively smooth flat surface with more small “holes”
Summary from WRI

• A preliminary study on a very limited sampling, still on going at WRI

• However some interesting findings:
  ▪ REOB not inert – affects microstructure, properties and aging
  ▪ Microstructure: 2-phase structures (at least) – “holes” occurring and expanding over aging
Binders’ and Mixtures’ Engineering Properties
Two Modification Approaches

• Softening an unmodified PG to another PG

PG64-22

6% REOB*

PG58-28

*with a single REOB sample
Two Modification Approaches

• Softening an unmodified PG to another PG

\[ \text{PG64-22} \downarrow \quad 6\% \text{ REOB}^* \]
\[ \text{PG58-28} \]

• Diluting a unmodified PG

\[ \text{PG69-24} \]
\[ +20\% \text{ PG100-0} \quad +15\% \text{ REOB}^* \]
\[ \text{PG58-28} \quad \text{PG58-28} \]

\[ \text{PG58-28} \quad \text{PG58-28} \]
\[ +2.5\% \text{ REOB}^* \]

*with a single REOB sample
- **DSR High Temp**
  ~9% REOB per PG Grade Drop
- DSR High Temp
  ~9% REOB per PG Grade Drop
- BBR m-Value
  ~21% REOB per PG Grade Drop
• **DSR High Temp**
  ~9% REOB per PG Grade Drop

• **BBR m-Value**
  ~21% REOB per PG Grade Drop

• **BBR Stiffness**
  ~9% REOB per PG Grade Drop
**BBR** $\Delta T_{\text{critical}}$ **Spread:** $\text{PG}^{(S)}$tiffness – $\text{PG}^{(m)}$-creep

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<th>Final Blends</th>
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<td>PAV</td>
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<td>PAV</td>
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<tr>
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<td>-2.0°C</td>
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<td>-0.8°C 0% / 20% 69-24</td>
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**Note:**
- PAV: Pennsylvania Association of Testing Laboratories
- BBR: Brookfield Rheometer
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**BBR ΔT_{critical} Spread:** \( PG^{(S)}_{\text{tiffness}} - PG^{(m)}_{\text{creep}} \)
DSR Fatigue: Linear Amplitude Sweep (LAST)
Notched Tension: Cracking Strain Tolerance

![Graph showing DENT Strength vs. DENT CTOD with various markers and annotations.]

Holly 58-28
15% REOB
20% PG100-0

Better Cracking Performance

P

δ

BP 64-22
6% REOB

Holly 58-28
2.5% REOB
Ongoing Mixtures’ Experimental Design

• “Moisture Damage”
  – Granite- Occoquan, VA
  – Tensile Strength Retained TSR
  – Hamburg Wheel Tracking
  – Repeated With & Without Liquid Amine Anti-strip or Hydrated Lime

• “Structural Performance”
  – ALF 22% RAP Mix
  – Flow Number; confined NCHRP 9-30A
  – Dynamic Modulus, |E*|
  – Uniaxial Fatigue - Short and Long-Term Aged (loose mix 5 days @ 85°C)
  – Thermal Stress Restrained Specimen TSRST - Short and Long-Term Aged (loose mix 5 days @ 85°C)
- Holly 58-28 Control + Antistrip
- Holly 58-28 Control + 15% REOB +20% Pitch + Antistrip
- BP 64-22 + 6% REOB + Antistrip
- Holly + 2.5% REOB + Antistrip
Conclusions (1 of 4)

1. You can readily detect REOB presence
2. You cannot tell exactly how much is there
3. Effect of REOB depends on base binder (like PPA)
4. Variation between REOB suppliers & their samples
   – Same concentration can produce different PG grades
Conclusions (2 of 4)

6. 2 X PAV is a reasonable approximation of 5 years - where anecdotal concerns lie

7. REOB softens and reduces tensile strength
   - Binder notched tension (DENT)
   - Decreases mix wet and dry IDT strength

8. In 2 of 3 cases, REOB improved binder intermediate temperature parameters for fatigue / strain tolerance
   - 6% and 2.5% REOB blends
   - CTOD and LAST
Conclusions (3 of 4)

9. Rheological “disruption” occurred w/ highest %REOB
   - Differences in Low Temperature m&S
   - Did Not occur in blend with PG100-0
   - Did occur in blends with high-REOB
   - Made worse by continued aging
   - Alludes to performance deterioration
   - Corroborated by DENT CTOD & LAST & Stripping
   - Forces the issue of compatibility (extenders, rejuvenators, RAP / RAS, WMA...)

10. Consider specification change to BBR m & S
Conclusions (4 of 4)

10. REOB effects on Moisture Sensitivity

- TSR ratio, strength and Hamburg performance decreases with increasing REOB when no anti-strip is added
- REOB did not interfere with liquid anti-strip which improved TSR and Hamburg performance
- Consistent results from T283 and Hamburg
  - different conditions: hot/no-freeze and cold-freeze
- Notably, liquid ant-strip (0.4%) alters IDT strength and Hamburg deterioration more than REOB (2.5%-15%)

Ongoing experiments on mixture performance will be finishing February-March 2015
Thank You.

Questions?
Awareness of long-term performance

- Utility of PAV to approximate 5-years age
- Poor performance after 5-years *anecdotally* attributed to REOB
- Data from FHWA ALF test sections
  - Top and bottom 1-inch of core extracted & recovered binder
Exploratory practices using 2 x PAV is a good step in the right direction.
Aging study: High Pressure DSC

Typical PDSC results - Heat flow rate overlay

PDSC Conditions
- 150°C
- 550 psi
- O₂

Heat Flow, W/\text{g}

Time, minutes

AAG-1
MN1-4
AZ1-1
Overlap of Phosphorous and Sulfur Peaks

Sulfur Kα