Water susceptibility of asphalt mixtures: Testing the effect of adhesion agents on different binder/aggregate mixtures

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ABSTRACT: One of the objectives of the Norwegian PROKAS project 1998-2004 was better durability of asphalt pavements. In a laboratory study of water susceptibility of asphalt mixtures, tests were conducted on asphalt mixtures containing three common aggregates and bitumen 160/220. The aim of the study was to: a) compare three different test methods b) study the effects of different additives (liquid amine, hydrated lime, limestone and combinations of these). The test methods were:

1) The "*Cantabro test*" after water conditioning of the asphalt specimen as described in prEN 12697-12.

2) "The Rolling bottle test" on the aggregate fraction 5.6/8.0 mm.

3) The Swedish *"Shaking abrasion test"*, a modified version of the prEN 12274-7. Hot mixed asphalt specimens with maximum aggregate size 2 mm were tested after water conditioning.

The three test methods showed reasonably good agreement on the tested materials, even though they may be considered as complementary methods. The tests results showed that the use of adhesion agent should be recommended for certain aggregate/bitumen mixtures. For one of the binder/aggregate combinations none of the used adhesion agents were effective. All of the three test methods are recommended for durability testing for production control and for research and development.

KEY WORDS: Asphalt test methods, water susceptibility, adhesion agents.

1 INTRODUCTION

In the Norwegian PROKAS project 1998-2004, the main objective was to develop or adopt improved systems for mix design and quality control of asphalt mixes and pavements. One of the activities in the PROKAS project was study of durability of asphalt mixtures.

By durability it is meant the permanence of the properties of the asphalt mixture during its functional lifetime, under the stresses from climate (air, water and temperature), traffic and road maintenance. In the rather cool and humid Norwegian climate, it is important to avoid stripping prone or water susceptible asphalt pavements. Quite soft bitumen grades (e.g. 160/220) are frequently used due to the need for flexible pavements. Most of the current tests methods used for the assessment of water susceptibility are considered insufficient. Improved

test methods both for the assessment of raw materials (aggregates, binders and additives) and of asphalt mixtures are welcomed. The test methods should be affordable and give reliable test results.

The aim of this study is to compare three test methods for water susceptibility that may give complementary information (Jørgensen, 2004). Three aggregates with and without adhesion agents were tested. The experimental works were carried out in the laboratories of NCC Roads in Lier and Skanska Asfalt in Bærum.

2 TEST METHODS

The Indirect Tensile Strength Ratio (ITSR) after water conditioning, the European standard EN 12696-12, is a reference method for bituminous mixtures (CEN, 2003 a). Our experiences with the ITSR were not always good. In this study we preferred to use the Cantabro test, EN 12696-17 (CEN, 2004a), on water conditioned asphalt concrete specimens. The rolling bottle Test, EN 12696-11 (CEN, 2003b), was used on the coarse aggregate and bitumen/additive combinations. The Swedish Shaking abrasion test, based on the drafted prEN 12274-7 (CEN 2004b), was used on fine graded asphalt mixtures.

2.1 The Rolling Bottle Test

The Rolling Bottle Test (RBT) is used to evaluate the adhesion of aggregates and binders for hot asphalt mixtures (Jørgensen, 2002). Effect and dosage of adhesion agents, including fillers can be evaluated with this method.

The coarse fraction of the aggregates is tested (8/11 mm or 5.6/8.0 mm). The aggregates and bitumen with/without additives are mixed at 150 °C. After storage at room temperature overnight, three part samples of 150 g are transferred to 250 mL bottles with a fixed glass rod inside, filled with cold distilled water and closed with a screw cap. The bottles are rotated at 60 rpm, and the degrees of binder coverage of the chippings are estimated after 6 h, 24 h, 48 h and 72 h rolling time. The average binder coverage of three bottles is reported.

2.2 The Swedish Shaking Abrasion Test

The shaking abrasion test was developed in Sweden for durability testing of the fine aggregates in hot mixed asphalt specimens (Ulmgren, 2004). The apparatus and procedures follow by and large prEN 12274-7.

A fine aggregate 0/2 mm curve is composed and heated to 150 °C before mixing with bitumen (binder content 7 %). The asphalt mixture is compacted in a preheated mould by a static press. The cylindrical test specimens have a diameter of 30 mm and a height of 27 mm. After cooling and storage of the test specimens at room temperature for 5-14 days, the dimensions and air voids are determined. Normally air voids in the range of 15-20 % are obtained.

Three test specimens are selected for the water susceptibility test. They are conditioned in water according to EN 12697-12, but only for two days at 40 °C. The test specimens are transferred to a water bath at 25 °C for 30 min before determining their dimensions and mass. Each test specimens is placed in a shaking cylinder filled with 750 mL of water. The cylinders are closed with a cap and mounted in a mechanical shaker (figure 1). The cylinders are rotated 20 rpm for 3 hours (3600 revolutions). The abrasion in percent loss of mass on each of the test specimens is determined, and the average is reported.



Figure 1: Mechanical shaker for the shaking abrasion test.

2.3 The Cantabro Test

The Cantabro test was developed in Spain for the testing of porous asphalt and cold asphalt mixtures (Pérez et al. 1989). It is frequently used in Europe for testing of porous asphalt.

In this study the Cantabro test is used on water conditioned asphalt specimens. The water conditioning was done in accordance with EN 12697-12. The asphalt samples were mixed at 135 °C and compacted with 2×25 blows. The target air void content was 8 %. After vacuum saturation (residual pressure 6.7 kPa) and water storage for three days at 30 °C, the test specimens were allowed to bench dry for 30 minutes. Each specimen was weighed and placed in the Los-Angeles-machine, and run for 300 turns. The test temperature was 22 ± 2 °C. After the test, the test specimen was weighed and the particle loss in percent calculated. The average of four determinations is reported.

3 MATERIALS AND METHODOLOGY

3.1 Materials

A Venezuelan crude bitumen 160/220, was used in the study, see test data given in table 1.

Table 1: Bitumen test data

Method	Unit	160/220	160/220
		original	after RTFOT
Penetration at 25 °C	mm/10	177	114
Viscosity at 60 °C	Pa·s	65	124
Softening point	°C	36,9	41,3
Acid value	mgKOH/g	3,8	

An overview of the aggregates used in the study is given in table 2. The aggregate material was crushed rock, and the fractions 0/4 mm and 4/11 mm were used. Mechanical characteristics of the aggregates are given in table 3.

Table 2: Mineralogy of the aggregates

Code	Aggregate	Mineralogy of fraction 4/11 mm	Comments
Li	Lierskogen	Hornfels (fine grained), less than 1 % limestone and granitic material, traces of pyrite. The 0/2 mm fraction was mainly hornfels, but enriched in calcite, fine grained silica and feldspar.	Need adhesion agent.
		Mica contents of 125/250 µm fraction: 1 %	
St	Steinskogen	Basalt (fine grained). Some dust on the surfaces of the coarse particles, mainly epidot.	Good adhesion expected
		Mica contents of 125/250 µm fraction: 0 %	
Sv	Svingen	Fine to medium grained pink granite. Granodioritic and tonalitic gneiss. Micas (biotite, some chlorite, some muscovite)	Need adhesion agent
		Mica contents of 125/250 μm fraction: 25 %	

Table 3: Mechanical properties of the aggregates

Code	Aggregate	Los-Angeles value	Nordic abrasion value	Density g/cm ³	Rigden void of material < 125 μm, %
Li	Lierskogen	9	4-5	2,79	43
St	Steinskogen	10	7-9	2,90	43
Sv	Svingen	16	10	2,71	44
LF	Limestone filler			2,7	32
HL	Hydrated lime			2,4	62

The adhesion agents used were:

- Liquid amine, derived from amidoamines (dosage in % of the binder).

- Hydrated lime used as a mineral adhesion agent (dosage in % of the total aggregate).

Limestone filler (dosage in % of the total aggregate) is not considered an adhesion agent, but may nevertheless improve the durability properties.

3.2 Test plan

Table 4 shows the different material combinations and coding of the test samples. For the Cantabro test, asphalt concrete mixtures (AC 11) with maximum particle size of 11 mm and

binder content of 6.0 % was prepared. When limestone filler or hydrated lime was used, the same amount of aggregate filler was taken out of the mixture.

Table 4: Material combinations used in the Rolling bottle test, the Shaking abrasion test and the Cantabro test

	Aggrega	te filler (AF)	Limeston	e filler (LF)	Hydrated lime (HL)		
Aggregate	1.0 % AF	0.5 % AF 0.25 % amine	1.0 % LF	0.5 % LF 0.25 % amine	1.0 % HL	0.5 % HL 0.25 % amine	
Lierskogen	Li-1	Li-2	Li-3	Li-4	Li-5	Li-6	
Steinskogen	St-1	St-2	St-3	St-4	St-5	St-6	
Svingen	Sv-1	Sv-2	Sv-3	Sv-4	Sv-5	Sv-6	

In addition, mixtures of the three aggregates (without filler) and bitumen without and with 0.4 % amine were tested with the rolling bottle test.

4 **TEST RESULTS**

4.1 Rolling Bottle Test

The results from the RBT are given in table 5 and figure 2. Mixtures without adhesion agent show rather poor results, while combinations with adhesion agent show improved binder coverage.

Table 5: Results of the rolling bottle test for b itumen 160/220 with different additives.

	Per cent binder coverage								
Aggregate	L	ierskog	en	Steinskogen			Svingen		
Rolling time	24 h	48 h	72 h	24 h	48 h	72 h	24 h	48 h	72 h
No additive	37	16	8	47	21	13	18	2	0
0.4 % amine	64	48	35	75	53	38	71	46	28
1.0 % LF *	34	17	12	62	36	29	47	17	10
0.25 % amine, 0.,5 % LF	62	46	33	78	63	52	80	48	45
1.0 % HL *	65	52	40	89	77	58	88	75	60
0.25 % amine, 0.5 % HL	88	65	65	88	78	55	93	67	48
0.4 % amine, 1.0 % HL	75	65	44	88	70	55	88	77	44
1 % AF *	28	20	9	48	32	23	21	7	0
0.25 % amine, 0.5 % AF	58	43	37	73	60	47	75	48	40

* LF = Limestone filler HF = Hydrated lime AF = Aggregate filler



Figure 2: Results from the rolling bottle test. Bitumen 160/220 with different additives (LF = Limestone filler, HF = Hydrated lime, AF = Aggregate filler).

4.2 Shaking abrasion test

Results from the shaking abrasion test are given in table 6 and figure 3. Mixtures of 0/2 mm limestone and bitumen were also tested.

Table 6: Results from the shaking abrasion	test (LF = Limestone filler, HF = Hydrated lime).
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		Density	Air void	Swelling	Abrasi	on, %
Aggregate	Diagram	g/cm ³	%	%	Average	Std. dev.
Lierskogen no additive	Li-1	2.014	19.3	4.7	81.1	0,86
Lierskogen + 0.4 % amine	Li-2	2.013	19.4	5.1	66.7	0,94
Lierskogen + 1.0 % LF	Li-3	2.033	18.6	6.0	76.3	0,98
Lierskogen + 0.25 % amine, 0.5 % LF	Li-4	2.013	19.4	5.9	74.0	1,70
Lierskogen +1.0 % HL	Li-5	2.014	19.3	4.2	43.0	0,49
Lierskogen + 0.25 % amine, 0.5 % HL	Li-6	2.006	19.6	4.6	47.2	3,95
Steinskogen no additive	St-1	2.111	18.5	2.6	20.8	0,20
Steinskogen + 0.4 % amine	St-2	2.092	19.1	2.3	12.0	0,51
Steinskogen + 1.0 % LF	St-3	2.103	18.8	2.1	19.2	0,72
Steinskogen + 0.25 % amine, 0.5 % LF	St-4	2.084	19.5	2.0	15.0	0,50
Steinskogen +1.0 % HL	St-5	2.062	20.4	2.9	9.7	1,37
Steinskogen + 0.25 % amine, 0.5 % HL	St-6	2.077	19.8	1.6	12.2	0,35
Svingen no additive	Sv-1	1.962	19.2	3.2	44.3	0,59
Svingen + 0.4 % amine	Sv-2	1.969	18.9	2.1	13.7	0,24
Svingen + 1.0 % LF	Sv-3	1.973	18.8	3.3	32.3	0,48
Svingen + 0.25 % amine, 0.5 % LF	Sv-4	1.964	19.2	1.3	24.3	1,00
Svingen +1.0 % HL	Sv-5	1.957	19.5	1.9	10.6	0,88
Svingen + 0.25 % amine, 0.5 % HL	Sv-6	1.984	18.3	0.81	11.5	0,55
LF no additive	LF-1	2.194	9.3	2.9	2.0	0,13
LF + 0.4 % amine	LF-2	2.199	9.2	3.1	1.7	0,19



Figure 3: Results from the shaking abrasion test, given as averages of three test specimens.

Note that the air voids of the two mixtures with limestone were only 9 %. The air voids of the mixtures with the other aggregates were in the range of 15-20 %. The test has no requirement for the degree of swelling. It can be noted that the samples with highest degree of swelling (Lierskogen) also had the highest abrasion values.

4.3 Cantabro Test

Results of the Cantabro test are shown in table 7 and figure 4. The air voids varied from 7.4 % to 11.3 %. The target air void was 8 %. The degree of swelling varied from 0.7 to 1.7 %, which is within the maximum limit of 2.0 %.

		Density	Air void	Swelling	Particle	loss, %
Aggregate *	Diagram	g/cm ³	%	%	Average	Std. dev.
Lierskogen no additive	Li-1	2,312	8,9	1,4	53	9,2
Lierskogen + 0.4 % amine	Li-2	2,306	9,2	1,4	28	1,9
Lierskogen + 1.0 % LF	Li-3	2,306	9,2	1,7	48	7,7
Lierskogen + 0.25 % amine, 0.5 % LF	Li-4	2,308	9,1	1,4	34	4,8
Lierskogen +1.0 % HL	Li-5	2,281	10,2	1,5	34	2,7
Lierskogen + 0.25 % amine, 0.5 % HL	Li-6	2,294	9,6	0,92	38	5,7
Steinskogen no additive	St-1	2,355	10,6	1,2	26	5,0
Steinskogen + 0.4 % amine	St-2	2,348	10,9	1,3	14	2,9
Steinskogen + 1.0 % LF	St-3	2,348	10,9	1,5	31	3,1
Steinskogen + 0.25 % amine, 0.5 % LF	St-4	2,349	10,8	1,0	17	1,1
Steinskogen +1.0 % HL	St-5	2,335	11,3	1,0	25	1,9
Steinskogen + 0.25 % amine, 0.5 % HL	St-6	2,243	11,1	1,2	16	4,2
Svingen no additive	Sv-1	2,266	8,3	1,4	28	3,1
Svingen + 0.4 % amine	Sv-2	2,277	7,8	1,3	12	2,0
Svingen + 1.0 % LF	Sv-3	2,278	7,8	1,3	27	4,4
Svingen + 0.25 % amine, 0.5 % LF	Sv-4	2,285	7,5	1,3	12	2,1
Svingen +1.0 % HL	Sv-5	2,265	8,3	0,71	15	3,0
Svingen + 0.25 % amine, 0.5 % HL	Sv-6	2,288	7,4	0,78	13	0,4

Table 7: Results from the Cantabro test on different mixtures of AC 11.

* LF = Limestone filler

HF = Hydrated lime





5 DISCUSSION

It is of interest to rank the test results and compare them to limits for acceptable water susceptibility. In table 8 the different material combinations are ranked in decreasing order with respect to their water susceptibility based on the results of the tests. Material combinations not fulfilling the requirements are given on shaded background. RBT samples without filler are coded Li-/St-/Sv-01 (no additive) and Li-/St-/Sv-02 (0.4 % amine). Tentative limits for acceptable test results are:

- Rolling bottle test, binder coverage after 72 h: min. 15 %
- Shaking abrasion test, loss of mass: max. 15 %
- Cantabro test, particle loss: max. 25 %

Rolling bottle test, 72 h *			Shakir	ng abrasion	ı test	Cantabro test					
Lierskogen	Stein- skogen	Svingen	Lierskogen	Stein- skogen	Svingen	Lierskogen	Stein- skogen	Svingen			
Li-6	St-5	Sv-5	Li-5	St-5	Sv-5	Li-2	St-2	Sv-2			
Li-5	St-6	Sv-6	Li-6	St-2	Sv-6	Li-4	St-6	Sv-4			
Li-2	St-4	Sv-4	Li-2	St-6	Sv-2	Li-5	St-4	Sv-6			
Li-02	St-2	Sv-2	Li-4	St-4	Sv-4	Li-6	St-5	Sv-5			
Li-4	St-02	Sv-02	Li-3	St-3	Sv-3	Li-3	St-1	Sv-3			
Li-3	St-3	Sv-3	Li-1	St-1	Sv-1	Li-1	St-3	Sv-1			
Li-1	St-1	Sv-1									
Li-01	St-01	Sv-01									

Table 8: Ranking of the materials based on the three test methods in decreasing order. Shaded areas indicate unsatisfactory water susceptibility.

* Samples without filler are coded Li-/St-/Sv-01 (no additive) and Li-/St-/Sv-02 (0.4 % amine).

The results show that limestone filler (1 %) didn't work as an adhesion agent with any of the test methods. Hydrated lime or hydrated lime/amine gave the best results in general, with the exception of the Cantabro test on the mixtures of Steinskogen and Svingen, where amine gave the best result.

The aggregate Lierskogen could fulfill the requirements for neither the shaking abrasion test nor the Cantabro test. The batch of fine aggregate from Lierskogen was considered inadequate. Other batches from the Lierskogen quarry tested with the shaking abrasion test show acceptable abrasion values. This discrepancy is yet to be clarified, but may be due to different mineralogy of the fines, different surface activity, different particle shape, etc.

There was a reasonable overall correlation between the shaking abrasion test and the Cantabro test ($R^2 = 0.711$). The two methods test similar asphalt mortars and the test specimens are conditioned in a similar way. There were a poor overall correlation between the RBT and the shaking abrasion test ($R^2 = 0.239$) or the RBT and the Cantabro test ($R^2 = 0.254$). The differences in sample constitution and conditioning practice imply that complementary properties are tested.

6 CONCLUSIONS

The three test methods may be suitable for routine testing (e.g. production control), but would also be useful for studies of any asphalt material/additive combination.

The study shows that there can be differences in mineralogy and adhesive properties between the coarse and fine fraction of the same aggregate. An adhesion problem can be caused by the coarse aggregate, the fines or both.

In the RBT, limestone was not sufficient to prevent the coarse fraction from becoming prone to stripping. Both amine and hydrated lime were effective to avoid stripping. The combination of amine and hydrated lime was most effective.

In the shaking abrasion test both amine and hydrated lime reduced the abrasion value. Mixtures with hydrated lime had the lowest abrasion. One of the aggregates (Lierskogen) had very poor abrasion values for all combinations.

In the Cantabro test the adhesion agents were also effective. Amine gave somewhat better results than hydrated lime.

An important observation is that ad hesion properties must always be judged with the actual constituents of the asphalt mixture. Different types (crude oils) of bitumen or different batches of aggregate can give different properties in the mixture. Moreover, differences in production and laying conditions of the asphalt mixture, together with differences in climate and traffic, will influence the durability of the asphalt. All of the three test methods are recommended for testing of water susceptibility. The Cantabro test could be an alternative to the ITSR method. The shaking abrasion test and the Cantabro test need validation based on field experience, and hopefully more experience will be gained in the coming years.

REFERENCES

- CEN, 2003a. EN 12697 Bituminous mixtures Test methods for hot mix asphalt Part 12: Determination of the water sensitivity of bituminous specimens. European standard, CEN, Brussels, Belgium.
- CEN, 2003b. EN 12697 Bituminous mixtures Test methods for hot mix asphalt Part 11: Determination of the affinity between aggregate and bitumen. European standard, CEN, Brussels, Belgium.
- CEN, 2004a. EN 12697 Bituminous mixtures Test methods for hot mix asphalt Part 17: Particle loss of porous asphalt specimen. European standard, CEN, Brussels, Belgium
- CEN, 2004b. *prEN 12274-7 Slurry surfacing Part 7: Shaking abrasion test*. Draft European standard, CEN, Brussels, Belgium
- Jørgensen, T., 2002. *Testing adhesion between bitumen and aggregate with the Rolling Bottle Test and the Boiling Test*. Proceedings of the Sixth International Conference on the Bearing Capacity of Roads, Railways, and Airfields, Lisbon, Portugal.
- Jørgensen, T., 2004. *PROKAS Project report no. 14: Testing water susceptibility of asphalt mixtures using the Cantabro Test, Shaking Abrasion Test and Rolling Bottle Test* (in Norwegian). Norwegian Public Roads Administration, Technology Department, internal report no. 2367.
- Peréz, J. et al., 1989. New Procedure for filler characterization based on the effect of filler properties on aggregate-binder adhesiveness. 4th Eurobitume Congress, Madrid.
- Ulmgren, N., 2004. *The influence of the mastic on the durability of asphalt pavements as studied by the shaking abrasion test.* 3rd Euroasphalt and Eurobitume Congress, Vienna.