Binder performance and testing

Aging of rejuvenated RAP binder – a RILEM inter-laboratory study

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Abstract

The growing use of reclaimed asphalt (RAP) results in a continuous increase in the percentage of added RAP to hot mix asphalt. Within the asphalt pavement, the asphalt binder is the component which is affected the most by the ageing during service life, resulting in considerable hardening of the RAP binder. Therefore, the aged binder needs to be reactivated to meet the requirements for new asphalt mixtures. For high recycling amounts above 50%, often rejuvenating agents have to be added when no conventional binders are available with the required low viscosity. Such additives are of very different chemistry reflecting the base materials used in production which includes petroleum-based oils, but more often side products from industrial process which are very different from bitumen. Consequently, the ageing behaviour may differ from that of pure bitumen, which has been observed already in a number of pavement

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construction sites. For this reason, the RILEM task group 3 of Technical Committee (TC) RAP decided to examine the ageing behaviour of bitumen rejuvenator blends in more detail. An extracted RAP binder was mixed with varying amounts of virgin bitumen and a rejuvenating agent to simulate different amounts of RAP concentrations in the hot mix. These binder blends were next subjected to laboratory short and long term ageing. At every stage, defined target characteristics have been determined using complex modulus, penetration value, softening point ring and ball, and low temperature creep stiffness. The first results of this research effort are presented in this paper.

1 INTRODUCTION

Asphalt pavement materials are one of the most recycled and reusable materials in the world [1]. They are considered as 100 % recyclable with different techniques, cold or hot, in-plant or in-situ. In the last decades, the reuse of Reclaimed Asphalt (RA) into new Hot Mix Asphalt (HMA) has seen a continuous increase due to the economic and environmental benefits. This is further supported by the European Commission's vision towards the concept of Circular Economy [2]. Currently the use of 30 % RA into new HMA is widely accepted [3]; however, the need for higher recycling levels, beyond the common practice, may prompt the challenge of pushing the process to a RA content up to 70-100 %. For this reason, the use of rejuvenators is foreseen to help recycling going to the next level, as successfully demonstrated by their use in Japan with 80 % RA [4].

The main limiting factor for a higher RA content is the aging of the binder. The aging mechanism is complex with negative effects coming from oxidation [5]. Due to aging, the material becomes harder and more brittle [6]; on one hand, the increase in stiffness is positive, as the pavement becomes less prone to rutting; on the other hand, the material is more brittle, more prone to cracking, a negative aspect. This represents a considerable constraint for pavement engineers and road authorities as more and more RA will have to be recycled in the upcoming years, possibly affecting the pavement performance beyond acceptable service levels. Hence, a technology, that aims to use higher RA percentages, must be able to restore the lost properties of the aged binder without affecting the benefits gained with aging. For this, rejuvenators have to restore the flexibility at intermediate and low temperature, without compromising the high temperature behaviour against rutting, and ensuring long-term benefits [7].

In view of the fundamental importance on the use of RA in asphalt pavement, RILEM established a specific Technical Committee, TC 264-RAP. Within the framework of this TC, the Task Group 3 on Asphalt Binder for Recycled Asphalt Mixtures is devoting its activities on the investigation of binders and additives that can restore lost properties of the aged RA binder. The goal of TG3 consists in the implementation of an experimental plan to organise an inter-laboratory test among different laboratories to address and to characterise the effect of rejuvenators. A total of 20 laboratories have committed to participate.

This paper reports the first outcomes of the work conducted by the TG3 and focuses mainly on analysis and interpretation of the basic properties of different blend of RA, treated with a bio-based rejuvenator, through aging. Further outcomes including in-depth analysis of full rheology will be published in a later stage.

2 EXPERIMENTATION

2.1 Materials

Granulated RA material was collected from a stockpile from Germany and RA binder was then extracted, with automated extraction method, and recovered according to EN 12697-3, by one single laboratory. This ensured having a single source of raw material. The RA binder was first characterised by basic properties according to EN 12591 showing values in the domain of hard binders. FTIR was run to detect any presence of residual solvent from the extraction or eventually any polymer modification. Neither solvent nor polymer was detected. Complementary to the RA binder, a paving bitumen with a pen grade 50/70, according to EN 12591, was used as the virgin binder in this study. Table 1 displays the main basic properties of the RA and virgin binders.

Table 1. RA and virgin binders properties

Binder	Penetration value at 25 °C (0.1 mm)	Softening point temperature (°C)	Penetration index
RA binder	14	71.0	0.3
Virgin binder	62	49.6	-0.8

The rejuvenator used was a bio-based rejuvenating additive. It is a liquid additive, which, with its specific amphipathic chemical structure, disperses the highly polar fractions limiting the agglomeration of asphaltenes [8]. It has been already used in different research studies [9] [10] and projects [11]. Table 1 presents the main properties of the used rejuvenator.

Table 2. Properties of rejuvenator RP1000

Flash point	Viscosity at 60 °C	Cloud point	Density
> 280 °C	22 10^{-3} m^2/s	< - 25 °C	0.93 Mg/m^3

2.2 Testing program

The testing program consisted of evaluating different blends of RA, treated with the rejuvenator, and virgin binder in a ratio of 60 %, 80 %, and 100 %. Each blend was further subjected to aging through Rolling Thin Film Oven Test (RTFOT) according to EN 12607-1 and Pressure Aging Vessel according to EN 14769, which respectively are considered as short-term aging occurring during asphalt production, transport and application, and long-term aging under road conditions. The evaluation included empirical testing with penetration value at 25 °C (EN 1426), providing the consistency at ambient temperature, and softening point temperature (EN 1427), returning the consistency at high temperature. More fundamental characterisation was performed using Dynamic Shear Rheometer (DSR) in different conditions of temperatures and frequencies and Bending Beam Rheometer (BBR) for low temperature evaluation.

A total of 20 laboratories committed to participate, six from North America, twelve from Europe, one from Brazil and one from Japan. Each laboratory implemented a specifically selected blending ratio: eight for 60 %, five for 80 % and seven for 100 % of RA. Due to the limited amount of RA binder available, some tests requiring larger amounts of binder (penetration and BBR) were not performed by all laboratories. Finally, 16 laboratories have provided results for softening point tenperature, 11 for penetration and 5 for BBR. Table 3 displays the testing matrix as provided by each laboratory.

Table 3. Testing matrix

Lab#	Blend	Pen	St Pt	DSR	BBR	RTFOT	PAV
1	100 %	X	X	X		X	X
2	60 %	X	X	X		X	
3	100 %	X	X	X		X	X
5	60 %		X	X		X	
6	60 %	X	X	X		X	X
7	80 %	X	X	X	X	X	X
8	60 %	X	X	X		X	X
9	80 %	X	X	X		X	X
10	80 %		X	X		X	X
12	60 %		X	X		X	X
14	100 %		X	X		X	
15	100 %		X	X		X	X
16	60 %	X	X	X		X	X
18	80 %	X	X	X	X	X	X
19	80 %	X	X	X		X	X
20	100 %	X	X	X	X	X	X

3 PRE-EVALUATION

Prior to launching the study, a pre-dosage study was conducted to characterise the RA and virgin binder and determine the optimum dosage to recover binder properties of the RA binder similar to a pen grade 50/70 bitumen [12]. Two initial dosages of rejuvenator were used, 5 % and 10 % by weight of RA binder. The evaluation was based on basic properties with penetration value and softening point temperature. Based on penetration values, a dosage chart was generated to determine the optimum content of rejuvenator to target a 50/70 bitumen.

Figure 2 displays the effect of the additive at different dosages, with penetration value in the vertical axis, in logarithmic scale, and softening point temperature on the horizontal axis. The boxes, within the plot, provide an indication of pen grade specifications in accordance with EN 12591. While the RA binder presented very hard properties, the effect of the rejuvenating additive helped to restore it back to a wider range of pen-grade even at dosages below 10%. This can span from 20/30 to 50/70 pen grade with an additive dosage that varies between 3 % and 10 %. On average, a dosage of 5 % restores the properties by 2 pen grades, which is in agreement with previous studies [8].

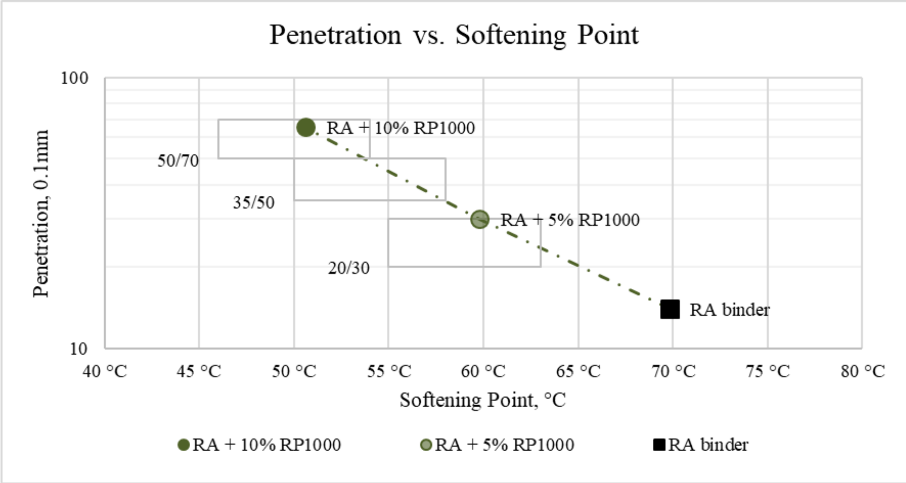

Figure 1. Pre-study, effect of rejuvenator on RA binder on empirical properties

The final dosage was determined to restore the lost properties in terms of penetration value, while still controlling that the softening point is not negatively affected. For this purpose, and to target a like 50/70 pen grade binder, a final dosage of 9 % was selected in order to match most of the three RA ratio blends 60 %, 80 % and 100 % RA.

4 RESULTS OF THE INTER-LABORATORY STUDY

Datasets from the 16 laboratories were used for data analysis; this includes 10 with penetration results (results from one laboratory have been dismissed because of a defective device) and 16 with softening point temperatures. Table 4 displays the whole results for the basic properties. Along the study the term blend refers to the blend of the RA binder treated with the rejuvenator and the virgin binder. It is worth noticing that not all laboratories have performed both penetration and softening point measurements and thus may make the analysis more complex. For the blend with 60 % RA binder a total of five complete sets of data were available, four for the 80 % RA blend and only two for the 100 % blend. In this condition, statistical analysis with repeatability or standard deviation is biased and not meaningful. From the first analysis of the data set, already some deviations for certain labs were observed. Lab 1, 3 and 5 had high softening point temperatures as compared to the others. It may depend on various parameters such as sample preparation, including the blending protocol, test methods or apparatus. This is sometimes experienced during inter-laboratory studies.

Table 4. Results of inter-laboratory study with basic properties

Lab#	Penetration value at 25 °C (0.1 mm)			Softening point temperature (°C)		
	Virgin	RTFOT	PAV	Virgin	RTFOT	PAV
	Blend with 60 %RA binder					
Lab 2	58	41		51.6	57.0	
Lab 5				56.4	61.1	
Lab 6	61	41	25	48.0	55.5	63.0
Lab 8	63	41	24	50.4	57.8	65.0
Lab 12				53.9	59.8	64.0
Lab 16	63	44	27	50.6	56.2	64.2
	Blend with 80 % RA binder					
Lab 7	64	44	29	51.4	58.8	65.4
Lab 9		42	27	52.1	58.2	65.3
Lab 10				51.5	57.3	64.1
Lab 18	56	41	28	52.2	57.6	63.7
Lab 19	57	38	24	52.2	59.0	69.3
	Blend with 100 % RA binder					
Lab 1	54	38	20	57.8	62.4	70.7
Lab 3				57.9	63.0	74.5
Lab 14				53.1	57.8	63.6
Lab 15				55.0	59.0	62.0
Lab 20	57	37	22	51.8	60.2	69.4

Additionally, some laboratories performed measurements on the RA and virgin binders respectively, five for the virgin binder with three having the full set of penetration and softening point, and four for the RA binder with 3 full data set.

The average values are presented in Table 1. This enables having a baseline between some laboratories identifying any deviation from measurement procedures or due to a difference in equipment.

4.1 Effect of rejuvenation at different RA content

One of the first analysis consisted of evaluating and comparing the effect of the rejuvenator between the different blends. Figure 2 shows the results for the different RA ratio blends at 60 %, 80 %, and 100 % along with the RA binder and the virgin binder as measured by some laboratory. The error bars are for the maximum and minimum values of each binder. A dotted line, between the RA and virgin binder, is added with intermediate points as it should result from the blending rules between RA and virgin binder (no rejuvenator) as defined in EN 13108-8. The green dash-dotted line is for the blends between the RA binder, the three blends of the treated RA binder and the virgin binder.

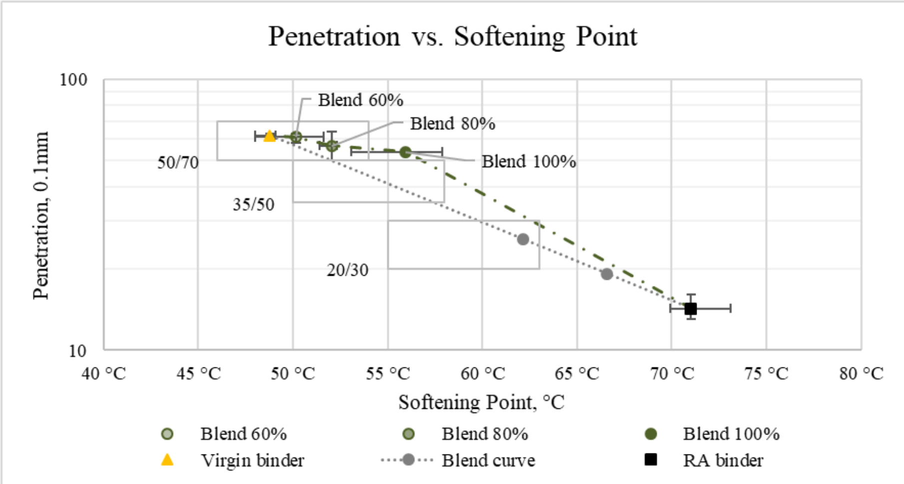

Figure 2. Effect of rejuvenator at different RA ratio

From this first analysis, it can be observed that the pre-defined dosage for the rejuvenator was enough to restore the penetration value in a range of a 50/70 pen grade bitumen. As this later is close to the one from the virgin binder, there was a minimal impact of the ratio 60, 80 or 100 % on the penetration value. The softening point was still slightly higher, which is a benefit coming from the aged RA binder, and was mostly affected by the ratio of RA binder in the final blend. As compared to the theoretical blending curve with no rejuvenator, the effect of the rejuvenator is recordable to restore the lost properties consistently, especially the penetration value at 25 °C.

4.2 Aging of the different blends

The first requirement for a rejuvenator is at least to restore the lost properties of aged binder in the final blend. However, it is also crucial that this effect will remain over time. The data set of the different binder blends, aged through RTFOT, short-term, and PAV, long-term aging, was analysed to qualify and quantify the long-term effect.

First, the aging was analysed for the RA and the virgin binder. Not all laboratories conducted the aging on those binders but at least two for the virgin binder and one for the RA binder performed the entire characterisation on the three conditioning levels and both penetration value and softening point temperature. Figure 3 displays the results for both binders in all aging conditions (unaged, RTFOT and PAV) along with the error bars as the minimum and maximum values when appropriate.

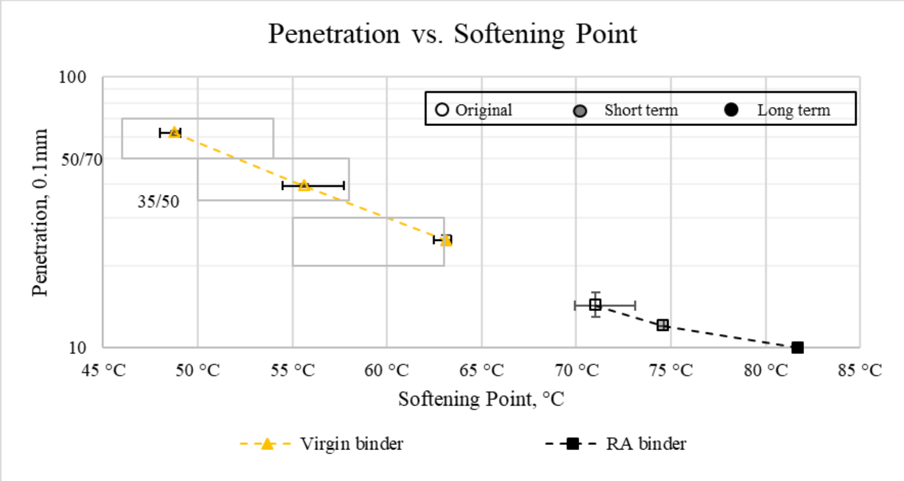

Figure 3. Aging of virgin and RA binders

As expected the virgin binder is losing one grade for each aging stage, ending as a binder like a 20/30 but still not as hard as the RA binder. For the RA binder, the aging effect was less pronounced as it can be seen considering the changes in basic properties between the unaged and PAV aged conditions. In total between the PAV and unaged binder, the increase in softening point temperature was 11 °C for the RA as compared with 14 °C for the virgin binder. Similarly, the retained penetration value was respectively 63 % for the RA binder and 40 % for the virgin binder. This is a positive effect of already aged binder, the aging is, in relative, less than a virgin binder.

Later, would the rejuvenator will lose its effect over aging, the end properties would be between the virgin / RA aged binder, at least harder than the aged virgin binder. Thus, the analysis for each blend has been made with the data set of each individual laboratory and compared with the virgin binder.

Figure 4 shows the blend with 60 % RA with the results of four laboratories (Lab 2 did not have PAV data). The trend between each lab is not significantly different. Three of them are very close and one slightly out. Overall the relative change in properties is in the same magnitude of range as the virgin binder and does not reach the RA binder point. The effect of the rejuvenator in the 60 % blend remained over aging as recorded by the four laboratories.

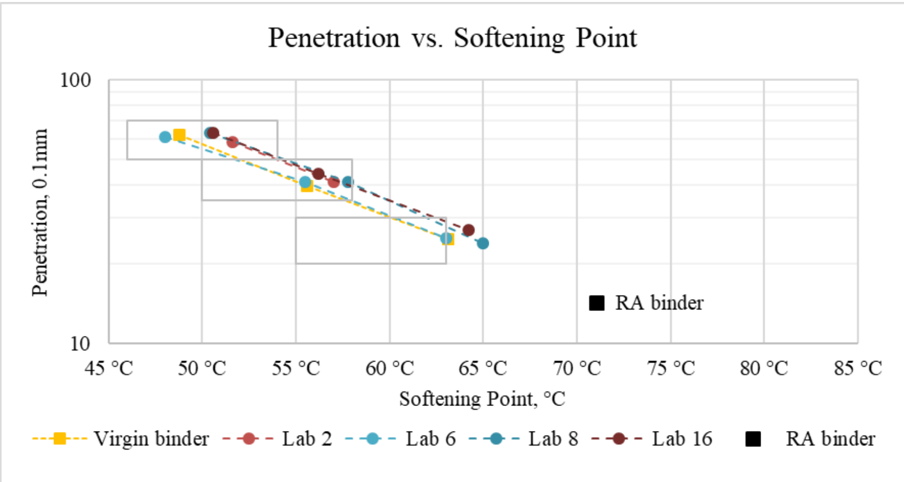

Figure 4. Aging of 60 % (RA+rejuvenator) blend

Figure 5 shows the blend with 80 % RA with the results of four laboratories, all of them having conducted RTFOT and PAV aging. The trend between each lab is not significantly different although they are a bit more dispersed than the 60 % blend. One laboratory, Lab 19, reported significantly more aging than the other and Lab 7 had a starting point for the original blend a bit softer than the other. Overall the relative change in properties is in the same magnitude of range as the virgin binder and does not reach the RA binder point. The effect of the rejuvenator in the 80 % blend remained over aging as recorded by the five laboratories.

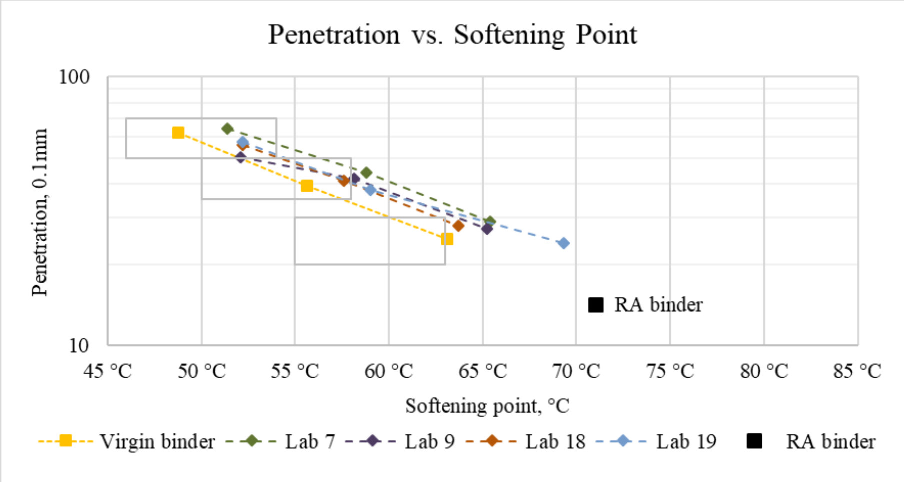

Figure 5. Aging of 80 % (RA+rejuvenator) blend

Figure 6 shows the blend with 100 % RA and rejuvenator with the results of two laboratories, having completed both penetration value and softening point temperature on unaged, RTFOT and PAV binders. With the limited data point, the analysis is less relevant and both may be seen as significantly different. In any case, the relative changes in properties were similar to the virgin binder whilst considered it was only treated RA binder with no virgin binder at all. At the end of the full aging conditioning, the points did not reach the RA values. Would have the effect of the rejuvenator disappeared over aging, the points should have been over the RA binder point. The effect of the rejuvenator in the 100 % blend remained over aging as recorded by the two laboratories.

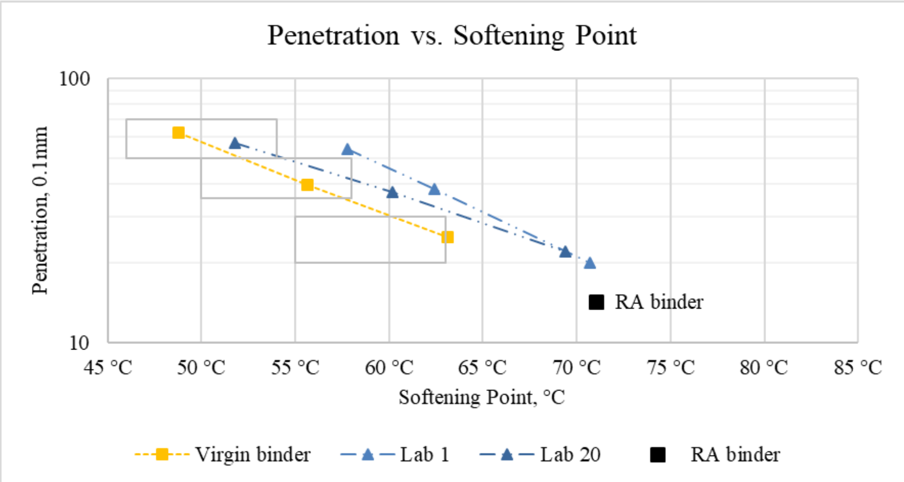

Figure 6. Aging of 100 % blend (RA+rejuvenator)

Overall, no significant difference in basic properties was observed between the blends containing 60%, 80% or 100% of RA binder at all aging stages. This is an interesting observation, as progressively the rejuvenator content was increased at the expense of the virgin binder. Hence, the aging behaviour of the blends was not negatively influenced by this type of rejuvenator.

4.3 Evaluation for low temperature

In addition to the empirical testing with penetration value and softening point temperature, more fundamental testing was run to characterise the rheology of the different blends at different aging stages. Amongst others, DSR measurements were performed; however, this will be analysed and reported in a separate publication. The low temperature behaviour was assessed using the Bending Beam Rheometer (BBR) on binder samples, having been conditioned after RTFOT and further PAV. BBR testing provides two key parameters, the creep stiffness, and the m-value, which represents the rate at which the creep stiffness changes over the test duration. The stiffness critical temperature is determined when the stiffness modulus is below 300 MPa, while the m-value critical temperature is determined when the m-value is above 0.300. The ΔTc, between creep stiffness and m-value critical temperatures, is foreseen as a parameter addressing the binder flexibility [13]. In the case of evaluating aged binder and rejuvenator, it provides a way to characterise the rejuvenating effect. The minimal accepted value is usually set at -5 °C. Four laboratories performed the BBR test at a minimum of three temperatures -6 °C, -12 °C and -18 °C. At the end of the test, these temperatures were too high to reach the creep stiffness and m-value critical values. Extrapolation was made to determine the critical temperatures respectively in arithmetic and logarithmic way for the m-value and the creep stiffness. Figure 7 displays the results for the four laboratories on the three blends and the two binders.

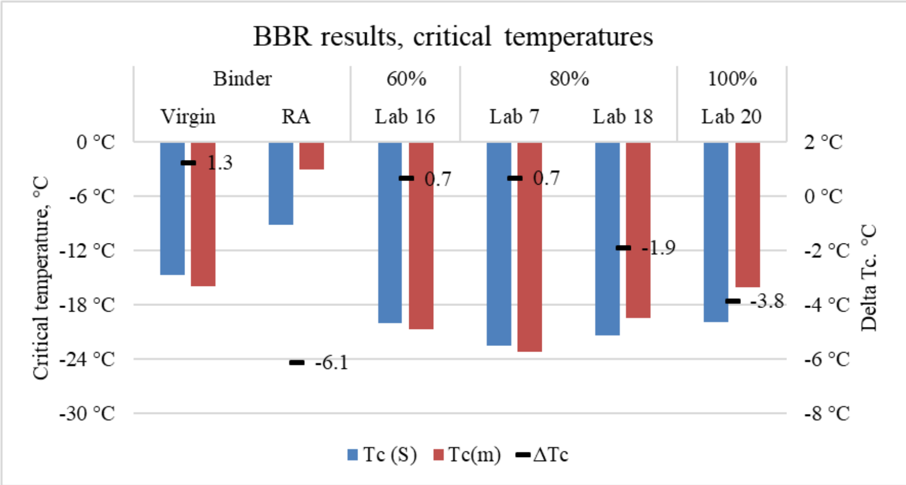

Figure 7. BBR, critical temperatures

The virgin binder had a maximal critical temperature of -15 °C with a ΔTc of +1.3°C, which should be graded as PG XX-22 as per the US PG grading ASSHTO M320. The RA binder had a higher critical temperature and should have been graded as PG XX-10. In this case the ΔTc was very low -6 °C, below the threshold foreseen in the US [13] and the binder should not be suitable for paving applications. With the 100 % RA blend, no virgin binder at all, the final critical temperature was -16 °C, equivalent to PG XX-22 and the ΔTc was improved from -6 °C, without the rejuvenator, to -3.8 °C, with the rejuvenator, above the threshold value. With the two other blends at 60 % and 80 % RA, the results were in the same magnitude of range with a critical temperature of – 20 °C / -22 °C and ΔTc higher than the critical value, the blends were less m-value controlled or even balanced between creep stiffness and m-value. The difference between the blends is mostly due to the higher ratio of RA binder as compared to the virgin binder. With limited data, the trend was consistent and the use of rejuvenator helps to both, restore the critical low temperature, even below the original binder 50/70, and keep the ΔTc above the critical threshold towards flexibility.

5 CONCLUSIONS

With the constant increase in environmental and economic constraints, recycling of RA is becoming a common practice. When the RA content is increased, when the RA displays very aged properties, its reuse in a Hot Mix Asphalt, at at-least-equal-to-normal-performance, may be challenging and requires special attention. The use of rejuvenator is foreseen as overcoming these issues, as it is currently used in Japan up to 80 % RA. However, the full effect under various conditions over time may not be fully evaluated and understood.

Under these conditions the RILEM has set up a specific Technical Committee, TC-264-RAP, to further focus more on asphalt recycling. Task Group 3 concentrates on the binder and the use of rejuvenator. An inter-laboratory working program has been conducted with the participation of twenty research institutes around the world, from industry and academia. The aim was to evaluate three blends of 60 %, 80 % and 100 % of RA treated with rejuvenator, through laboratory short-term, RTFOT, and long-term, PAV, aging. Testing included empirical testing and more fundamental rheological evaluation.

A pre-study was performed and concluded that the RA binder had very hard properties, much harder than expected from a virgin 50/70 pen grade bitumen. The dosage of rejuvenator, per weight of RA binder, was determined in order to restore the consistency at intermediate temperature based on penetration value at 25 °C.
The blends with 60 % and 80 % RA treated with rejuvenator gave consistent results between laboratories, while the 100 % blend was tested by only two labs. Overall the effect of the rejuvenator was not lost after aging and did not exceed the aging of the virgin binder regardless of the rejuvenator content.

Additionally, four laboratories performed low temperature testing using BBR covering the three different blends. While the RA binder had high critical low temperature and very low ΔTc, the rejuvenator helped to recover consistently, for the three blends at the same time, both the absolute critical low temperature and ΔTc. Further analysis with DSR measurements is ongoing and will be reported in a future publication.

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