Complex Bituminous Binders, Are Current Test Methods Suitable for?

Complex bituminous binders, are current test methods suitable for?

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Abstract. The asphalt industry is constantly working to enhance the performances of asphalt materials, introducing innovative and more sustainable solutions. In this context, the incorporation of materials, such as additives, polymers, is more and more used to improve the properties of neat bitumen. This leads to even more complex bituminous binders, raising the question, are the current specifications and test methods appropriate for complex materials? To deal with this, the RILEM Technical Committee 272-PIM 'Phase and Interphase behaviour of innovative bituminous Materials' with its Task Group TG1 is looking at the efficiency of various test methods for complex binders with an extensive inter-laboratory program with 17 laboratories. It includes seven different binders, two neat bitumen, two polymer modified bitumen and three binders with liquid additives, emphasising on compositional and physical changes at different conditions. The focus is low temperature; while a complementary experimental program encompasses as well as testing at intermediate and high temperatures. The outcomes of the work will provide indications on how robust the current binder characterisation techniques are and establish technical recommendations for future test methods specially designed for complex binders. Some first results are presented hereby.

Keywords: Bitumen, Polymer modified Bitumen, asphalt additive, BBR, Rheology, Chemistry, Test protocol

1 Introduction

The worldwide demand for long-lasting pavement is leading the asphalt industry to incorporate various chemical substances, such as polymers or additives, in order to enhance the sustainability of pavement materials. Thus, performance-based binders need, together with the implementation of new modification technologies in bitumen, accurate standardised characterisation protocols and in-depth understanding of phase and interphase phenomena of these binders.

Within this framework, RILEM initiated the Technical Committee 272-PIM '*Phase and Interphase behaviour of innovative bituminous Materials*' on assessing the performance of complex binders. It aims to provide recommendations for the experimental tools and protocols used by the asphalt research and engineering community on bituminous binders. For this purpose, the Task Group 1 (TG1) of RILEM 272-PIM is specifically focusing on binders, while TG2 is looking at asphalt mix and TG3 on pavement structure. Herein, some preliminary results of TG1 activities are presented.

The current characterisation protocols have been initially developed for conventional binders and applied for modified bitumen. Penetration value and softening point temperature, amongst others, are empirical properties used in specifications [1]; while, since the initiation of the Strategic Highway Research Program (SHRP) Superpave program, more fundamental properties using Dynamic Shear Rheometer (DSR) were introduced [2]. There was already some fundamental work, within the RILEM, on characterising the physico-chemical properties of bituminous binders [3] [4]. Furthermore, the activities of TG1 are extending to complex binders.

In this research, focus was given on elucidating the low temperature performance of fresh and aged complex binders formulated with polymers and liquid additives by using amongst others, Fraass breaking point, Bending Beam Rheometer (BBR), 4-mm plate DSR, Differential Scanning Calorimetry (DSC). A complementary testing program focuses on intermediate and high temperature domains as well. A total of 17 laboratories expressed interest in this experiment, 13 from Europe and 4 from the US. The experiment started earlier in 2019 and about 50% of the results have been delivered by the end of 2019. Examples of preliminary analyses are presented in this paper.

2 Experimental plan

The way to consider complex binders is wide and includes various materials, liquid or viscous additives either bio-based or hydrocarbon-based, polymers, solid particles. The amount used can vary between dopes from 0.1 to 1.5 %, or additives from 2 to 7-10 %, or extender from 20 to 40 %, or replacement above 50 %, all per weight of bitumen. In order to restrain the scope, only two groups of complex binders were considered, polymer modified binders and additive blends, and compared with neat bitumen.

Group 1 is for assessing Polymer modified Bitumen with:

- Bit1, a standard 35/50 pen grade bitumen used as a reference
- PmB1, a standard commercial Polymer modified Bitumen
- PmB2, a highly polymer modification with 7.5 % of high vinyl SBS in Bit2

Group 2 is to assess liquid additives in bitumen with:

- Bit2, a standard 70/100 pen grade bitumen used as a reference
- Blend1, 4% of commercial bio-based recycling additive in Bit1
- Blend2, 8% of Refined Engine Oil Bottom (REOB) in Bit1
- Blend3, 4% of paraffinic oil typically not used as paving additive in Bit1

For Group 1, the reference bitumen and PmBs were selected having the same consistency at intermediate temperature; PmB1 was industry produced and composition is proprietary information; PmB2 was lab produced. For Group 2, the dosage per blend was determined with the aim at resulting in properties as close as possible to the properties of the reference bitumen, Bit2; all blends being lab produced.

As the goal of the TG was not to run a round robin on testing, the experimental program was agreed and defined as a toolbox, where each laboratory could select the most appropriate way to assess the binders and depending on the available equipment of each laboratory. Table 1 shows the experimental matrix with, for each binder and test, the number of laboratories, and, in brackets, the numbers of results provided by end of 2019.

Table 1. Rilem PIM TG1 - Experimental matrix

Sample	Bit 1	PmB 1	PmB 2	Bit 2	Blend 1	Blend 2	Blend 3
Aging conditioning							
RTFOT	7 (5)	8 (5)	9 (6)	9 (8)	7 (6)	8 (7)	7 (6)
PAV	7 (5)	8 (5)	9 (6)	9 (8)	7 (6)	8 (7)	7 (6)
Low temperature testing							
Fraass	3 (2)	4 (2)	5 (3)	3 (3)	2 (2)	2 (2)	2 (2)
BBR	7 (2)	8 (2)	9 (3)	9 (5)	8 (4)	8 (4)	8 (4)
ABC	1 (0)	1 (0)	1 (0)	1 (0)		1 (0)	
3 notch beam	2 (0)	2 (0)	2 (0)	2 (0)	2 (0)	2 (0)	2 (0)
4mm DSR	6 (3)	7 (3)	7 (3)	6 (3)	5 (3)	6 (3)	5 (3)
DSC	5 (2)	5 (2)	5 (2)	5 (2)	4 (1)	5 (2)	4 (1)
Intermediate and high temperature testing							
Penetration	8 (4)	9 (4)	10 (5)	9 (6)	8 (5)	8 (5)	8 (5)
Softening Point	7 (3)	8 (3)	9 (4)	8 (5)	7 (4)	7 (4)	7 (4)
Force ductility	2 (1)	2 (1)	3 (2)				
Elastic recovery	2 (1)	2 (1)	3 (1)				
DSR	12 (5)	13 (5)	14 (6)	14 (7)	12 (6)	13 (6)	12 (6)
MSCR	7 (0)	7 (0)	8 (1)	10 (3)	8 (2)	9 (2)	8 (2)
Other testing							
Storage stability	2 (1)	3 (2)	4 (2)	3 (1)	2 (1)	2 (1)	2 (1)
FTIR	8 (6)	8 (6)	9 (7)	10 (9)	8 (7)	9 (8)	8 (7)
Microscopy	3 (0)	3 (0)	4 (0)	4 (0)	3 (0)	3 (0)	3 (0)

3 Results

As shown in Table 1, the experiment is still ongoing, with 50 % of data available, therefore, only partial analysis is presented here. As a first attempt, the different binders, from Group 1 or Group 2, were analysed towards current specifications. Fig 1 displays them in the pen grade system, as used in Europe (EN 12591, EN 14023), with penetration value at 25 °C and softening point temperature. Fig 2 displays them in the Performance Grade (PG) system, as used in the US (AASHTO M320) with the exact low, medium and high temperature criteria. For each figure, the points are for the mean value and the error bars representing the maximum and minimum values.

With pen grade system, depending on the binder, the analysed data set was from one to maximum three laboratories. The Group 1 binders, Bit1, PmB1 and PmB2 were displaying similar penetration value at 25 °C, while the softening point temperatures were much more discriminant. For the Group 2, Bit2, Blend 1 to 3 were all in the 70/100 pen box and, considering the error bars, there was no significant difference among them. For all binders, the variability between labs was relatively low except for the PmBs with high value of softening point temperature showing variation between 2 and 3 °C. However, the number of data points is still limited.

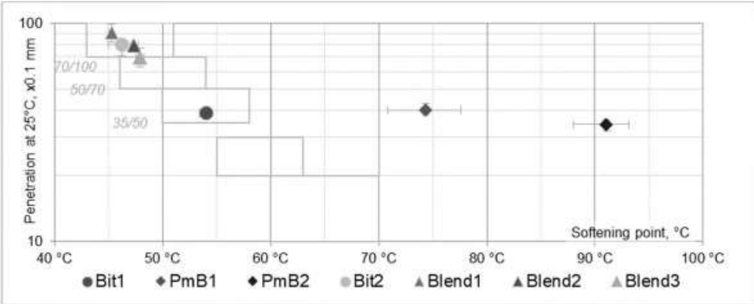

Fig 1. The seven binders in the pen grading system

Depending on the binder, the PG grading data set analysed was from one (Bit1, PmB1) to maximum four laboratories (Bit2). With Group 1, as compared to the neat bitumen, the temperature span for PmB was extended, especially at high temperatures. The PmB2, using Bit2 as base bitumen, maintained the low and intermediate temperatures and increased significantly the high temperature. For Group 2, all blends used the Bit1 as base bitumen and displayed similar shift to the left either for low, intermediate and high temperatures. Overall, the properties were not significantly different from the reference bitumen Bit2. The variability between labs, as shown with error bars, was relatively low except for PmB2. However, more data is needed here.

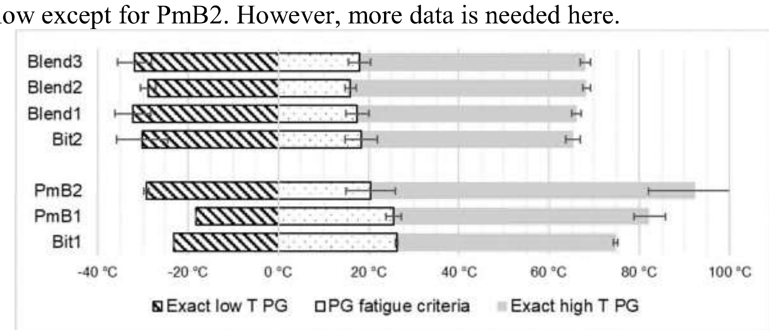

Fig 2. The seven binders in the PG grading system

Between the pen and PG grade system, the binders from the different groups disclosed the same grading. However, considering penetration value at 25 °C and PG intermediate critical temperature, addressing similar temperature domain, there were interesting differences. The pen values for Group 1 binders were similar and lower than Group 2, while for the PG intermediate T, PmB2 value was lower by almost 5 °C than PmB1 and Bit1 and in the same magnitude of range as the Group 2 binders. The PG grading system helps to better distinguish between complex bituminous binders.

Further analysis is ongoing including ductility, DSR data analysis with broader parameters and low temperature properties. Herein, a preliminary analysis of low temperature testing results is provided as an example. Fig 3 displays, in a bar chart, the Fraass breaking point temperature with the BBR temperatures when the creep stiffness $S \leq 300$ MPa and the relaxation m-value ≥ 0.300, including the delta between them, known as ΔTc. Depending on the binder, the sets of data were from between two and four laboratories. As expected, the trend was similar at least, the lower the Fraass temperature, the lower the BBR critical temperatures were, although for the Blend1, the Fraass seems to underestimate the low temperature performance as indicated by BBR.

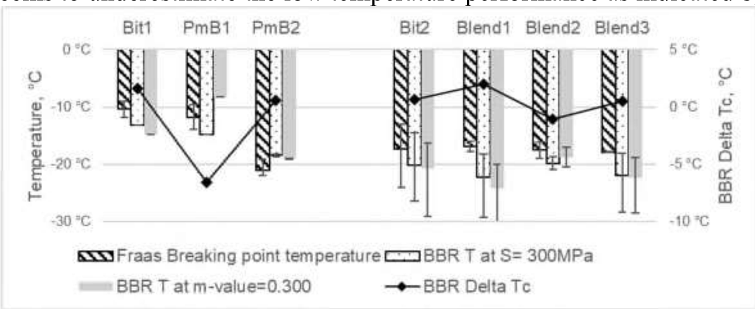

Fig 3. Low temperature Fraass and BBR for the seven binders

A comparison by pair is made in Fig 4 between (a) the two BBR temperatures and (b) the Fraass breaking point and the BBR continuous (as the max of T_S and $T_{m\text{-value}}$) temperatures; the diagonal line is for equi-temperature. For the BBR, considering variability within the labs, there is a good balance between both criteria except for PmB1. At comparing the Fraass and BBR temperatures, for the Group 2, the Fraass is significantly higher than the BBR and all ranking in the same magnitude of range. The BBR continuous temperature seems to better discriminate among them, within the variability of the set of data. With more data including 4 mm plate DSR and DSC, further analysis will be presented in a future study.

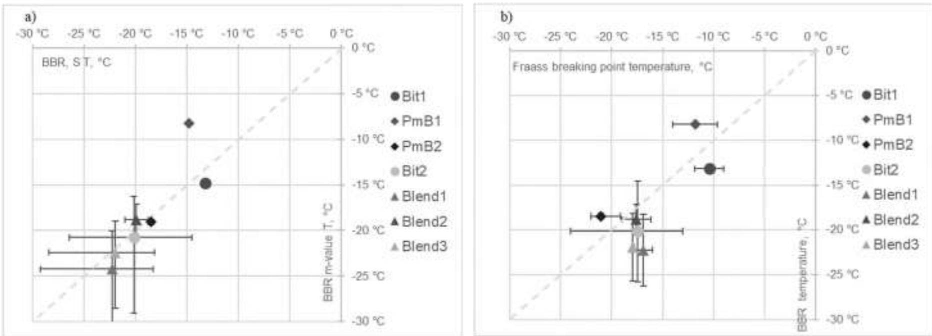

Fig 4. Comparison of (a) BBR S and m-value critical temperatures, (b) Fraass and critical BBR

4 Conclusion

The RILEM 272-PIM has set up a specific Task Group, TG1, looking at phase and interphase of complex bitumen. Two groups of binders, Polymer modified Bitumen and liquid additive blends, are included in an exhaustive experimental program encompassing physical and chemical properties. Seventeen laboratories are involved, providing a unique data set and characterisation approach on complex binders. The activities are still ongoing, and some preliminary results are presented in this paper, as example.

In terms of standard specifications, PmB binders clearly display very different properties, and the additive group shows all binders within a similar grading, in either pen grade or PG grade systems. Thanks to the PG system and by recording properties at low, intermediate and high temperature, some binders were already distinguished more efficiently at intermediate temperatures as compared with pen grade system.

Low temperature properties were further analysed based on empirical testing with Fraass breaking point and more fundamental rheology characterisation with BBR testing. While Fraass can distinguish between the two groups, within each group such as between additive blends, it does not enable to differentiate binders. By combining both creep stiffness and m-value critical temperatures, BBR provides a more efficient diagnosis of different complex bituminous binders.

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References

[1] BitVal, "Analysis of Available Data for Validation of Bitumen Tests," Report on Phase 1 of the BitVal Project, 2006.

[2] D. Anderson, D. Christensen, H. Bahia, R. Dongre, M. Sharma, C. Antle and J. Button, "Binder characterization and evaluation Volume 3: Physical characterization," Washington, 1994.

[3] H. Soenen, J. Besamusca, H. Fischer, L. Poulikakos, J. Planche, P. Das, N. Kringos, J. Grenfell, X. Lu and E. Chailleux, "Laboratory investigation of bitumen based on round robin DSC and AFM tests," *Materials and Structures,* vol. 47, no. 7, pp. pp 1205-1220, 2013.

[4] L. Poulikakos, B. Hofko, A. Cannone Falchetto, L. Porot, G. Ferrotti and J. Grenfell, "Recommendations of RILEM TC 252-CMB: relationship between laboratory short-term aging and performance of asphalt binder," *Materials and Structures,* vol. 52, no. 69, 2019.