LABORATORY TESTING OF ORGANIC AND CHEMICAL WARM MIX ASPHALT TECHNOLOGIES

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ABSTRACT
Warm Mix Asphalt (WMA) technologies allow significant lowering of the production and paving temperature of the conventional Hot Mix Asphalt (HMA), which promise various benefits, e.g. lowering the greenhouse gas emissions, reduction of energy consumption, improved working conditions, better workability and compaction, etc. However, in order to reach widespread implementation of WMA, it is necessary to prove that it has the same or better mechanical characteristics and long-term performance as HMA.

This article presents a laboratory study that has been conducted to evaluate two different WMA technologies – chemical (using Rediset WMX®) and organic (using Sasobit®). The aim of the research is to investigate the changes in bitumen consistency after the modification with WMA additives, to determine the physically-mechanical properties of asphalt after reduction of compaction temperature and to compare the qualities of WMA with those of conventional HMA.

The properties of two types of bitumen after modification with two different dosages of each WMA additive have been tested by traditional empirical test methods and with the Dynamic Shear Rheometer for a wide temperature range. The results suggest that the binder, containing Sasobit®, at in-service temperatures and short loading times that are typical for traffic, has improved elasticity and lower viscosity if compared to pure bitumen. Rediset WMX®, however, had only minor effect on the binder properties suggesting that the additive changes the interaction between bitumen and the aggregates rather than bitumen itself and therefore should be evaluated in context with the designed asphalt mixture.

Asphalt testing has been performed for Stone Mastic Asphalt mixture. At first, the necessary changes in testing conditions were determined by means of asphalt stiffness – the results suggested that for adequate comparison with reference HMA, two hour asphalt aging is essential before preparing test specimens. The properties of asphalt were determined for specimens that were prepared at four different compaction temperatures by means of two compaction methods – Marshall hammer and gyratory compactor. The test results showed poor correlation between the compaction methods in most of the tests. However, in general the results of compactibility and density suggest that modifying the bitumen with WMA additives allows reducing the compaction temperature by at least 30°C and still reaching similar compaction level as for HMA. The properties of stiffness and resistance to permanent deformations also show that it is possible to reduce the compaction temperature of 155°C for HMA to 125°C for both WMA products with maintaining similar mechanical characteristics as for HMA.

KEYWORDS: Warm Mix Asphalt, rheology, stiffness, aging, permanent deformations
INTRODUCION
The modern Warm Mix Asphalt (WMA) technologies have the potential to reduce the production temperature by 20°C up to 50°C from the conventional Hot Mix Asphalt (HMA) without compromising the performance of asphalt (1). This can be achieved by different production principles which depending on the technology can be categorized in three groups:

1. Foaming technologies;
2. Organic or wax technologies;
3. Chemical additives.

The foaming technologies usually include strictly technological operations that are hard to simulate in laboratory, therefore this laboratory study has been conducted to evaluate Fischer-Tropsch wax and one of the available chemical additives.

Sasobit® (Figure 1 (a)) is a Fischer-Tropsch process wax that reduces the viscosity of bitumen above the melting point of wax (~90°C), thus improving the coating of aggregates and workability of the mix.

Rediset WMX® (Figure 1 (b)) is a chemical additive in flaked form with a melting point of 110°C. It is a combination of cationic surfactants and rheology modifier based on organic additives. It modifies the bitumen chemically and encourages active adhesion that improves the coating of the aggregates by binder. Other components of the additive reduce the viscosity of the binder at the production temperature.

(a) Sasobit®, (b) Rediset WMX®

FIGURE 1  WMA additives for evaluation.

The article contains data from book (2) and is based on the work done by M.Zaumanis for his Master thesis at the Riga Technical University.

Tasks of the research
The aim of the research is to investigate the changes in bitumen consistency after modification with WMA additives, to determine the physically-mechanical properties of asphalt after reduction of compaction temperature and to compare the characteristics of WMA with those of conventional HMA. To achieve this aim, the following tasks have been set:

1. Investigation of the changes in bitumen consistency at different temperatures after modification with WMA additives.
2. Determining the necessary adjustments in the mixture preparation, testing conditions and compaction method for evaluation of WMA properties and their adequate comparison with HMA.
3. Determining the physical and mechanical properties, including stiffness, resistance to deformations and compactibility of the asphalt modified with WMA additives and comparing the results with conventional HMA.
BITUMEN TESTING

Methodology
In order to determine the visco-elastic behavior of bitumen after modification with WMA additives, testing has been performed with conventional for Europe testing methods and the Dynamic Shear Rheometer (DSR) according to the testing plan provided at Figure 2. Two different types of bitumen were tested – 50/70 and 40/60. To evaluate the effect of additives on bitumens’ resistance to hardening, empirical tests were performed also after Rolling Thin Film Oven Test (RTFOT).

FIGURE 2  Experimental Plan for Testing Bitumen.

The stirring of additives with bitumen was performed at ~175°C. Testing with traditional tests was performed by means of the test methods according to EN 12591. For the DSR methodology from AASHTO TP5 was used at temperatures ranging from 100°C to 30°C with a 10°C step, at frequencies within each temperature of 0.01, 0.0215, 0.0464, 0.1, 0.215, 0.464, 1, 1.59, 2.15, 4.65, and 10 Hz, for 25 mm diameter samples with 1 mm gap between parallel plates, at unaged state.

Experimental results and discussion

Testing with empirical test methods
The test results (Table 2) for the binder containing Sasobit® compared to pure bitumen show the tendency of the consistency reduction at temperatures above the melting point of the additive and increase after crystallization of the wax. As expected, the degree of viscosity changes depends on the amount of the additive in the bitumen. If different types of Sasobit® modified binders are compared, it can be concluded that relatively to the pure bitumen the influence on the tested properties is similar, with the exception of dynamic viscosity. The results for initially softer bitumen (50/70) in this test show comparatively greater increase in viscosity than for harder bitumen (40/60).

The addition of Rediset WMX®, however, has small effect on the bitumen consistency characteristics at any temperature suggesting that the difference in viscosity is not the explanation of the warm mix effect. This chemical additive according to (3) has surface active formulation that allows the bitumen to disperse and coat the aggregates also making it easy to compact asphalt. At the same time hydrophilic amine group absorbs on the aggregate surface, which prevents water from entering the aggregate and bitumen interface. The amine group also encourages passive adhesion by bounding to siliceous surface.
The Fraass breaking point temperature is significantly increased by using WMA additives. However in general, the properties of original bitumen are irrelevant, because during the production process it oxidizes. It is more important to evaluate bitumen in the state in which it occurs in the mixture. The aging process was simulated by the RTFOT and the influence of this procedure on Fraass temperature is significantly different for pure and modified bitumen. The breaking point temperature after the RTFOT for the reference bitumen has increased by notable 5°C, only by 1°C for Sasobt modified binder and it has even dropped by 2°C for bitumen modified with Rediset WMX which suggests some anti-aging effect on bitumen of the chemical additive. This shows that the general concern that wax technology significantly worsens the low temperature behavior may not be true for all types of bitumen and has to be verified. It must also be taken in consideration that the effect of oxidative hardening in actual production process would be smaller for bitumen in WMA than for HMA because lower temperatures would be applied, therefore possibly even greater flexibility of bitumen in WMA can be attained.

The analysis of the consistency results after RTFOT suggests that aging has similar effect on the change of mass and retained penetration for both WMA modified binders and pure bitumen. The changes in softening point for Sasobit are relatively smaller than for other binders, but this is logical considering that already initially it had a significantly higher value in this test.

### TABLE 2  Traditional Bitumen Test Results

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Bitumen 50/70</th>
<th>Bitumen 40/60</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ref. 50/70</td>
<td>+3% Sas.</td>
</tr>
<tr>
<td>Penetration at 25°C,</td>
<td>65,0</td>
<td>45,2</td>
</tr>
<tr>
<td>Softening point, °C</td>
<td>50,4</td>
<td>78,4</td>
</tr>
<tr>
<td>Dynamic viscosity at 60°C, Ps-2</td>
<td>340</td>
<td>2379</td>
</tr>
<tr>
<td>Kinematic viscosity at 135°C, mm²/s</td>
<td>607</td>
<td>485</td>
</tr>
<tr>
<td>Fraass breaking point, °C</td>
<td>-25</td>
<td>-20</td>
</tr>
</tbody>
</table>

After RTFOT aging at 163°C

|                                | Bitumen 50/70 | Bitumen 40/60 |
|                                |              |               |
| Change in mass, %              | -0,10        | -0,09         | -0,12   |
| Retained penetration, %        | 70,8         | 72,0          | 69,0    |
| Softening point, °C (change)   | 56,8 (+6,4)  | 80,3 (+1,9)   | 63,7 (+5,6) |
| Fraass breaking point, °C (change) | -20 (-5) | -19 (-1) | -23 (+2) |

Performance-related testing

The DSR was used to measure the rheological properties of the binder. The test parameters (complex shear modulus ($G^*$) and the phase angle ($\delta$)) are used to characterize both the viscous and elastic behavior at intermediate to high temperatures which are the main ranges that are affected by the WMA additives. The relative comparison of the complex modulus ($G^*$) for modified and unmodified binders (Figure 5) show that after crystallization, Sasobit® increases the stiffness of the binder and improves the resistance to deformation at test temperatures. The relative comparison between two Sasobit® modified bitumens show...
a logarithmical increase when the wax content changes from 2% to 3% which indicates that 3% is the best alternative for further testing. The illustration (Figure 5) also demonstrates the crystallization range of wax, which is between 80°C and 90°C, meaning that in the construction object compaction should be finalized before this temperature is reached. At this temperature the additive creates a shear sensitive binder, to which the consistency depends both on the temperature and the frequency of loading. The evaluation of complex modulus for Rediset WMX®, however, suggests that this chemical additive has almost no effect on this property.

The summary of changes in phase angle, in comparison with pure bitumen, provided at Figure 6, shows that binders containing Sasobit® have improved elasticity, but addition of Rediset WMX®, like with G*, shows almost no effect on δ at any given temperature. The large phase angle variations within 80°C and 70°C compared to other temperature ranges for 3% Sasobit® may be attributed to the process of wax crystallization while the test was being performed.

Both of the results (G* and δ) somewhat explain the different reports of increased resistance to rutting for the Sasobit® modified bitumen, which is especially important for high in-service temperatures (~60°C) and short loading times that are typical for traffic.
ASPHALT TESTING

Methodology
Testing of the mixture has been performed according to testing plan at Figure 3. Based on bitumen testing results 2% Rediset WMX and 3% Sasobit was used for mixing WMA. Three different WMA compaction temperatures were compared with the reference HMA temperature which was deducted according to EN standard for the 40/60 grade bitumen. All of the testing was performed according to respective EN standard procedures.

The differences in WMA production temperature and technology include modification of bitumen consistency, different bitumen and aggregate interaction and changes in the binder aging processes. This may result in different strength gain of the WMA compared to HMA during a short period of time (4), therefore, a part of the testing plan was to determine whether short-term aging is necessary before performing tests. Short-term hardening simulates the initial strength gain processes that would occur during actual asphalt storage in the silo and transportation of the mix to paving site (5). Asphalt aging was performed according to AASHTO PP2 in a forced draft oven at the proposed compaction temperature. The mechanical effect of asphalt aging was examined by means of the indirect tensile test, which characterizes the stiffness of asphalt and is proven to be sensitive to stiffness of binder.

Compaction was performed by means of two different methods – Marshall hammer and gyratory compactor. Impact (Marshall) compaction was performed according to EN 12697-30 at the desired temperature with 50 blows from each side. Gyratory compactor allows evaluating the compaction of mixture in all of the densification range which is especially important for assessment of WMA properties. However, there are concerns that it is insensitive to temperature changes (6). To evaluate wide range of compaction force, 200 gyrations at 600kN for 1.25º external angle were applied. Molds of 100mm diameter were used.

Maximum density of the mixture was determined for unaged reference samples according to EN 12697-5 procedure A (volumetric) by using water.

<table>
<thead>
<tr>
<th>Mixture composition</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
<th>Compaction temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original</td>
<td>155°C</td>
<td>135°C</td>
<td>125°C</td>
<td>115°C</td>
<td>Aging</td>
<td>Aging</td>
<td>Aging</td>
</tr>
<tr>
<td>Sasobit 3%</td>
<td>0h</td>
<td>2h</td>
<td>4h</td>
<td>0h</td>
<td>2h</td>
<td>4h</td>
<td>2h</td>
</tr>
<tr>
<td>Rediset WMX 2%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**FIGURE 3** Experimental plan for testing asphalt mixture.
Experimental results and discussion

Asphalt aging

The densification data from gyratory compactor was expressed as a function of density at particular number of gyrations with a reference maximum density of 2532.2 kg/m³. The results show significant changes in densification at different times of aging. The compactibility data for specimens with no aging confirm that the compaction requires less energy for both WMA compared to HMA. However, after hardening for two and four hours, the compaction characteristics level out and are very similar for both WMA and HMA.

The stiffness modulus and the number of air voids at different aging times are presented at Figure 7. The results show an increase of the stiffness after extending the aging time for all specimens, except for Sasobit® at 4 hours which is considered to be connected to the excessive density of this core. The strength gain however is different for WMA products, compared to the reference HMA. Whereas specimens initially have a similar stiffness modulus, already after two hours of aging the stiffness has a variation of 2089 MPa between the lowest (Rediset WMX®) and the highest (Sasobit®) of the obtained results.

The stiffness test results suggest that initial aging is essential for adequate comparison of mixes, however further research is required to determine the optimum oxidation time. All subsequent samples for the purposes of this research were compacted after two hour aging.

Density

The results of bulk density for both compaction methods that are shown at Figure 8 do not correlate. The density of the reference HMA at 155°C for gyratory specimens was lower than for WMA, whilst for Marshall specimens it was higher in all cases. This is probably due to different compaction energies used, but the different temperature sensitivity of each compaction method could be another explanation. However, numerically the difference between all of the WMA specimens and HMA, except for Marshall at 115°C, is minor and the cores can be attributed to have a similar density.
The compaction data from gyratory compactor in percent to maximum density for both WMA products and the control mix at different temperatures is illustrated at Figure 9. It can be seen that compactibility at temperatures of 125°C and 135°C is similar to the reference mix for both WMA products. WMA at 115°C, however, has noticeably different compaction characteristics for both products. The density at the first part of the compaction is significantly higher than for other samples and reaches its final compaction level at about 100 gyrations for Sasobit and 70 gyrations for Rediset WMX. The compaction energy of about 70 gyrations is considered to relate to the actual field compaction, meaning that with this compaction effort, higher in-situ density than for HMA would be achieved. This behavior can be attributed to the reduced hardening of binder, due to the lower aging temperature.

![Graph showing compaction characteristics of Sasobit and Rediset WMX at different temperatures.](image)

**FIGURE 9 Compaction characteristics of Sasobit and Rediset WMX at different temperatures.**

**Stiffness**

The comparison between the stiffness modulus of Marshall and gyratory cores has not shown a good correlation (Figure 10) in relation to control mix at 155°C. Therefore, the evaluation of the stiffness modulus of WMA depends not only on the type of additive used and the compaction temperature, but also on the compaction method and/or the applied compaction force. Nonetheless, the results show that the stiffness of Sasobit® is higher than for Rediset WMX® at all compaction temperatures for both compaction methods. It is also clear that the difference between stiffness of both WMA at 135°C and 125°C is not significant, thus allowing to assume that the temperature can be lowered to at least 125°C while maintaining the relatively highest possible stiffness modulus for both WMA products. Further lowering of the temperature is considered to reduce stiffness of the mixture.

![Graph showing stiffness modulus test results for gyratory and Marshall specimens.](image)

**FIGURE 10 Stiffness modulus test results for gyratory and Marshall specimens.**

**Permanent deformations**

**Marshall test.** The Marshall test results are presented in Table 3. The Marshall stability results show a tendency to decrease with the reduced temperature and are generally lower than for the control mix at 155°C, meaning that the rutting resistance is worse than for the reference mix at 155°C. The results of Marshall flow also show the tendency of decreasing with lowering the temperature. This means less
deformation in the pavement under the critical stability load. The Marshall quotient values are calculated as the ratio of stability to flow and represent an approximation of the load ratio to deformation under the particular test conditions. Therefore, the results can be used as a measure of materials in service resistance to shear stresses, permanent deformation and, hence, rutting. The results show that the WMA at 125°C has approximately the same value as the reference. Therefore, the results obtained from the Marshall test allow concluding that both WMA products compacted at 125°C in terms of rutting would perform approximately the same as the control HMA compacted at 155°C.

However, although the Marshall test is widely used for mix design, it is important to recognize its limitations. The research (1) for conventional HMA shows that the Marshall test is a poor measure of permanent deformations of asphalt, especially for SMA which is evaluated in this research.

### TABLE 3  Marshall test results

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Stability, kN</th>
<th>Flow, mm</th>
<th>Quotient, kN/mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ref</td>
<td>Sas</td>
<td>Red</td>
</tr>
<tr>
<td>155°C</td>
<td>9.3</td>
<td>5.4</td>
<td>1.7</td>
</tr>
<tr>
<td>125°C</td>
<td>8.0</td>
<td>7.2</td>
<td>4.5</td>
</tr>
<tr>
<td>115°C</td>
<td>6.2</td>
<td>7.4</td>
<td>3.9</td>
</tr>
</tbody>
</table>

**Dynamic creep test.** The dynamic creep test has been performed only for the WMA samples that according to all other results were considered to have the best ratio of temperature reduction versus performance. Consequently samples compacted at 125°C were used. The maximum strain results at the end of test (3600 sec.) are presented in Figure 11 and show similar levels of WMA for specimens compacted with both compaction methods, but the results of the reference sample differ by 30%. These differences for the HMA specimens are attributed to different compaction levels because of changed compaction methods and densification force. Nonetheless, in general, the results are considered to show good resistance to rutting, proving that reduction of the compaction temperature by 30°C for both WMA products is possible without having an increased susceptibility to permanent deformations.

Elastic behavior, which is measured as the recovery after the relaxation period, has proportionally shown almost identical data for WMA and HMA, meaning that both WMA products are capable of recovering after the applied stress, as well as the control mix.

**FIGURE 11  Maximum strain for dynamic creep test.**

**FINDINGS AND CONCLUSIONS**

1. Addition of Sasobit® reduces viscosity of bitumen at high temperatures and increases it at intermediate temperatures. At in-service temperatures, Sasobit® provides higher resistance to deformations and improved elasticity of bitumen. Addition of Rediset WMX® has minor effect on bitumen viscosity properties.
2. The low temperature properties after RTFOT aging are similar for pure and modified bitumens. The use of Rediset WMX® reduced oxidative hardening compared to other samples and decreased the Fraass breaking point temperature after RTFOT. 
3. Oxidative hardening has different effects on WMA and HMA. Therefore, for the laboratory mixed samples, changes in mix preparation method should be considered, by performing asphalt aging before carrying out the compaction.
4. Use of both tested WMA products allows reducing the compaction temperature to at least 125°C, with the density remaining similar to HMA. The compactibility at this temperature is similar as for HMA.
5. The analysis of mechanical properties of asphalt show that reduction of the compaction temperature at least to 125°C for both WMA products is possible while maintaining similar stiffness and without having an increased susceptibility to permanent deformations.

References