Binder performance and testing

# Correspondence between the state of aging of a PmB and its state in asphalt mix

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Abstract

PmB binders are well-known to improve AC mix characteristics and durability. But they often present a very complex evolution with time and temperature, which can have a significant effect on their characteristics. So it is unclear if the sample prepared for testing is in the same state as it is in the asphalt mix, even if the coating step has been simulated. On the other hand, in order to evaluate a binder of an asphalt mix a recovery steep is needed, which may also change the state of the binder. To get a comparison of the modified binder with a given and controlled thermal history to the one as it is in an asphalt mix is therefore almost impossible directly. This study presents detailed characteristics of three modified binders from industrial production facilities in different aging states : fresh, after RTFOT and after RTFOT+ PAV. Some of these binders are also extracted from an AC mix. Empirical binder tests are conducted and as well as a complete rheological binder characterisation. Three AC mixes produced with these binders are characterized from an asphalt mix rheological point of view. The behaviour of both the binders and the asphalt mixes is modelled according to the 2S2P1D model, and the best “correspondence” between the parameters determined on the binders at different aging states and those measured on AC mixes are sought. That helps to define a more adapted state to work on the binders. This will hopefully also shed some light on the relevance of some requirements based on the characterisation of recovered binders, and help indicate which aging and thermal history is the most relevant for possible future durability specifications.

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## 1. INTRODUCTION

Bitumen suppliers and contractors work to improve quality of their solutions for better and long lasting pavements. It is unclear yet how to quantify and control this increase. So new requirements appear in contracts to control this point. It is an opportunity, as it can promote innovation. But sometimes some requirements, expected to warrant good performances under traffic, are not the right ones or the best adapted. This comes partly from the fact that the behaviour of the product itself, is not fully understood. PmBs are a well known and good solution for roads, with large positive feedback from in-situ behaviour: e.g. improvement in rutting and fatigue resistance and in low temperature characteristics. But the behaviour of the Pmb, the compatibility between bitumen and polymer, and its evolution with temperature is not fully understood [1]. For some, storage at high temperature is an issue, but even with storage-stable binders, some evolution appears with an impact on the characteristics of the binder. This is mainly linked to the fact that the blending of polymer into a bitumen gives a very complex medium, where stability strongly depends on the components themselves and on temperature [2]. Quality control of AC mixes is based on checking grading curves and binder contents at the plant, and void contents and pavement thicknesses on site. Working on recovered binders is the easiest way to try to capture some interesting performances of mixes. And this is really well adapted for neat binders, where their characteristics can be checked: for example, the effect due to possible overheating and ageing, or estimating the modulus of the mix. But it is an issue when working with PmBs, as the recovery step will necessarily affect the binder. How, such complex blending of polymer and bitumen, could be affected after dissolution into a solvent and subsequent recovery through a heated extraction step? Is it expected that this recovered product still correctly reflects the behaviour of the binder present in the mix on road? This paper presents an analysis of three different binders in different states, to try to better understand how we should work for QC on binders. The mechanical behaviour of an AC mix made with these three binders is also characterized. The objective is to improve the global comprehension in a relevant way so as to correctly characterize a binder as it performs in the 'end-product', the asphalt mixture.

## 2. MATERIALS AND METHODS

2.1. Binders

Three different binders have been selected for this study, all sampled in industrial units. Two are 25/55-65 (PmB 1 &2) and one is a 45/80-60 (PmB 3) according to EN 14023. One is a crosslinked PmB, while two others are obtained by physical blending. Standard characteristics have been measured, in three different states: fresh, after RTFOT and after RTFOT + PAV. A complete rheological characterization with DSR has also been conducted at these different ageing states.

2.2. AC Mix

A common AC 10 mix design was selected. Three different mixes were produced, made with the 3 binders and diorite aggregates at a binder content of 5.4%. Large slabs were compacted and cut in order to conduct modulus measurements in 2PB-TR, according to EN 12697-26, annex A. Mean void content of 4 samples, tested for each mix were respectively 8.6, 8.2, 9.4% for mixes with PmB 1, PmB 2 and PmB 3.

### 2.3. Methods

2.3.1. Recovery

One of the main purposes of this study is to compare the viscoelastic behaviour of a recovered binder with the one obtained on the same product in different states. Recovering step alone is a very complex test, chemically and physically, as the binder is first dissolved and then the liquid phase obtained is heated under low pressure to regain a binder. This test is well described in EN 12697-3. It is difficult to believe that such a complex preparation history for the binder will at the end give a binder sample in exactly the same state as it is in AC mix. The effect of the recovery step has been largely studied. Possible effects of some residual solvent, even at very low level can strongly affect the results [3]. The effect of the nature of the solvent alone is unclear as behaviours can differ with PmB: Ring and Ball values after extraction and recovery can sometimes be erratic [4]. For this project, the solvent used was dichloromethane and binders recovered from the slabs in which trapezoidal samples for modulus measurements have been cut. PmB 1, was also recovered after only dissolution, to check any possible effect of the dissolution step alone.

2.3.2. Rheological measurements

Rheological tests they have been conducted with Anton Paar rheometers with a plate plate geometry.

- 4 mm plate tests were conducted from 0°C (6°C) up to -30°C, every 6°C a frequency sweep was recorded (10Hz-0.01Hz strain 0.02%)

- 8 mm plate tests were conducted from 0°C up to 40°C, every 10°C a frequency sweep was recorded (10Hz - 0.01Hz strain 0.05%)

- 25 mm plate tests were conducted from 40°C up to 90°C, every 10°C a frequency sweep was recorded (10Hz -0.01Hz strain 1%)

DSR measurements were recorded on all the samples. For the 4 mm plate tests, the original and the binders after RTFOT+PAV were recorded twice, the other plate geometries were repeated for a few randomly selected samples, since for these tests the repeatability is typically very good (below 5%). In addition, some stress sweeps were conducted to check the linear viscoelastic range. An example is given in Figure 1 on fresh binders, that confirms that test conditions selected where well adapted.

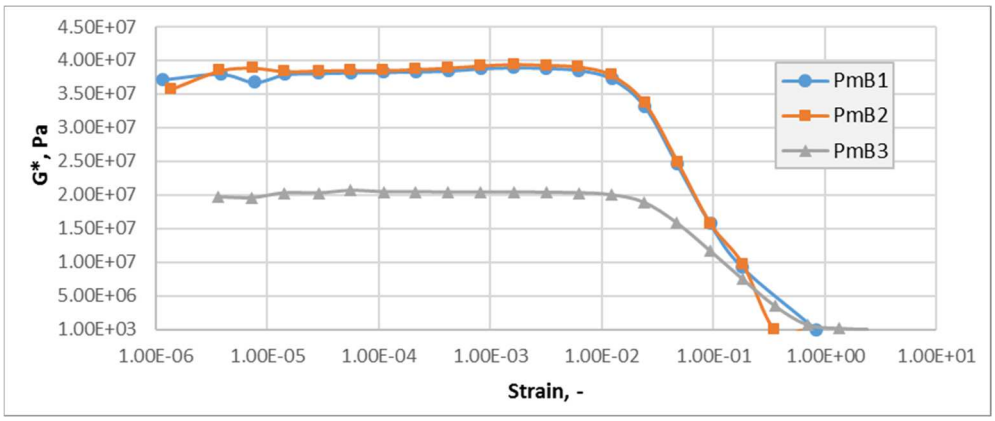


Figure. 1: Stress sweeps at 0°C and 0.1Hz, (4 mm plate test setup)

## 3. RESULTS

3.1 Results on binders

3.1.1 conventional tests

Penetration, Ring and Ball temperature, Elastic recovery, and Fraass breaking point have been determined on the three binders. Results are given in Table 1.

Table 1: Results of conventional test on binders

| Binder | Original Fresh | RTFOT | RTFOT + PAV | Recovered AC mix |
|---|---|---|---|---|
| | Penetration at 25°C, 0.1 mm (EN 1426) | | | |
| PmB1 | 38 | 26 | 15 | 27 |
| PmB2 | 44 | 28 | 19 | 30 |
| PmB3 | 55 | 45 | 26 | 46 |
| | Softening point R&B, °C (EN 1427) | | | |
| PmB1 | 70.8 | 74.4 | 79.6 | 72.4 |
| PmB2 | 77.2 | 75.6 | 78.2 | 71 |
| PmB3 | 75.2 | 61.8 | 69.2 | 62.4 |
| | Elastic recovery at 25°C, % (EN 13398) | | | |
| PmB1 | 91 | 81 | | 88 |
| PmB2 | 96 | 87 | | 88 |
| PmB3 | 97 | 83 | | 90 |
| | Fraass breaking point, °C (EN 12593) | | | |
| PmB1 | -13 | | | -10 |
| PmB2 | -16 | | | -13 |
| PmB3 | -14 | | | -20 |

Penetration is decreasing with ageing, which is expected. The penetration of recovered binders is between original and RTFOT. But R&B evolution is more erratic, increasing for PmB 1, decreasing after RTFOT and increasing again after

PAV for PmB 2&3. The R&B values measured on recovered binder seem not to be so far from the values determined on fresh or after RTFOT. In between for PmB 1 and 3, it is under all the results for PmB 2. For the Fraass breaking point also, values on recovered binders do not all seem consistent, as they increase for PmB 1 and 2, and significantly decrease for PmB 3. The storage stability of these binder was measured according to EN 13399, after three days at 180°C, and results are given in Table 2

**Table 2: Storage stability of three PmBs studied**

| | Storage stability, EN 13399, °C | | |
|---|---|---|---|
| Binder | Top | Bottom | Δ |
| PmB 1 | 80.2 | 70 | 10.2 |
| PmB 2 | 70.8 | 71 | -0.2 |
| PmB 3 | 71 | 67.8 | 3.2 |

Values of R&B on top and bottom parts can differ from the original ones measured on fresh binder after long term storage at 180°C. Even if the binder seems homogeneous with similar R&B on top and bottom, as for PmB 2 and 3, these values may differ from the initial ones. There is no direct link between storage stability and performances of mixes, as long as the industrial process with continuous stirring keep the binder homogeneous [2]

BBR tests, according to EN14771, were conducted on recovered and on aged binders (aging procedure RTFOT+PAV) and on one sample of original binder. The limiting temperatures LST and LmT were derived according to ASTM D7643 – 16. These data are represented in Table 3, including the tests temperatures and the number of repeats.

**Table 3: Limiting temperatures derived from BBR tests, the test temperatures are indicated as well as the number of repeats in brackets**

| | Bending beam Rheometer | | | EN 14771 (°C) |
|---|---|---|---|---|
| Recovered | LST | LmT | ΔTc =LST-LmT | Measured temp (nr beams) |
| PmB1 | -17.9 | -15.3 | -2.6 | -18 °C (1), -12°C (1) |
| PmB 2 | -18.9 | -18.9 | 0 | -18°C (1) and -24°C (1) |
| PmB 3 | -21 | -23.3 | 2.3 | -18°C (1) and -24°C (1) |
| PmB 3 original | -19.8 | -20.4 | 0.5 | -18°C (1) and -24°C (1) |
| RTFOT + PAV | | | | |
| PmB 1 | -17.1 | -7 | -10.1 | -18°C (2), -12°C (1) -6°C (1) |
| PmB 2 | -16.9 | -13.4 | -2.7 | -18°C (2) and -12°C (2) |
| PmB 3 | -19.1 | -19.4 | 0.3 | -18°C (2) and -24°C (2) |

After RTFOT + PAV ageing, PmB 1 gives a very low ΔTc value, -10.1°C, while the others are -2.7 and 0.3. Thresholds for neat binders are defined as -2.5 and -5°C even if these levels are beginning to be discussed as they do not seem adapted for PmBs [5]. Same measurements on recovered binders lead to significantly higher values for ΔTc. This is expected as ageing is supposed to be less severe after mix production alone, but evolutions differ between binders, as ΔTc increase by about 3°C for PmB 2 and 3 and 7.5°C for PmB 1.

3.1.2 Rheological data

Black diagrams and master curves are built with data recorded with 4mm, 8mm and 25mm plate tests, on the original, RTFOT and RTFOT+PAV aged binders. In Figure 2&3 master curve results on PmB 3 are plotted, for a reference temperature of 10°C . The shape of the Black curves is typical for PmBs. The effect of aging is similar for all the binders, with a shift towards lower phase angle and stiffer results. The fact that isotherms do not form a unique curve in Black space indicates that these binders do not fully respect the time-temperature superposition principle, which is one of their well-known properties [6] [7].

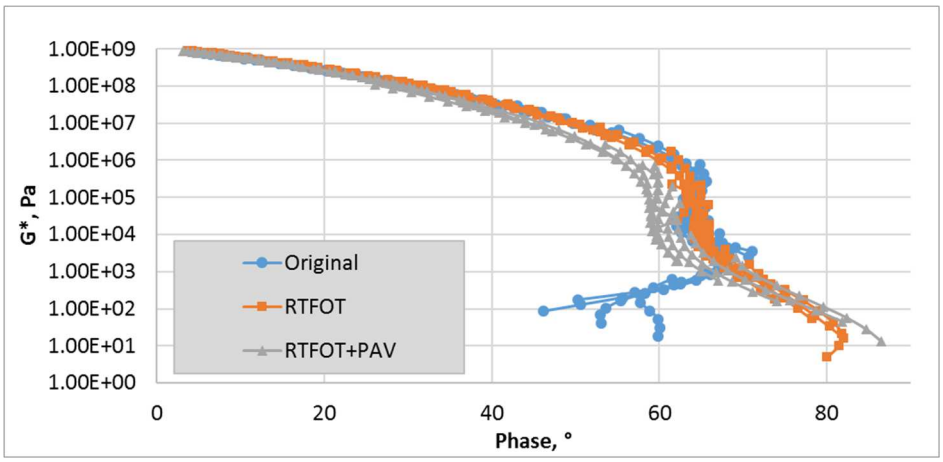


**Figure 2: Black curve for PmB3**

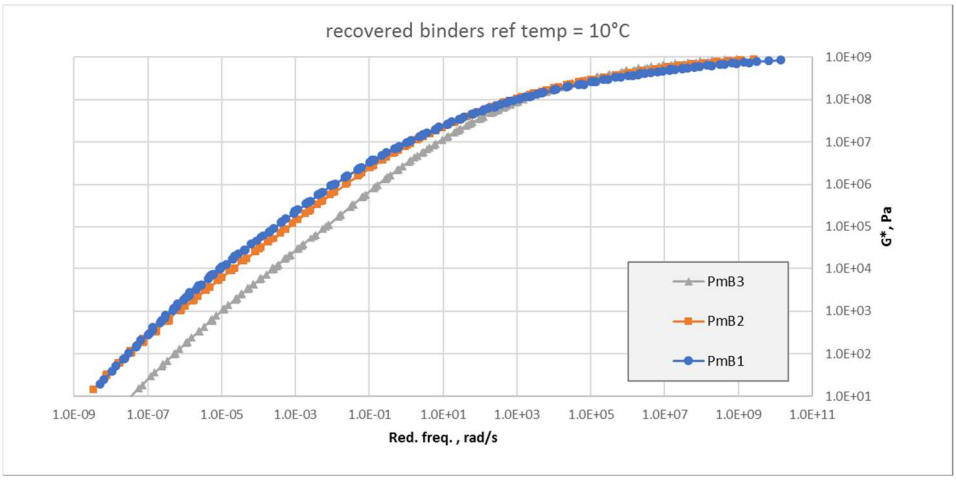


**Figure 3: Master curves at 10°C of the three recovered binders**

All these measurements allow to calculate rheological parameters, like R index, Crossover frequency, ΔTc and Glover-Rowe parameter, which are supposed to reflect the behaviour of the binders, ageing sensitivity through decrease in ability to relax stress, and cracking susceptibility. They are all calculated with Rhea software, according to their definitions given in [8]. Some of them are reported in Table 4, while the variation of the Glover-Rowe parameter is plotted in a Black diagram on Figure 4.

**Table 4: evolution of rheological parameters with state of ageing**

| | | | | |
|---|---|---|---|---|
| R index | fresh | RTFOT | RTFOT+PAV | Recovered |
| PmB1 | 2.09 | 2.46 | 2.95 | 2.38 |
| PmB 2 | 1.96 | 2.21 | 2.42 | 2.17 |
| PmB 3 | 1.68 | 1.71 | 1.98 | 1.73 |
| Crossover freq (Hz) | | | | |
| PmB 1 | 6.47E-03 | 5.96E-04 | 5.92E-05 | 1.71E-03 |
| PmB 2 | 8.06E-03 | 1.38E-03 | 2.36E-04 | 4.83E-03 |
| PmB 3 | 6.11E-01 | 6.15E-01 | 1.04E-01 | 6.26E-01 |
| ΔTc (from DSR) °C | | | | |
| PmB 1 | -3.7 | -6.4 | -8.3 | -6.0 |
| PmB 2 | -2.5 | -4.2 | -5.0 | -3.7 |
| PmB 3 | -0.2 | 0.4 | -1.0 | -0.7 |

The evolution of the parameters with ageing is as expected, with an increase in R index and a decrease of the crossover frequency. Again we can notice that the values of the different rheological parameters measured on recovered binders are most of the time in between results obtained on fresh and RTFOT aged samples.

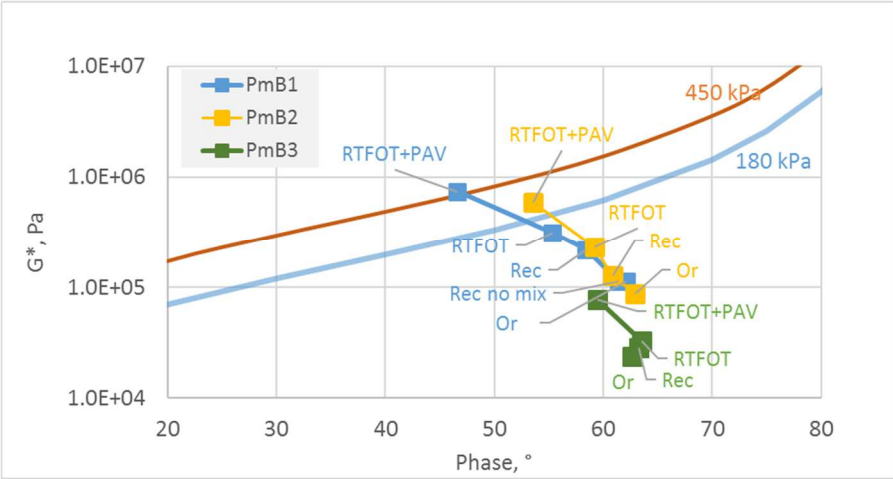


Figure 4 Evolution of G-R parameters of 3 PmBs

In figure 4, the evolution is also the one expected. PmB 1 after RTFOT and PAV seems to come close to the limits proposed for G-R thresholds. PmB 3 seems to be the less sensitive to ageing.

3.2 Results on AC Mixes

Looking for modelling with 2S2P1D model, master curve was determined on mixes with as much data as possible. Example of results obtained are detailed in Figure 5. Void content of the samples are respectively 8.6, 8.2 and 9.4 % for mixes with PmB 1, 2 and 3. Modulus were measured with two points bending tests, on eleven isotherms from -20°C to 20, in step of 5°C, and also at 30 and 40°C, at 5 frequencies (3,6,10, 25 and 40 Hz) . Half of the slabs were kept for tension compression test, which results are not presented here.

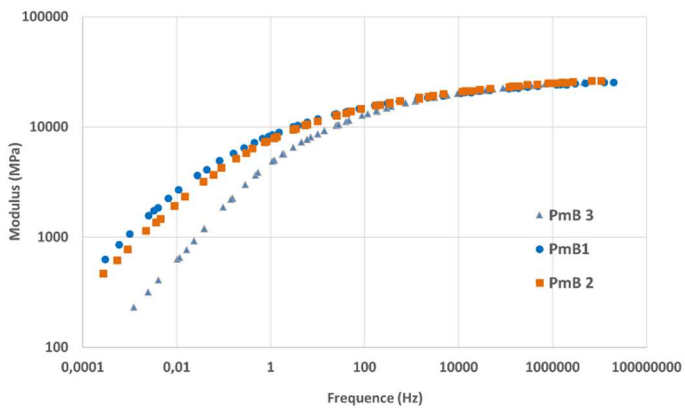


**Figure 5: Master curves of the mixes with the 3 PmBs**

For low temperatures/high frequencies modulus are all close to an equivalent value. For high temperatures/low frequencies, PMB 3 leads to lower modulus in line with its grade, softer than the two others.

## 4. MODELLING AND ANALYSIS

4.1 2S2P1D Model

The objective was to use the 2S2P1D rheological model applied to the mix and to compare parameters determined on AC mix and binder in different state of ageing. The 2S2P1D model was selected, as it gives parameters which are supposed to be equivalent between the binder and the mix [6]. For binders, this model was initially based on results from Metravib and annular shear rheometer, was later also based on data from DSR [9]. It has also been used to model the effect of ageing on neat bitumen, with increasing time exposure at high temperature with RTFOT [10].

Various kinds of PmBs, made with different bitumen origins, blended with EVA or SBS, unaged and aged, and also mastics, were modelled with 2S2P1D with some limitations for highly modified binders with EVA [11]. Determining values of the 8 parameters for the mixes, and comparing it to all those obtained for all different states of the binders, included those from recovered binder, should allow to identify for PmB which state of the lab aged binder is closer to its state in AC mix.

## 4.2 Results

Data from measurements on binders and AC mixes were used to determined parameters of the model. DSR results with 4 and 8 mm plate geometry only were used for binders. No fixed values were selected for parameters except $G_{00}$ which was chosen to be zero, as commonly admitted for binders. An example of the results is given in figure 6 for PmB1. It has been done for all measurements on fresh and aged binders, and also recovered. The same has been done for three AC mixes. Results obtained on mix made with PmB 1, are presented Figure 7. This allow to get a full set of parameters for all the data, and a direct comparison between binders and mixes. Best fitting between the model and experimental data is obtained for numerical values presented Table 5.

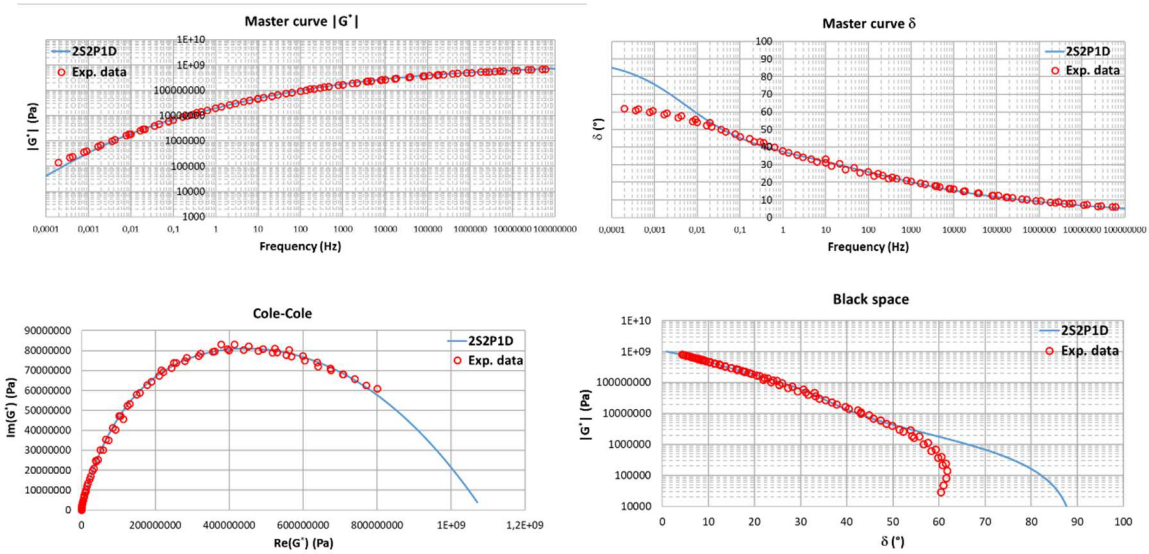


**Figure 6: Master curve, Cole-Cole diagram, Black curve for PmB 1**
**Comparison between experimental and 2S2P1D model.**

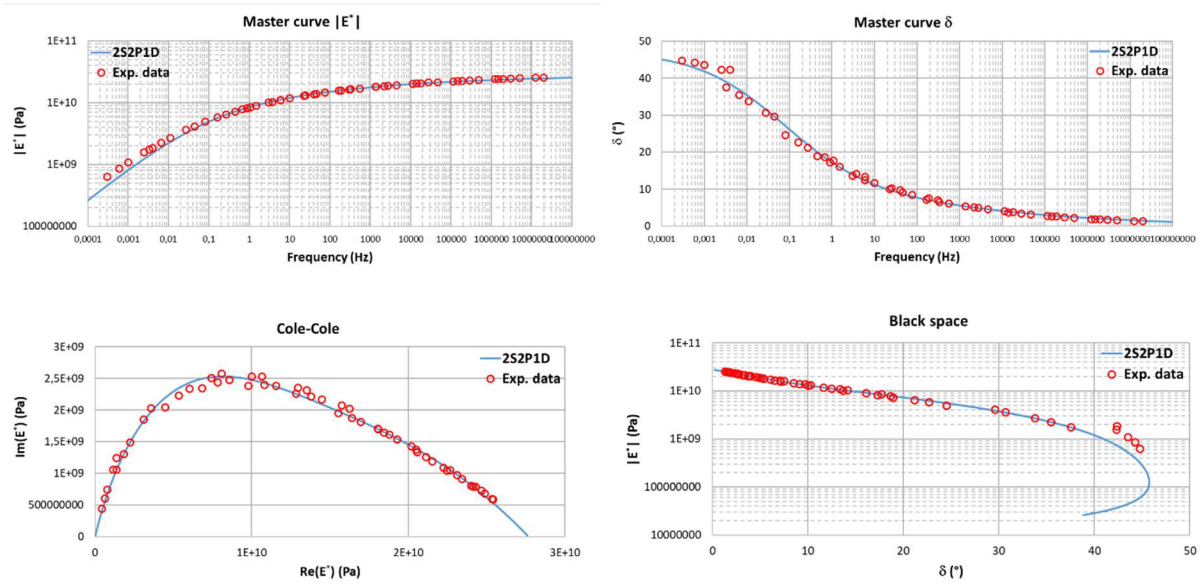


**Figure 7: Master curve, Cole-Cole diagram, Black curve for Ac mix with PmB 1.**
**Comparison between experimental and 2S2P1D model.**

**Table 5: values of the different parameters of 2S2P1D model for all the binders and AC mixes tested.**

| PMB 1 | $G_{00}$ (Pa) | $G_0$ (Pa) | k | h | δ | $\tau_0$ | β |
|---|---|---|---|---|---|---|---|
| **original** | 0 | 1.09E+09 | 0.17 | 0.460 | 2.8 | 0.000048 | 1400 |
| **Recovered** | 0 | 9.00E+08 | 0.17 | 0.460 | 2.9 | 0.000062 | 1400 |
| **Recovered (mix)** | 0 | 1.04E+09 | 0.17 | 0.430 | 2.8 | 0.000035 | 3400 |
| **RTFOT** | 0 | 9.90E+08 | 0.17 | 0.440 | 3.1 | 0.000080 | 3300 |
| **RTFOT+PAV** | 0 | 1.01E+09 | 0.17 | 0.395 | 3.1 | 0.000055 | 12 000 |
| **Asphalt mix** | $E_{00}$ (Pa) | $E_0$ (Pa) | k | h | δ | $\tau_0$ | β |
| **AC mix PmB 1** | 1E+07 | 2.77E+10 | 0.16 | 0.53 | 1.8 | 0.28 | 1400 |

| PmB 2 | $G_{00}$ (Pa) | $G_0$ (Pa) | k | h | δ | $\tau_0$ | β |
|---|---|---|---|---|---|---|---|
| **original** | 0 | 1.05E+09 | 0.19 | 0.495 | 3.0 | 0.000085 | 675 |
| **Recovered (mix)** | 0 | 1.05E+09 | 0.19 | 0.460 | 2.8 | 0.000050 | 1100 |
| **RTFOT** | 0 | 1.00E+09 | 0.19 | 0.470 | 3.0 | 0.000150 | 1400 |
| **RTFOT+PAV** | 0 | 1.18E+09 | 0.19 | 0.430 | 3.0 | 0.000150 | 2400 |
| **Asphalt mix** | $E_{00}$ (Pa) | $E_0$ (Pa) | k | h | δ | $\tau_0$ | β |
| **AC mix PmB 2** | 1E+07 | 2.84E+10 | 0.18 | 0.55 | 2.05 | 0.22 | 675 |

| PmB 3 | $G_{00}$ (Pa) | $G_0$ (Pa) | k | h | δ | $\tau_0$ | β |
|---|---|---|---|---|---|---|---|
| **Original** | 0 | 1.08E+09 | 0.21 | 0.520 | 2.4 | 0.000026 | 280 |
| **Recovered (mix)** | 0 | 9.75E+08 | 0.21 | 0.515 | 2.2 | 0.000028 | 430 |
| **RTFOT** | 0 | 1.10E+09 | 0.21 | 0.520 | 2.3 | 0.000030 | 390 |
| **RTFOT+PAV** | 0 | 1.06E+09 | 0.21 | 0.490 | 2.3 | 0.000035 | 870 |
| **Asphalt mix** | $E_{00}$ (Pa) | $E_0$ (Pa) | k | h | δ | $\tau_0$ | β |
| **AC mix PmB 3** | 3E+07 | 2.71E+10 | 0.22 | 0.65 | 2.05 | 0.042 | 280 |

There is not a significant evolution with the state of the ageing when we focus on parameters $G_{00}$, k and δ obtained on binders. Variation of $G_0$ is limited to 10% max, and we can also notice a slight decrease in h after RTFOT and PAV. In some previous research these parameters were even kept with constant values. $\tau_0$ and β are the two main parameters for which there is a significant evolution, both increasing with ageing. For PmB 1, difference between binder extracted and recovered compared to values obtained for the binder only dissolved, is limited to $\tau_0$ and β. If we look to results obtained

on binder recovered from AC mixes, $G_0$ and h present small variations, while there is a significant increase in β and a decrease in $\tau_0$. Rheological parameters are not so far from results obtained after RTFOT, but decrease in $\tau_0$ is opposite to the trend normally observed with ageing. These results are in line with some others previously published [11].

Initial assumption was to compare values of the parameters obtained on AC mixes to all the ones measured on binders, including those after recovery. In [6] it was even concluded that the parameters on fresh binders were strictly equivalent to those measured on AC mixes, which would mean that the coating at high temperature had no effect. Here we wanted to compare parameters for all states of the binder (fresh RTFOT aged and also recovered from an AC mix, to select which one would be the closest to results from AC mix.

Values obtained for h are always higher on mixes than on binders, and the opposite is observed for δ. That means that in a Cole-Cole diagram, the normalized data on mixes and binders which should have the same shape, does not. This is plotted Figure 8, with normalized values for E and G. β is always close to the value determined on original binders.

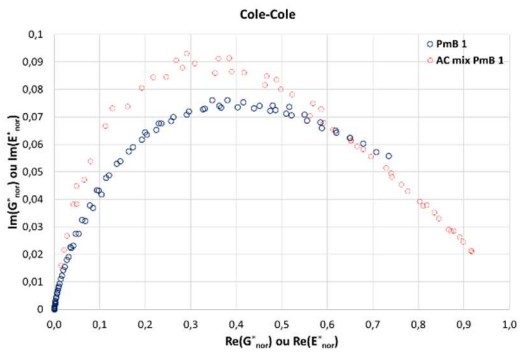


**Figure 8: Normalized Modulus of AC Mix with PmB 1 and PmB 1 in Cole Cole diagram.**

Additional results on mixes, based on traction compression test, still ongoing and not presented here, will allow to identify if this could come from limits of the temperature and frequency domain covered with 2 points bending test, or others artefacts. Another possible explanation could come from data with DSR. Lots of studies are conducted on rheological measurements made with DSR. But some significant differences in results at low temperature, between data from DSR and Metravib on the same binder have been recently noticed [12]. That could also explain the difference between mix and binders in our case.

Despite this we have also looked for possible correlation between β or $\tau_0$, parameters which are known to be representative of the evolution and ageing of the binder, and try to connect them with R index or G-R. On Figure 9, the relation between β and R index is plotted. Some more data should be added to confirm the trend but we can expect good correlations between these parameters.

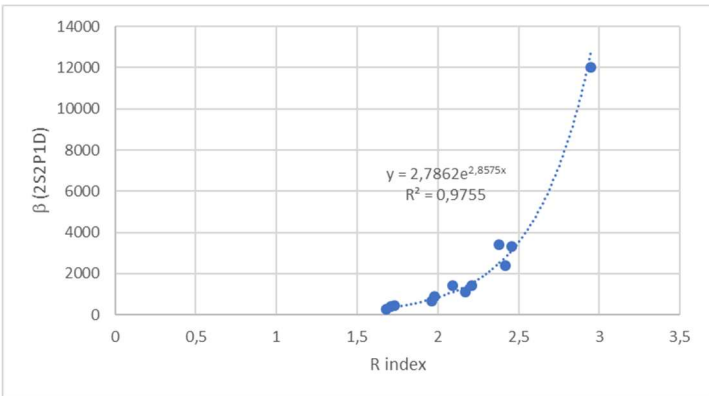


**Figure 9: Correlation between β from 2S2P1D and R index**

We also looked at possible correlations between $\beta*\tau_0$ defined through 2S2P1D and G-R value, as they are both supposed to be representative of the ageing of the binder. Results obtained on the three binders are plotted Figure 10, with a quite good $R^2$. Here again, further research is necessary. But it would allow to define thresholds like for G-R. It is also based only on results on binders after ageing, and it should be also verified on aged AC mixes. But we have a link between paths followed in Europe through 2S2P1D model and rheological parameters from the US,

which is in fact not so surprising as we are all looking for tools to capture the rheological behavior of binders, always starting from master curves.

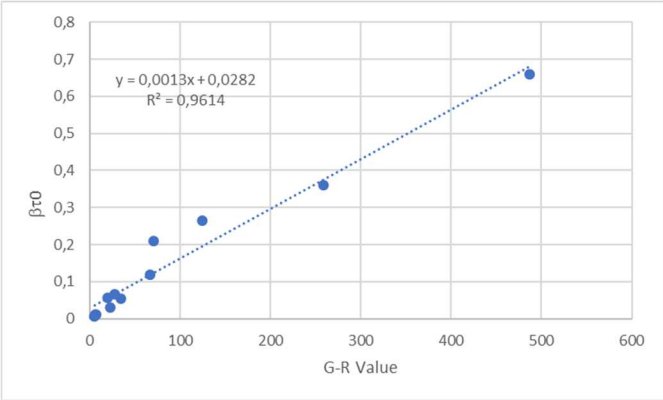


**Figure 10: Correlation between G-R and $\beta*\tau_0$ from 2S2P1D**

Parameters on binders recovered from AC mixes seem really close to values on fresh binders or after RTFOT for k, h and δ. The most noticeable variations are for $\tau_0$ and β. $\tau_0$ is lower to its initial value on original binders for PmB 2 and 3, while β always increases. This is not representative of the variations usually observed with ageing of binders in labs through RTFOT and PAV as both these parameters should increase. Recovery can have a slight effect of the characteristics of the binder. And as usual for bitumen, its properties depend on the origin ad composition of the raw materials used to produce PmB. So, it is necessary to be careful on results on recovered binders. It seems that with DSR measurements we can capture useful information on the binder. But simple test like R&B seems to be erratic to define relevant thresholds on recovered binders.

## 5. CONCLUSION

This study, limited to three binders was focused on the characterization of PmBs in various ageing states, on the characterization of the respective asphalt mixes, and on a comparison of the same binders recovered from these AC mixes.

The 2S2P1D model was used to try to capture the rheological behaviour of the binders in their different stages, and also the behaviour of corresponding AC mix. It was not possible to get exactly similar values for the parameters of 2S2P1D model on binders and mixes. Some specific variations after recovery were noticed. We can assume it is linked to the thermal history of the polymer modified binder in the recovery process.

Significant evolution of the parameters $\tau_0$ and β were observed. Interesting possible correlations of those parameters with parameters usually selected in the US were identified. Further research is needed to confirm this observation. As 2S2P1D is supposed to also capture rheological behavior of the mix, some interesting thresholds could be defined through parameters determined on AC Mixes. Improvement in ageing process of AC mixes would be a good way to confirm these last assumptions.

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