Development of a new direct adhesion test method

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ABSTRACT

The principal task of a binder is the bonding (adhesion) of the used components. The adhesion is affected by the adhesive strength of the binder and the characteristics of the aggregate surface. The climatically conditions can additionally influence the durable adhesion. Many asphalt damages can be traced back on a lack of bonding between aggregate and binder.

A new test method developed at the Ruhr-University of Bochum analyzes the adhesion between "aggregate and binder" with a direct test at the area of contact in the shear-, oscillation and/or tensile loading mode in combination with an exact temperature control system to allow an objective rating. The testing can be realized in a wide temperature range, with or without the presence of water.

For this test method the Dynamic Shear Rheometer is used, which is able to record the binder viscosity continuously for measuring under equivalent viscosity conditions. The test conditions are related to the sort of binder, aggregate, binder film thickness, temperature, water quality and loading are variable. With this test method the adhesion properties are quantifiable at the relevant area of contact (aggregate-binder-aggregate) with an expected high precision and testing variability.

The paper will describe the test method developed and the results and experiences gained until now.

Keywords: adhesion, dynamic shear rheometer, aggregate, binder

1. INTRODUCTION

A strong and permanent bonding between mineral aggregates and bitumen is essential for a long durability of a road [1]. The first studies about adhesion were done in the nighteen-thirties [2] and still this matter is not sufficiently explored.

Describing the bonding between mineral aggregates and bitumen is only possible with different theories and simultaneous processes [3]. There are two different kinds of failures – a break due to adhesion or cohesion. If there is a cohesion break the aggregates are separated from each other while the bonding between aggregate and bitumen stays intact. The adhesion failure is due to a fractured surface between mineral aggregate and bitumen that happens only in the presence of water. The water infiltrates, causes a displacement and finally a detachment of the bitumen from the mineral aggregate [4].

In the past more than 100 test methods for the bonding have been developed [1]. In 2004 a CEN Ad-Hoc group was founded. The aim of this group was to find a test method that allows an indication of adhesion or more precisely determine the loss of adhesion in the presence of water. However the outcome of this working group was: No easy to use test method was found to characterize the adhesion / stickiness of bitumen and it is not likely that one will emerge in the near future [5]. One of the reasons is that many test methods cannot exclusively test the adhesion, but are influenced by other parameters, e.g. the rolling bottle test, where results are influenced by the abrasion because of mechanic-dynamic stress. Therefore, the aim of this project is the development of a test method to describe adhesion behaviour of different bitumen taking into account many influencing factors. Thereby the following requirements by [5] should be considered:

- simple use
- differentiation between different kinds of bitumen
- reproducibility
- duration < 7 days
- the test samples must allow access of water to interface voids
- the test samples should be capable to include the development of ageing

2. DEVELOPMENT OF THE NEW DIRECT ADHESION TESTING METHOD

The newly to develop testing method should allow the use of existing testing instruments. For this reason and because of the several possibilities to change the boundary conditions the dynamic shear rheometer (DSR) has been selected. It is possible apply an oscillating stress at different frequencies and a tensile loads. The chosen test installation is shown in figure 1.



Figure 1: Schematic construction "direct adhesion testing method"

A stone sample (\emptyset 25 mm) is fixed with special glue to the bottom of a water tank (Figure 2). For the first tests samples of silica glass were used, as it's a matter of common knowledge that acidic stones (silica content > 70 %) have a bad influence on the adhesion [6]. They are hydrophilic (water affine) and consequently adhesion problems occur [3]. The adhesion is also influenced by surface roughness of the mineral aggregates. To eliminate this influence the first tests have been made with polished surfaces to be able to evaluate the adhesion behaviour of different bitumen. In further steps the influence of the mineral aggregate can also be tested using different stone samples. The influence of the surface roughness can be determined leaving the samples in their original, unpolished condition. To study one individual influencing factor the other factors must be constant during one series of tests otherwise the changing of the adhesion behaviour can not clearly be matched to the right source.

The water tank with the stone sample is clamped on the lower fixed plate of the DSR (Figures 3 and 4). Another stone sample (Ø 25 mm) is fixed on the upper oscillating plate (Figure 5). The bitumen sample is fixed into the middle of the lower stone sample (Figure 6) after installation of the water cup and the upper test geometry. This bitumen sample has a diameter of 8 mm. For the reproducibility of the test it is very important that the bitumen samples have the same weight

 $(0,17 \text{ g} \pm 0,02 \text{ g})$. Afterwards the DSR can be moved to the measure position and the tank can be filled with water. At the moment the used water is deionized to eliminate the influence of the pH-value.



Figure 2: Stone sample in water tank



Figure 4: Clamped water tank



Figure 3: Cup holder for the water tank



Figure 5: Upper test geometry with stone sample



Figure 6: Sample of bitumen insert in the test instruments

The boundary conditions of this new testing method are resumed in table 1.

| Boundary condition | Value |
|--------------------|----------------------------------|
| gap distance | 2 mm |
| test duration | 24 h |
| temperature | equivalent viscosity temperature |

| Table 1: | Boundary | conditions o | f the | testing | method |
|----------|----------|--------------|-------|---------|--------|
|----------|----------|--------------|-------|---------|--------|

The gap distance between the stone samples was fixed to 2 mm based on pre-testing. The temperature of the device when the bitumen is put on the lower plate and the DSR is moved to the measure position must be chosen such that the softening point ring and ball is reached to assure a strong bonding between mineral aggregate and bitumen at the beginning of the test. Figure 7 shows the good bonding between stone and bitumen. For the picture the gap was increased and as figure 7 shows the bonding is still good.



Figure 7: Good Bonding between stone and bitumen at the beginning of the test

The DSR-testing-temperature depends on the type of bitumen because the testing should be done at an equivalent viscosity temperature to avoid the influence of the bitumen viscosity. The DSR can determine the dynamic viscosity according to DIN EN 13702-1 and the complex shear modulus according to DIN EN 14770. The viscosity is determined for temperature ranges between 60 and 170 °C. Pre-testing showed that a paving grade bitumen 30/45 should be tested at a temperature of 20 °C. For this temperature the viscosity could not be determined but as figure 8 shows there is a functional relation between the viscosity and the complex shear modulus.



Figure 8: Functional relation between dynamic viscosity and complex shear modulus

The complex shear modulus was examined also for temperatures below 60 °C. Therefore the complex shear modulus is chosen to define the test temperatures. In figure 9 the determination of the testing temperatures for paving grade bitumen 20/30, 30/45, 50/70 and 70/100 is shown. These temperatures are given in table 2.



Figure 9: Determination of the testing temperatures

| Type of paving grade | Testing temperature |
|----------------------|---------------------|
| bitumen | [°C] |
| 20/30 | 26,0 |
| 30/45 | 20,0 |
| 50/70 | 17,0 |
| 70/100 | 15,5 |
| 160/220 | 7,5 |

Table 2: Testing temperatures of different paving grade bitumen

The test installation is left at rest for 30 minutes before bringing up an oscillating sinusoidal stress. The frequency is 1 rad/s and the deformation is 10 %. The development of the complex shear modulus and the normal force over time is used for the analysis and evaluation of the bitumen adhesion. A tensile load is not used because not every DSR has this function and the aim was to find a new test method which most of the laboratories can make with their existing instruments.

3. Results and Discussions

With this new test method it is possible to evaluate different bitumen as to their adhesion behaviour. The complex shear modulus results of two types of paving grade bitumen are shown in the plots of Figure 10. In this testing the type of the bitumen was the only thing which was changed. The other boundary conditions were fixed. So Figure 10 shows the influence of different paving grade bitumen on adhesion. As figure 10 shows the complex shear modulus of the 30/45 decreases very fast. After five hours the complex shear modulus stagnates at a bottom level so that you can assume that there is a loss of bonding between stone sample and bitumen. The process of adhesion failure runs much slower with the bitumen 50/70. This bitumen reaches the same complex shear modulus as the bitumen 30/45 after almost 30 hours. The bitumen 50/70 shows a higher resistance to adhesion failure than the bitumen 30/45. The viscosity of the both bitumen is equal so that the difference is due to the chemical composition of the bitumen.

In the next months it will be proved which value is the best one to describe the adhesion failure. Maybe it could be also the phase angle or a combination of phase angle and complex shear modulus. But figure 10 already shows the possibility to describe the adhesion failure and the differences between bitumen with this test method.



Figure 10: Results of new testing method for different kinds of road bitumen

Furthermore different types of aggregate can be tested and samples with a diameter of 25 mm must be taken. To determine the effect of the petrographical, mineralogical and chemical composition of the mineral aggregates the samples must have polished surfaces. If additionally the influence of the surface roughness should be evaluated then the surfaces must be left unpolished. The effect of the water characteristics can be tested by making the test with water with different pH-values instead of deionized water. According to [5] the filler has an important influence on the durability of asphalt. With this test method also mixtures of bitumen and filler can be tested. Furthermore aged bitumen samples can be used. In order for the results to be reproducible a very exact sample preparation and experimental procedure is necessary. The requirements of the CEN Ad-Hoc group for a new test method as mentioned in paragraph 1 seem to be

complied with this test method but it is necessary to make more tests to validate this testing method. Maybe improvements on the boundary conditions are still possible. This has to be clarified with future tests.

4. Conclusions

A new test method for measuring adhesion was developed. The planned test series have not been completed yet but the results obtained until now are promising and it is expected that the test will deliver good results in creating a background of the adhesion behaviour of different bitumen and mineral aggregates. Furthermore many influencing factors can be analyzed with this test. In the coming months more tests will be done to validate this test method and to optimize the boundary conditions.

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