Developing a Fundamental Understanding of the Chemistry of Warm Mix Additives

TxDOT Project 6591

Education Material
Handouts summarizing the project goals, methods, and findings

Please use presentation mode to review the slides and contents

Project Objectives and Research Approach

Evaluate influence of WMA additives on:
1. Chemical properties of the binder
2. Rheological properties of the binder

Synthesize information to assess expected mixture properties

Limited validation based on:
1. Engineering properties of the mixture
2. Mixture performance

Synthesize information to provide final recommendations
Tasks

Task 1. Review on state of the art and literature search
Task 2. Selection of materials, WMA additives, and processes
Task 3. Chemical and physio-chemical properties
Task 4. Rheology of binders and performance
Task 5. Performance of mixtures
Task 6. Recommendations for the use of WMA mixtures
Task 7. Final report

Task 1: Review on state of the art and literature search

• Summary of literature review
• “Quick reference” table of findings

Refer to interim report for a full summary from the review
**Task 1: Review on state of the art and literature search**

**Summary**

<table>
<thead>
<tr>
<th></th>
<th>Mixing and compaction</th>
<th>Rutting resistance</th>
<th>Moisture damage resistance</th>
<th>Fatigue cracking resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical (Evotherm)</td>
<td>Similar</td>
<td>Similar to lower</td>
<td>Mostly lower</td>
<td>Not reported</td>
</tr>
<tr>
<td>Wax based (Sasobit)</td>
<td>Similar</td>
<td>Similar to better</td>
<td>Mixed results</td>
<td>Better</td>
</tr>
<tr>
<td>Particle foaming (Aspha-min)</td>
<td>Similar</td>
<td>Mixed results</td>
<td>Mixed results</td>
<td>Not reported</td>
</tr>
<tr>
<td>Direct foaming</td>
<td>Similar</td>
<td>Similar to lower</td>
<td>Similar to lower</td>
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</tbody>
</table>

Note 1: In some cases WMA did not perform as well as HMA but still met the specification criteria
Note 2: Most results are based on mixture performance

**Tasks**

Task 1. Review on state of the art and literature search

Task 2. Selection of materials, WMA additives, and processes

Task 3. Chemical and physio-chemical properties

Task 4. Rheology of binders and performance

Task 5. Performance of mixtures

Task 6. Recommendations for the use of WMA mixtures

Task 7. Final report
Task 2: Selection of materials and additives

Selection of Binders

Objective:
Select binders to have diversity in terms of chemical makeup and potential chemical interactions

Diversity in chemical makeup:
(i) Based on wax content
(ii) Based on natural acid content

Total of 32 different asphalt binders from Texas were included in the screening process
### Task 2: Selection of materials and additives

#### Selection of Binders: 1 of 2 Based on wax content

**Differential Scanning Calorimeter Thermograph**

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Crystallization Onset °C</th>
<th>Crystallization Exotherm J/g</th>
<th>Melt Midpoint °C</th>
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<th>Remarks</th>
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<tr>
<td>1001-38 G</td>
<td>47.44</td>
<td>1.71</td>
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<td>Valero</td>
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<td>5.33</td>
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<td>High wax</td>
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<td>1001-40 K</td>
<td>46.16</td>
<td>3.27</td>
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<td>31.22</td>
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- **High wax**: Indicates a high wax content.
- **Low wax**: Indicates a low wax content.

**Legend**

- **Exotherm**: Indicates the onset of crystallization.
- **Melt**: Indicates the midpoint of the melt phase.
- **Endotherm**: Indicates the end of the melt phase.

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**Task 2: Selection of materials and additives**

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Task 2: Selection of materials and additives

**Selection of Binders: 2 of 2 Based on acid content**

- 0.1M TBAH was used as the titrant
- Titrant was standardized with benzoic acid every day before titration experiments
- Samples were dissolved in an organic solvent (a mixture of ethyl alcohol and chlorobenzene)
- A pH electrode was used to measure the potential change of the system during a titration experiment
- A titration curve was curve-fitted with five-parameter sigmoid functions and then took first derivative to determine inflection points
- Acid numbers were calculated by the amounts of titrant consumed at the inflection points

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Task 2: Selection of materials and additives

**Selection of Binders: 2 of 2 Based on acid content**
Task 2: Selection of materials and additives

Selection of Binders: 2 of 2 Based on acid content

Total Acid Numbers

Asphalt binders with high acid content:
PG 76-28 from Valero
PG 64-22 from Calumet

Asphalt binders with low acid content:
PG 64-22 from Valero
PG 70-22s from Valero
Task 2: Selection of materials and additives

Selection of Aggregate

Previous studies had shown that siliceous aggregates lose surface water at WMA temperatures, BUT
Do porous aggregates retain absorbed moisture at the same rate when dried at these two temperatures?

Results suggest that the aggregates lose approximately the same amount of moisture at the same rate when dried at these two temperatures.
Task 2: Selection of materials and additives

Selection of Additives

Additives for binders with different wax contents
- Controls A & B – Neat Binders
- Sasobit®
- Cecabase RT 945
- Evotherm DAT
- Evotherm 3G
- Rediset® WMX

Additives for binders with different acid contents
- Controls A & B- Neat binders
- Advera WMA
- Cecabase RT 945
- Dehydrated Advera WMA

Note: Additives mixed with acid based binders were selected based on their alkalinity.
Task 2: Selection of materials and additives

Mixing of additives to the asphalt binder

- RW 20 Digital Overhead Mixer.
- Manufacturers recommendation
  - Dosage of additives
  - Mixing temp
  - Mixing time

Digital controller for mixing speed

Temperature jacket that fits around quart can with temperature controller

Tasks

Task 1. Review on state of the art and literature search
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Task 3. Chemical and physio-chemical properties
Task 4. Rheology of binders and performance
Task 5. Performance of mixtures
Task 6. Recommendations for the use of WMA mixtures
Task 7. Final report
Task 3: Chemical properties

Main Objectives

- Select asphalt binders based on their chemical make up
- Identify chemical interactions between asphalt binders and WMA additives
- Investigate reasons behind observed performance

Test Methods

- DSC (wax content)
- Acid titration (acid content)
- FTIR (carbonyl area as a measure of oxidative aging)

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Task 4: Rheological properties of binder

Two test plans: (i) Binders with varying wax content
(ii) Binders with varying acid content

Binders (4)
PG76-28 HW  PG76-22 HW  PG64-22A LW  PG64-22B LW

WMA Additives (7)
Controls A&B  Sasobit  Cecabase RT 923  Evotherm DAT  Evotherm 3G  Rediset WMX

Un-aged  RTFO-aged (170 min)  RTFO-aged (85 min)

Viscosity  Viscosity  Viscosity  PAW-aged  Complex Modulus using DSR  MSCR Using DSR

Viscosity  Viscosity  Viscosity  PAW-aged  Complex Modulus using DSR  MSCR Using DSR

S and m-value using BBR  Complex modulus Using DSR  Tensile Strength Using Tension-Compression DMA

Workability  Long haul or storage  Low temperature cracking

High temperature rutting  Intermediate temperature fatigue

Intermediate temperature fatigue

Low temperature cracking

Tensile Strength Using Tension-Compression DMA
**Task 4: Rheological properties of binder**

**Two test plans:**

(i) Binders with varying wax content

(ii) Binders with varying acid content

Binders (4)
- PG76-28 HA
- PG64-22C HA
- PG70-22s LA
- PG64-22D LA

Modifiers (5)
- Controls A&B
- Cecabase RT 923
- Advera Dehydrated-Advera

RTF0-aged (85 min)

Viscosity

PAV-aged

Complex Modulus using DSR

MSCR Using DSR

High temperature rutting

Intermediate temperature fatigue

Workability

S and m-value using BBR

Complex modulus Using DSR

Low temperature cracking

Intermediate temperature fatigue

High temperature rutting
Task 4: Rheological properties of binder

**Controls and terminology**

- Control A = Binder RTFO aged at 163°C (current standard)
- Control B = Binder RTFO aged at 143°C
- **WMA binder** = Binder + Additive RTFO aged at 143°C

Control A vs. Modified binders → Influence of additives + reduced mixing temperatures
Control B vs. Modified binders → Influence of additives

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### Task 4: Rheological properties of binder

**Viscosity**

1. Viscosity of unaged binder (mixing workability) - *some or no reduction in viscosity*

2. Viscosity of RTFO aged binder (compaction workability) – *some or no reduction in viscosity*

3. Viscosity of extended RTFO aged binder (long hauls or silo storage) – **WMA additives were very beneficial especially for the two PG 76 binders** that had very high viscosities if stored at HMA temperatures for long durations of time
Task 4: Rheological properties of binder

1. Results are based on $G^*/\sin \delta$ and MSCR non-recoverable compliance

2. Sasobit typically compensated for the reduced stiffness associated with reduced aging (3 out of 4 cases); this was consistent for both parameters.

3. WMA additives other than Sasobit typically had a $G^*/\sin \delta$ that was 80% or less than that for a corresponding HMA binder (distribution based on 24 binder-additive pairs shows in Figure).

![Diagram of Stiffness vs. Permanent Strain](image-url)
Task 4: Rheological properties of binder

<table>
<thead>
<tr>
<th>Viscosity</th>
<th>Rutting</th>
<th>Fatigue Cracking</th>
<th>Low Temp Cracking</th>
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4. **WMA additives** (other than Sasobit) **further aggravated the reduction in stiffness due to reduced aging** (in other words for about half the additive-binder combinations the stiffness loss was due to the additive itself and not due to reduced aging).

5. Results based on MSCR were similar to results based on G*$/sin\delta$ but more sensitive.

6. **PG 76 binders (high wax)** were very sensitive to reduced aging and additives (other than Sasobit); Jr increased by 2 to 8 times due to reduced aging and 5 to 50 times due to addition of additives and reduced aging!!

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Task 4: Rheological properties of binder

1. All combinations were evaluated using G*$sin\delta$ at intermediate temperatures and a subset were also evaluated using direct tensile fracture test.
### Task 4: Rheological properties of binder

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1. All combinations were evaluated using $G\sin\delta$ at intermediate temperatures and a subset were also evaluated using direct tensile fracture test

2. **Sasobit modified WMA binders had similar stiffness as HMA** (3 of 4 cases) at intermediate temperatures; therefore a WMA with Sasobit and RAP may be at risk for cracking

3. WMA additives other than Sasobit typically had a $G\sin\delta$ that **less than that for a corresponding HMA binder** (distribution based on 24 binder-additive pairs show in Figure)

![Ratio of $G\sin\delta$ of WMA binder to corresponding HMA binder](image)

**Desired**

4. Results based on fracture / strength testing of binder **were very different**

5. In most cases the **WMA binders had a reduced strength** (energy until failure) compared to the corresponding HMA (distribution based on 16 binder-additive pairs show in Figure)

![Ratio of $G\sin\delta$ of WMA binder to corresponding HMA binder](image)

**Desired**
Task 4: Rheological properties of binder

<table>
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</table>

1. All combinations were evaluated using stiffness and m-value at low temperatures
2. WMA binders were very similar in terms of stiffness and m-value to HMA binders
3. WMA binders with Sasobit showed a slight increase in susceptibility to low temperature cracking based on these parameters

Why is there no influence of reduced short-term aging temperature after long-term?
Will addition of RAP to WMA increase susceptibility to low temperature cracking?
Task 4: Rheological properties of binder

Viscosity  Rutting  Fatigue Cracking  Low Temp Cracking

CA or carbonyl area is a semi-quantitative measure of the extent of oxidation in a binder.
**Task 4: Rheological properties of binder**

Results suggest that extent of oxidation in WMA binders is catching up to the extent of oxidation in the control binders over time. A note of caution here is that for each binder-additive pair the relationship between oxidation and stiffness could be different.

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**Task 4: Rheological properties of binder**

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*Why is there no influence of reduced short-term aging temperature after long-term?*

*Will addition of RAP to WMA increase susceptibility to low temperature cracking?*
**Task 4: Rheological properties of binder**

**Influence of RAP on WMA binder rheology**

The use of artificial RAP allows testing and interpretation of results in the "best case scenario" where the RAP binder and original binder are chemically compatible and completely co-mingle.

**Influence of RAP on WMA binder rheology – LOW TEMPERATURE**

- PG 64-22
- Low temperature grade based on m-value and estimated temperature sensitivity
- Best case scenario (25% RAP binder fully blended and same base binder)
Task 4: Rheological properties of binder

Influence of RAP on WMA binder rheology – LOW TEMPERATURE

- PG 64-22
- Low temperature grade based on m-value and estimated temperature sensitivity
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Task 4: Rheological properties of binder

Influence of RAP on WMA binder rheology – LOW TEMPERATURE

- PG 76-28
- Low temperature grade based on m-value and estimated temperature sensitivity
- Best case scenario (25% RAP binder fully blended and same base binder)
Task 4: Rheological properties of binder

Influence of RAP on WMA binder rheology – LOW TEMPERATURE

- PG 76-28
- Low temperature grade based on m-value and estimated temperature sensitivity
- Best case scenario (25% RAP binder fully blended and same base binder)

Task 4: Rheological properties of binder

Side note on MWV data (Evotherm) …

Claim (implicit): With WMA you could use RAP and the low temperature grade is preserved and with HMA you need to use one grade lower because the low temperature grade increases

1. HMA binder could have started out as a -29 and ended up as a -27.8
2. WMA binder could have started out as a -27.5 and ended up as a -26.1
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Task 5: Performance of mortars / mixtures

How we plan to test the mortar?

- Compaction in a Superpave gyratory compactor
- Ends of the compacted sample are removed using a saw
- About 15 to 20 test specimens are cored from a single SGC compacted sample
Task 5: Performance of mortars / mixtures

How we plan to test the mortar?

Task 5: Performance of mortars / mixtures

Why test the mortar?

1. There is no binder test for moisture damage
2. Validate the observations and expected performance for fatigue cracking based on binder tests
3. Allows for larger number of test replicates
Task 5: Performance of mortars / mixtures

Mix design for mortar or FAM

Fine Aggregate Mixtures

1. How do WMA additives affect fatigue cracking?

- Controls were cured at mixing temperature and others at (mixing temperature – 20°C)
- Four hour curing time was used for all mixes
- ★ indicates significantly different at α=0.05 compared to HMA using Fisher’s LSD
- All tests are stress controlled
Task 5: Performance of mortars / mixtures

Fine Aggregate Mixtures

2. What happens to fatigue cracking resistance after long-term aging?

- Long-term aging of mixtures or mortars is not straightforward
- We used PAV level of aging as a benchmark
- For thin films of binder, about 30 days at 60C reaches the same level of oxidative aging as the PAV
- Loose mix was aged for 30 days

![Graph showing fatigue cracking resistance after long-term aging]

- Controls were cured at mixing temperature and other at mixing temperature – 20C; all mixes were then aged as loose mixes at 60C for 30 days
- ★ indicates significantly different at α=0.05 compared to HMA using Fisher’s LSD
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Task 5: Performance of mortars / mixtures

Fine Aggregate Mixtures

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- ⭐ indicates significantly different at α=0.05 compared to HMA using Fisher’s LSD
- All tests are stress controlled

3. Does the rank order change after long-term aging?

- Based on mortars with one type of aggregate only
- Other studies on mixtures indicate that for a given aggregate different binders aged at consistent rates in mixtures (Morian et al. 2011)
Task 5: Performance of mortars / mixtures

Fine Aggregate Mixtures

4. What about moisture sensitivity?

![Graph showing moisture sensitivity](image)

Task 5: Performance of mortars / mixtures

Full Asphalt Mixtures

1. What is the influence of WMA on rutting and moisture damage at high temperatures?

2. What is the influence of WMA on stiffness at intermediate temperatures?

3. What is the influence of WMA on fracture at low temperatures?
Task 5: Performance of mortars / mixtures

Full Asphalt Mixtures

1. What is the influence of WMA on rutting and moisture damage at high temperatures?

![Graph showing number of load cycles vs rut depth for different asphalt mixtures. The graph compares Control, Sasobit, Evotherm 3G, and Advera for PG 64-22 and PG 76-28 asphalt mixtures. The x-axis represents the number of load cycles, and the y-axis represents rut depth (mm). The graph shows the performance of the mixtures under varying conditions, indicating the effectiveness of WMA in managing rutting and moisture damage.]
**Task 5: Performance of mortars / mixtures**

**Full Asphalt Mixtures**

1. What is the influence of WMA on rutting and moisture damage at high temperatures?

![Bar chart showing number of cycles to reach 12.5 mm rut depth for various binders.]

- Jnr was lower compared to control.
- Jnr was similar compared to control.
- Jnr was slightly lower compared to control.
- Jnr was much higher compared to control (this was the only exception in binder testing with Sasobit).

**2. What is the influence of WMA on stiffness?**

![Graph showing load and micro strain for various conditions.]

- Load vs Time in seconds.
- Micro Strain vs Time in seconds.
2. What is the influence of WMA on stiffness?

**Task 5: Performance of mortars / mixtures**

**Full Asphalt Mixtures**

Percentage reduction in $E^*$ compared to HMA control
(all measurements were at 24C)

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**Task 5: Performance of mortars / mixtures**

**Full Asphalt Mixtures**

Percentage reduction in $E^*$ compared to HMA control
(all measurements were at 24C)
Task 5: Performance of mortars / mixtures

Full Asphalt Mixtures

2. What is the influence of WMA on stiffness?

![Graph showing percentage reduction in $E^*$ compared to HMA control.](image)

PG 64-22
PG 76-28
Percentage reduction in $E^*$ compared to HMA control (all measurements were at 24°C)

Task 5: Performance of mortars / mixtures

Full Asphalt Mixtures

3. What is the influence of WMA on fracture at low temperatures?

![Graph showing load and displacement data.](image)
Task 5: Performance of mortars / mixtures

Full Asphalt Mixtures

3. What is the influence of WMA on fracture at low temperatures?

![Graph showing peak load to failure in kN for different binders and temperatures.]

- PG 64-22
- PG 76-28

Broader conclusions – Binder testing

Viscosity / Workability
- WMA additives seem to be very helpful, especially for longer storage/haul of mixes with PG 70 or 76 grade

Early age rutting resistance:
- Except for Sasobit (in most cases), WMA mixtures may be susceptible to rutting unless specifically designed
- Reduced rutting resistance was not just due to reduced aging but also due to the additives themselves
Broader conclusions – Binder testing

**Long-term aged stiffness and cracking resistance**

- After long-term aging, stiffness and relaxation of WMA modified binders tend to be similar to conventional binders

**Implications of using RAP**

- Use of WMA + RAP with WMA can help mitigate initial risk for rutting but may not provide any significant benefits over HMA + RAP.

Broader conclusions – Mortar and mixture testing

**Fatigue Cracking**

- WMA additives reacted differently with different binders in terms of their fatigue cracking resistance after long-term aging
  - PG64 showed reduced fatigue cracking resistance
  - PG76 showed no significant change
  - Consistent with binder fracture for PG64 measured at similar temperature

- Indirect tensile strength of short-term aged mixtures either did not change or reduced slightly depending on the binder – WMA additive pair

- General trends reported in the literature for different additives and binder combinations
  - WMA improved cracking resistance (Johnston et al. 2006)
  - WMA resulted in similar cracking resistance (Kristjansdottir 2007, Gandhi 2008, Johns 2009, D’Jones 2010)
  - WMA has mixed influence on cracking resistance (Haggag 2011)
Broader conclusions – Mortar and mixture testing

**Modulus / Stiffness**

- Stiffness of WMA mixtures was similar or lower compared to HMA at different frequencies
- WMA additives were more sensitive to stiffness reduction at lower rates of loading (equivalent to higher temperatures)
- Binder absorption in aggregate had a significant effect on mixture stiffness

**Rutting**

- WMA mixtures were generally more susceptible to rutting
- Rutting results from Hamburg were qualitatively consistent with binder testing with WMA additives

**Moisture induced damage**

- Based on mixture and mortar testing WMA mixtures had similar or less resistance to moisture induced damage compared to the control; this does not necessarily imply that the WMA mixtures will fail requirements
- The above findings are generally consistent with the literature