



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

Laurent Porot, Hilde Soenen, Alex Apeagyei, James Grenfell, Stefan Vansteenkiste, Emmanuel Chailleux

► To cite this version:

Laurent Porot, Hilde Soenen, Alex Apeagyei, James Grenfell, Stefan Vansteenkiste, et al.. Recommendation of RILEM TC 237-SIB on affinity between aggregates and bituminous binders. Materials and structures, 2018, 51 (6), 7 p. <10.1617/s11527-018-1297-6>. <hal-03257666>

HAL Id: hal-03257666

<https://hal.science/hal-03257666v1>

Submitted on 6 Jul 2021

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



HAL Authorization

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

Laurent Porot¹, Hilde Soenen², Alex Apeagyei³, James Grenfell³, Stefan Vansteenkiste⁴, Emmanuel Chailleux⁵

¹ Kraton Chemical, Almere, The Netherlands

² Nynas, Machelen, Belgium

³ University of Nottingham, Nottingham, UK

⁴ Belgium Road Research Center, Brussels, Belgium

⁵ LUNAM Université, IFSTTAR, Bouguenais, France

Abstract These recommendations are based on the results of a round robin test organised by the RILEM technical committee TC 237-SIB “Testing and characterization of sustainable innovative bituminous materials and systems”. The Task Group 1 (TG1) focused on binder testing. The aim of the 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 binders to aggregate surfaces. A total of 13 laboratories participated, running different test methods. Three bituminous binders and four aggregate types were selected for testing. These recommendations focus on the main aspects related to testing procedure, measurement and presentation of results. While some qualitative trends are observed, there is still a wide scattering in results both within the same test method and between test methods. This is not only related to a visual interpretation of the degree of bitumen coverage, but also theoretical and numerical analyses of test results. The overall recommendation is to consider the results within qualitative classes.

Keywords Bituminous binder, aggregate affinity, round robin test, water sensitivity, durability, surface energy, adhesion, rolling bottle test, boiling water test, bitumen bond test

This recommendation was developed by the task group TG1 within the RILEM TC 237-SIB consisting of Laurent Porot, Hilde Soenen, Alex Apeagyei, James Grenfell, Stefan Vansteenkiste, and Emmanuel Chailleux. The draft recommendation was submitted for approval to the full TC and subsequently approved by RILEM TIC 237-SIB.

TC Chairman: Manfred N. Partl (EMPA – Swiss Federal Laboratories for Materials Science and Technology, Dübendorf, Switzerland)

TC Secretary: Emmanuel Chailleux (IFSTTAR – French Institute of Science and Technology for Transport, Development and Networks, Nantes, France)

TG1 Group Leader: Laurent Porot (Kraton Chemical, Almere, The Netherlands)

TC Members: Gordon Airey (United Kingdom), Alex Apeagyei (United Kingdom), Ignacio Artamendi (United Kingdom), Hassan Baaj (Canada), Hussain U. Bahia (USA), Wojciech Bankowski (Poland), Maurizio Bocci (Italy), Yves Brosseau (France), William G. Buttlar (USA), Francesco Canestrari (Italy), Armelle Chabot (France), Emmanuel Chailleux (France), Eshan Dave (USA), Joëlle De Visscher, (Belgium), Hervé Di Benedetto (France), Augusto Cannone Falchetto (Germany), Gilda Ferrotti (Italy), Tomas Gabet (France), Marcin Gajewski (Poland), Vincent Gaudefroy (France), Andrea Graziani (Italy), James Grenfell (United Kingdom), Andrea Grilli (Italy), Ferhat Hammoum (France), Bernhard Hofko (Austria), Martin Hugener (Switzerland), Illan Ishai (Israel), Kim Jenkins (South Africa), Nicole Kringos (Sweden), Greet A. Leegwater (The Netherlands), Eyal Levenberg (Israel), Andreas Loizos (Greece), Davide Lo Presti (United Kingdom), Xiaohu Lu (Sweden), Paul Marsac (France), Anne Millien (France), Konrad Mollenhauer (Germany), Francisco Morea (Argentina), Virginie Mouillet (France), Patrick Muraya (Norway), Marco Pasetto (Italy), Manfred N. Partl (Switzerland), Daniel Perraton (Canada), Christophe Petit (France), Katherine Petros (USA), Laurent Porot (The Netherlands), Simon Pouget (France), Lily D. Poulikakos (Switzerland), Christiane Raab (Switzerland), Safwat Said (Sweden), Cesare Sangiorgi (Italy), Cédric Sauzéat (France), Tom Scarpas (The Netherlands), Hilde Soenen (Belgium), Dariusz Sybilsky (Poland), Gabriele Tebaldi (Italy), Ann Vanelstraete (Belgium), Stefan Vansteenkiste (Belgium), Michael Wistuba (Germany), Ludo Zanzotto (Canada), Adam Zofka (Poland)

1 Introduction and scope

Asphalt materials are sustainable materials used for pavement and roofing application thus subject to weather conditions especially rainfall, moisture and freeze thaw. These conditions lead to pavement damage. Water sensitivity of asphalt materials is influenced by contact between bituminous binder and aggregates, including complex phenomena such as wetting, cohesion and adhesion of the binder in the presence of moisture. This has been a subject for much research over the years [1] [2] and different test methods exist to characterise it [3] [4]. However, consensus is lacking in terms of the most effective method to characterise the water sensitivity phenomenon and hence the need for establishing recommended procedures based on consensus among experts.

These recommendations are based on the results of a round robin test (RRT) organised by the RILEM technical committee TC 237-SIB “Testing and characterization of sustainable innovative bituminous materials and systems”. The Task Group 1 (TG1) focused on binder testing. The aim of the TG1 was to further understand the fundamental mechanism of water interaction in asphalt mixture application, and to review the various test methods available to evaluate the affinity of bituminous binder to aggregate. These recommendations focus on the main aspects related to testing procedure, measurement setup, data analysis and presentation of results.

A total of 13 laboratories participated, running different test methods. Three bituminous binders were selected, two unmodified from different sources and a polymer modified binder. Four aggregate types with different mineralogy were used. The use of adhesion promoter, also known as anti-stripping agent, was not included in the experiment to avoid complications and also because some of the common antistripping agents are proprietary materials. The test methods considered in this study were limited to those on loose aggregates coated with binder and included:

- the Rolling Bottle Test with eight laboratories,
- the Boiling Water Stripping Test with three laboratories,
- the Bitumen Bond Strength Test with three laboratories,
- and a more fundamental measurement of surface energy for two laboratories.

The first three tests used different approaches to quantify the degree of stripping resistance. The Rolling Bottle Test is a visual observation of the degree of coating. The Boiling Water Stripping Test involves using a chemical titration procedure, while the Bitumen Bond Strength Test uses a mechanical measurement. Additional details on the results of the RRT have been already published [7].

2 Rolling Bottle Test

The Rolling Bottle Test (RBT) was performed by eight laboratories, based on the EN 12697-11 standard clause 5 [8] and the best practice of each laboratory. During the test, coated aggregates are immersed in a bottle of water and rolled for 6 h and 24 h. The results are the percentage of remaining coating of binder on the aggregates, as assessed by visual observation.

Out of the combination of the four aggregates and the three different binders, the outcomes were that the results are more discriminant after 24 h and more selective between aggregates than between the binders, with slightly better results for the polymer modified binder [5]. Nevertheless, an important scattering of the results was seen between laboratories. The extreme results, either bad with granite or good with limestone, were more pronounced. The intermediate results displayed a lot of variation between 20 % and 80 %. With additional cross-survey between the laboratories using the same pictures, the visual observation did not lead to important differences in the values.

Thus, the recommendation for this test is to stick precisely to the test procedure especially

- when considering the preparation of the aggregates to be washed,
- the EN standard allows doing the mixing procedure of bitumen and aggregates by hand; however, this can result in differences in energy and time used for the coating of the aggregates,
- the test temperature is certainly one of the main influencing factors of the results as bituminous binder is temperature susceptible. The EN standard stipulates to fill the bottle with water at 5 °C at the beginning of the test and then to run the test at a temperature between 15 to 25 °C. It is recommended to reduce the test temperature range to 21 °C +/- 2 °C.

Finally, for the results, the outcomes suggest reporting the results after 24 h and to consider classes as already in use in EN 13614 for the adhesivity of bituminous emulsion.

3 Boiling Water Stripping Test

The Boiling Water Stripping test was performed only by three laboratories, two of them based on the EN 12697-11 standard clause 7 [8] and the third one on the French standard XP T 066-043 [9]. Similarly, to the Rolling Bottle Test, coated aggregates are immersed in water and boiled for 10 minutes, in case the procedure from the standard EN 12697-11 was applied, or alternatively kept at 60 °C, following the French methodology. At the end of the test the percentage of stripping is recorded by titration with acid (EN

12697-11 procedure only). This is anticipated to be a more objective assessment as compared to the rolling bottle method.

The outcomes of the test gave slightly different results than the Rolling Bottle Test [6]. The test can distinguish aggregates with the granite having the worst results, but both limestone and basalt recording very good results above 80 % coating. It was also discriminating with better results for the modified bitumen.

As only three laboratories conducted this test, it is difficult to draw conclusions and recommendations for this test procedure. However, the usage of aggressive acids makes the test less attractive to use. As a result, of this shortcoming, it is recommended to consider computer-based image analysis techniques to obtain measurements that are more accurate.

4 Bitumen Bond Strength Test

The Bitumen Bond Strength Test was performed by three laboratories according to AASHTO TP 91 [10] using the Pneumatic Adhesion Tensile Testing Instrument (PATTI ®). The test consists of a bond between an aggregate substrate, made from bulk stone, with a binder under controlled conditions of temperature and humidity. The force to pull off is recorded as a result. Moisture damage is evaluated as the ratio of results from dry sample and water-immersed sample prior to the test.

The outcomes were different as well compared to the two other tests. The granite still displayed the lowest results, while the greywacke and limestone had similar very good results. It showed as well some differences between bituminous binders.

Again, at this point of time it is difficult to establish a recommendation. Special attention is needed for sample preparation especially in order to control the binder thickness and the way the stub is placed on the stone surface. This test uses bulk stone with a ground surface, which affects the surface adhesion with bitumen. In addition, in the case of the wet conditioning, the water needs to penetrate through the aggregate to reach the interface between aggregate and binder, thus introducing an extra parameter.

5 Surface energy measurement

The measurement of surface energies is a more fundamental test to determine intrinsic properties of aggregates, bituminous binders and a combination of both. Two laboratories were able to perform these measurements using different methodologies. Three experimental devices were used to determine the surface energy of the aggregate and bitumen samples. They included a dynamic contact angle (DCA) analyser, a

dynamic vapour sorption (DVS) system and a Drop Shape Analyser (DSA). Surface energy measured for the binder gave similar results for both laboratories. For aggregates, the surface energy values appear significantly different for both laboratories. There is no explanation for this at this moment but it must be noted that the theoretical framework used by both laboratories were different.

A first recommendation could be to work on the same theoretical framework or model. Another recommendation is to assess the influence of the surface state, roughness and porosity.

6 Overall outcomes and recommendations

From the different test methods used, i.e. the Rolling Bottle Test, the Water Boiling Stripping Test and the Bitumen Bond Strength Test, the conclusion is that the aggregate type mostly affects the results. The binder impacts the results significantly, mostly with the boiling water, only when the temperature is raised at least to the softening point level of the binder. While for granite, the three tests displayed the worst results consistently, for the others, the ranking from one test to another changed as shown in Table 1. Hence, it is not obvious to conclude that the three methods are equal and provide similar results. They all discriminate granite to have the worst affinity with a bituminous binder in the presence of water. For limestone, considering the absolute values, they all rank it as having good results in terms of affinity. These results are consistent with existing literature and field performance, as many past studies have shown granite mixtures as most susceptible and limestone mixtures least susceptible to moisture. However, for intermediate values, the ranking is not consistent, and the absolute values are not comparable.

Table 1. 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	3	2	1
Boiling Water Stripping Test	Average coating	0-25 %	75 %	50 %	75-85 %
	Ranking	4	1	3	2
Bitumen Bond Strength Test	BSR	30-40 %	50-90 %	100 %	85-100 %
	Ranking	4	3	1	2

Despite the reasonably good level of participation in the round robin test, the number of results was not enough to determine statistically valid repeatability and

reproducibility. Still, some recommendations can be made to consider values in terms of classes rather than absolute values. Typically, four different classes are recommended, as in Table 2, for the degree of remaining coating after the test, from 1 being the best and 4 the worst.

Table 2. Recommended classes for affinity between aggregate and binders

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

7 Conclusions

Within the RILEM TC 237-SIB, the Task Group 1 (TG1) worked for a better understanding of the adhesion and cohesion between bituminous binder and aggregate, especially in presence of water. For this purpose, 13 laboratories conducted a Round Robin Test on affinity between aggregates and bituminous binders in presence of water. Three methods – Rolling Bottle Test, Boiling Water Stripping Test and Bitumen Bond Strength Test – were used with four different aggregate types and three bituminous binders. In addition, more fundamental investigation, considering surface energy, was included.

The most common test run by eight laboratories was the Rolling Bottle Test. It consists of recording the remaining percentage of bituminous binder coating aggregates after being rolled in a water bottle. The outcomes show that the test starts to be really discriminating after 24 h. The reproducibility is to date rather fair and depends on the degree of coating, worst for intermediate values. The visual evaluation did not appear as the most important cause of poor repeatability. Other possible causes include the test conditions and sample preparation.

The Boiling Water Stripping Test consists of more accurately measuring remaining coated bitumen on aggregates after being left in hot or boiling water. The test results are discriminating towards aggregate type. As the test is run at high temperature, the impact of the bitumen type may depend largely on differences in the softening point.

The Bitumen Bond Strength Test consists of measuring the bonding strength of bitumen on a block of aggregate stone. The test addresses more cohesive failure in the dry state and adhesive or cohesive failure (depending on aggregate-binder combination) in the wet state. The Bonding Strength Ratio of wet over dry strength values produced similar rankings as compared to the other tests. However, with a limited number of laboratories, the reproducibility was difficult to determine, and absolute results have shown a wide variability between laboratories.

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 values, 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. As an alternative to represent the results, it is suggested to provide results within four classes from class 1, the best, to class 4, the worst.

Acknowledgements

The authors want to thank Jeroen Besamusca, Vincent Gaudefroy, Preeda Chaturabing, Crsitina Tozzo, Ignacio Artamendi, Darius Sybilski, Francisco Barcelo Martinez, Said Safwat, Manfred Partl, Francesco Canestrari, Elisabeth Hauser, and Michael Wistuba for their valuable contribution.

Compliance with ethical standards

Conflict of interest: The authors declare that they have no conflict of interest.

Bibliography

- [1] W. Rieder and H. Weber, "Adhesion of Bituminous Binders to Stone," *Asph.u. Teer*, 1933, 37-41.
- [2] D. H. Mathews, D. M. Colwill and R. Yüce, "Adhesion tests for bituminous materials," *J. Appl.Chem.*, 15, September, 1965..
- [3] NCAT, "Stripping in HMA Mixtures: State-of-the-art and critical review of test methods," NCAT Report No 88-02, September 1988..
- [4] BitVal, "Analysis of Available Data for Validation of Bitumen Tests," Report on Phase 1 of the BitVal Project, 2006.
- [5] L. Porot, H. Soenen, J. Besamusca, A. Apeagyei, J. Grenfell and D. Sylbilski, "Bitumen / Aggregate affinity – Rilem Round Robin Test on rolling bottle Test," in *RILEM SIB Symposium*, Ancona Italy, 7-9 October 2015.

- [6] S. Vansteenkiste, J. Grenfell, L. Porot, A. Apeagyei, H. Soenen and E. Chailleux, "Bitumen aggregate affinity: a comparison between rolling bottle and boiling water test," in *7th EATA Symposium*, Dubendorf Switzerland, 2017.
- [7] M. Partl, L. Porot, H. Di Benedetto, F. Canestrari, P. Marsac and G. Tebaldi, Testing and Characterization of Sustainable Innovative Bituminous Materials and Systems - State-of-the-Art Report of the RILEM 237-SIB, vol. 24, RILEM, Ed., Switzerland: Springer, 2018.
- [8] EN, "CEN (Comité Européen de Normalisation), EN 12697-11 - bituminous mixtures – test methods for hot mix asphalt – part 11: determination of the affinity between aggregate and bitumen," EN 12697-11.
- [9] NF, "XP T 066-043 - Essai d'adhésivité passive des liants anhydres en présence d'eau," Norme Française.
- [10] AASHTO, "AASHTO TP 91 - Method Of Test For Determining Asphalt Binder Bond Strength By Means Of The Asphalt Bond Strength," 2011.