Asphalt mixture performance and testing

Outcomes of RILEM TC 237-SIB on the affinity between aggregates and bituminous binders

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Abstract

Water damage is one of the key aspects related to pavement durability. It leads to ravelling and loss of integrity especially for asphalt pavements. There are numerous test methods that aim to address this, either with respect to binder, aggregates or the combination of both, as loose or compacted mixture. The RILEM technical committee, TC 237-SIB, focused on testing and characterisation of Sustainable Innovative Bituminous (SIB) materials and systems. Within this TC, Task Group 1, TG1, focused on bituminous binder testing and more specifically on the affinity between aggregates and bituminous binders and test methods. One of the main purposes was to run a Round Robin Test to evaluate the reproducibility and repeatability of these test methods and to give recommendations for improvement. A total of 13 laboratories participated from Europe and North America. This paper summarises the main results comparing, amongst others, the rolling bottle test and boiling water stripping test according to EN 12697-11. Three asphalt binders were selected, two unmodified and one polymer modified binder, along with four aggregate types with different mineralogy. This paper provides the key results on the evaluation of the test methods related to test procedure, measurement and interpretation of the results. The outcomes show that, while some qualitative trends are seen, there is still a large scatter in results both within the same test method and between different test methods. Thus, it is hard to consider these methods as giving essential characteristics for defining the specifications of bituminous materials. This does not come only from visual interpretation. The overall recommendation is to relate the results to qualitative classes.

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1 INTRODUCTION AND SCOPE

The ingress of water is one of the most important factors for asphalt pavement deterioration [1]. It leads to loss of the material characteristics and thus durability of the pavement [2]. The water sensitivity of asphalt mixture is more than complex. It depends on the nature of the aggregates, the cohesion and adhesion of bitumen, the environmental conditions such as temperature or moisture, the layer type, amongst other things. This subject matter has been researched for a very long time [3] and is still not precisely understood [4].
There are a lot of test methods available to address water sensitivity or the affinity between aggregates and bitumen [5]. They can be based on the presence or absence of water during the test procedure, on the type of sample, whether it be loose aggregate coated with a bitumen or a compacted asphalt mix sample. Moreover the individual components of bitumen and aggregate can be tested separately by measurement of their intrinsic properties.
The RILEM (Réunion Internationale des Laboratoires et Experts en Matériaux) had as specific technical committee, TC 237-SIB "Testing and characterization of Sustainable Innovative Bituminous materials and systems", which, with its Task Group 1 (TG1), focused on the affinity between aggregates and binder. The aim of TG1 was to further understand the fundamental mechanism of water interaction in asphalt mixture, and to review the various test methods available to evaluate the affinity of bituminous binder to aggregate. For this purpose a Round Robin Test (RRT) was conducted between 13 different laboratories from Europe and the US running different test methods [6].
Three bituminous binders were selected, two unmodified from different sources and one polymer modified binder (PmB). Four aggregate types with different mineralogy were included. The test methods considered in this RRT were carried out on loose aggregates coated with bitumen and included the affinity between aggregates and bituminous binder as described in EN 12697-11. Eight laboratories considered the rolling bottle test, whilst the boiling water stripping test was undertaken in three laboratories. Additional tests were also performed on the same bitumen and aggregates using the bitumen bond strength test as well more fundamental assessment with surface energy measurements. The full results were published in a comprehensive publication including all the results and interpretation [7] and recommendations were made [8].
This paper provides the key results and outcomes on the evaluation of the EN 12697-11 test method with regards to the Rolling Bottle Test and Boiling Water Stripping Test.

2 EXPERIMENTAL PLAN

2.1 Materials

A total of three bitumens from two different sources were used, two unmodified 50/70 paving grade bitumen and one Polymer modified Bitumen (PmB) graded as 45/80-60 according to EN 14023. Table 1 displays the basic properties with penetration value at 25°C, in accordance with EN 1426, as the consistency of the bitumen at ambient temperature and softening point temperature, in accordance with EN 1427, as the consistency of the bitumen at high temperature. The three bituminous binders had similar values in term of penetration values at 25 ºC meaning that their consistency / viscosity at ambient temperature were of a similar magnitude. Therefore, tests run at ambient temperature should not be affected by the binder consistency. On the other hand, the softening point temperature for the Polymer modified Binder was higher, around 60 ºC, than those for the neat binders. Therefore, the tests run at high temperature may be expected to have different results if viscosity is affecting the cohesion / adhesion of the bitumen with aggregates.

Table 1. Basic properties of the used bituminous binders

	Unit	Standard	Bit 1	Bit 2	Bit 3
Bitumen type			50/70	50/70	PmB 45/80-60
Penetration value at 25 °C	0.1 mm	EN 1426	51	57	50
Softening point temperature	°C	EN 1427	51.2	50.8	65.4

The four aggregates were granite, basalt, greywacke and limestone and supplied from different quarries located in the UK. They were selected to have different colour and water sensitivity based on the differences in mineralogy or composition. They can be ranked according to their degree of stripping, from slight to severe [9]. The aggregates were supplied as crushed with a nominal aggregate size of 8/11 mm.

2.2 Test methods

The affinity between aggregates and bitumen was assessed using the EN 12697-11 standard [10].
The Rolling Bottle Test (RBT) was conducted according to clause 5, with ad hoc adjustment based on each laboratory's experience and habits. The test consists of leaving coated aggregates in bottles of water and rolling for 6 h and 24 h. The degree of coating is reported after each period as the percentage of remaining coating of bitumen on aggregate, with 100 % being fully coated and 0 % being no more residual bitumen remaining. This test was the most widely used test within the round robin test with a total of eight laboratories.

The Boiling Water Stripping (BWS) test is related to the clause 7 of the standard. It consists of coating aggregates with bitumen and immersing in water which is then boiled for 10 minutes. At the end of the test the percentage of stripping is measured by an acid/base titration using a calibration curve. The titration is expected to be more accurate than visual observation as carried out in rolling bottle test. Only two laboratories conducted the boiling water stripping test according to the European standard. Another performed the test according to the French standard which keeps the coated aggregate in water at 60 °C for 16 h and then records the percentage of remaining coating of bitumen on aggregate by visual observation and report by classes.
Another test method performed by three laboratories was the Bitumen Bond Strength Test [11]. It uses the Pneumatic Adhesion Tensile Testing Instrument, PATTI ®, adapted from the paint and coatings industry. The test consists of a bond between an aggregate substrate, made from bulk stone, with a binder under controlled conditions of temperature and humidity. A bitumen sample is placed on the surface of a stub and pressed into the aggregate surface (Figure 1). The stub is pulled off and the force is recorded. The test is performed on dry samples at different temperatures. For wet conditioning, the samples are immersed in water prior to the test. The moisture damage is evaluated using the Bond Strength Ratio (BSR) between wet and dry bond strength at 22 °C.

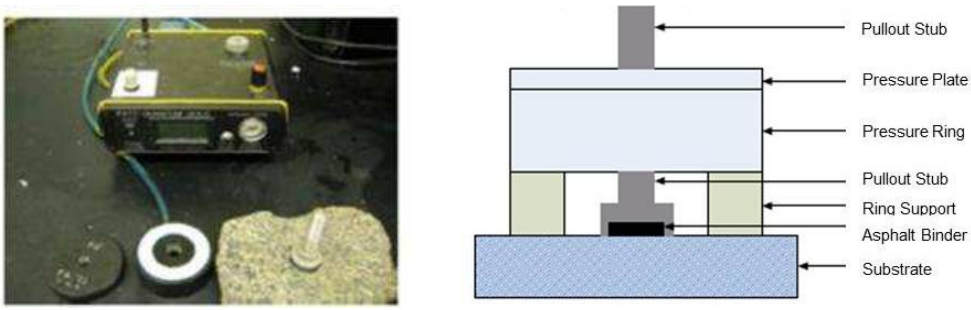

Figure 1. Bitumen Bond Strength test

3 RESULTS

3.1 Results for the Rolling Bottle Test

The results were analysed after 6 h and 24 h for the 12 combinations of aggregates and bitumen for the 8 laboratories. A total of 192 results were analysed. Figure 2 displays the average results after 6 h and after 24 h, the error bars are for min and max values. The results were more discriminating after the 24 h period and more selective between aggregate type/bitumen than as compared after 6 h. Also, it is worth noticing that no significant differences can be seen between bitumen type, whilst aggregate type can be considered as significant. The extreme results, bad results for granite and good results for limestone, were more pronounced. Aggregates such as basalt and greywacke, had intermediate values with a high variation between 10 % and 75 %.

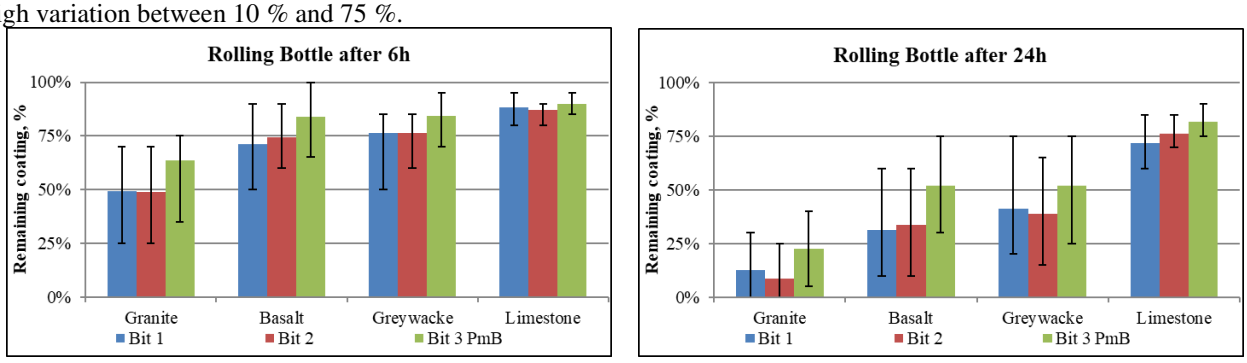

Figure 2. Rolling Bottle test – results after 6 h and 24 h

However, the test results displayed variability between the different laboratories. The highest variation being for basalt and greywacke aggregates, which showed the intermediate values, regardless of the bituminous binder. To some extent this is aligned with the precision statement of the EN 12697-11 standard. A reproducibility of 30% is given in the standard with the note: *"The obtainable precision may depend on the level of the result as determinations close to 0 or 100 are easier visually to determine than 'mid-range' results between 25 % and 75 %"*. The standard deviation was between 6 % and 21 % (absolute values) with lower scatter for high values compared to intermediate ones. The reproducibility was difficult to determine within this Round Robin Test as the number of laboratories was not high enough to provide a reliable statistical analysis.
Considering the scatter of results, the question was raised as to whether the visual observation was already too subjective to give a value. However, as some pictures were made available from laboratories, it was possible to compare the results of three laboratories having recorded significantly different results for granite and PmB, for the same bitumen/aggregate

pair. Figure 3 displays the results from left to right with one lab having recorded 5 % coating, the next 20 % and the last a 40 % coating. The comparison could lead to significant differences with the same visual observation.

Figure 3. Pictures of different rolling bottle test for same bitumen / aggregate

Furthermore, the same pictures for the 12 combinations of bitumen and aggregates were sent the participants. A total of 10 laboratories did their own independent visual observation in a blind test. Figure 4 shows the outcomes. Each lab having the same pictures, the visual observation gave significantly consistent and independent values for each pair of the 12 combinations. The reproducibility is not as bad, and does not seem to come solely from the visual observation. Maybe there are some underlying reasons for this, including the test conditions themselves.

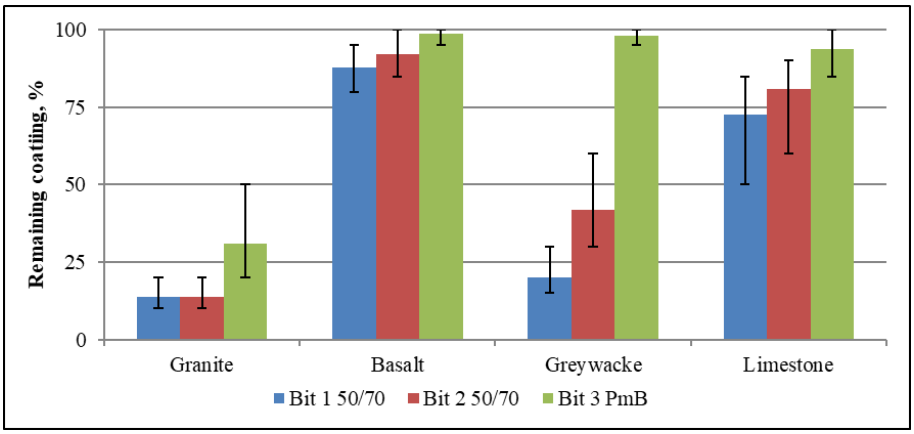

Figure 4. Results of visual observations from different laboratories on the same pictures

The outcomes for this test are:

- The results are more discriminating after 24 h compared to 6 h,
- The type of aggregates has a significant influence
- The type of bitumen has limited influence, with slightly better results for the PmB
- Record and use consistent methodology when performing the test
- Consider viscosity of the binder when performing a comparative analysis

Special attention should be made when performing the test itself. For the size of the aggregates, the 8/11 size is recommended as used by most of laboratories. The smaller the fraction, the higher the specific surface area; and this may affect the results. The aggregates have to be washed as the presence of dust affects the results with a better degree of coating at end of the test. The degree of coating depends on the amount of binder and energy put in to mix. The mixing procedure from the standard is by hand and it is difficult to define a better mixing procedure. At least the amount of bitumen used has to be adjusted accordingly with a factor to correlate with aggregate density. And while keeping the mixing time as constant as possible, the mixing temperature has to be adjusted according to the grade of the binder. The test temperature is an influencing factor as bitumen is temperature susceptible. The EN standard stipulates to fill the bottle with water at 5 ºC at the beginning of the test and then to run the test at a temperature between 15 to 25 ºC. During the RRT, the test temperature was between 17 to 23 ºC. A temperature range of 21 ºC +/- 2 ºC is recommended. The visual observation does not necessarily lead to inappropriate results, but Digital Image Processing could help [12].

3.2 Results for the Boiling Water Stripping Test

Only two laboratories performed the test according to clause 7 of EN 12697-11 and one lab with the French standard. (XP T 066-043) Thus, a statistical analysis was not possible. Figure 5 displays the results for the two laboratories having used the same protocol, the error bars are for the minimum and maximum values. The granite displayed the worst results,

while both limestone and basalt have the best results with above 80 % coating. Also, it is worth noticing a recordable difference between modified and paving grade bitumen, especially the neat unmodified bitumen Bit 1 shows higher stripping percentages compared to the other bitumens tested. The scattering of test results increased in cases where aggregate was more prone to stripping and consequently the degree of bitumen coverage decreased (e.g. granite/bitumen combinations).

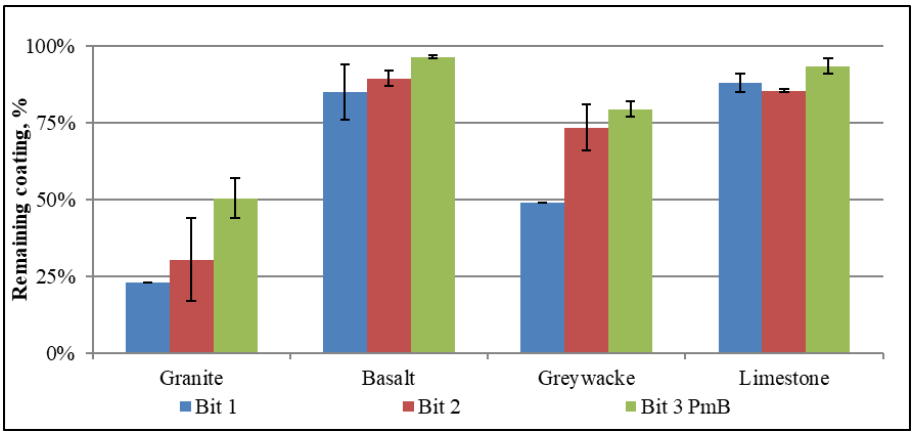

Figure 5. Results for the Boiling Water Stripping Test

At this point of time, as there were limited laboratories performing the test, statistical analysis remains impossible. However, it is worth noticing that as the test is carried out at boiling water temperature, 100 °C, the viscosity of the bitumen could play an important rule. This may explain the more discriminating results between the neat bitumen and the PmB, the latter having a higher softening point temperature. On the other hand the test condition is maybe more relevant to real conditions on the road.

3.3 Bitumen Bond Strength

Additionally three laboratories performed the bitumen bond strength. Figure 6 displays the overall results. It shows significant more variability for the results of moisture sensitive aggregate such as granite and basalt as compared to the less sensitive aggregate. The higher the value above 80 %, the lower the variability is.

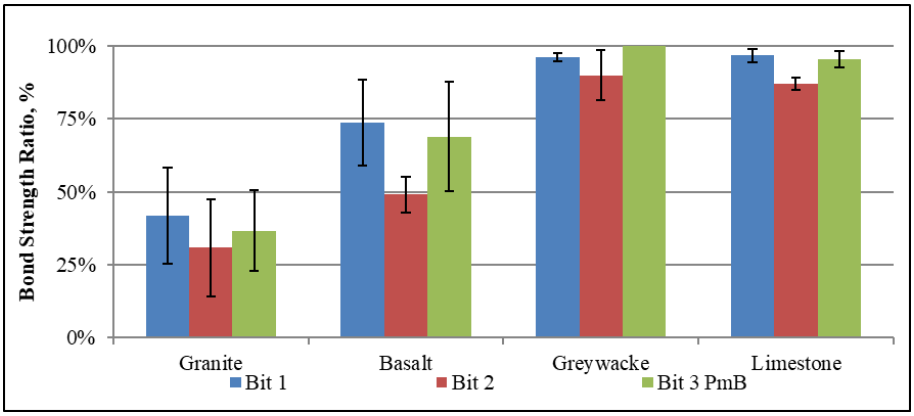

Figure 6. Results of the Bitumen Bond Strength Ratio at 22 °C (%)

At this point of time it is difficult to establish a recommendation. The sample preparation requires special attention to control the binder thickness and the way the stub is placed on the stone surface. Moreover, the test is using bulk stone with a ground surface, the texture of which can influence the surface adhesion with bitumen. And for wet conditioning, the water needs to penetrate through the aggregate to reach the interface between aggregate and binder, thus introducing an extra parameter.

4 OVERALL OUTCOMES

From the different test methods used, the Rolling Bottle Test, the Boiling Water Stripping Test or the Bitumen Bond Strength Test, the aggregate type had the largest effect on the results. The bitumen had a lesser impact, although the viscosity can affect the results as was seen in the boiling water stripping test. Table 2 shows the different ranking as

obtained from the different tests. The three test methods did not rank the aggregates in the same way, only for the worst case. The granite was always ranked as the worst, but for the other aggregates it was method dependant.

Table 2. Ranking between laboratories for each test methods

		Granite	Basalt	Greywacke	Limestone
Rolling Bottle Test after 24 h	Average coating	10 %	30 %	40 %	75 %
	Ranking	4	2.5	2.5	1
Boiling Water Stripping Test	Average coating	0-25 %	75 %	50 %	75-85 %
	Ranking	4	1	3	2
Bonding Bond Strength Test	BSR	30-40 %	50-90 %	100 %	85-100 %
	Ranking	4	3	1	2

The reasons for the different ranking may be linked with the specific properties tested and how the water affects the adhesion. For the rolling bottle and boiling water tests, rough aggregates are used, while in the BBS it is controlled-ground surface, therefore the specific surface is not the same. For the BBS, cohesion vs adhesion also impacts the results. For the rolling bottle test adhesion is mainly involved, but also sometimes aggregate abrasion. And in the boiling water test the temperature conditions are very different, so the binder viscosity may have a more pronounced effect as the temperature is closer to the softening point.
The visual observation did not seem to have an important impact on the final results, but some other means such as an automatic image analyser can improve the accuracy of the results.
Nevertheless, some recommendations can be made to consider values in terms of class from 1 to 4 rather than an absolute value, as suggested in Table 3. However, at this point of time it does seems suitable to be used as essential characteristics defining the specifications of bituminous materials.

Table 3. Recommended classes for affinity between aggregate and binders

Class	1	2	3	4
Ratio	100% - 75%	75% - 50%	50% - 25%	25% - 0%

5 CONCLUSIONS

As water damage is one of the most important parts of asphalt pavement durability, RILEM TC 237 SIB Task Group 1 worked on the affinity test between aggregate and bitumen. A Round Robin Test was conducted with 13 laboratories on three test methods – the rolling bottle test, the boiling water stripping test and the Bitumen Bond Strength test - with combinations of four different aggregate types and three bituminous binders.
The most common test run by eight laboratories was the rolling bottle test. It consists of recording the remaining percentage of bitumen coating on aggregates after being rolled in a water bottle. The outcomes show that the results are discriminating after 24 h. While the reproducibility was fair, it did not come solely from visual observation. Other possible causes include the test conditions and sample preparation.
The boiling water stripping test is a more accurate way measuring of the remaining bitumen coated on aggregates after being left in boiling water for 10 minutes. It was used only by three laboratories. The test results are discriminating towards aggregate type and may exhibit differentiation for bitumen having differences in viscosity
The Bitumen Bond Strength test consists of measuring the bonding strength of bitumen on a substrate of aggregate. The Bonding Strength Ratio of wet over dry strength values has been shown to be in similar ranking than the other tests. Certainly, other parameters bring additional artefacts in the results and their interpretation.
The final recommendations from this study are that the different tests display similar results for extreme values, best or worst combinations of aggregate binder. However, for intermediate performing combinations, the correlation between tests is less obvious. While reproducibility was difficult to determine due to insufficient data sets, it is in line with what is reported in the literature and the current standard. While it looks interesting test for comparative evaluations between different combinations of material, it does not address essential characteristics that can be used further in specifications for bituminous materials. In order to better improve the accuracy of the results, it is suggested to provide results within four classes from class 1 for the best, to class 4 for the worst.

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