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AFFINITY BETWEEN BITUMEN AND AGGREGATES: IMPROVEMENT OF THE EN STATIC TEST METHOD

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ABSTRACT

Stripping is a major problem in many countries and can lead to severe pavement damage. Although adhesion properties between aggregate and binder is only one of the possible failure criteria in terms of stripping, it is regarded as important enough to develop test methods for their assessment. In 2005 the revised European standard 12697-11:2005 [1] was introduced offering a choice of three completely different methods. After the evaluation of these methods none was regarded as suitable for the definition of requirements for adhesion properties.
The static method described in part B has some similarities with the replaced Swiss standard for the determination of the affinity between bitumen and aggregates [6]. But a first trial series on Swiss aggregates showed some major problems and the limits of the original procedure of part B. As part of a research project, the static method was further developed including some procedures already used in the other methods of the standard [7]. An increase of the precision was achieved with the help of graphics for the evaluation of the amount of bitumen coverage. Some of the major impact factors like the mixing conditions and the temperature for water conditioning have then been studied in more detail. The resulting modified method looked promising and was validated with different binders and the most common Swiss aggregate types. The outcome of the research project will be considered in the current revision of the European Standard 12697-11:2005.

Keywords: Stripping, adhesion, European standard method

1. INTRODUCTION

2.2 Background

Stripping is a major problem in pavement construction, especially in countries with wet climate. Different mechanisms contribute to the stripping phenomenon [4-6], but one of the key parameter is the adhesion property between aggregate and binder. It was regarded important enough to develop specific test methods for their assessment. In the review on water sensitivity by Airey and Choi [7] the most popular tests methods are characterized and discussed.

There are two major principles to evaluate the water sensitivity of pavements and asphalt mixtures. Tests are performed either on bitumen coated aggregates or on compacted asphalt specimens. The assessment of compacted asphalt specimens or cores is composed of some kind of water conditioning, followed by a relative comparison of conditioned and unconditioned samples using a standard mechanical test like Duriez, Marshall or indirect tensile test. However, with asphalt mixes the degree of water sensitivity cannot be separated from other influences like void content or aggregate size distribution, which have a significant impact on the test result.

For this reason, in many countries tests on loose coated aggregates, generally referred to as immersion or boiling tests, are preferred. Working group WG 1 of CEN TC 227 had the task to harmonize the different existing national standards dealing with adhesion properties and in 2003 prepared the first version of the European Standard 12697-11 “Determination of the affinity between aggregate and bitumen“. The revised version of 2005 contains three different methods, part A, B and C [1]. Each country has to choose one of these methods for their national testing and requirements, if they consider adhesion as a problem.

Switzerland initiated a research project to evaluate the different methods and to compare them with the former Swiss national standards [2]. In addition, it answers the question, whether new requirements have to be formulated or the existing values from the national standard can be adopted. After the evaluation of the three different methods none of them was regarded as suitable for Switzerland [3]. This paper deals mainly with the improvement of the test method described in part B of the European standard, which is closest to the previous Swiss standard.

2.3 Basic principles of the test methods

In all the methods of EN 12697-11:2005 and the Swiss National Standard SN 670 460 [2] minerals of a specific aggregate size are selected and coated with hot bitumen. After cooling down, the aggregates are immersed in water for a defined time and a specific water temperature. If the adhesion strength is not sufficient, water is infiltrating between the thin bitumen layer and the mineral surface, resulting in a weakening or even partial removal of the bitumen film. This can be evaluated by assessing the degree of coating for each mineral aggregate and the adhesion property is characterized by the so called coating index. In table 1 the procedures of the four test methods for the assessment of the adhesion property are compared.

Table 1: Differences between European and Swiss standards for adhesion properties

	EN 12697-11:2005 [1]			SN 670460 [2]
	Part A	**Part B**	**Part C**	
Aggregate coating				
Number of samples	3	1 or 4	2	2
Binder amount	16g (3.1%)	4% (±x·0.5%)	31.5 g (2.1%)	8 g (5.3%)
Aggregate size	8/11.2 (5.6/8, 6.3/10) mm	6/10 mm	7/14 mm	8/11.2 mm
Aggregate amount	510 g for 3 sets	150 particles	1500 g	150 g
Mixing temperature	variable, according to EN 12697-35 + 25°C [8]	130°C	variable, according to EN 12697-35 [8]	variable, according to EN 12697-30 [9]
Cooling time	12 h	60 min	Immediate quenching in cold water	5 min
Water conditioning				
Conditioning type	rolling bottle	static	static	static
Water temperature	20 ±5°C	19 ±1°C	boiling water (90 - 100°C)	60 ±0.5°C
Conditioning time	24 ±1h	48 ±1h	10 min	60 ±1 min
Assessment of the coating degree				
Condition of the aggregates	wet	dry	in hydrochloric or hydrofluoric acid	wet
Validation of the result	visual estimation of the coating proportion after 6 and 24 h	number of not completely coated aggregates (visual)	titration of the reaction solution	visual estimation of the coating proportion with graphical aids
Result	coating coefficient in 5% steps	passing yes/no	coating coefficient in 1% steps	coating coefficient in 1% steps

2. EXPERIMENTAL

2.1 Materials

Five different bituminous binders including PmBs with penetration values in the range of 10 to 200 [0.1 mm] were used for the tests covering the most important range of bituminous binders used in Switzerland.

Mineral aggregates representing the Swiss petrographic types and the Swiss reference mixture which consists of 5 typical Swiss aggregate types (Table 2) are used. Aggregates have been sieved to 8/11.2 mm, washed and dried before mixing with bitumen.

Table 2: List of mineral aggregates

Sample name	Origin	Petrography
Mineral M1	Ambrosini (Lodrino TI)	Granitic gneiss
Mineral M2	St-Léonard (VS)	Quarzite
Mineral M3	Gasperini (Attinghausen UR)	Alpine limestone
Mineral M4	Balmholz (Sundlauenen BE)	Alpine limestone
Mineral M5	Comibit (Sigirino TI)	Limestone/Dolomite
Mineral M6	Aebisholz (Oensingen SO)	Alluvium
Mineral M7	Reference mixture of 5 aggregates with different petrography	
Mineral M8	Weiach (ZH)	Alluvium
Mineral M9	Oldis (Haldenstein GR)	Alluvium
Mineral M10	Bartenheim (F)	Alluvium

2.2 Evaluation of the static test according EN 12697-11 part B

Switzerland used for many years a static water immersion test with success. Because the method described in EN 12697-11 part B resembles the Swiss method, a preliminary study was carried out using the original European method part B to see whether this method could replace the Swiss national standard. The pre-study was performed on five bituminous binders including two PmBs with the reference mixture mineral M7. For the bitumen 70/100, the adhesion was tested additionally with the minerals M1 and M4.

The test was not successful for the polymer modified binders and the hard bitumen 10/20, because complete coating was not possible and in some cases the coated particles did stick together and formed lumps (Table 3). The assessment of the coating efficiency, which is done particle by particle, is not feasible with particles sticking together. In the case of incomplete coverage, the European Standard suggests to increase the amount of binder in steps of 0.5% (w/w). This approach might work for unmodified bitumen to a certain degree, but fails in the case of “sticky” polymer bitumens with high viscosity.

In part B of the EN the mixing temperature of the bituminous binder and aggregates is 130°C and constant for all binders irrespective of the binder viscosity. This temperature is too low for hard binders and higher modified PmB. However, this inadequacy is only present in part B. In part A and C the mixing temperature is depending on the viscosity and is chosen among the reference temperatures T_R for the production of hot mixes according to EN 12697-35 [8]. Theoretically, the equiviscous temperature is the correct solution but for practical reasons hardly feasible. The mixing temperature as function of the softening point ring and ball or the mixing temperature for hot mixes is less precise but is regarded as an acceptable compromise. Trying to avoid the lump forming, the adhesion tests were repeated using the reference mixing temperature T_R for hot mixes, which is equivalent to the mixing temperature defined in the method of part C.

With this change of the coating procedure each particle is completely covered and separated from adjacent particles after the water conditioning step. However, with the exception of the softest binder, all tests lead to a complete coverage of the particles, even for the two mineral types M1 and M4 with very different petrography (Table 3). The reason for this outcome is the low water conditioning temperature of 19°C. At this temperature the debonding of bitumen is very slow for binders with medium to high viscosity.

Table 3: Results of the preliminary study using the original method of part B

Binder type	Aggregate type	Number of not completely coated particles	
		Mixing temperature 130 °C	Mixing temperature T_R = 135 … 180 °C according EN 12697-35
Bitumen 70/100	Mineral M7	**12**	**0**
Bitumen 70/100	Mineral M4	**8**	**0**
Bitumen 70/100	Mineral M1	**7**	**0**
Bitumen 10/20	Mineral M7	Lumps	**0**
Bitumen 160/220	Mineral M7	**25**	**5**
PmB 10/30-70	Mineral M7	Lumps	**0**
PmB 70/100-65	Mineral M7	Lumps	**0**

The results of the pre-study pointed out three main shortcomings of EN 12697-11 part B:

- invariable mixing temperature
- low water conditioning temperature
- insensitive evaluation of the result (pass / fail type)

The first two parameters were studied in more detail and are discussed in the second part of this paper.

2.3 Evaluation of some modifications of the method part B

For the assessment of the results the calculation and expression of results according to EN 12697-11 part A was used in addition with a graphical help showing the different coating degrees of a particle (Figure 1). This graphical help was developed in a previous national research project and proved to be very helpful for the estimation of the coating degree of a single particle [12] The coating coefficient is calculated as the mean value of the coating degree of all particles.

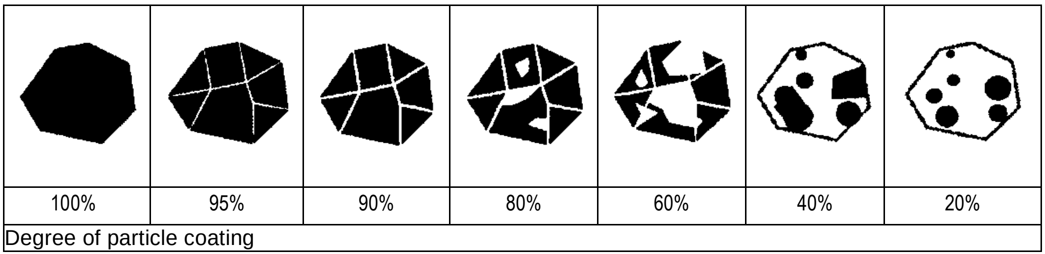

Figure 1: Graphical help for the assessment of the degree of coating

The results of the pre-study showed clearly, that a water conditioning temperature of 19°C as described in the static method B causes no bitumen debonding from the aggregate for the bitumen investigated. If a coating coefficient is asked as final result, the thermal stress has to be increased to get a better differentiation of the binder and mineral types. This raises the question of the right water conditioning temperature, which was addressed in a first series of tests by changing the water conditioning temperature in a range between 19 and 60°C. Two non-modified binders with different stiffness were selected for the evaluation of the water conditioning temperature. The mixing temperature was set to 150°C for the bitumen 50/70 and to 145°C for bitumen 70/100 in accordance to the reference temperature T_R for the production of hot mixes as defined in EN 12697-35 [8].

Table 4: Mixing temperature of binders according EN 12697-35 [8]

Binder type	Temperature of softening point ring and ball [10,11] [°C]	Mixing temperature T_R (according to EN 12697-35) [°C]	Mixing temperature = T_R - 15°C [°C]
10/20	58 - 78	according producer (ca. 180-190)	
20/30	55 - 63	180	165
35/50	50 - 58	165	150
50/70	46 – 54	150	135
70/100	43 – 51	145	130
100/150	39 – 47	140	125
160/220	35 – 43	135	120

Table 5: Mixing temperature of modified binders as chosen by the authors

PmB type	Temperature of softening point ring and ball [°C]	Mixing temperature T according to producer [°C]	Mixing temperature T - 15°C [°C]
10/30-70	71	180	
70/100-65	65	180	
E 25/55-65	64	160	150
E 65/105-60	75	150	135
C 25/55-55	59	155	130
C 45/80-50	56	145	125

Figure 2 shows the coating degree of the two binders as a function of conditioning temperature and conditioning time. With increasing conditioning temperature, the coating degree declines and the difference between the two binders grows. At 50°C a good differentiation is obtained, but this temperature is above the softening point ring and ball of the binder. At this temperature the binder is already fluid, which doesn't reflect the real condition in a pavement in middle Europe.

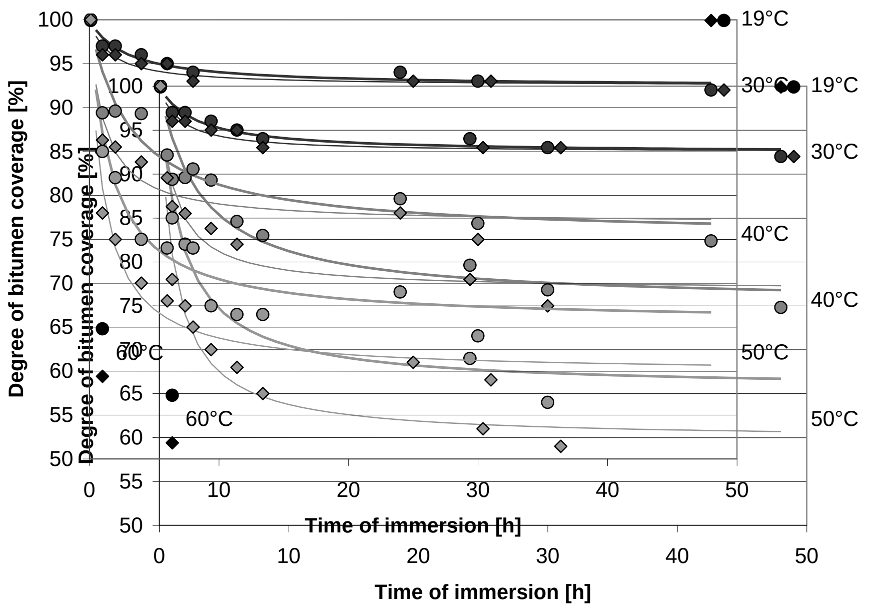

Figure 2: Bitumen coverage as a function of water conditioning temperature and time of immersion; bitumen 50/70: circles, bitumen 70/100: diamonds.

A conditioning temperature which is below the softening point ring and ball for all binders used for surface layers in Switzerland must be equal or below 40°C. As a good compromise a conditioning temperature of 40°C is suggested. For 90% of the binders used in Switzerland the softening point ring and ball is below 40°C. Furthermore, it is a surface temperature which may last for some time after rain has cooled down the pavement and therefore is more realistic than 50°C. For hotter climates a higher conditioning temperature might be useful, as the pavement temperature is higher.

The mixing temperature is different for all methods of the European standard. Two of them are referring to the reference temperature T_R (Table 4 and 5) of the hot mix production (in part C, $T = T_R$; in part A, $T = T_R + 25°C$). A mixing temperature of $T_R + 25°C$ results in temperatures close to 200°C for hard bitumens like bitumen 20/30 and harder. This is above the tolerable maximum temperature for the warming of bituminous binders, which is defined as the temperature of softening point ring and ball + 100°C [13]. This reveals a contradiction between the standard for bitumen sample preparation [13] and preparation of hot mixes [8].

In figure 2 the results for the two binders are very close except for the immersion temperature at 50°C. In order to increase the sensitivity of the test method it was decided to carry out additional tests at a mixing temperature of T_R-15°C (Table 4 and 5). The results illustrated in figure 3 demonstrate indeed a marked influence of the mixing temperature on bitumen coverage, the effect being more pronounced with the higher grade bitumen 70/100. The use of the higher mixing temperatures T_R results in a more effective adhesion between bitumen and aggregate and in a less perceptible difference between the paving grades. Lower mixing temperatures T_R-15 on the other hand result in a lower affinity and a clearer differentiation between different binder stiffnesses. Hence, a mixing temperature of T_R-15°C is suggested for Switzerland.

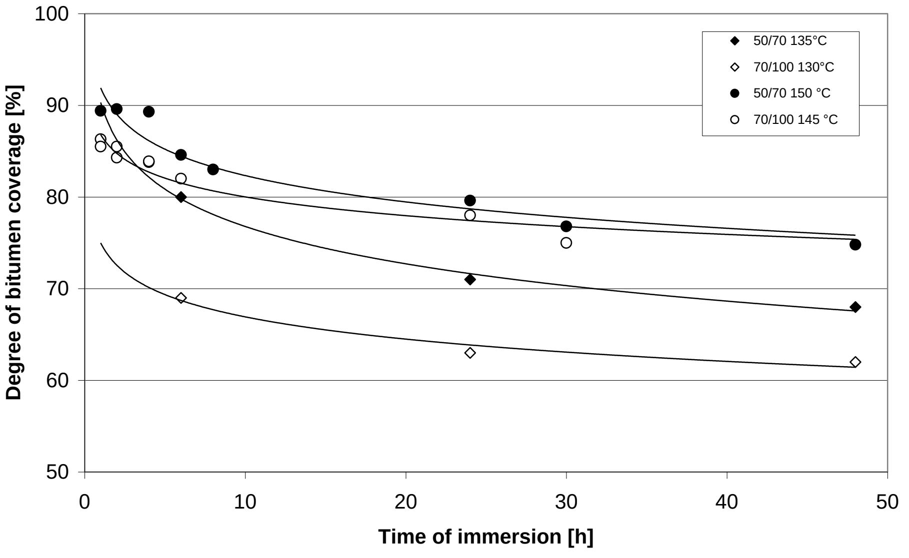

Figure 3: Bitumen coverage as a function of mixing temperature and time of immersion; bitumen 50/70 in black; bitumen 70/100 in white; water conditioning temperature 40 °C.

2.4 Validation of the improved method B with different binders and aggregates

The improved method B was validated with a set of different binders and aggregates. Different binder types showed a good differentiation in the case of non-modified binders. PmB resulted in almost complete coverage. It is not clear whether this can be attributed fully to the superior adhesion properties of polymer modified binders.

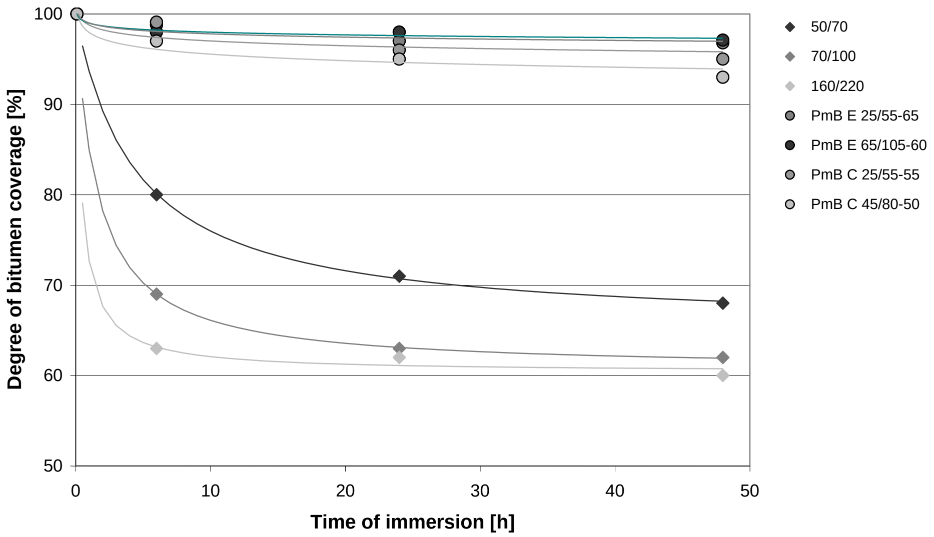

Figure 4: Bitumen coverage of different binders and the reference mixture M7.

As well as for the set of bitumens, the different aggregate types show a reasonable differentiation, although no adhesion coefficient lies below 50%. This observation in particular justifies the question whether the sensitivity of the test is sufficient. The former Swiss Standard [2] used a water conditioning temperature of 60°C and a cooling time of 5 minutes which resulted in adhesion coefficients below 30% in some cases. Accordingly, when the coated aggregates first come into contact with water, their temperature is much higher compared to the EN procedure and the early stripping is enhanced. The lower conditioning temperature together with the 60 minutes cooling time of the European standard may be the explanation for the rather high degrees of bitumen coverage which are observed in figure 5.

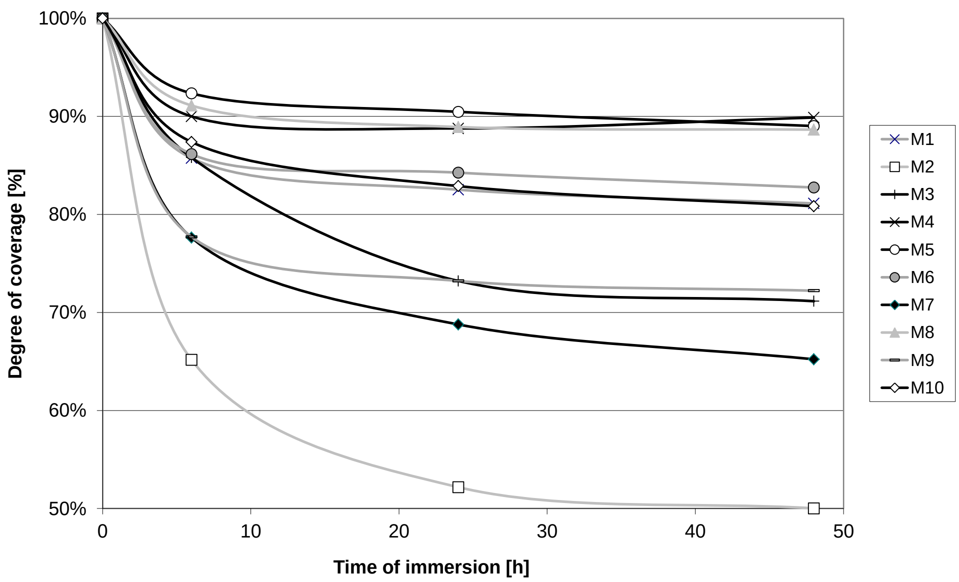

Figure 5: Bitumen coverage of different aggregate types and bitumen 70/100.

3. CONCLUSIONS

The static method for the assessment of the adhesion between binder and aggregate as defined in EN 12679-11 can be improved by following changes:

- Definition of the mixing temperature as a function of binder stiffness or actually binder viscosity. For practical reason, the binder viscosity is not measured directly, but the reference temperature T_R for hot mix production is used. For better differentiation of the results a mixing temperature of T_R - 15°C is suggested.
- Increasing the water conditioning temperature to 40°C instead of 19°C. This could be different depending on the climate.
- Expressing the result as a coating coefficient like in part A of the standard and adopting the use of a graphical aid.

With these improvements the adhesion test is a good tool to evaluate different binders or aggregates with different petrography. However, additional aggregates not found in Switzerland should be evaluated as well, for example in a European round robin test. The outcome of the research project has a direct influence on the current revision of the EN 12697-11:2005.

4. ACKNOWLEDGEMENT

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