Effect of ageing on the low temperature properties of bitumen

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ABSTRACT

Properties of aged binder are believed to be relevant for low temperature cracking of asphalt. The properties of bitumen at low temperatures have been determined for many years with Fraass fracture temperatures of fresh bitumen. Since Fraass braking point test has several shortcomings, additional parameters like stiffness and creep rate were introduced with Bending Beam Rheometer (BBR) method. The BBR method has been standardized (EN 14771) but it is still not widely used in middle Europe, mostly because it is intended for the investigation of aged instead of fresh bitumen, and secondly, the limit values for evaluation of the quality of bitumen are still not set.

In our study six samples of original bitumen B70/100 were extensively tested. The purpose of the study was to determine the impact of aging on the most commonly used types of paving grade bitumen B70/100 in Slovenia and to propose criteria for bitumen quality based on evaluation of obtained test results. On original bitumen the usual scope of bitumen tests (R&B, Penetration, Fraass) was performed and additionally BBR test. These bitumen were also tested after short term aging (RTFOT method) and long term aging (PAV method). In this study, the sensitivity to aging and temperature, as well as low temperature behaviour of these bitumen was evaluated on the basis of the results of the tests.

Additionally we determined some correlations between results of BBR test and conventional bitumen tests. Correlations between all test results were visualized with principal component analyses (PCA) method and then the most adequate model of BBR results in dependency to all other test results was determined with Multivariate linear regression.

Keywords: Functional specifications, Low-Temperature, Rheology, Standardisation, Thermal Cracking

1. INTRODUCTION

Research into the low temperature cracking of asphalt pavements is an important priority in asphalt testing laboratories worldwide. Several different types of test are used in the laboratory in order to simulate the process of low temperature cracking on asphalt samples. Standardised test methods such as the Thermal Stress Restrained Specimen Test (TSRST) and the Uniaxial Tensile Stress Test (UTST) are used the most frequently. Both tests are descibed in the corresponding European standard [1]. Due to the fact that the resistance of asphalt to low temperature cracking depends mainly on the bitumen, it is assumed that low temperature cracking of asphalt can be predicted (indirectly) if the realistic low temperature properties of bitumen are sufficiently well known. For many years these properties have been determined on the basis of Fraass fracture temperatures [2]. Since the Fraass breaking point test has several shortcomings, additional parameters such as stiffness and creep rate were introduced, using the Bending Beam Rheometer (BBR) method [3]. The BBR method has been standardized, but it is not yet widely used in Central Europe. In the European standard for bitumen specifications [4, 5] the Fraass breaking point test is a normative test performed on the original bitumen, whereas in the American specifications for bitumen [6] the BBR method is a normative test performed on aged bitumen, and not on the original bitumen.

In ZAG Ljubljana's Laboratory for Asphalts and Bitumen-Based Products, over the years an extensive data base about the Fraass breaking-point testing of original bitumen samples has been established. In this study an attempt was made to find out whether there is any correlation between the BBR and Fraass test results. Six samples of original B 70/100 bitumen were extensively tested. The initial purpose of the study was to determine the impact of ageing on the most commonly used types of paving grade B 70/100 bitumen in our region, and to propose criteria for bitumen quality based on an evaluation of the obtained test results. Bitumen tests of the usual scope (i.e. R&B, Penetration, and Fraass) were performed on six original bitumen samples, as well as the BBR test. These bitumens were artificially aged using the RTFOT method, and all the tests were repeated on the short-term aged bitumens. In the last stage of the investigation the bitumen samples were artificially aged using the RTFOT and PAV methods, and then re-tested. In this paper the results of sensitivity to ageing are presented for the six different bitumens, as well as the correlations found for the original bitumens.

2. EXPERIMENTAL PROCEDURE

Bitumen tests were performed at ZAG's Laboratory for Asphalts and Bitumen-Based Products according to the procedures defined in the European standards. Paving grade B 70/100 bitumens from six different producers were tested. The results when performing the usual scope of tests (R&B, Penetration, and Fraass) on the original bitumen samples are presented in Table 1. BBR tests were additionally performed at temperatures from -4°C to -34°C, using steps of 6°C, in order to establish the limiting temperatures by plotting stiffness and m-value versus temperature at a loading time of 60 s according to the American specifications for bitumen [6]. The limiting temperatures, i.e. the temperature at a 300 MPa stiffness - creep stiffness at a 60 s loading time ($T_{(S)60}$), and the temperature at an m-value of 0.300 at a 60 s loading time ($T_{(m)60}$), were determined for each sample. From Fig. 1a and Fig. 1b it can be seen that there is a weak correlation between the Fraass breaking point and the BBR $T_{(S)60}$ results for the original B 70/100 bitumens.

Table 1: Results of the tests performed on the original B70/100 bitumen samples

Sample	Penetration	$T_{R\&B}$	T_{Fraass}	$T_{(S)60}$	$T_{(m)60}$
No.	mm/10	°C	°C	°C	°C
1	84	45.8	-13	-19.0	-22.0
2	77	47.4	-14	-22.0	-24.5
3	78	46.4	-13	-21.0	-23.0
4	70	48.3	-16	-21.5	-23.0
5	79	45.8	-10	-17.5	-21.0
6	81	46.4	-10	-20.0	-24.0

a. **b.**

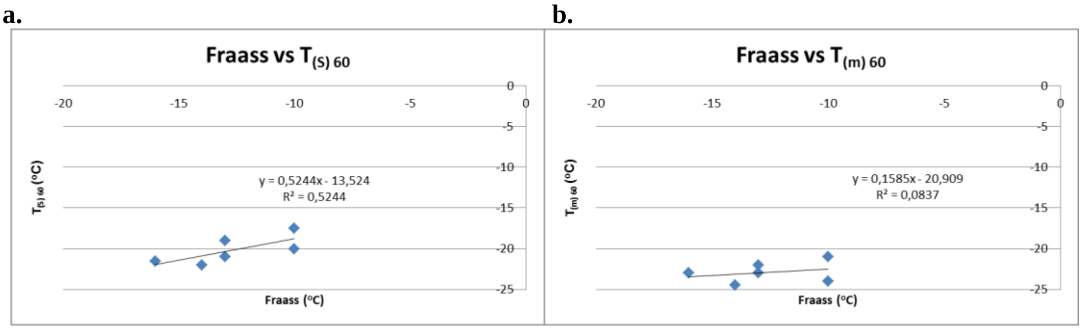

Figure 1: The Fraass breaking point results plotted against the results for BBR (a) $T_{(S)60}$ and (b) $T_{(m)60}$ for the original bitumen samples

The results of the tests performed on the short term aged bitumen samples (with RTFOT) are presented in Table 2. A comparison of the results of the low temperature tests in Table 2 with the results in Table 1 shows that the short term ageing procedure had only a moderate effect on the low temperature properties of these bitumens. On average, the Fraass breaking point temperature increased by 1ºC, whereas $T_{(S)60}$ increased by 0.8ºC, and $T_{(m)60}$ by 2.9ºC. It can be concluded that in fact only the $T_{(m)60}$ values increased due to the RTFOT ageing ($\Delta T_{(m)60}$). Surprisingly, the softening point temperature (R&B) increased on average by 8.2ºC ($\Delta T_{R\&B}$) after RTFOT ageing, whereas the low temperature parameters were almost unaffected (ΔT_{Fraass}). If only these two tests are taken into consideration, then it would appear that such ageing improved the low temperature behaviour of B 70/100 bitumen samples.

Table 2: Results of the tests performed on B70/100 bitumens after short-term (RTFOT) ageing, and the differences from the original bitumen characteristics

Sample	**Pen.**	**Δpen.**	$T_{R\&B}$	$\Delta T_{R\&B}$	T_{Fraass}	ΔT_{Fraass}	$T_{(S)60}$	$\Delta T_{(S)60}$	$T_{(m)60}$	$\Delta T_{(m)60}$
No.	mm/10	mm/10	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C
1	51	-33	50.8	5	-13	0	-18.5	0.5	-20.0	2.0
2	44	-33	58.2	10.8	-14	0	-21.0	1.0	-21.0	3.5
3	39	-39	56.9	10.5	-11	2	-20.5	0.5	-20.0	3.0
4	47	-23	54.8	6.5	-14	2	-20.5	1.0	-20.0	3.0
5	50	-29	51.3	5.5	-9	1	-15.0	2.5	-17.0	4.0
6	45	-36	52.8	6.4	-10	0	-19.0	1.0	-22.0	2.0

The results of the tests which were performed on the long term (using RTFOT+PAV) aged bitumens are presented in Table 3. From this table it can be seen that long term ageing had a more significant effect on the low temperature properties. A comparison of the results of the low temperature tests given in Table 3 with those in Table 1 shows that, on average, the Fraass breaking point temperature increased by 5.8ºC, and the limiting temperatures $T_{(S)60}$ by 2.8ºC, and $T_{(m)60}$ by 8.9ºC. The Fraass breaking point and $T_{(m)60}$ increased significantly after the RTFOT+PAV ageing. In Fig. 2a and Fig. 2b no significant correlation can be found between the Fraass breaking point and the BBR test results for the RTFOT+PAV aged B 70/100 bitumens.

Table 3: Results of the tests performed on the B70/100 bitumens after long term (RTFOT+PAV) ageing, and the differences from the original bitumen characteristics

Sample	**Pen.**	**Δpen.**	$T_{R\&B}$	$\Delta T_{R\&B}$	T_{Fraass}	ΔT_{Fraass}	$T_{(S)60}$	$\Delta T_{(S)60}$	$T_{(m)60}$	$\Delta T_{(m)60}$
No.	mm/10	mm/10	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C	⁰C
1	30	-54	59.2	13.4	-17	4	-17.0	2.0	-17.0	5.0
2	27	-50	70.8	23.4	-13	5	-19.5	2.5	-13.0	11.5
3	22	-56	70.5	24.1	-9	10	-18.0	3.0	-9.0	14.0
4	29	-41	63.6	15.3	-13	6	-18.0	3.5	-13.0	10.0
5	29	-50	56.4	10.6	-16	2	-15.0	2.5	-16.0	5.0
6	23	-58	63.6	17.2	-16	8	-17.0	3.0	-16.0	8.0

a. b.

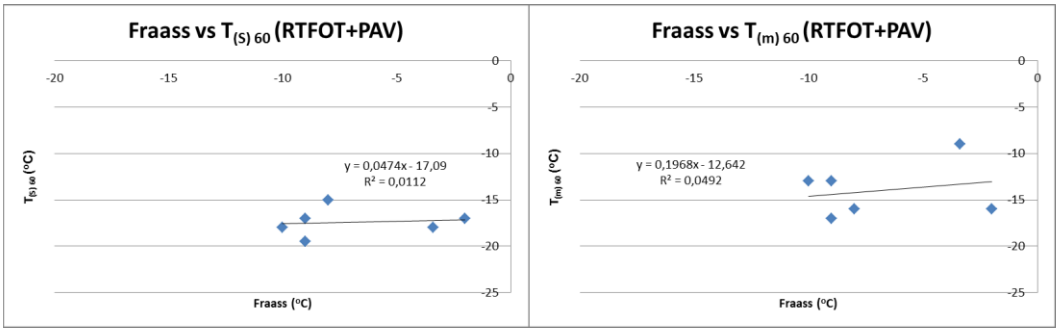

Figure 2: The Fraass breaking point results plotted against the results for BBR (a) $T_{(S)60}$ and (b) $T_{(m)60}$ for RTFOT+PAV aged B70/100 bitumens

3. LITERATURE OVERVIEW

The number of tests performed in ZAG's laboratory was too low for statistical analyses, so data from the literature [7] were added in order to enable multiple linear regression analyses of the results of the low temperatures tests. The results of tests performed on 8 different B 35/50 penetration grade bitumens, obtained from the literature [7], are presented in Table 4. The following bitumen types were involved: three distilled bitumens from different origins (B_1, B_2 and B_3), 1 semi blown bitumen B_4 , 1 bitumen B_1 modified with paraffin wax (B_{11}), 1 bitumen B_1 modified with phosphoric acid (B_{12}), 1 bitumen B_1 modified with crosslinker free SBS (B_{13}), and 1 bitumen B_1 modified with crosslinked SBS (B_{14}). From Fig. 3a and Fig. 3b it can be seen that there are weak correlations between the Fraass breaking point and the BBR results for the original bitumens.

Table 4: Results of the tests performed on different types of original bitumen [7]

Sample	Penetration	$T_{R\&B}$	T_{Fraass}	$T_{(S)60}$	$T_{(m)60}$
No.	mm/10	^0C	^0C	^0C	^0C
B_1	38	53.6	-9.0	-15.9	-16.2
B_2	44	56.4	-12.5	-20.9	-20.8
B_3	40	54.0	-14.5	-16.0	-17.3
B_4	36	60.7	-13.5	-21.1	-18.3
B_{11}	30	67.4	-7.0	-13.9	-13.4
B_{12}	28	62.8	-14.0	-16.2	-16.1
B_{13}	39	55.8	-10.0	-17.7	-16.5
B_{14}	35	64.3	-13.0	-17.0	-16.6

a. b.

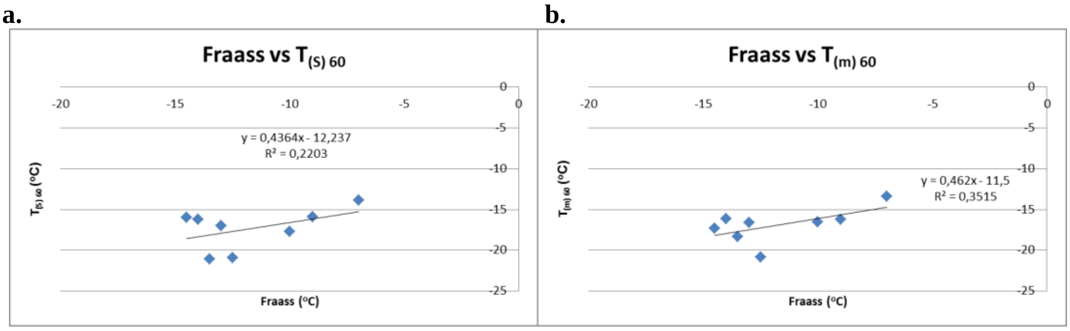

Figure 3: The Fraass breaking point results plotted against the results for BBR (a) $T_{(S)60}$ and (b) $T_{(m)60}$ for original bitumen samples (results obtained from the literature [7])

The authors of the paper [7] assumed that modified PAV ageing at 100°C and 2.1 MPa for 25 hours (prolonged) has the same effect on bitumen as the ordinary procedure of long term ageing (RTFOT followed by PAV ageing at 100°C and 2.1 MPa for 20 hours). The results of the tests performed on modified PAV aged bitumen [7] are presented in Table 5. In the case of the modified long term aged bitumens, too, only weak correlations were found between the Fraass breaking point and the BBR results (see Figure 4).

Table 5: Results of tests performed on modified PAV aged bitumen [7], and the difference from the original bitumen(s)

Sample	Pen.	ΔPen.	$T_{R\&B}$	$\Delta T_{R\&B}$	T_{Fraass}	ΔT_{Fraass}	$T_{(S)60}$	$\Delta T_{(S)60}$	$T_{(m)60}$	$\Delta T_{(m)60}$
No.	mm/10	mm/10	^{0}C	^{0}C	^{0}C	^{0}C	^{0}C	^{0}C	^{0}C	^{0}C
1	16	-22	66.6	13.0	-3	6	-12.7	3.2	-8.6	7.6
2	17	-27	75.6	19.2	-7	5.5	-17.9	3.0	-10.7	10.1
3	17	-23	66.8	12.8	-8	6.5	-13.2	2.8	-13.4	3.9
4	17	-19	77.0	16.3	-8.5	5	-19.7	1.4	-7.1	11.2
5	13	-17	83.5	16.1	-5	2	-11.1	2.8	-4.6	8.8
6	12	-16	87.5	24.7	-4.5	9.5	-14.3	1.9	-9.9	6.2
6	18	-21	69.4	13.6	-4	6	-14.3	3.4	-9.4	7.1
6	16	-19	75.2	10.9	-6	7	-14.3	2.7	-9.8	6.8

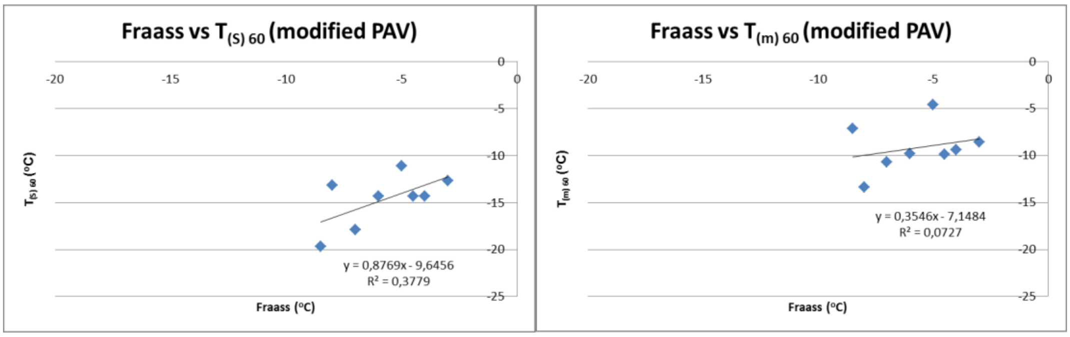

Figure 4: The Fraass breaking point results plotted against the results for BBR (a) $T_{(S)60}$ and (b) $T_{(m)60}$ for modified PAV – long term aged bitumen samples (results obtained from the literature [7])

If the results of the low temperature tests presented in Table 5 are compared with those in Table 4 it can be seen that, on average, the Fraass breaking point increases by 5.2°C, and the limiting temperatures $T_{(S)60}$ by 2.1°C and $T_{(m)60}$ by 6.7°C. As expected, the Fraass breaking point and $T_{(m)60}$ increased significantly due to the modified PAV ageing. From Figures 2 and 4 it can be concluded that the ageing effect on the low temperature properties of B 35/50 bitumens from the literature is similar to that indicated by the results obtained in ZAG's laboratory.

4. STATISTICAL ANALYSES

The available data on the six investigated B 70/100 bitumens and the eight samples from the literature [7] were merged in order to try to find correlations by means of multiple linear regressions. Univariate (simple) linear regression is usually used when a correlation between two variables is being searched for, but when there are more than two variables there is no reason to avoid multiple linear regressions. If a dependent variable is to be explained by two or more independent variables, then it is important to have enough experimental points (degrees of freedom), and that the variety of the experimental points is wide enough and spread out as evenly as possible in the experimental space.

Although our experiments only covered B 70/100 types of bitumen, data from the literature [7] made it possible to widen the variety of the experimental points to other types of bitumen. The final results of the performed multiple linear regression consisted of statistical models for the output (dependent) variables BBR $T_{(S)60}$ and BBR $T_{(m)60}$ as functions of the input (explanatory) variables. Two promising linear models were found. The equations with two input variables with the highest correlation coefficient for BBR $T_{(m)60}$ of the original bitumen have the following form:

Model 1 BBR $T_{(m)60\ (original\ bitumen)} = -7.333 - 0.137 \cdot (Penetration) + 0.388 \cdot (T_{Fraass})$ (1)
($R^2 = 0.885$)

and surprisingly

Model 2 BBR $T_{(m)60\ (original\ bitumen)} = -16.917 - 0.121 \cdot (Penetration) + 0.074 \cdot (T_{R\&B})$ (2)
($R^2 = 0.816$)

From the above it can be seen that the second equation does not contain the Fraass breaking point as a factor. All other correlations for the output variable BBR with two input variables (Fraass or penetration or R&B) were less significant. For validation of the proposed two models we looked at independent data from the literature [8]. The results of tests performed on three different penetration grade bitumens, and stated in the literature [8], are presented in Table 6, as well as the results of both models - the limiting temperatures at an m-value of 0.300 and a 60 s loading time ($T_{(m)60}$).

Table 6: Results of tests performed on original bitumen samples [8] – experimental and calculated values

Sample	Pene-tration	$T_{R\&B}$	T_{Fraass}	$T_{(m)60}$ (experimental)	$T_{(m)60}$ (calculated from model 1)	$T_{(m)60}$ (calculated from model 2)
	mm/10	^{0}C	^{0}C	^{0}C	^{0}C	^{0}C
B 160/220	174	42.5	-23	-27.4	-40.1	-34.9
B 70/100	64	50.0	-16	-22.4	-22.3	-21.0
B 50/70	50	51.5	-15	-18.8	-20.0	-19.2

From Table 6 it can be seen that the predictions of both models for bitumen of penetration types B 70/100 and B 50/70 are relatively good. For the bitumen of penetration type B 160/220 the predicted $T_{(m)60}$ is too high. In the case of the B 160/220 bitumen one should be aware that none of the bitumens used to construct the models had similar properties, so that such an assumption is in fact an extrapolation of the model. However, by adding more different types of bitumen to the training set it should be possible to improve the models, and widen their range of applicability.

5. CONCLUSIONS

In ZAG Ljubljana's Laboratory for Asphalts and Bitumen-Based Products, over the years an extensive data base about the Fraass breaking-point testing of original bitumen samples has been established. In the present study at attempt was made to determine sensitivity to ageing for six different bitumens, and to find any correlations between the limiting temperatures obtained by the BBR test ($T_{(m)60}$ and $T_{(S)60}$) and the Fraass breaking point (T_{Fraass}). Six samples of original, short term and long term aged B 70/100 bitumen were extensively tested.

In the first part of the study it was found that short term ageing (with RTFOT) has only a moderate effect on the low temperature properties of bitumen. Long term ageing (with RTFOT+PAV) has a significant effect on $T_{(m)60}$ and a somewhat smaller effect on T_{Fraass}. The least affected by long term ageing was $T_{(S)60}$.

In the second part of the study models for the prediction of the low temperature properties of bitumen were defined. Results from the literature [7] were merged with our data in order to ensure a sufficient variety of bitumen types. Two promising models with two input variables for the prediction of BBR $T_{(m)60}$ of original bitumen were defined. Surprisingly, the second equation includes penetration and the softening point as input variables, without the Fraass breaking point. The performed validation showed that the correlation is relatively good for penetration grade bitumen of types B 70/100 and B 50/70. However, by adding more different types of bitumen to the training set it should be possible to improve the models, and widen their range of applicability. For the aged bitumens no significant correlations between the limiting temperatures obtained by the BBR test ($T_{(m)60}$ and $T_{(S)60}$) and the Fraass test results (T_{Fraass}) was found.

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