Low temperature and aging properites of polymer-modified binders

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ABSTRACT

The search for a “universal” (applicable to any kind of bituminous binder) test describing the performance of bituminous binders at low temperatures is still ongoing. Although several test methods exist, they remain questionable and discussed, be it in relation to their reliability (reproducibility / universality of Fraass testing) or for phenomenological reasons (e.g. no crack phenomenon for BBR testing).

Our paper presents the results of a study devoted to the validation of different methods measuring the low temperature behaviour of bituminous binders. This validation was performed in reference to the restrained specimen thermal shrinkage test (TSRST) performed on an standard asphalt mix-design. In addition to both Fraass and BBR testing, a newer method (ABCD test) for the assessment of cracking behavior has been evaluated as well. The investigation focused on pure bitumen and binders modified at several levels of content with an elastomeric polymer. The majority of the modified binders have been obtained by a technique of crosslinking but comparisons were also made with equivalent un-crosslinked physical mixture binders. Finally, an important part of the investigation was devoted to a concomitant analysis of the sensitivity of these test methods to the degree of aging of both binders and asphalt mixes.

Keywords: Low-Temperature, Modified Binders, Polymers, Rheology, Thermal Cracking
1. **INTRODUCTION**

The search for truly performance related binder tests, which means in particular test methods and performance criteria which are equally applicable to both modified and unmodified bituminous binders, is more than ever an important topic for binder and asphalt industry [16, 17]. The purpose of the work presented here was to compare and validate different possible binder tests on their ability to predict low temperature failure behaviour of asphalt as measured through the Tensile Strength Restrained Specimen Test (TSRST) performed on a standard reference mixture. Beside the Fraass low temperature brittleness test and flexural creep stiffness data from the Bending Beam Rheometer (BBR), the more recent ABCD (Asphalt Binder Cracking Device) procedure developed in the USA has also been evaluated. The study has been based on several unmodified bitumens which have further been modified at different rates with an elastomer polymer. Most of the modified binders have been obtained through cross-linking but they have also been compared to equivalent simple physical blends.

2. **TEST EQUIPMENT AND PROCEDURES**

2.1 **Low temperature behaviour of asphalt - TSRST**

The TSRST is performed in a temperature controlled test chamber. The vertically mounted cylindrical asphalt sample is clamped at both ends and kept at a fixed length while temperature is dropped at a given constant rate. As a consequence, thermal stress builds up in the sample until it ultimately breaks. Development of stress with time, failure stress and failure temperature are recorded. The test does mimic “single event” low temperature failure associated to important drops in temperature. A particular interest of this test is that it generates a uniform average stress distribution over the cross-section of the sample, which is a favourable condition for the obtaining of correlations with binder characteristics.

The tests have been performed on an MTS electro-hydraulic servo-loop test rig following the AASHTO TP10 procedure [1] with however some small deviations. The samples had a diameter of 57.5mm for a height of 250mm. Testing started at an initial temperature of 5°C after a temperature equilibration period of 4 hours. Temperature has been dropped at a rate of 10°C/h. Three replicates have been tested per mixture.

2.2 **Low temperature tests on binders**

The Fraass test [6] is performed on a thin film of binder (0.5mm) coated on a thin metal plate which is cooled down at a fast rate (1°C/min) and repeatedly flexed until failure is obtained. The temperature at failure is recorded as the Fraass breaking point temperature. The procedure has the advantage of characterizing failure behaviour but is often blamed for poor reproducibility (6°C for unmodified bitumen and probably more for modified binders). This is ascribed to difficulties in test sample preparation and differences in the various possible operating procedures (manual, semi-automatic, fully automatic). In the frame of this study, automatic BPA 5 equipment has been used and all tests have been performed by a single laboratory so as to escape reproducibility artefacts.
The **Bending Beam Rheometer (BBR)** [9], which is an outcome of the SHRP (Strategic Highway Research Program) program in the USA, characterizes flexural creep stiffness at low temperature while applying a constant load (100g) on a beam sample (127 x 12.7 x 6.4mm) for 240s. At a loading time of 60s, two characteristic temperatures are determined with this test:

- \( T_S = 300 \text{ MPa} \), which is the temperature at which stiffness becomes equal to 300 MPa
- \( T_m = 0.3 \), which is the temperature at which the slope \( m \) (absolute value) of the log(S) versus log(time) curve becomes equal to 0.3. The \( m \)-value can be seen as indicating the propensity of the material to deform under an applied stress.

It is to be mentioned that, unlike Fraass which is a failure test, the BBR procedure only characterizes stiffness evolution under a constant load. A relationship to failure is then a matter of correlation, which may not be universal (binder dependent).

BBR testing has been performed at 3 temperatures (2 repeats per temperature) for each binder, again by a single laboratory. To avoid repeated reheating, and contrarily to EN 14771 which requests testing to be done within 4 hours after sample preparation, all test samples have been prepared simultaneously 24 hours in advance.

The **ABCD (Asphalt Binder Cracking Device)** test (AASHTO TP92-11) [2] has been developed in the USA as the binder counterpart of the TSRST (Tensile Strength Restrained Specimen Test) procedure on bituminous mixtures. The bituminous binder is poured into a silica mould around an invar steel ring (outer \( \Phi \approx 54\text{mm} \)). The cross-sectional area of the binder is 0.5” x 0.25” (12.7 x 6.35 mm). The mould is then placed in an environmental chamber and cooled down at a fixed rate (20°C/h starting from 20°C in the case of AASH TO TP92-11). As it tends to shrink, the binder compresses the invar ring and the resulting stress is obtained from strain gauges mounted on the invar rings. The location of the failure is controlled via a protrusion (reduction of cross-sectional area) placed at a given point of the circumference. Theoretical considerations (stress concentration factor) [3] allow calculating the actual failure stress at the protrusion from the recorded average thermal stress at failure.

Previous research proved the correlation of the ABCD test with cracking tests on mixes and showed a good ability of this test to discriminated polymer-modified bitumen from unmodified ones [5].

For our study, the ABCD tests have been contracted to Western Research Institute (WRI, Laramie, WY). To be as close as possible to the practice of the TSRST test on bituminous mixtures, the starting test temperature has been set at 5°C and the cooling rate slowed down to 10°C/h.

Others tests exist to characterize the failure of asphalt binders at low temperature [11, 12, 13, 16]. But these fracture tests were not included in this study because they are difficult to run and are impractical for specification purposes.
3. TESTED MATERIALS

3.1 Asphalt mix

To minimize possible bias due to coating and compacting problems and to ensure a good homogeneity in the quality of the asphalt mixtures prepared from the different bituminous binders, a continuously graded wearing course formulation, with a maximum aggregate size of 10 mm (AC 10 surf) and complying with NF EN 13108-1 has been selected. Compositional data are given in Table 1.

3.2 Investigated binders and available test results

The study had to cover a wide range of pure and modified binders likely to be used in asphalt mixes. The final selection was as follows.

- Five unmodified paving grade bitumen (penetration classes 20/30, 35/50 and 50/70 according to EN 12591[10]) of which the three 35/50 bitumen, identified as A, B and C were of different origins. The bitumen noted 20/30-A, 35/50-A et 50/70-A came from the same refinery.
- Eleven “Styrelf®” cross-linked elastomer modified binders which have been made on the basis of the above mentioned bitumen at different levels of polymer content (2, 3.5 and 5%). Bitumen 35/50-B and 35/50-C have however only been modified at 3.5% polymer content.
- Two “physical blends” at 3.5% polymer made from bitumen 35/50-A and 50/70-A. The used SBS polymer had however a higher molecular weight than the polymer used for the Styrelf® products, this so as to compensate for non-cross-linking.

Table 2 gives an overview of the results which have been generated for these various binders.

<table>
<thead>
<tr>
<th>Bitumen</th>
<th>Styrelf®</th>
<th>PB</th>
<th>Fraass-o</th>
<th>BBR-o</th>
<th>BBR-p</th>
<th>ABCD-o</th>
<th>ABCD-r</th>
<th>ABCD-p</th>
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<td>x</td>
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<td>x</td>
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<td>x</td>
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<tr>
<td></td>
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<tr>
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<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>

Styrelf® : cross-linked elastomer modified bitumen
PB : “physical blends” with an SBS elastomer
- o : fresh binder
- r : after RTFOT+PAV ageing (EN 12607-1 followed by EN 14769)

Table 1: Asphalt formulation

<table>
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<th>AC 10 surf</th>
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<tr>
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<tr>
<td>Binder</td>
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</tr>
<tr>
<td>Voids</td>
<td>5 - 8</td>
</tr>
</tbody>
</table>

In the following, we will successively address the results obtained on pure and modified binders. In each case, we will first discuss the bare TSRST results and then attempt correlations with measured binder properties.
4. EXPERIMENTAL RESULTS – UNMODIFIED BITUMEN

4.1 TSRST results

For a same penetration grade (35/50), marked differences are seen depending on the origin of the bitumen (Figure 1). One may also question the ranking of bitumen 35/50-A versus 50/70-A. The difference between the corresponding fracture temperatures, slightly to the advantage of 35/50-A, may possibly be ascribed to experimental uncertainty and to the low penetration value of 50/70-A which is on the low side of the specification range (see table 3).

4.2 Search for correlations with binder properties

4.2.1 “Failure” properties – Fraass and ABCD

When plotting Fraass breaking point against TSRST fracture temperature, we notice that Fraass does well differentiate bitumen according to penetration classes but seems unable to reflect the differences evidenced by TSRST between bitumen of the same penetration grade (35/50) but coming from different origins (Figure 2a). ABCD results are only available for origins A and C. ABCD failure temperatures (shown for fresh bitumen in Figure 2b) are fairly well in line with TSRST fracture temperatures although the TSRST temperature obtained for bitumen 50/70-A is higher than what would be suggested by the ABCD result. It is further to be noted that Fraass values are “pessimistic”, in the sense that Fraass predicts failure at a higher temperature compared to TSRST. On the other hand, ABCD values are “optimistic” relatively to TSRST.

4.2.2 Flexural creep stiffness – BBR

Figure 3a shows the evolution of creep stiffness S in relation to temperature in the BBR test. The different curves are essential parallel (however with a slightly different slope for bitumen 35/50-C) and do also show a close behaviour between bitumen 35/50-A and 50/70-A. Similar evolutions are obtained for the m parameter (Figure 3b) for which only bitumen 50/70-A shows a somewhat different evolution.

These curves validate the use of the critical temperatures $T_{S=300}$ MPa and $T_{m=0.3}$ for the search of correlations to TSRST fracture temperatures since binder ranking stays essentially the same whatever the chosen level for S and m. Figure 4a and 4b show a quite good alignment of both critical BBR temperatures with the TSRST fracture temperature.
It is further to be noticed that the gap between TSRST fracture temperatures and the $T_S=300 \text{ MPa}$ values is in the range of 6 to 8°C and thus not so far away from the 10°C shift on which the SHRP SUPERPAVE binder specifications have been established. At the TSRST fracture temperature, the BBR stiffness values are in a range of 600 to 700 MPa.

4.2.3 Interpretation

Considering that ABCD test conditions are as close as possible to those applied in the TSRST, it is quite encouraging to find a certain relationship between both tests (Figure 2b) and that their respective failure temperatures are also reasonably close. Those measured with the ABCD procedure tend to be more “optimistic” which seems logical considering the much thinner and more heterogeneously distributed binder film in the asphalt test sample, leading to higher stress concentrations at micro level than in the ABCD test sample.

The cooling rate in the Fraass test (1°C/min) is much quicker than in the TSRTST and ABCD tests (10°C/h). In addition, the test is performed on a thin film which is repeatedly flexed (fatigue component). Moreover, the Fraass breaking point corresponds to crack initiation whereas TSRST and ABCD failure occur after a certain crack propagation time. The Fraass test is thus particularly severe and these considerations could explain why Fraass breaking temperatures are much higher and not well correlated to TSRST and ABCD failure temperatures.

In the case of the investigated bitumen, both $T_S=300 \text{ MPa}$ and $T_m=0.3$ turn out to be good predictors of the TSRST fracture temperature. This could be surprising considering that these indicators are measured under small strain and not failure conditions but may be explained by a “rheologically simple” behaviour in which failure would be directly related to stiffness (the stiffer, the quicker it gets brittle). Whether this reasoning is applicable whatever the origin and production mode of the bitumen is however an open question.
5. EXPERIMENTAL RESULTS – MODIFIED BITUMEN

5.1 TSRST results

The evolution of TSRST fracture temperature and fracture stress in relation to polymer content is shown in Figure 5. Following comments can be made.

- For the crosslinked elastomer-modified binders made from the “A” bitumen, fracture temperature drops with increasing polymer content yet grows again after a minimum at 3.5% of polymer. Maximum gain in fracture temperature ranges from 5°C to 8°C, depending on the penetration class of the base bitumen.
- In the case of the 35/50 bitumen, the differences in behavior which have been evidenced in relation to bitumen origin (Figure 1) tend to disappear for the 3.5% crosslinked elastomer-modified binders.
- The two physical blends at 3.5% of polymer are no better than the corresponding base bitumen. Similar observations have already been made in previous studies [14].
- Except for the binders made from the softer 50/70 bitumen, fracture stress does not show large variations in relation to polymer content and tends to culminate at a value around 4.5 to 5MPa.

5.2 Search for correlations with binder properties

5.2.1 “Failure” property – Fraass breaking point

An interesting observation in the case of the 20/30-A and 35/50-A bitumen is the parallel evolution with polymer content of Fraass breaking point and TSRST fracture temperature. This could however not be confirmed in the case of 50/70-A for which we could unfortunately not obtain consistent Fraass values at 5% polymer content (Figure 6).

All the results generated for the the crosslinked elastomer-modified binders are gathered on Figure 7a. We recognize again the good alignment of Fraass breaking point and TSRST fracture temperature for the blends made from 20/30-A, 35/50-A and, possibly (only 2 results available), 35/50-C which seem to belong to a same “family” of behavior. The blends made from 35/50-B are on a somewhat different line. The blends made from 50/70-A show a markedly different behavior, their fracture temperatures being much better than would be expected from the Fraass test.

The two physical blends do not show any improvement over their base bitumen in the TSRST test. This is confirmed by the Fraass test results which are even worse than for the base bitumen (Figure 7b).
These findings do thus confirm that there is no unique correlation between Fraass and TSRST, although both tests measure failure behaviour. As stated for pure bitumen, the main reason is probably the difference in loading conditions (much more severe in the Fraass test) to which the response may be different depending on the nature of the bitumen, type of modification and polymer content. Not to mention that Fraass tests may be biased due to the difficulty of mastering operating conditions, more particularly for highly modified binders.

5.2.2 “Failure” property – ABCD test

Although ABCD has not been run on all binders, a number of observations can be made. The most immediate is that the test does clearly differentiate the modified binders from pure bitumen. But modified binders and pure bitumen do not seem to belong to a same “family” in terms of behaviour. Figure 8a evidences two distinct lines, one for pure bitumen, the other one for the corresponding 3.5% crosslinked elastomer-modified binders. These two lines are quite “flat”, which suggests that TSRST is better in discriminating binders than ABCD. Furthermore, in comparison to TSRST, the ABCD results are very optimistic for the cross-linked 5% modified and the 3.5% physical blend.

Figure 7a and 7b: TSRST fracture temperature versus Fraass breaking point

Figure 8a and 8b: TSRST vs ABCD – Impact of polymer content

Figure 8b shows that, in the ABCD test, low failure temperature is generally associated to high fracture stress. Polymer modified binders are able to go to higher stress levels before break than pure bitumen. The various products do however not align on a unique stress/temperature curve and this is probably to be attributed to the origin and penetration class of the base bitumen as well as to the nature of polymer modification (physical blend versus cross-linking). The fact that the excellent performance obtained by some binders in the ABCD test (50/70-A with 5% cross-linked polymer and 35/50-A physical blend with 3.5% of polymer) is not reflected in the TSRST results is maybe due to the fact, for one or the other reason, these binders could not develop the same strength once in the asphalt mix.

5.2.3 Flexural creep stiffness – BBR
In § 4.2.2, it could be shown that both BBR temperature criteria did correctly classify the pure bitumen with regard to the TSRST results. When modifying with a cross-linked elastomer, the $T_{S=300\,MPa}$ temperature changes in a monotonous way with increasing polymer content (in general at a slower rate than the corresponding TSRST temperature). The BBR test did thus not reflect the optimum value at 3.5% polymer content obtained in the TSRST. As in TSRST, BBR data do not show any improvement over the base bitumen for the physical blends at 3.5% polymer. With regard to $T_m=0.3$, there is almost no impact of polymer addition on the measured values which suggests that, at least for the investigated binders, this criterion seems to be essentially controlled by the base bitumen (Figures 9a and 9b).

These findings lead logically to following observations on a first graph of TSRST fracture temperature versus $T_{S=300\,MPa}$ (Figure 10a):

- The data points for pure bitumen and for the 3.5% polymer modified physical blends are on a same line, since the latter do not show any significant evolution in neither the TSRST nor the BBR test.
- Except for the binder made from 35/50-C, the cross-linked blends at 3.5% of polymer do not belong to this line.

Figure 9a and 9b: BBR test – Evolution of $T_{S=300\,MPa}$ and $T_m=0.3$ with increasing polymer content

Figure 10a and 10b: TSRST vs BBR – Impact of polymer content
The correlation between a stiffness characteristic (BBR) and a failure property (TSRST) which could be shown in the case of unmodified bitumen appears thus as not applicable to these modified binders and thus as not “universal”. At the best, we may imagine to establish such correlations for “families of products” such as, in the case of this study, the family of cross-linked binders at 3.5% of polymer content (although this correlation seems to be relatively poor).

The “family” of cross-linked binders at 5% polymer content happens to be on the same line as the pure bitumen (Figure 10b). This is however more a coincidence than a true relationship. It is due to the fact that the T\textsubscript{S=300 MPa} values continuously decrease with polymer content while the TSRST fracture temperatures rise after the minimum at 3.5% of polymer content.

Considering the small evolution of m-values with polymer content (Figure 9b), there is of course no likelihood to find a relation between T\textsubscript{m=0.3} and TSRST fracture temperatures obtained at different polymer contents.

6. EXPERIMENTAL RESULTS – DISCUSSION

Table 3 gathers all the generated performance data. As indicated in Table 2, BBR and ABCD tests have also been performed after RTFOT + PAV ageing ([7], [8]). PAV ageing conditions being 20 hours at 100°C and under a pressure of 2.1 MPa. The impact of ageing is discussed in more details in another paper [4] but with regard to the search for correlations which is the subject of this study, two main comments can nevertheless be made when looking at the figures in Table 3.

- Ageing leads to a shift in the different critical temperatures but does not change the ranking of binders and correlations do generally not become better.
- The evolution of ABCD failure temperature as well as the evolution of T\textsubscript{S=300 MPa} after RTFOT+PAV appear as relatively small (often less than 2°C). Larger evolutions are obtained for T\textsubscript{m=0.3} (especially for the two physical blends). Those evolutions tend however to become smaller when polymer content is increased (crosslinked elastomer-modified binders).

Table 3: Summary of all test results

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Pen.</th>
<th>Soft. Pt.</th>
<th>TSRST</th>
<th>Fraass</th>
<th>BBR - T\textsubscript{S=300 MPa} [°C]</th>
<th>BBR - T\textsubscript{m=0.3} [°C]</th>
<th>ABCD Failure Temperature [°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td>[%]</td>
<td>[mm/10]</td>
<td>[°C]</td>
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Styrelf®: cross-linked elastomer modified bitumen
PB: “physical blends” with an SBS elastomer
- o : fresh binder
- r : after RTFOT ageing
- p : after RTFOT+PAV ageing

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Obviously none of the investigated binder tests (Fraass, BBR, ABCD) is able to predict the TSRST fracture temperature in a “universal” way (applicable to any kind of binder). Some explanations may be found when considering the nature of these tests.

- BBR is not a failure test [15], which implies that one has to assume a direct relationship between “stiffness” and “failure”. This may be true per families of products (pure bitumen, cross-linked binders, …) but not in a universal way.
- Fraass is a true failure test but, as stated in § 4.2.3, it is much more severe than the TSRST test. This would explain that Fraass breaking point temperatures are always higher than TSRST fracture temperatures. The difficulty to master operating conditions, especially in the case of modified binders, is another possible explanatory factor.

Such considerations apply less easily to ABCD since its loading conditions are quite close to those applied in the TSRST and since it is also fairly repeatable. ABCD failure temperature is closer to TSRST fracture temperature than Fraass breaking point but also lower. The ABCD test is thus more “optimistic” which could be explained as follows:

- The ABCD test applies a uniform stress pattern over a large cross-sectional area while the local heterogeneity of the stresses applied in a TSRST sample could explain earlier cracking.
- Another part of the explanation could also be that kinetics of temperature equilibration within the ABCD sample is different than within the TSRST asphalt sample.

To explain that one does not obtain unique correlation lines, one has to assume that the magnitude of the above mentioned effects differs depending on the type of binder. But this may even be more so for possible interactions between bituminous binders and aggregates which could impact the TSRST performance even when working with a constant formulation and the same aggregates. For example, one may think of improper coating and lack of adhesivity (due for instance to the high viscosity of a binder at mixing stage) which would not allow to take full benefit of the intrinsic performance of the binder. This could maybe explain why the TSRST performance measured for the 50/70-A / 5% polymer crosslinked and for the 3.5% polymer physical blend are much poorer that what would be expected from the ABCD results.

7. CONCLUSIONS

Referring to the intrinsic nature of the investigated test methods, it may be stated that:

- The stiffness characteristics measured by the BBR test can only be related to TSRST performance through correlation. Such a relationship can however not be universal and has to established per families of products which show a similar behaviour, such as for instance pure bitumen or modified bitumen of a same “family”.
- Although Fraass measures a failure characteristic, it appears as being particularly severe and thus prone to eliminate binders which show a satisfactory TSRST behaviour. Its known lack of precision and difficult operating (in particular with modified binders) is a further handicap for this method.
- The ABCD test seems more reliable from an operational point of view. Contrarily to Fraass, it gives however an evaluation which tends to be too optimistic and, also here, the relationship to TSRST is dependent on the type of binder.

Overall, due to differences in loading conditions and possible artefacts related to binder/aggregate interaction, it seems thus difficult, if not impossible, to find a good “parallelism” between a binder performance property and TSRST performance of asphalt mixes. When interpreted correctly, binder tests are however extremely useful evaluation tools. Correlations to asphalt performance are to be established per families of products which behave similarly. This will allow binder tests to become true binder selection tools. As a confirmation of previous works [18] our study suggests that cross-linked binders, probably due to their monophasic nature, are well suited for such correlations. Among all the tested binders, the crosslinked elastomer-modified were also those which improved TSRST performance most significantly.

More research is needed to determine whether these findings depend on the type and formulation of the asphalt mixture.
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Some publications on the same subject