QUALITY CHECKING OF POLYMER MODIFIED BITUMENS IN SLOVENIA

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Abstract

For many years the properties of bitumen have been determined based on mechanical tests as needle penetration, Ring&Ball and Fraass fracture temperature. For elastomer (styrene-butadiene-styrene) polymer modified bitumens these tests are not sufficient to show the important differences in bitumens. Elastic recovery and cohesion provide better insight, but rheological properties cannot be adequately described with conventional test.
The requirements of the polymer modified bitumens (PmB) in Europe were defined in EN 14023 in 2010 [1]. Since then several new tests were introduced in the research field and their procedures improved. In the European Standards Committee (CEN) TC 336, there is ongoing work to develop performance related specifications. New laboratory test methods from American standards were adapted and transformed into EN standards (bitumen laboratory aging methods and rheological tests). These test methods are not yet employed in the current PmB European standard, however, the draft prEN 14023, April 2020 [2] suggests these new tests. In the recent years at ZAG Laboratory for asphalts and bitumen-based products long-term aging of bitumen by pressure aging vessel (PAV) and rheological tests were introduced. Traditional bitumen test methods are performed together with new rheological tests e.g. Dynamic Shear Rheometer (DSR) testing, to characterize complex modulus and phase angle, and Multiple Stress Creep Recovery Test (MSCRT) in order to develop a preliminary data base on PmB's, which are frequently used in Slovenia.
The paper presents the current requirements for PmB's in Slovenia and test results on PmB 45/80-65, original, laboratory aged and extracted from produced asphalt mixtures.

Keywords: polymer modified bitumen, recovered bitumen, laboratory ageing, dynamic shear rheometer, multiple stress creep recovery test

1 Introduction

In Slovenia polymer modified bitumens (mostly type PmB 45/80-65) are regularly used on motorways for surface (SMA11) and binder (AC22bin) asphalt layers. In the past years many samples of PmB 45/80-65 were tested at ZAG Laboratory (original, laboratory aged and extracted from asphalt). ZAG laboratory for asphalt and binder products performs quality control of asphalt mixture production and asphalt layer compaction. The testing includes samples of original bitumen as well as recovered bitumen from produced asphalt mixtures. In this way, the test results can be linked to the performance of the corresponding road sections during their life span. According to CEN rules, each country may adopt suitable requirements for their climate region or other reasons in national standards. Based on EN 14023 the national delivery requirements for PmB's were set in Slovenian Institute for Standardization (SIST) document titled SIST 1035 in 2008.

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Since then the requirements, as presented in Table 1, have not been changed. There are no requirements for rheological characteristics however we started to perform these tests to develop a preliminary data base.

Table 1 National requirements for modified bitumen PmB

Conventional test methods	Standard	Unit	PmB 45/80-65
Needle penetration @ 25 °C	EN 1426	mm/10	45-80
Softening point R&B	EN 1427		≥65
Fraass breaking point	EN 12593	°C	≤ -18
Cohesion, Force ductility	EN 13589	[J/cm²]	≥ 2 at 25 °C
Elastic recovery @ 25 °C	EN 13398	%	≥80
Stability against hardening (RTFOT)	EN 12607-1		
Change of mass after RTFOT	EN 12607-1	M.-%	≤ 0.50
Change of softening point after RTFOT	EN 1427		no requirement
Retained penetration after RTFOT	EN 1426	%	≥60
Elastic recovery @ 25 °C after RTFOT	EN 13398		≥70
Additional test methods			
Dynamic Shear Rheometer (DSR)			
G* (30 °C to 90 °C) at 15 kPa (25 mm plate)	EN 14770		no requirement
Multiple stress Creep and Recovery Test			
MSCRT @ 60 °C Jnr 3.2 kPa after RTFOT	EN 16659	[1/kPa]	no requirement

2 Results of tests on original and laboratory aged bitumens PmB 45/80-65

On neat bitumen the conventional tests R&B [3], Penetration [4], Fraass [5] were performed as well as new tests MSCRT [6], and DSR [7], [8]. In our study DSR test with continuous increase in testing temperature, as described in a draft EN standard [7] and guidelines [9], was performed. According to proposed [2] temperature for G* =15 kPa and corresponding δ should be evaluated. In this research all tests were performed on 25 mm plates. Original bitumens have been laboratory aged with RTFOT [10] method and all tests were repeated. Rolling Thin Film Oven Test (RTFOT) is a laboratory method that simulates or short term ageing during mixing, transport and compaction. In the last step, the five bitumens were laboratory aged with RTFOT and PAV [11] method and then re-tested. The results are presented in Tables 2 and 3 and in Fig. 1 and Fig. 2.

Table 2 Results on neat (original) bitumen PmB 45/80-65

Sample	Pen	$T_{R\&B}$	T_{Fraass}	G* at 15 kPa	δ at $T_{G^*=15kPa}$	%R at 3.2 kPa	Jnr at 3.2 kPa
No.	[0.1 mm]	[°C]	[°C]	[°C]	[°]	[%]	[kPa^{-1}]
1	53	83.4	-18	54.0	62.9	77	0.203
2	51	80.4	-18	54.4	64.2	76	0.252
3	59	81.6	-20	53.5	58.4	96	0.028
4	47	77.0	-18	54.8	62.3	60	0.350
5	59	78.4	-19	53.6	55.5	95	0.030

The tests on original bitumen are primarily intended to control the conformity of the supplied bitumen with the indications on the declarations of performance.

Table 3 Results on laboratory aged (RFTOT) bitumen PmB 45/80-65

Sample	Pen	$T_{R\&B}$	T_{Fraass}	G* at 15 kPa	δ at $T_{G^*=15kPa}$	%R at 3.2 kPa	Jnr at 3.2 kPa
No.	[0.1 mm]	[°C]	[°C]	[°C]	[°]	[%]	[kPa^{-1}]
1	37	73.2	-12	58.6	61.6	60	0.198
2	37	74.0	-10	60.3	62.1	59	0.184
3	40	79.2	-14	59.9	55.1	91	0.033
4	34	74.8	-7	61.1	59.8	63	0.138
5	41	78.4	-12	57.6	54.4	92	0.034

The results of tests after RTFOT aging indicate the characteristics of the bitumen in freshly produced and transported asphalt mixture. Typically, PmB 45/80-65 penetration is between 30 and 40 [0.1 mm]. For Slovenia climate conditions $T_{R\&B}$ should be above 70 °C, T_{Fraass} preferably as low as possible. DSR should be performed at 1.59 Hz (10 rad·s–1). The DSR test results show that the temperature at which G* = 15 kPa, is usually between 50 °C and 60 °C. Two tested samples (MSCR test) had a recovery above 90 % at 60 °C and a non-recoverable creep Jnr at 3.2 kPa of less than 0.100 kPa^{-1}, which indicates good viscoelastic response.
For PmB 45/80-45 the conventional tests do not give relevant results for quality control. The softening point $T_{R\&B}$ of original PmB is close to 80 °C, therefore the tests should be performed both in water and in glycerol. The decrease in $T_{R\&B}$ after RTFOT ageing mainly depends on the type and characteristics of polymers used, and therefore depend on the producer of polymers. The $T_{R\&B}$ of original bitumens is much higher than the minimum required value ($T_{R\&B}$ › 65 °C). From 2003 till 2006 ZAG was included in BitVal project [12]. The Bitumen Test Validation (BiTVal) project was set up by the Forum of European National Highway Research Laboratories (FEHRL) in response to a request from TC 336, Bitumen and bituminous binders, of the CEN together with other stakeholders in the industry to assess the relevance of the results of bitumen tests on the required properties of asphalt mixtures. One of the conclusions of the BitVal project was that the $T_{R\&B}$ is not appropriate test method to designate permanent deformation for asphalt layers containing PmB.

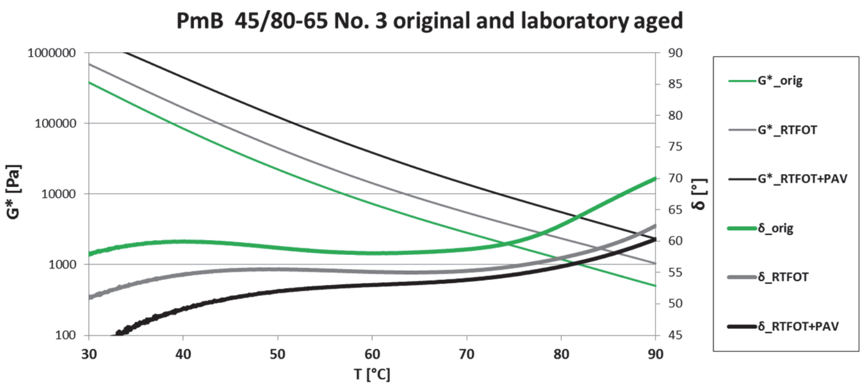

Figure 1 DSR – Temperature sweep results for a PmB 45/80-65

Fig. 1 shows results of DSR test (continuous increase of test temperature) for sample No. 3 in the range of 30°C to 90°C. The graph shows G* and δ as a function of temperature for original bitumen (_orig), after laboratory short term aging (_RTFOT) and after laboratory long term aging (_RTFOT+PAV). The bitumen gets stiffer with aging (G* increases), while δ decreases.

3 Results of rheological tests on naturally aged bitumens from SMA asphalt mixture

The same aforementioned bitumens (Samples No.1 to No. 5) were used in SMA asphalt mixtures produced in various asphalt plants. In ZAG laboratory bitumen PmB 45/80-65 extracted [13] and recovered [14] from asphalt mixtures (referred thereafter 'extracted') was investigated. For sake of clarity only results of extracted bitumens numbered No. 2, No. 3 and No. 4 (denoted by '_ex') are presented (Fig. 2). It was expected that the results would be similar to those after short-term aging (RTFOT). DSR results show that samples aged to the similar extent during mixing in the asphalt plant and during RTFOT ageing in the laboratory. However the rheological behaviour of PmB 45/80-65 of different bitumen producers can significantly differ after RTFOT ageing or mixing at asphalt plant.
We also compared the results of MSCR tests (@ 60°C) of these three bitumen samples. Comparison of non-recoverable creep compliance Jnr at 3.2 kPa and recovery R [%] at 3.2 kPa is shown in Fig. 3. The line shown in the graph is defined in AASHTO R 92-18 specifications [15] to assess elastic response for RTFOT aged samples. For heavy traffic loads MSCRT characteristics should be above this line. The results for RTFOT aged and extracted sample No. 3 are practically identical. The results for samples No. 2 and No. 4 show that the PmB's aged differently during mixing at the asphalt plant and transport than during RTFOT in laboratory. The shown differences in results of DSR and MSCRT tests may be due to the extraction and recovery process of PmB bitumen. From Fig. 3 it can be seen that all tested PmBs fulfilled the requirement in AASHTO R 92-18 specifications [15].

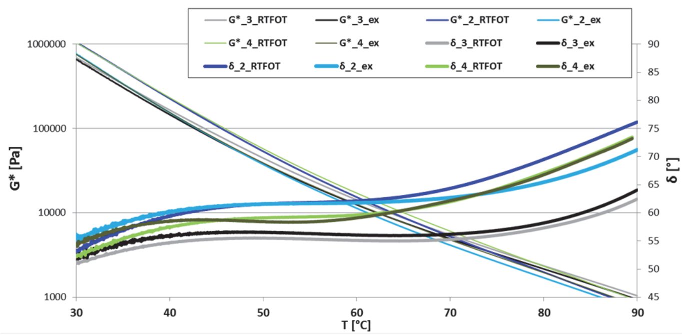

Figure 2 DSR for RTFOT aged and extracted samples No.2, No.3 and No.4

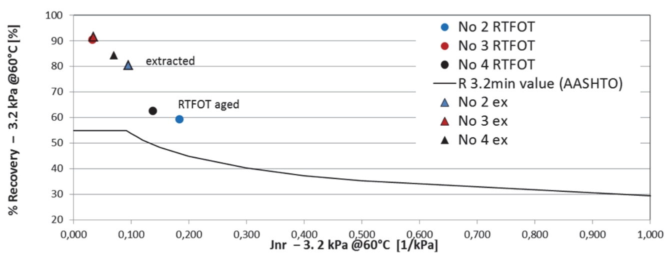

Figure 3 MSCRT results on RTFOT aged and extracted samples.

4 Results of rheological tests on naturally aged bitumens from different asphalt mixtures

In Table 4 and Fig. 4 and Fig. 5 are presented results of PmB 45/80-65 extracted in 2020 from eight different asphalt mixtures (ACbase, ACbin, SMA, PA). All asphalt samples were taken at construction sites at paver and tested in ZAG's laboratory. The results of DSR test present an impression of expected rheological characteristics G* and δ for aged PmB 45/80-65 bitumens. Fig. 4 and Fig. 5 provide a good insight into the rheological behaviour of aged PmB 45/80-65 bitumens. The complex modulus of bitumen labelled as A and C are very similar through the temperature range, while there are obvious differences for the phase angles. It should be noted that original bitumen was produced in different refineries.

Table 4 Results of extracted and recovered bitumen PmB 45/80-65 from different asphalt mixtures

Sample	Pen	$T_{R\&B}$	T_{Fraass}	G* at 15 kPa	δ at $T_{G^*=15kPa}$	%R at 3.2 kPa	Jnr at 3.2 kPa
Label	[0.1 mm]	[°C]	[°C]	[°C]	[°]	[%]	[kPa-1]
A AC22base	53	79.4	-17	53.8	60.8	93	0.052
B SMA 11	40	78.8	-19	59.8	55.9	86	0.044
C AC 22bin	57	85.0	-16	53.2	60.4	96	0.032
D AC22base	40	81.0	-18	55.5	58.7	93	0.040
E SMA11	35	71.8	-15	60.7	58.0	78	0.064
F AC22base	44	79.2	-19	54.7	61.2	91	0.065
G AC22bin	42	74.8	-17	56.7	60.2	86	0.077
H PA11	38	75.8	-18	59.2	57.5	N/A	N/A
average	42	78.1	-17	57.1	58.8	89	0.054
min	35	71.8	-19	53.2	55.9	78	0.032
max	57	85.0	-15	60.7	61.2	96	0.077

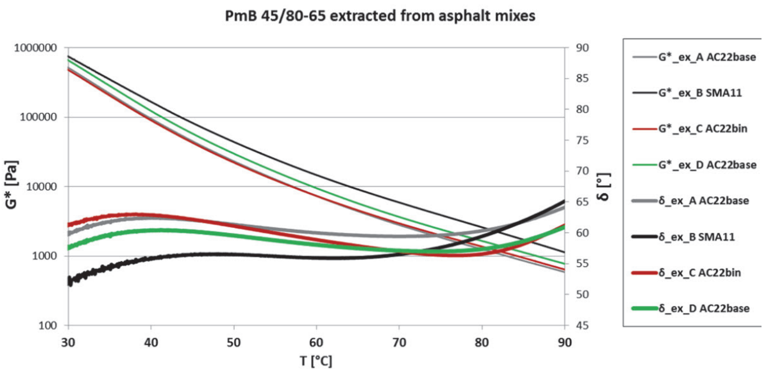

Figure 4 DSR results for PmB 45/80-65 extracted from asphalt mixes A to D.

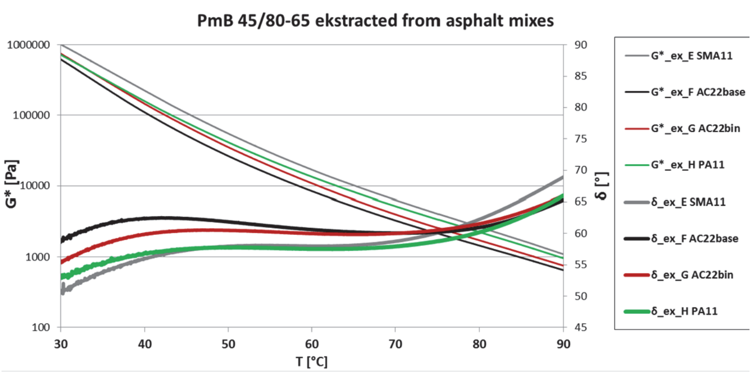

Figure 5 DSR results for PmB 45/80-65 extracted from asphalt mixes E to H.

In Fig. 6 are graphically presented MSCRT results of PmB 45/80-65 extracted from asphalt mixtures A to G in 2020. Results of MSCRT tests show that they all exhibit good elastic response, as their results lie above the AASHTO R 92-18 requirement. All PmBs included in our study fulfilled the requirement in AASHTO M 332-18 [16] specifications for extremely heavy traffic.

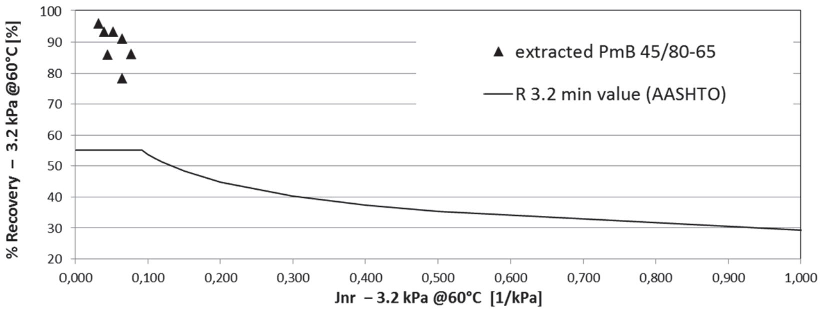

Figure 6 MSCRT results for PmB 45/80-65 extracted from asphalt mixes A to G.

5 Conclusions

From this study we concluded that test laboratories for asphalt should develop the relevant competences to perform the rheological tests, and at the same time the users (asphalt producers, road authorities, etc.) should become familiar with the results of these tests.
Goal of our study was to inform advanced and trusty paving contractors about the characteristics of the RTFOT aged bitumens, particularly PmBs, so they will be able to choose the appropriate bitumen for a specific project. Rheological DSR test, used in our study, provide good insight in bitumen behaviour through temperature range. MSCRT test provide information about elastic behaviour of bitumen, which is very important for polymer modified bitumens. The only requirement that we could rely on was AASHTO R 92-18 specification. All PmBs included in our study showed a significant elastic response for the associated value of non-recoverable creep compliance. All PmBs included in our study fulfilled the requirement in AASHTO M 332-18 [16] specifications for extremely heavy traffic. Rheological tests are thus a very good complement to conventional tests. The results show that rheological characteristics for a PmBs produced in the same refinery having an unchanging production exhibit similar rheological graphs for original bitumen and after RTFOT ageing. DSR results of extracted PmB often do not correspond to RTFOT results. We found out that results for RTFOT aged and

extracted sample No. 3 are practically identical, but the results for samples No. 2 and No. 4 show that the PmB's aged differently during mixing at the asphalt plant and transport than during RTFOT in laboratory.
For optimization of design of asphalt pavements long term monitoring of road sections should be performed. It is beneficial that a database of performance of laid asphalt and bitumen is formed. With sufficient quantitative data limit values will be set for the results of rheological tests, which will serve for quality control, for both original and (laboratory and naturally) aged bitumens.

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