Binder performance and testing

**Rheological characterization and comparison of aged polymer modified bitumens**

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Abstract

Investigations of low temperature and fatigue cracking in asphalt pavements are a priority in asphalt laboratories. Asphalt resistance to cracking depends mainly on the bitumen characteristics. The development of cracks can be indirectly predicted with the knowledge about bitumen properties, especially after aging. Contractors must ensure the required quality of produced asphalt mixes. To fulfil this obligation the characteristics of bitumens delivered to asphalt plants should be monitored. Investigations of such laboratory aged bitumens allow for their characterization and comparison between aged and non-aged bitumen. For many years the properties of bitumen at low temperatures have been determined based on the Fraass fracture temperatures. Since the Fraass breaking point test has several shortcomings, additional parameters like stiffness and creep rate were introduced in the Bending Beam Rheometer (BBR) method, which has been standardized, but it is still not widely used. Several samples of polymer modified bitumen PmB 45/80-65, which is widely used in our climate region, were extensively tested. The purpose of the study was to determine the impact of aging on bitumens. On neat bitumen the usual scope of bitumen tests (R&B, Penetration, Fraass) and BBR and DSR tests were performed. All tests were subsequently repeated on short term aged (RTFOT method) bitumens. In the last step the bitumens have been laboratory aged with RTFOT and PAV method and then re-tested. In the paper, the sensitivity to laboratory aging for samples of PmB 45/80-65, produced by different manufacturers, is presented.

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## 1. INTRODUCTION

Many factors affect asphalt resistance to low temperatures cracking, specially material characteristics (properties of bitumens) and structure related factors (type of asphalt mixture and compaction). The most important material property is the type of bitumen used in the asphalt mix. However there are differences between the characteristics of bitumens within the same bitumen type.

Contractors must ensure the required quality of produced and paved asphalt mixtures. To fulfil this obligation the characteristics of bitumens delivered to asphalt plants should be checked and monitored by asphalt mixture manufacturers. Investigations of these bitumens usually comprise only basic tests on bitumen penetration and softening point. However testing of laboratory aged bitumens allows for good characterization and comparison between the delivered bitumnes.

Several samples of polymer modified bitumen PmB 45/80-65, which is commonly used type of bitumen in our climate region for motorways, were tested at ZAG Laboratory for Asphalts and Bitumen-Based Products. In our climate region the temperatures in asphalt vary for up to 79°C. The lowest measured temperature in motorway network in the last five years was -18 °C, the highest temperature was +61 °C. Thermal cracking at low temperatures and rutting at high temperatures are common types of distress in pavements. Investigations of low temperature and fatigue cracking in asphalt pavements are a priority in asphalt laboratories. Asphalt resistance to cracking depends mainly on the bitumen characteristics. The likelihood of development of cracks can be indirectly predicted with the knowledge on properties of bitumen after aging.

## 2. TESTING OF LABORATORY AGED BITUMENS

### 2.1. Scope of testing

In this study the research of five different polymer modified bitumens PmB 45/80-65 (PmB) is presented. The original bitumens, sampled at delivery to asphalt plants, were tested, then laboratory short term aged and retested. Finally the samples were laboratory long term aged and tested again. All tests were performed in the same laboratory with the same personnel following the same procedures which allows for good repeatability of the results.

The purpose of the study was to determine the impact of aging on bitumens PmB 45/80-65, produced by different companies (and probably different sources). On original bitumen the usual scope of bitumen tests that is the softening point Ring and Ball (R&B) test [1], needle penetration at 25 °C (Pen) [2] and Fraass breaking point (Fraass) test [3] was performed. Fraass test characterises low temperature behaviour of bitumens as a measure of brittleness. It has been broadly used for a long time, however it is difficult to achieve good repeatability and reproducibility.

Less common test method for evaluation of low temperature characteristics is bending beam rheometer (BBR) test [4]. Research showed good correlation between bitumen limiting temperatures (at 300 MPa stiffness) and asphalt mixture cracking temperature [5]. Unfortunately for the tests relatively large amount of aged bitumen is required. The BBR tests were performed on long term aged bitumens at three temperatures from -22 °C to -10 °C with the temperature increment of 6 °C. The samples were left for one hour at the test temperature prior to loading. For BBR testing Cannon Thermoelectric BBR was used.

The five different PmB's were aged with the RTFOT [6] method and the tests were subsequently repeated. In the last step the five bitumens were laboratory aged with RTFOT + PAV method and then re-tested. For laboratory simulation of short term aging the binder was aged at 163 °C for 75 minutes through Rolling Thin Film Oven Test (RTFOT). For laboratory simulation of long term aging the binders were also aged in Pressure aging Vessel (PAV) [7] for 20 hours at 100 °C and 2.1 MPa pressure.

Up to now the Dynamic Shear Rheometer (DSR) tests (determination of complex shear modulus 'G*' and phase angle 'δ') [8] was not common in our region. The tests in our laboratory are normally performed according to EN standards. However for DSR testing the procedure Binder-Fast-Characterisation-Test (BTSV), described in German standard technical specifications FGSV [9] was utilised for original, short term aged and long term aged bitumen. In this test softening of bitumen sample is constant by applying a temperature rate of 1.2 °C/min (Temperature-sweep), at a constant shear stress of 0.5 kPa and oscillating mode with a test frequency of 10 rad/s. Complex shear modulus 'G*' and phase angle 'δ' were measured every 2.5 s and plotted versus temperature. The output parameter is temperature $T_{BTSV}$ at G*=15 kPa, and the corresponding phase angle $\delta_{BTSV}$. The test was performed in 25 mm plate-plate mode and a gap width of 1 mm for unaged and aged bitumen samples. During the tests the bitumen sample temperature was increased from 20 °C to 90 °C. The results of DSR T-sweep tests for original, short term aged and long term aged bitumen sample No.3 are shown in Figure 1.

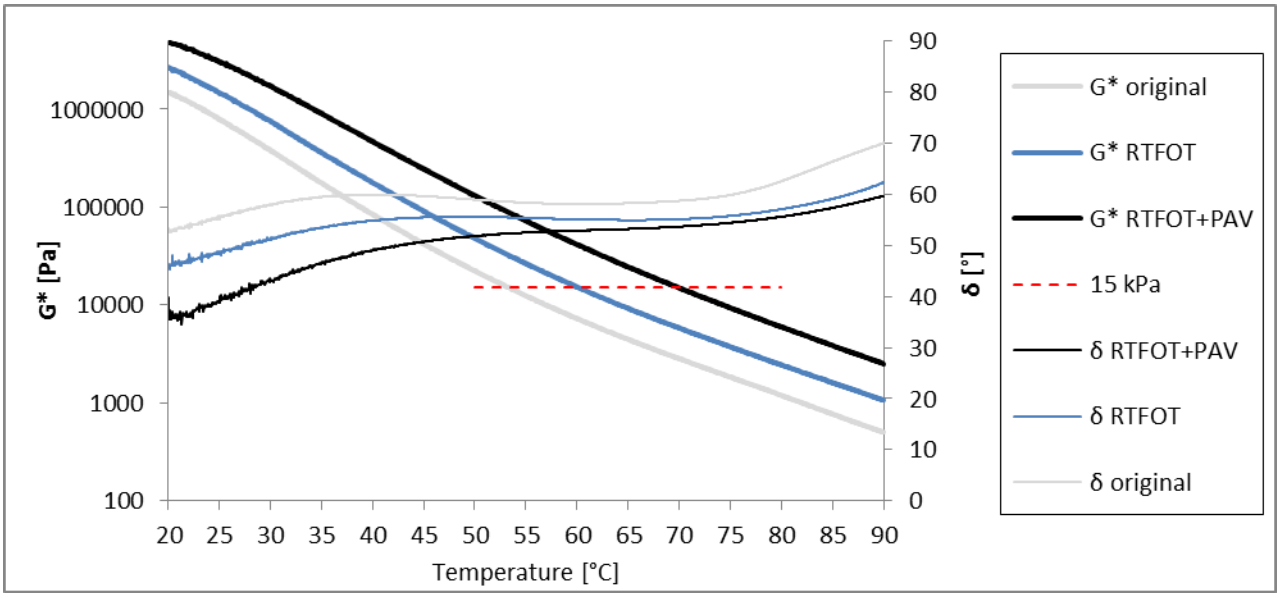


**Figure 1: Results of Binder-Fast-Characterisation-Test (BTSV) for sample No.3**

For DSR testing Anton Paar rheometer MCR302 was used. On RTFOT+PAV aged bitumens DSR tests were performed according to FGSV [9] and also according to EN standard [8] on 8 mm plate-plate mode and a gap width of 2 mm. The samples were sheared at four temperatures from 10 °C to 40 °C with the temperature increment of 10 °C.

Our binder specifications for polymer modified bitumen include basic tests for PmB (pen, R&B, Fraass) and criteria for original bitumen. However there are little criteria for RTFOT aged bitumen and no criteria for RTFOT+PAV aged or recovered bitumen. So results of this study could be used also as a base for new guidelines with criteria for RTFOT+PAV aged or recovered bitumen.

## 2.2. Results of tests on original bitumen (bitumen before aging)

In Table 1 are presented results of the usual scope of basic bitumen tests (R&B, Penetration, Fraass) on original bitumen samples as well as results of DSR T-sweep tests as temperature $T_{BTSV}$ at G* = 15 kPa and $\delta_{BTSV}$ at G*=15 kPa. In Table 1 is also shown the temperature at G*/sin δ = 1.0 kPa [10], calculated from the BTSV test results.

**Table 1. Results of tests on bitumen PmB 45/80-65 before laboratory aging (designated 'original')**

| Sample | Penetration | $T_{R\&B}$ | $T_{Fraass}$ | G* at 15kPa ($T_{BTSV}$) | δ at G*=15kPa ($\delta_{BTSV}$) | G*/sin δ = 1.0 kPa |
|---|---|---|---|---|---|---|
| No. | mm/10 | °C | °C | °C | ° | °C |
| 1 | 53 | 83.4 | -18 | 54.0 | 62.9 | 83.0 |
| 2 | 51 | 80.4 | -18 | 54.4 | 64.2 | 80.8 |
| 3 | 59 | 81.6 | -20 | 53.5 | 58.4 | 85.6 |
| 4 | 47 | 77.0 | -18 | 54.8 | 62.3 | 81.7 |
| 5 | 59 | 78.4 | -19 | 53.6 | 55.5 | 88.3 |

## 2.3. Results of tests on laboratory short term aged bitumen – after RTFOT

In Table 2 are presented results of basic bitumen tests on RTFOT aged bitumen samples as well as results of DSR T-sweep tests $T_{BTSV}$ at G* = 15 kPa and $\delta_{BTSV}$. In Table 2 is also shown the temperature at G*/sin δ = 2.2 kPa [10], calculated from the BTSV test results.

**Table 2. Results of tests on laboratory short term aged bitumen (designated 'after RTFOT')**

| Sample | Penetration | $T_{R\&B}$ | $T_{Fraass}$ | G* at 15kPa ($T_{BTSV}$) | δ at G*=15kPa ($\delta_{BTSV}$) | G*/sin δ = 2.2 kPa |
|---|---|---|---|---|---|---|
| No. | mm/10 | °C | °C | °C | ° | °C |
| 1 | 37 | 73.2 | -12 | 58.6 | 61.6 | 75.8 |
| 2 | 37 | 74.0 | -10 | 60.3 | 62.1 | 79.0 |
| 3 | 40 | 79.2 | -14 | 59.9 | 55.1 | 83.8 |
| 4 | 34 | 74.8 | -7 | 61.1 | 59.8 | 80.8 |
| 5 | 41 | 78.4 | -12 | 57.6 | 54.4 | 81.9 |

### 2.4. Results of tests on laboratory long term aged bitumen – after RTFOT+PAV

In Table 3 are presented results of basic bitumen tests on RTFOT+PAV aged bitumen samples as well as results of DSR T-sweep tests $T_{BTSV}$ at G* = 15 kPa and $\delta_{BTSV}$.

**Table 3. Results of tests on long term aged bitumen (designated RTFOT+PAV)**

| Sample | Penetration | $T_{R\&B}$ | $T_{Fraass}$ | G* at 15 kPa ($T_{BTSV}$) | δ at G*=15kPa ($\delta_{BTSV}$) |
|---|---|---|---|---|---|
| No. | mm/10 | °C | °C | °C | ° |
| 1 | 25 | 75.8 | -8 | 58.6 | 61.6 |
| 2 | 23 | 74.8 | -6 | 67.4 | 61.0 |
| 3 | 25 | 81.4 | -9 | 69.6 | 53.7 |
| 4 | 20 | 80.6 | -3 | 73.0 | 57.3 |
| 5 | 27 | 81.4 | -9 | 67.1 | 53.0 |

Additionally to these tests also BBR tests were performed to establish the low temperature characteristics of the aged bitumen. We established the limiting temperatures by plotting stiffness and m-value versus temperature at a loading time of 60 s. We determined the temperatures at 300 MPa binder stiffness - creep limiting stiffness temperature at 60 s loading time (LST) and temperature at m-value 0.300 at 60 s loading time (LmT). The results of BBR tests (Table 4) show that sample No.5 and No.1 are the most resistant to low temperature cracking.

Based on the results of DSR T-sweep tests we calculated the temperature relating to pavement resistance to fatigue cracking – intermediate temperature stiffness. The temperatures were determined at $|G^*|\cdot\sin(\delta) = 5$ MPa and $|G^*|\cdot\sin(\delta) = 6$ MPa [11] as shown in Table 4.

**Table 4. Results of test on long term aged bitumen for low temperatures and fatigue resistance**

| Sample | $T_{Fraass}$ | DSR T at $|G^*|\cdot\sin\delta = 5$ MPa | DSR T at $|G^*|\cdot\sin\delta = 6$ MPa | BBR LST | BBR LmT | BBR $|\Delta T|$ |
|---|---|---|---|---|---|---|
| No. | °C | °C | °C | °C | °C | °C |
| 1 | -8 | 19.9 | 18.8 | -18.3 | -15.8 | 2.5 |
| 2 | -6 | 19.2 | 17.9 | -18.2 | -13.1 | 5.1 |
| 3 | -9 | 17.3 | 15.6 | -21.2 | -14.4 | 6.8 |
| 4 | -3 | 20.5 | 19.0 | -17.9 | -11.4 | 6.5 |
| 5 | -9 | 17.9 | 16.5 | -19.6 | -18.9 | 0.7 |

## 3. EFFECTS OF LABORATORY AGING

Aging has a hardening effect on all types of bitumens. In Figure 2 are presented changes in penetration and in Figure 3 changes in Fraass breaking point. Results show that samples No.3 and No.5 exhibit less hardening (higher penetration after PAV) and better characteristics at low temperatures (lower $T_{Fraass}$ ).

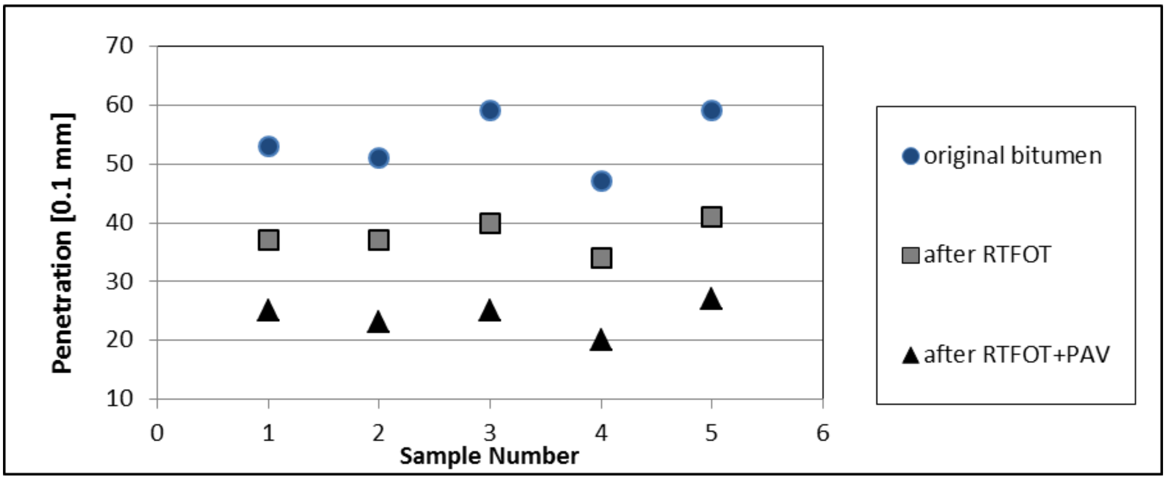


**Figure 2: Penetration of original and aged bitumens**

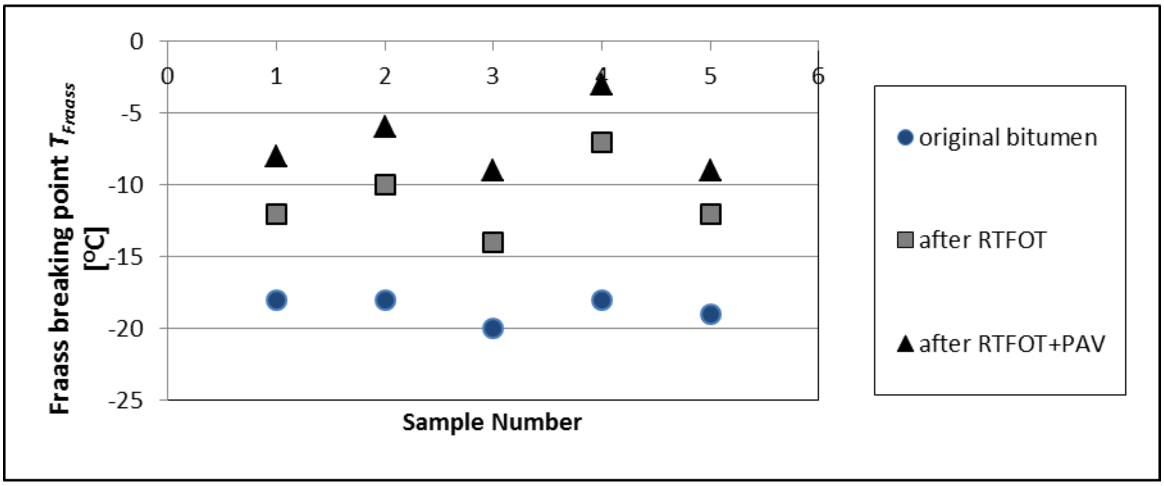


**Figure 3: Fraass breaking point of original and aged bitumens**

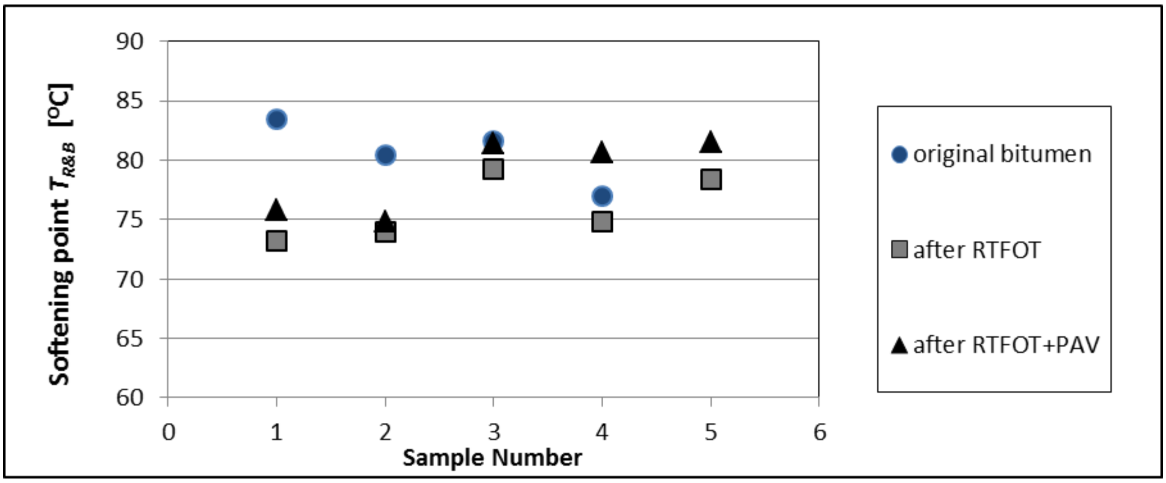


**Figure 4: Softening point of original and aged bitumens**

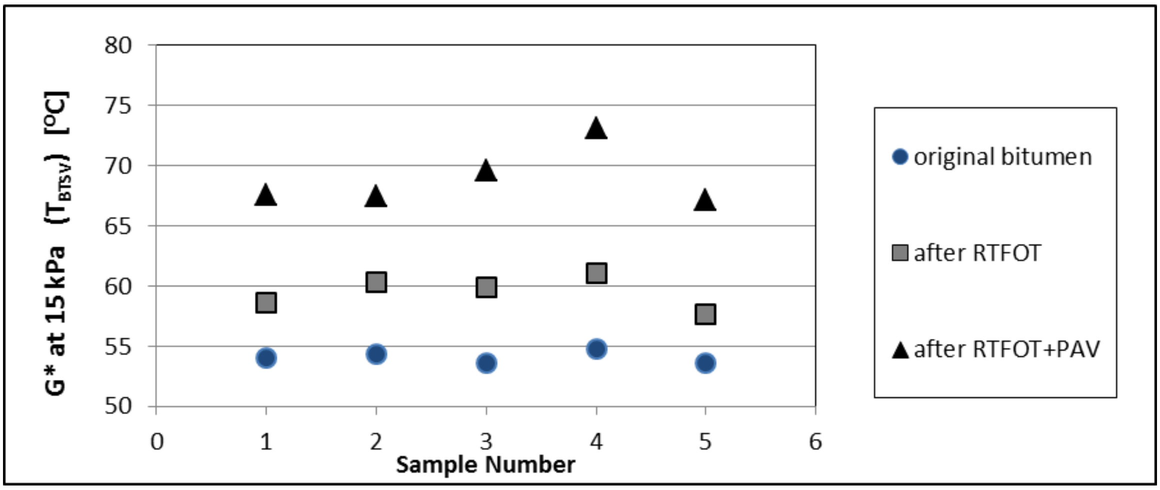


**Figure 5: G* at 15kPa ($T_{BTSV}$) of original and aged bitumens**

In Figure 4 are presented changes in softening point $T_{R\&B}$ and in Figure 5 changes in $T_{BTSV}$ after laboratory aging. Figure 4 shows, that samples No.1 and No.2 have exhibited more hardening ($T_{R\&B}$ is lower) than the other samples. However Figure 5 shows that the sample No.4 has the highest $T_{BTSV}$ and the samples No.1, No.2 and No.5 have the lowest $T_{BTSV}$ after PAV aging.

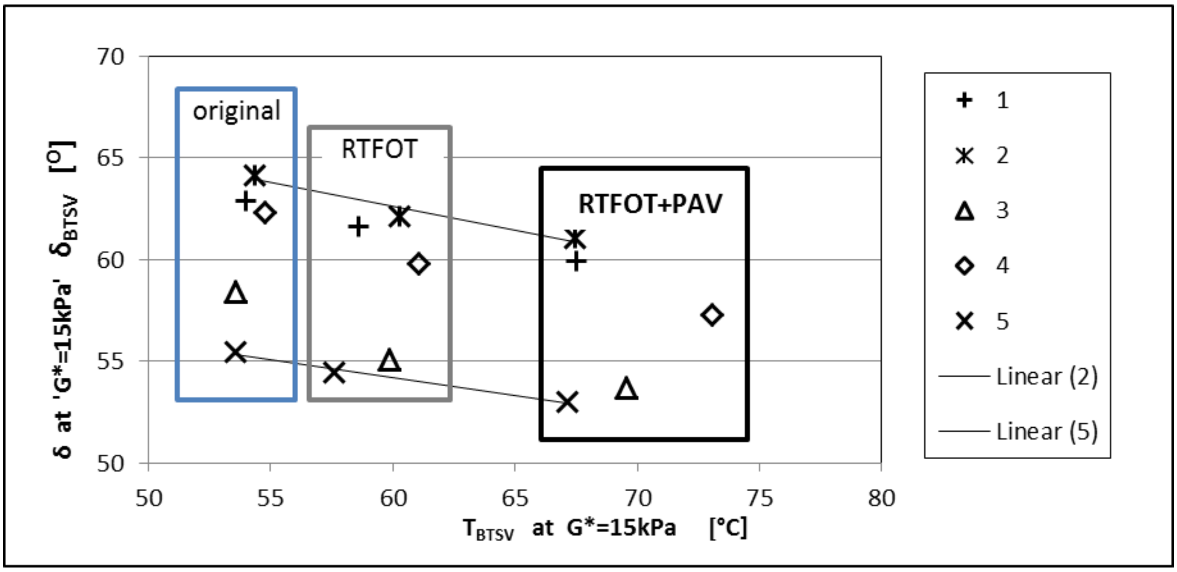


**Figure 6: Effect of laboratory aging on rheological characteristic G* and δ of five samples**

In Figure 6 are shown the $T_{BTSV}$ versus $\delta_{BTSV}$. Samples No.3 and No.5 exhibit lower δ in comparison to samples No.1 and No.2.

The results of basic tests, rheological tests and BBR tests clearly show that there are differences in the five bitumen characteristics at low temperatures. Sample No.4 has the lowest penetration after aging and the complex modulus G* reaches 15 kPa at higher temperature than the other samples. For sample No.4 also the LST and LmT values are at the highest. We can assume that sample no.4 is less suitable to harsh winter climate conditions then the other four samples of PmBs. However since there are no set criteria for laboratory aged bitumen and no comparison to the actual behaviour of paved asphalt mixtures, we assume that all five bitumens are appropriate.

## 4. CORRELATIONS

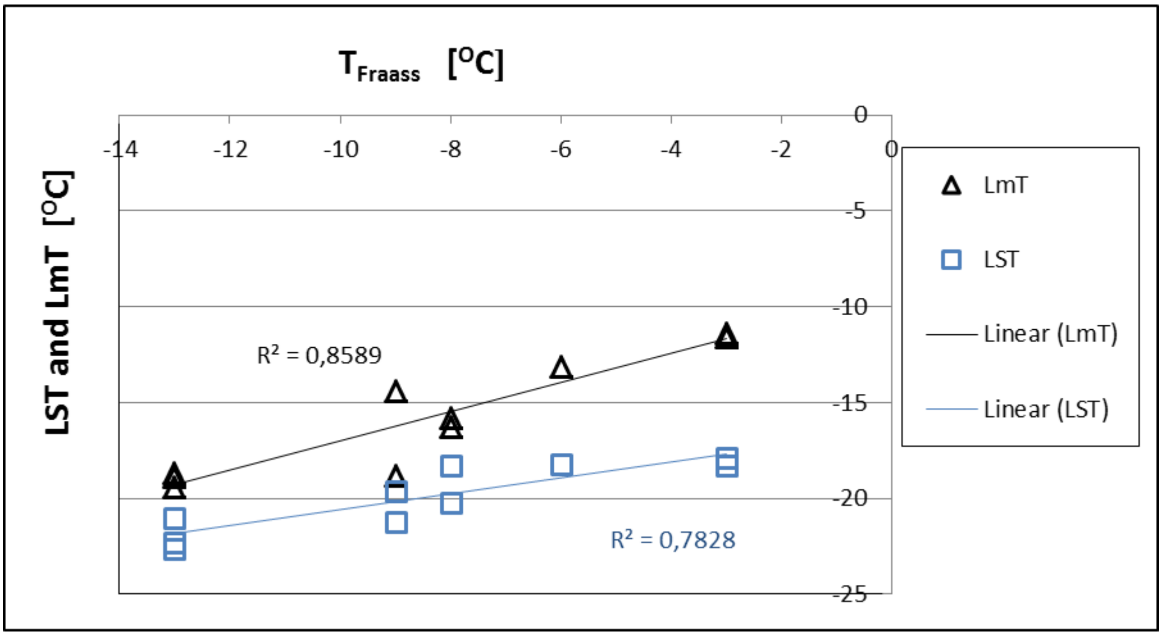


**Figure 7: Effect of laboratory aging (RTFOT+PAV) on low temperature characteristics**

Figure 7 presents the results of ten PmB 45/80-65 samples (the five described in previous chapters and five others) tested at ZAG laboratory by the same equipment over the timespan of two years (2017-2018). There is a moderate correlation between $T_{Fraass}$ and LST and LmT. The graph shows that lower values of LST and LmT correspond to lower values of $T_{Fraass}$. For all tested PmB 45/80-65 the LmT was higher that the LST. Moreover for the lower $T_{Fraass}$ the difference between LmT and LST was smaller than for bitumens having higher $T_{Fraass}$.

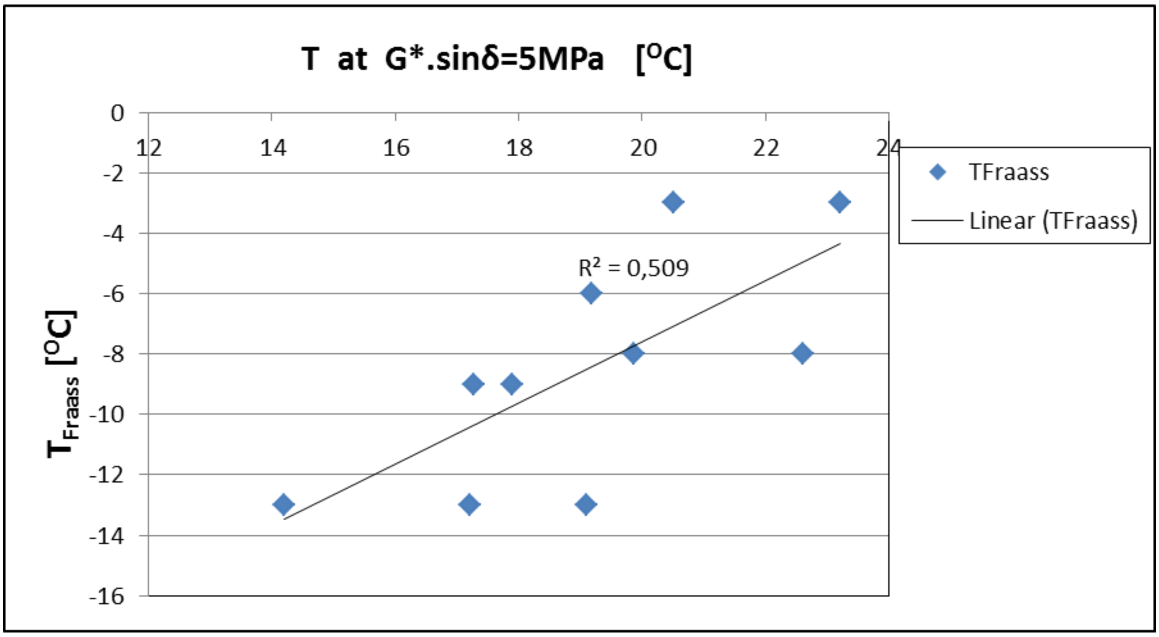


**Figure 8: Correlation T at G*sin δ=5MPa and $T_{Fraass}$ (RTFOT+PAV) of all tested PmB 45/80-65**

There was weak correlation between the temperature at T at G*.sin δ=5 MPa and $T_{Fraass}$ (Figure 8). The Fraass test is an empirically based standardised test having a poor reproducibility of 6 °C, still the testing within the same laboratory has shown a weak correlation with this rheological parameter which represent susceptibility to fatigue cracking.

For statistical evaluation and estimation of polymer modified bitumen behaviour over a pavement lifetime a much larger data set should be collected and analysed. In Germany extended bitumen testing started in 2013 [12], the publication of results of testing may help setting criteria for characteristic after short term and long term aging.

## 5. CONCLUSIONS

Asphalt mixture manufacturers must ensure the required quality of produced asphalt mixtures – resistance to low temperature cracking and rutting at high temperatures. For a specific structure of asphalt mixture the most important material property is the type of bitumen and its characteristics. The characteristics of bitumens delivered to asphalt plants should be checked and monitored. For polymer modified bitumens basic tests on bitumen penetration (Pen) and softening point (R&B) do not reveal appropriately their characteristics. Despite numerous disadvantages Fraass breaking point is a simple and widely used test method for checking characteristics at low temperatures.

BBR tests offer good characterisation of bitumen resistance to low temperature, but the amount of bitumen needed for testing at least two temperatures (sometimes three temperatures are required for testing) is quite large. Testing on dynamic shear rheometer (determination of complex shear modulus and phase angle and multiple stress creep and recovery test) enables quick and good characterisation of binders. The tests are well reproducible and require a small amount of binder; however asphalt mixture manufacturer's laboratories are normally not equipped with rheometers. Laboratory aging of bitumen takes time, skills and appropriate equipment therefore testing of aged bitumens is not suitable for everyday control.

Results of this study could be used also as a base for national guidelines with criteria for RTFOT+PAV aged or recovered PmB 45/80-65 bitumen. New criteria will be proven when a database on behaviour of laboratory aged polymer modified bitumen from a distinctive producer/source will be compared to the actual behaviour of paved asphalt mixtures. Using new criteria the appropriate bitumen could be chosen for each traffic and climate load situation.

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