ARE THE EUROPEAN STANDARDS prEN 12697-11 AND -12 ABLE TO LIMIT THE AMOUNT OF FLINTS IN ASPHALTIC MIXTURES?

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ABSTRACT

High amounts of flint in Dutch asphalt concrete mixes leads to poor adhesion between aggregate and bitumen. The Netherlands is largely depended on aggregate that contains flint. For this reason the amount of flint in Dutch mixes is limited to a maximum of 15%. However in the European asphalt concrete drafts (in the prEN 13108-serie) this restriction is not allowed anymore. For this reason, the CROW-working group CIENA carried out research to tackle this problem. In this paper the results of this test program is reported.
In this paper a correlation is made between the amount of flints and the European test standards 'Affinity between aggregate and binder' (prEN12697-11) and 'Water sensitivity' (prEN12697-12). In this study the influence of the amount of flints in asphalt concrete mixes is tested.
In this research in some cases relations have been found between the amount of flints and the results obtained by using the two mentioned European Standards. However, in most cases this correlation is not clear. It is concluded that for a limited number of cases only one test method is able to give the same result as observed in the road. Therefore, much work has to be done to have a relevant test method to predict the poor adhesion of flint in aggregate.

Keywords: adhesion, aggregate, ravelling, standardisation

1. INTRODUCTION

In the Dutch regulations for road materials it stated the amount of flints used in asphalt concrete should be lower than 15 %. This because in the past poor adhesion between aggregates and bitumen was found in mixes with a high amount of flints. The Netherlands is largely depended on aggregate that contains flint.
Using the new European asphalt concrete standards it will not be possible anymore to restrict the flint amount. The use of flint in asphalt mixes can only be limited by making demands with respect to functional European test methods like prEN 12697-11 'Aggregate and binder affinity' and prEN 12697-12 'Water sensitivity'.
With respect to the need for a good adhesion test, research is carried out in the Netherlands within the CROW working group 'CIENA'. In this study both test methods (prEN 12697-11 and prEN 12697-12) are used. In this paper attention will be paid to the results of this test program.

2. LABORATORY TESTS

2.1 Stripping tests

The following candidate test methods are used:

A. prEN 12697-11: Determination of the affinity between aggregate and bitumen [1]; Part B: Rolling Bottle Method (October 2001).
This European Standard describes procedures for the determination of the affinity between aggregate and bitumen by using a rolling bottle. The bitumen-coated aggregate is placed in a bottle filled with water. The bottle is sealed and placed on a rolling device. The bottles are rolled at ambient temperature for a total time of 6 h at a speed of 40 $^{-1}$ min. At several intervals the degree of bitumen coverage on the aggregate particles is visually estimated;

B. prEN 12697-11: Determination of the affinity between aggregate and bitumen [1]; Part D: Boiling Water Stripping Method.

Bitumen-coated aggregate is placed in boiling water during 10 min. After contact of acid with the uncovered aggregate, the consumption of which is proportional to the uncoated surface, the degree of bitumen coverage is determined with reference to a calibration curve;

C. prEN 12697-12: Determination of the water sensitivity of bituminous specimens [2] (June 2001).
A set of cylindrical specimens is divided into two subsets. One subset is maintained dry at room temperature while the other set is saturated and stored in water at a temperature of 40 °C for a period of 68 hours. After conditioning, the indirect tensile strength of the two subsets is determined in accordance with prEN 12697-23. The indirect tensile strength ratio of the two subsets, which is a direct number for the water sensitivity, is determined by dividing both strengths;

D. P-DWW-98055.
A Dutch (Road and Hydraulic Division) test method for the water sensitivity is used as a reference. This method is more or less identical to prEN 12697-12. However, asphalt specimens are saturated and stored in water at a temperature of 60 °C for a period of 48 hours.

2.2 Strength test

A. Determination of the indirect tensile strength of bituminous specimens according to prEN 12697-23 (June 2001) [3].
According to this European test cylindrical specimens (cores) are tested at a test temperature. The specimens are placed in the compressive testing machine and loaded diametrically along the direction of the cylinder axis with a constant speed of 50 mm/min until it fails.

2.3 Test Set-up

A. Stripping method according prEN 12697-11; Part B; Rolling Bottle method [4].
Mixes with pure Dutch river gravel, and mixes with 15, 30 and 100 % sea gravel containing 81% flints were made. After covering the aggregate with bitumen, the specimens were tested according to prEN 12697-11, Part B. After 6, 24, 48, 72, and 144 hours the degree of bitumen coverage on the aggregate particles was visually estimated;

B. Stripping method according prEN 12697-11; Part D; Boiling Water Method [5,6].
Mixes with pure Dutch river gravel, and mixes with 15, 30 and 100 % sea gravel containing 81% flints were prepared. Because of expected different adhesion performances quarry material (Quenast from Belgium) and Venezuelan and Arabic bitumen were used. After coating the aggregate with bitumen stripping with boiling water is applied according to prEN 12697-11, Part D;

C. Water sensitivity and stripping method according prEN 12697-12 [4]
Three asphalt concrete mixes were prepared with 0 %, 15 % and 30 % sea gravel containing 81% flints.
In the case of prEN 12697-12, retaining conditions of 40 °C during 68 hours are used and before and after stripping the indirect tensile strength of the specimens were measured according to prEN 12697-23 at 25°C;

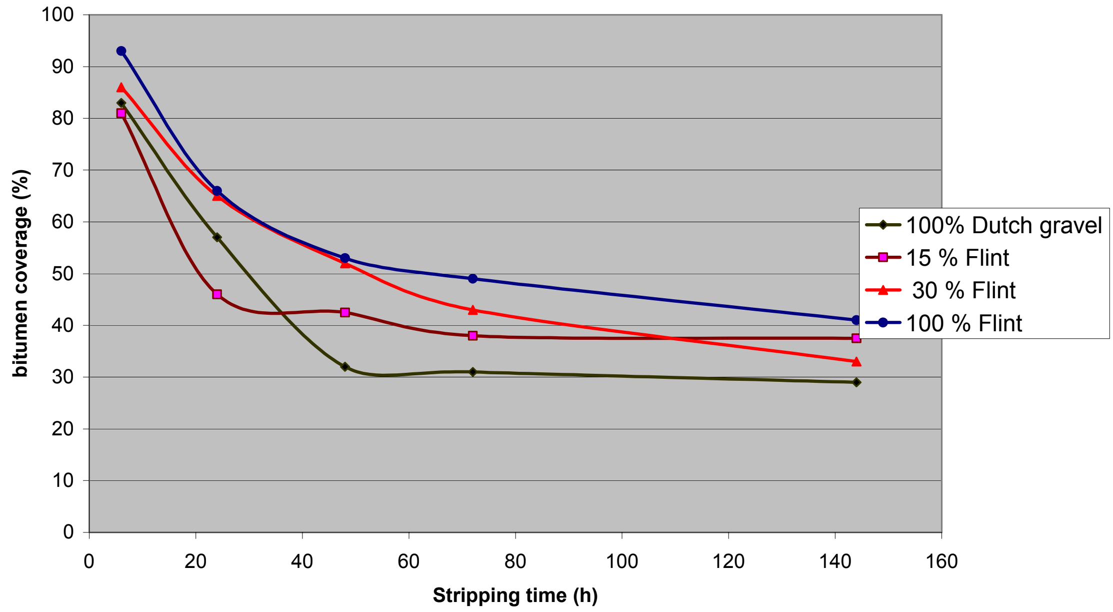
D. In the Dutch procedure a retain procedure of 48 hours at 60°C is applied. Porous asphalt specimens were tested at 0°C; the other specimens at 5 and 25°C [4].

3. RESULTS AND DISCUSSION

3.1 Stripping method according prEN 12697-11; Part B; Rolling Bottle method.

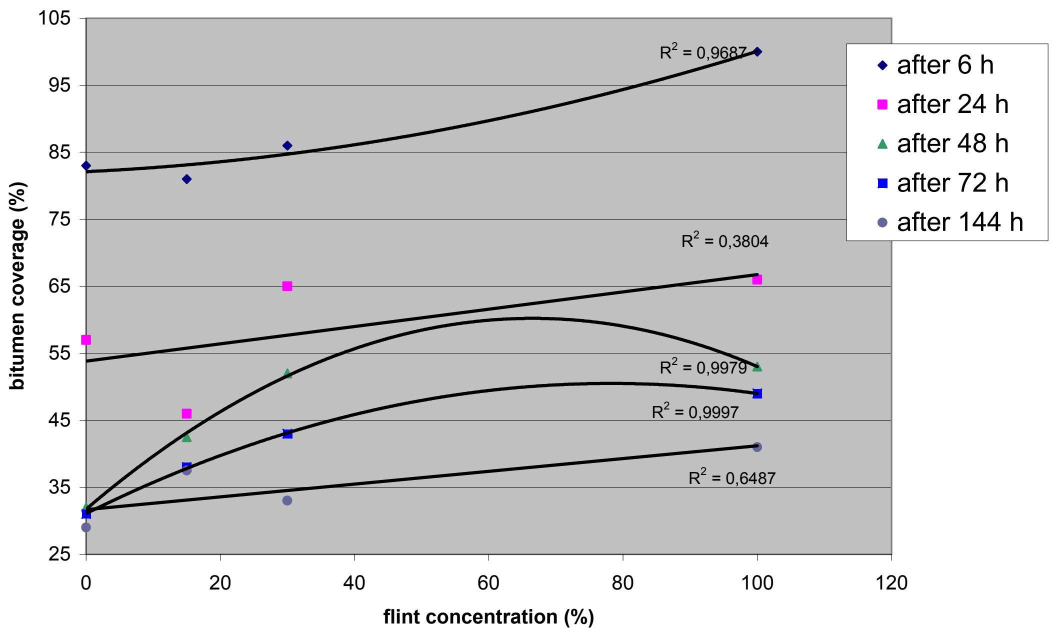
From figure 1 it is clear that after being exposed to the stripping test, the most covered aggregate is found for the specimen with the highest amount of flint. This is in contradiction with the observation on the road, where the higher flint amounts results in poor adhesion.
Moreover, the asphalt mixture which contains no flint at all (Dutch river gravel) is already stripped of most of the bitumen after 48 hours. The cause of this phenomenon is still not clear. The most reasonable explanation is that covered flint has the tendency of clogging together by which the accessibility is diminished. Also the formation of fines aggregate particles due to crushing during the rolling process is different for gravel and flint might play a role.

Figure 1. Coverage of aggregate (Dutch gravel/flint) with bitumen after stripping according prEN 12697-11; Part B



In figure 2 the coverage percentage is plotted against the flint concentration In this way it is possible to see whether there is a good relation between the flint amount and the stripping concentration and the duration of the stripping test. Here the same effect is seen as described above.

Figure 2. Coverage of aggregate mixtures (Dutch gravel/flint) from figure 1; flint concentration versus bitumen coverage



3.2 Stripping method according prEN 12697-11; Part D; Boiling Water method.

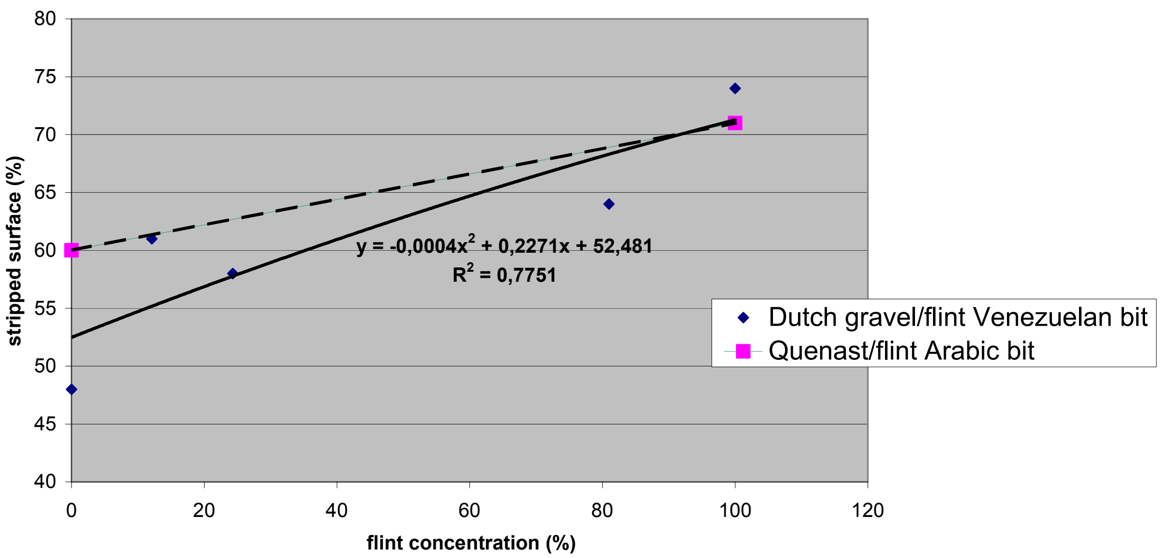
From table 1 and figure 3 it can be seen that there is a weak relation between the flint concentration and the amount of stripped surface. The stripping of mixes with 15 % flint is even higher than mixes, which contains 30% flint.

Number	Mixes	% stripped uncovered surface
1	100 % Dutch gravel. (Venezuelan bit.)	48 %
2	85 % Dutch gravel + 15 % flint (Venezuelan bit)	61 %
3	70 % Dutch gravel + 30 % flint (Venezuelan bit)	58 %
4	100 % Quenast aggregate (Venezuelan bit)	53 %
5	Sea gravel with 81 % flint (Venezuelan bit)	64 %
6	100 % flint from sea gravel (Venezuelan bit)	74 %
7	100 % Quenast aggregate (Arabic bit)	60 %
8	100 % flint from sea gravel (Arabic bit)	71 %

Table 1: Stripping results of aggregate/bitumen mixes according prEN 12697-12; Part D

Table 1 indicates also that Venezuelan bitumen adheres better to Dutch gravel than to Quenast aggregate. Also in this test the adhesion of Quenast to Venezuelan bitumen is better than Quenast to Arabic bitumen. Furthermore, the adhesion of Arabic or Venezuelan bitumen to pure Flint is poor.
The results of this test must be handled with care because the calibration curve for the uncovered surface material is different than for the uncovered surface, which developed during boiling on the aggregate. Sometimes a very thin layer of bitumen is considered as a uncovered surface. Also it is possible that during boiling uncontrollable phenomena like cracks in the surface occur by which the surface becomes larger.

Figure 3. Relation of flint concentration versus bitumen (two types) coverage of aggregates (Dutch gravel, Quenast, flint) after stripping according prEN 12697-11;Part D



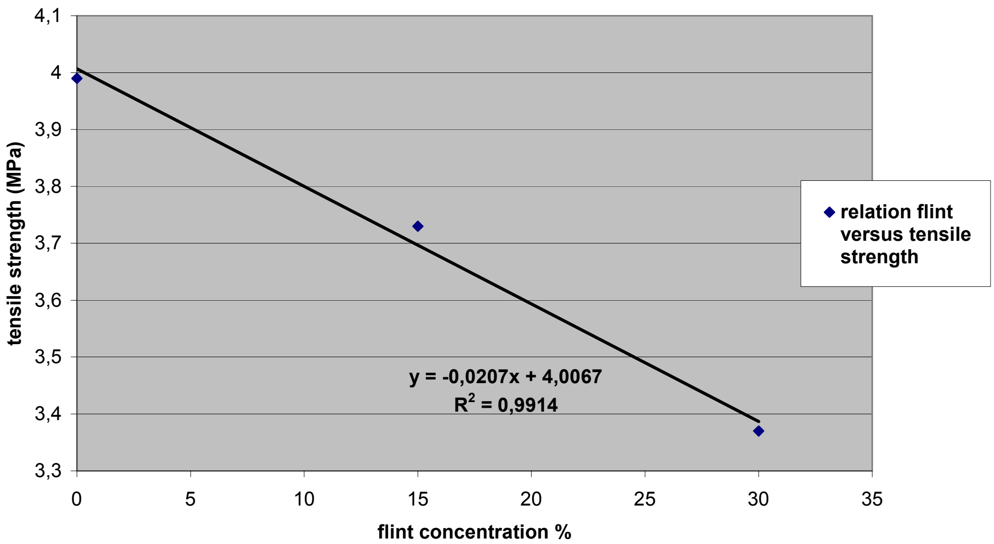
3.3 Water sensitivity and stripping methods according prEN 12697-12 and the Dutch procedure.

In most cases (table 2) no relation has been found between the flint concentration and the indirect tensile strength. Only for the binder layer tested by the Dutch stripping procedure, a positive correlation has been found between the tensile strengths (before and after) and the flint concentrations (see figure 4). The same asphalt mix, which is tested according to the prEN 12697-12 procedure, gives this positive correlation as well, however in a minor degree.

Asphalt mixes	Tensile strength before (MPa)	Tensile strength after (MPa)	Retain ratio (%)	Tensile strength before (MPa)	Tensile strength after (MPa)	Retain ratio (%)
	PrEN 12697-12 (25°C)			Dutch procedure (5°C)*		
binder layer 0% flint	1,58	0,59	37,3	3,99	1,78	44,6
binder layer 15% flint	1,48	0,54	36,5	3,73	1,67	44,8
binder layer 30% flint	1,47	0,56	38,1	3,37	1,51	44,8
Dense layer 0% flint	0,87	0,48	55,1	3,19	1,63	51,1
Dense layer 15% flint	0,87	0,48	55,2	3,19	1,62	50,8
Dense layer 30% flint	0,79	0,50	63,3	3,21	1,76	54,8
Porous A.C 0% flint	0,38	0,28	73,3	1,53	0,85	55,6
Porous A.C 15% flint	0,43	0,28	65,1	1,54	0,96	62,3
Porous A.C 30% flint	0,40	0,23	57,5	1,69	1,06	62,7

Table 2: Results of stripped asphalt concrete according prEN 12697-12 and the Dutch retain procedure
(* In this case the tensile strength of porous asphalt is measured at 0°C)

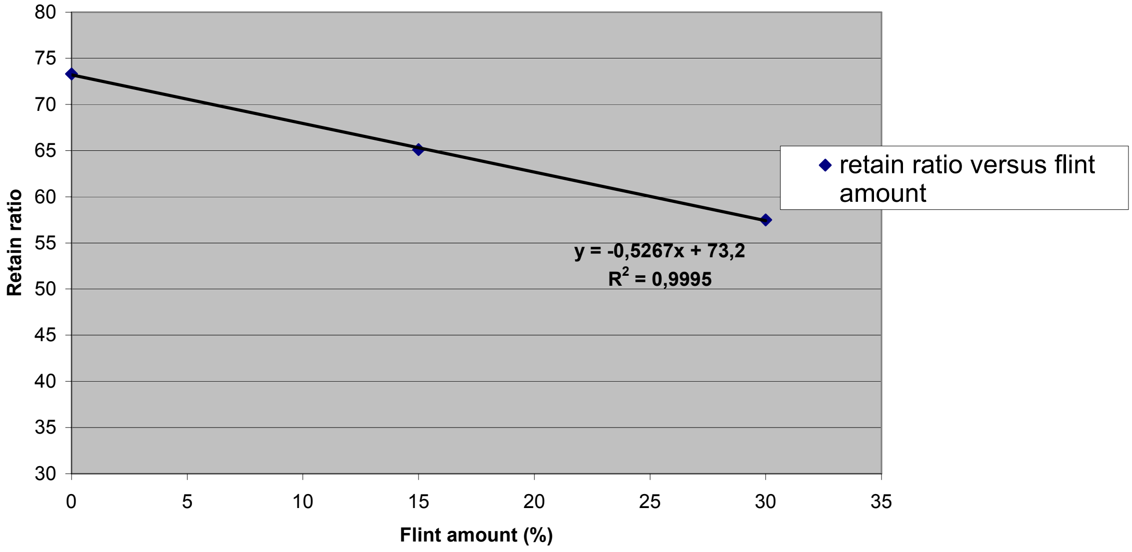
Figure 4. Flint amount versus tensile strength of binder layer specimen (containing different flint amounts) after stripping according a Dutch procedure



For the other mixes sometimes even an opposite effect is found; the tensile strength is higher for higher flint concentrations. This indicates good adhesion to flints. This strange phenomenon is already found in the results of the Rolling Bottle method.

Only the retain ratio's of porous asphalt indicates that by an increasing flint concentration the tensile strength retain ratio decreases. This indicates bad adhesion between flint and bitumen (see figure 5). This is expected because when the tensile strength after the stripping procedure declines in accordance to the flint concentration due to the bad adhesion, the retain ratio also decreases.
Other asphalt mixtures do not show such a trend in retain ratio's. In most cases the retain ratio's are identical or increases a little with higher flint concentration. This aspect is strange because the flint rich aggregate should adhere poor.
In general the retain ratio's are low which means that both stripping procedures are apparently severe.

Figure 5. Flint amount versus retain ratios of porous asphalt specimen (containing different flint amounts) after stripping according prEN 12697-12



4. CONCLUSIONS

Based on the results of the research program the following conclusions can be drawn:

1. The stripping procedure according European standard prEN 1297-11; Part B; Rolling Bottle Method apparently cannot be used to recognize poor adhering materials like flint. In a number of cases there is even an opposite effect; i.e. increasing the amount of flint content increases the adhesion;
2. For the stripping method according prEN 12697-11; Part D; Boiling Water Method, a moderate correlation between the amount of flints and the stripped aggregate surface is found for a mixture of Dutch gravel/flint and Venezuelan bitumen. After additional research it might be possible to incorporate the use of this method instead of the existing regulations with regard to the maximum flint concentration in aggregates.
3. Stripping methods according prEN 12697-12; Water sensitivity and the Dutch procedure, give a positive correlation between the indirect tensile strength and the amount of flint only for one type of asphalt mix (binder layer). A direct correlation between the retain ratio and the flint concentration is found for only one asphalt mix (porous asphalt). The other asphalt mixes do not show these correlations. These methods do not seem fit to determine the negative amount of flint in aggregate.

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