

# Development of a laboratory test for characterization of asphalt-aggregate adhesion

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**ABSTRACT:** The mechanical properties of bituminous mixtures depend on the adhesion between mastic and aggregate, as well as on the cohesion within the mastic. These properties vary as a function of the composition of the bituminous mixture, of the chemical and physical features of the aggregate and bitumen and of the condition, under which the mixing is carried out.

It is thus fundamental to develop an adhesion test, which is of easy execution and, at the same time, represents correctly the bituminous mixture and its mechanical behaviour, essentially the aggregate-mastic adhesion.

The test described here simulates the behaviour of a small representative specimen of the bituminous material subject to increasing tension or shear force, inducing rupture of the aggregate-mastic bond.

The specimens have a prismatic shape and comprise a single large piece of aggregate between two layers of mastic.

The efficiency of the aggregate-mastic bond will be evaluated by the necessary force to rupture the specimen and by the form of rupture.

The results obtained from this test will not only allow the features which most influence the adhesion of mastic to aggregate to be established, but also reveal the best aggregate-fines-binder combinations as a function of the type of aggregate and bituminous mixture.

## 1 INTRODUCTION

A new laboratory test for the characterization of the coarse aggregate adhesion to the asphalt mastic that surrounds it is being developed at the University of Minho. The present paper intends to show the studies done to date.

Since the mechanical mix behaviour depends on the adhesion, the new test can be used to evaluate not only the adhesion itself but also the performance of the whole mixture.

Two tests of adhesion have been developed in order that one can simulate the adhesion resistance to fatigue (tension test) and the other can simulate the adhesion resistance to permanent deformation (shear test at constant normal stress).

By using these tests, the influence of the binder content of the mastic on the bond resistance has already been observed, but other variables have also been studied to understand their influence on the mixture behaviour.

Each specimen contains a layer of coarse aggregate particles at mid-height. The rest of the specimen is filled with asphalt mastic.

These tests can show the optimum combination of bitumen, fines and coarse aggregate. Thus, they can be considered as fundamental (or simulative) tests, which can be used for the design of bituminous mixtures. For this, a simple way has been proposed to use the results of these tests in the mix design.

Finally, pure mastics with different binder contents (based on this study) have been tested in both tension and shear, in order to evaluate the mastic cohesion.

## 2 STUDY OF THE ADHESION – COARSE AGGREGATE AND MASTIC INTERACTIONS

In many countries there are references concerning adhesion tests. In these tests, the interaction is measured between the bitumen and the aggregate in order to increase the adhesion. The chemical and physical characteristics of both are measured in order to maximize the absorption and adsorption of the bitumen by the aggregate. Thus, the bigger the absorption and the adsorption are, the better this bond is. In this way, the adhesion will increase and stripping is

unlikely to occur when the mixture is in contact with water.

Thus, most adhesion tests consist in observing the effect of water in the diminution of the mechanical characteristics of the bituminous mixtures. The specimens are tested before and after their immersion in water during a certain period of time. Then, they are tested and the ratio between their characteristics before and after the immersion is measured. In this way, the effect of adhesion is determined.

Other tests (among which the most well-known is the Net Adsorption Test) consist in the evaluation of the bitumen adhesion by measuring its dispersion by water. In these, the quantity of bitumen removed from the mixture is measured after its contact with water. The less the quantity of removed bitumen is, the greater the absorption and adsorption are – i.e. the adhesion is greater.

In both cases, the aim is to evaluate the bond between the bitumen and the aggregate to avoid it being broken. In such a case, stripping would hardly occur, avoiding the harmful effects of water in the pavement (especially in the aggregates) and the break-up of bituminous material.

The following paragraphs describe tests of the types referred to above.

Ingo Nösler (2000) uses the following method to measure adhesion strength. Compacted Marshall specimens are used to determine the resilient modulus at 25 °C by means of the non-destructive dynamic indirect tensile test. The loss of resilient modulus after a defined water immersion, compared to the initial measured modulus, is used to quantify the adhesion strength between aggregate and bitumen. If the loss of resilient modulus is more than 30%, the adhesion strength is considered to be too low and, in consequence, either the type of aggregate should be changed, or polymer-modified bitumen or adhesion additives should be used.

This type of test is of the first category noted above. The Net adsorption test and the Cantabro test are examples of the second category.

Woodside *et al.* (1996) used these tests in order to observe in which ways the results varied, when modifying certain characteristics of the bituminous mixtures. They tested three types of aggregate and they observed that the bond between aggregate and bitumen was greatest for the old red sandstone, followed by the silurian greywacke and the worst results were for the tertiary basalt. They concluded that further work on the gradings used for these two methods could prove invaluable for predicting likely in-service performance. The authors believed that these two test methods can be used to give an early indication of problems that may be encountered during the life of the road at a minimum of cost and time. In terms of minimising the likelihood of in-service failure, such methods could alert the engineer to consider the use of alternative materials such

as adhesion agents to improve the adhesive bond between the aggregate and bitumen and so improve the prediction of likely in-service performance.

Other authors have experimented with new tests for the characterization of the bond between bitumen and aggregate. Among these, Scholz and Brown (1996), who developed a test in the dynamic shear rheometer to measure the rheological characteristics of bitumen in contact with mineral aggregate in the presence of water.

Observation of specimens tested in fatigue or permanent deformation shows that very often the rupture of specimens occurs in the bonding zone between the mastic and the aggregate. The tests described in this paper were developed based on the fact that bitumen does not exist in a pure form in a mixture; it is always mixed with fines, thus forming a bituminous mastic.

Moreover, because of the form of rupture of the specimens, it is important to verify whether the adhesion only avoids stripping or if it actually increases the mechanical properties of the bituminous mixture, inhibiting both fatigue and permanent deformation (in the rupture zone).

Therefore, it was decided to develop a mechanical adhesion test, which will be described in the following sections.

### 3 CHARACTERIZATION OF THE MATERIAL USED

Before the development of the adhesion test described in the present paper, it was necessary to study the composition of the mastic for use in the test specimens. This mastic should be representative of the mixture being investigated and also appropriate for the specimen's dimension.

The largest dimension of the aggregate should not exceed a quarter of the dimension of the specimen. Since it was decided that the specimens would have a dimension of 4 cm, the coarse aggregate was therefore restricted to 1 cm, which corresponds to the 3/8" sieve.

The specimens were formed by two mastic layers, between which there was a layer of coarse aggregates.

Thus, aggregate retained on the 3/8" sieve was used and the composition of the mastic (Figure 1), which surrounds these aggregates, was determined from the Portuguese standard (APORBET) for wearing courses.

The material used for this study was that most commonly utilized in the North of Portugal. The binder material was an unmodified bitumen with a penetration grade of 51 dmm and the softening point (ring and ball) was 53 °C. This bitumen has good thermal behaviour at low temperatures, but it shows some problems at higher temperatures. In spite of

this, for the observed temperatures in the North of Portugal, this bitumen was completely appropriate.

The aggregate used was one of the most commonly utilized from the same region. It was a mechanically crushed granitic rock (both fine and coarse fractions) and it was stored in two stock piles: the 0/4 stock pile with the necessary fines to produce the mastic and the 11/16 stock pile, from which the coarse aggregate was sieved. This material has a good durability.

In Figure 1 the design grading curve of the mastic (which surrounds the coarse aggregates of the 3/8'' fraction, (Silva et al, 2002)) is presented, termed "target gradation". This was determined through analysis of the mastic composition. The actual mastic gradation used is also shown in this Figure and it was obtained through a simple sieving of the material stored in the 0/4 stock pile.

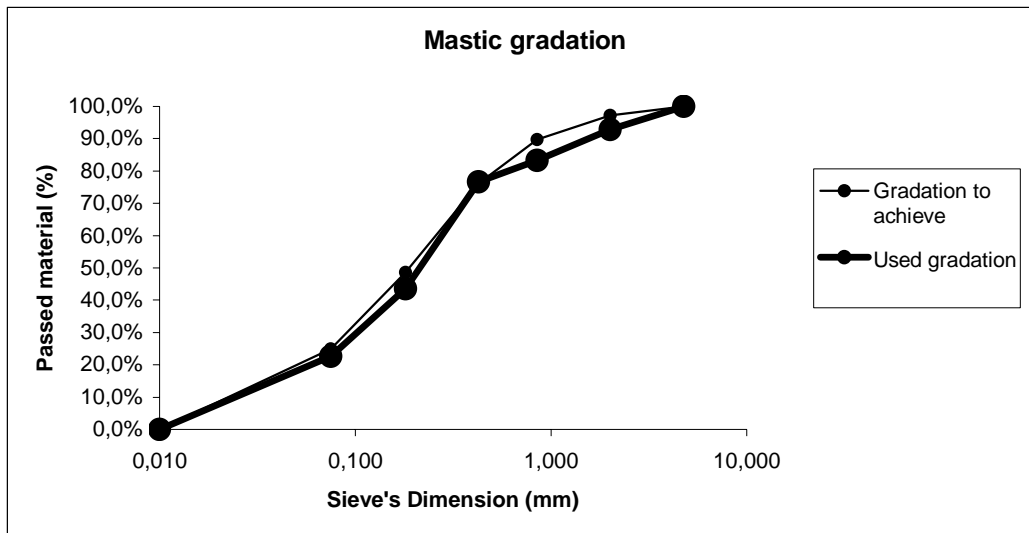


Figure 1 – Mastic gradation after separation of coarse aggregate in 3/8" sieve

#### 4 LABORATORY PROCEDURE AND TEST METHODS

The first step consisted of evaluating the mastic composition.

Specifically, mix design was carried out using the grading curve indicated in the Portuguese standard for wearing courses and the Marshall method for the determination of the optimum binder content.

Based on this, a mixture was made and divided into different fractions. Each fraction was burnt in order to determine the binder content and grading curve. The retained fraction of the mixture on the 3/8'' sieve was then evaluated to derive the mastic composition, which was used in the present adhesion study.

The adhesion test specimens were prepared once the mastic composition was known.

In order to obtain the grading curve of the mastic, the material in the 0/4 stock pile was sieved using the n° 40 sieve and the material passing was then mixed with 20% of the retained material.

The coarse aggregates were obtained from the 11/16 stock pile, using material which passed the 1/2" sieve but was retained on the 3/8'' sieve.

The binder contents used to prepare the mastic were based on the study of its composition. The values used were of 10.5%, 13%, 14% and 15%.

For each binder content, sufficient mixture was made in order to prepare 16 specimens for the tensile

adhesion test and 12 specimens for the shear adhesion test. Firstly, the fine material was mixed with the bitumen, forming the mastic. A small part of it was also mixed with the coarse aggregates to surround them.

The mould, where the specimens were prepared, was filled to half its height with the mastic. Then, a layer of coarse aggregate particles was placed uniformly on the mastic. The rest of the mould was filled with the rest of the mastic. During the whole process, the mould was kept warm to avoid the loss of temperature in the mix.

The specimens were then compacted by a cylindrical compactor with vibration. According to SHRP recommendations, this is a method which represents the *in situ* compaction.

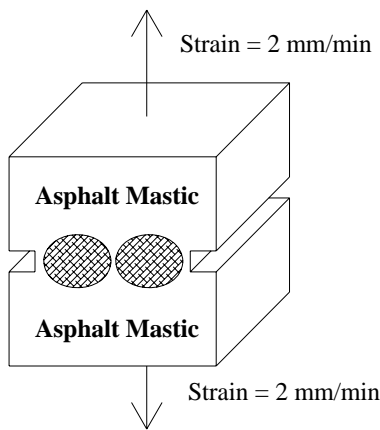
When the specimens had cooled, an incision was made at half height in the specimens for the tensile adhesion test in order to force the rupture to occur in the adhesion area between the mastic and the coarse aggregate layer.

Finally, some of the specimens were tested under a simple tension test in order to measure the adhesion performance of a mixture subjected to fatigue and others were tested under a simple shear test, at constant axial stress, in order to measure the adhesion performance of a mixture subjected to permanent deformation, as in Figure 2. The results were then analysed.

In Figure 3, both the tension and shear specimens with the coarse aggregate in the middle can be observed. Prior to test, these specimens are glued to

cylindrical platens used in the test machine, as shown in Figures 4 and 5.

### Tension test



### Shear test at constant axial strength

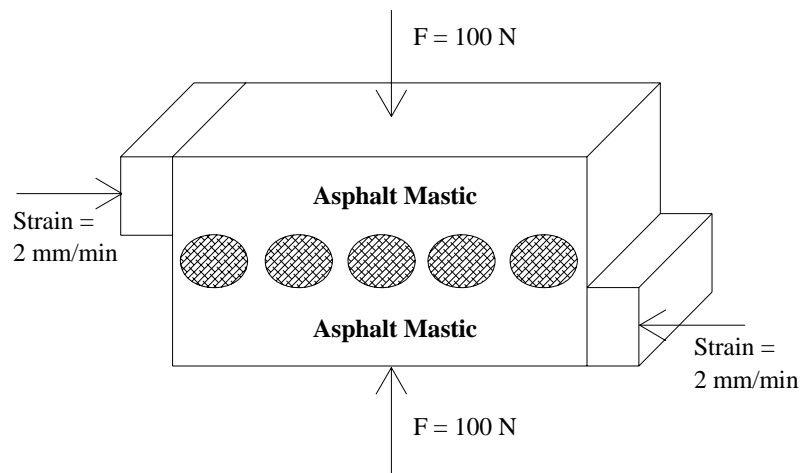


Figure 2 – Representation of the tension and shear adhesion tests

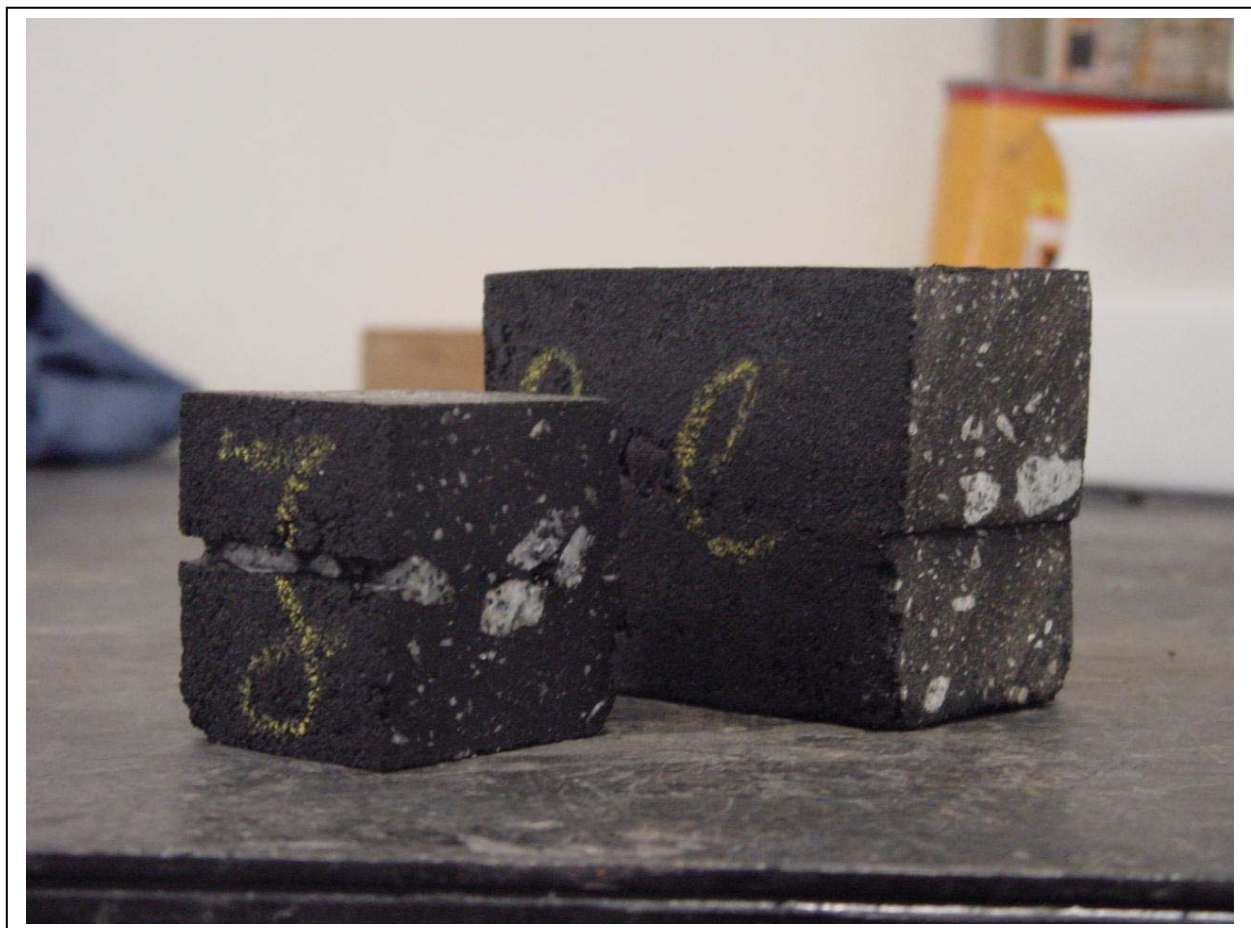


Figure 3 – Test specimens used in tension and shear adhesion tests

## 5 RESULTS

In Figure 6, one can observe the typical results of a tension test in specimens with different binder contents. The specimens were tested in simple tension at a constant deformation rate of 2 mm/min, quickly reaching rupture and the maximum value of resis-

tance. This value can be termed the adhesion performance relating to fatigue.

Some specimens ruptured at their base, caused by relatively poorer compaction in the lower part of the mould. Other specimens gave rise to peculiar results, because, after reaching an initial maximum, they

subsequently reached a higher value. Sometimes the recorded stress value varied very quickly. All these

specimens were rejected during the analysis.

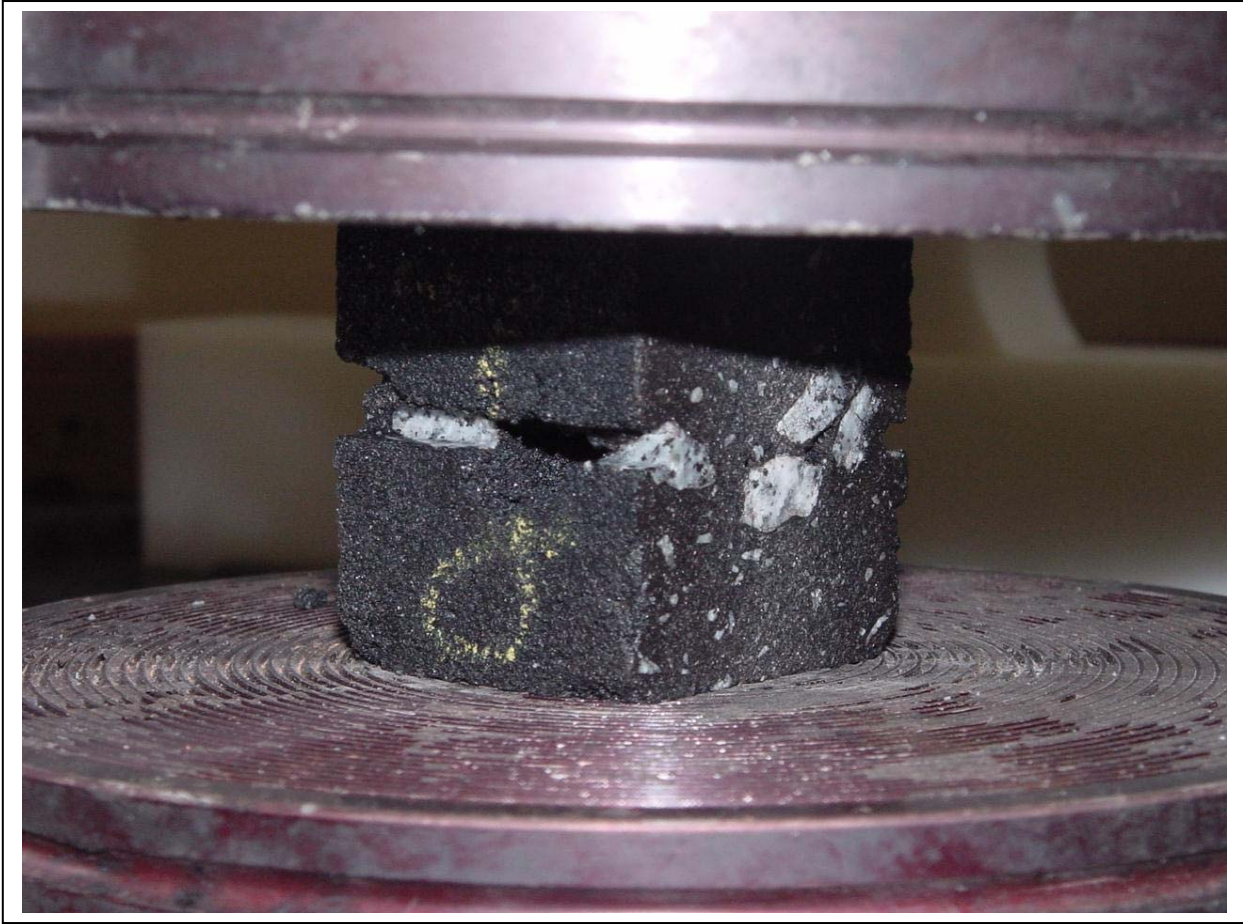


Figure 4 – Test specimen used in tensile adhesion test glued to the machine platens



Figure 5 – Test specimen used in shear adhesion test fitted in the machine platens

All the others specimens had a regular form of rupture, which was as expected, in the adhesion area between the coarse aggregate particles and the mastic.

In Figure 7 the overall results of the tension tests are presented. The results correspond to the resistance of each specimen to tension as a function of the binder content. The presented trend line was po-

sitioned through the average values of resistance for each binder content.

In Figure 8, one can observe the typical results of a shear test at constant axial stress. The specimens were tested in simple shear at a constant axial stress and at a constant shear deformation rate of 2 mm/min.

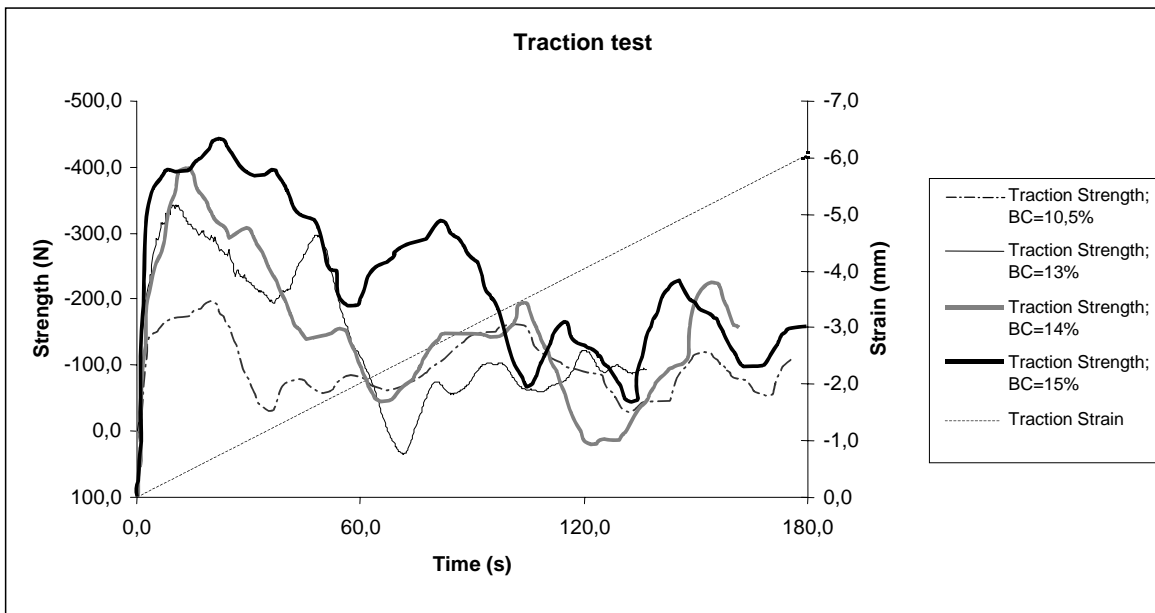


Figure 6 – Results of tensile adhesion tests on specimens with different binder contents

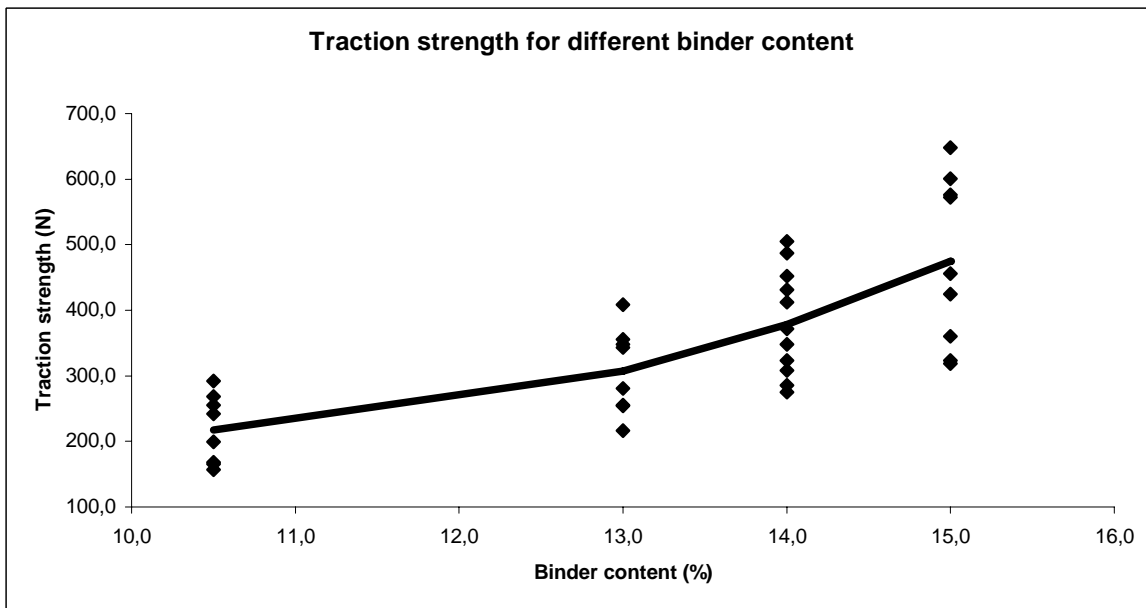


Figure 7 – Tensile strengths of specimens with different binder contents

The form of result from the shear tests (Figure 8) shows that there is an irregular development of shear

resistance, which is due to the fact that the constant axial confining force is too low (100 N). This causes

a sliding of the specimens when they are sheared. This sliding generates a rapid loss of resistance and the irregular form of the graph.

In spite of this, the resistance recovers again and the maximum value obtained can be read off. This value can be termed the adhesion performance relating to permanent deformation.

Some specimens presented a different form of result, because, instead of reaching a maximum resis-

tance and then reducing, this resistance never decreased. Sometimes the measured stress value varied very quickly. All these specimens were rejected during the analysis.

All the specimens had a regular form of rupture, in the adhesion area between the coarse aggregates and the mastic.

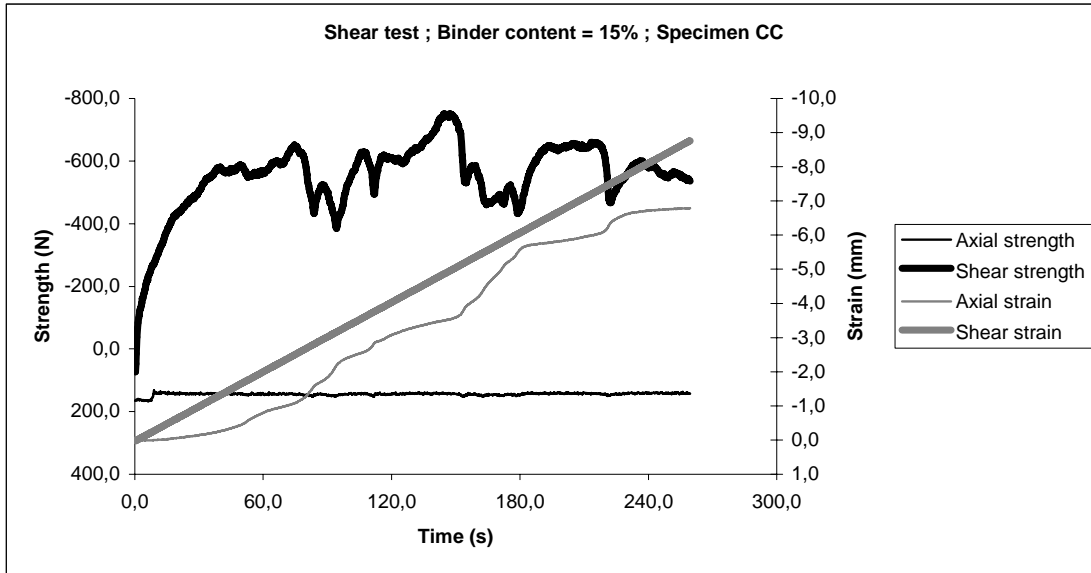


Figure 8 –Result of a shear adhesion test with a constant axial load (100 N) on a specimen

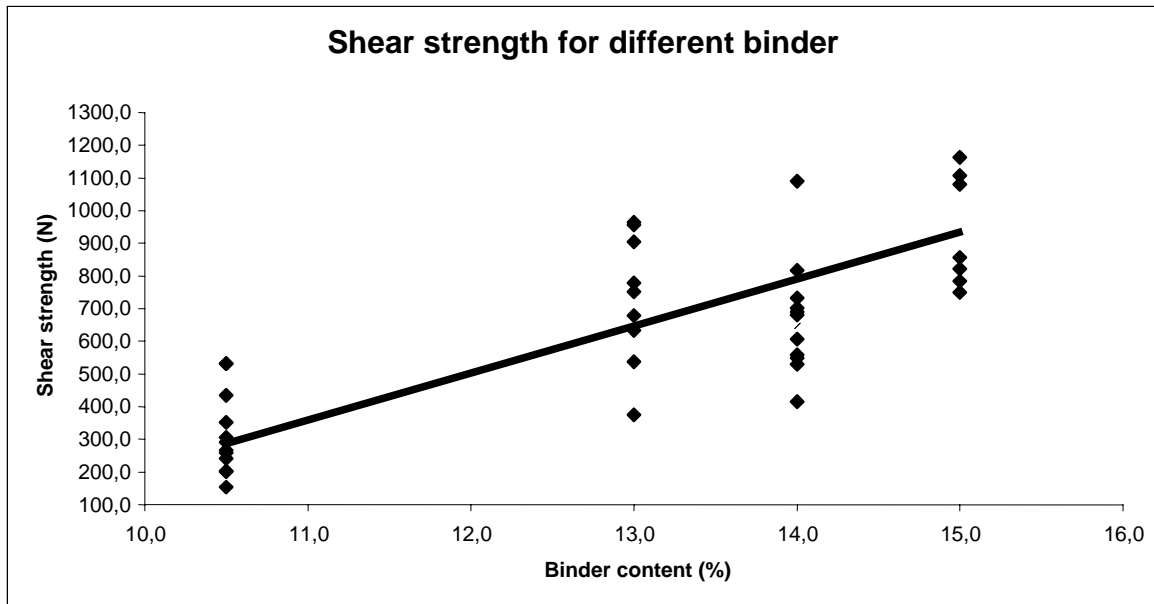


Figure 9 –Results of the shear adhesion test at constant axial stress on specimens with different binder contents

In Figure 9 the overall results of the shear test are presented. The results correspond to the resistance of each specimen to shear as a function of the binder content. The presented line is a linear best fit obtained through the data.

Finally, in order to know the bituminous mixture behaviour not only with regard to adhesion but also in general terms, tests were done to study the mastic cohesion.

These tests were similar to the adhesion tests (the main difference was the fact that the specimens were totally formed by mastic without any coarse aggregate), consisting of a tensile strength test, which simulated the mastic behaviour relating to fatigue, and a shear test at constant height, which simulated the mastic behaviour relating to permanent deformation.

In Figures 10 and 11 the results of the tests on the asphalt mastic are shown. As the main objective was not to study the mastic cohesion, only two binder contents and relatively few specimens were tested.

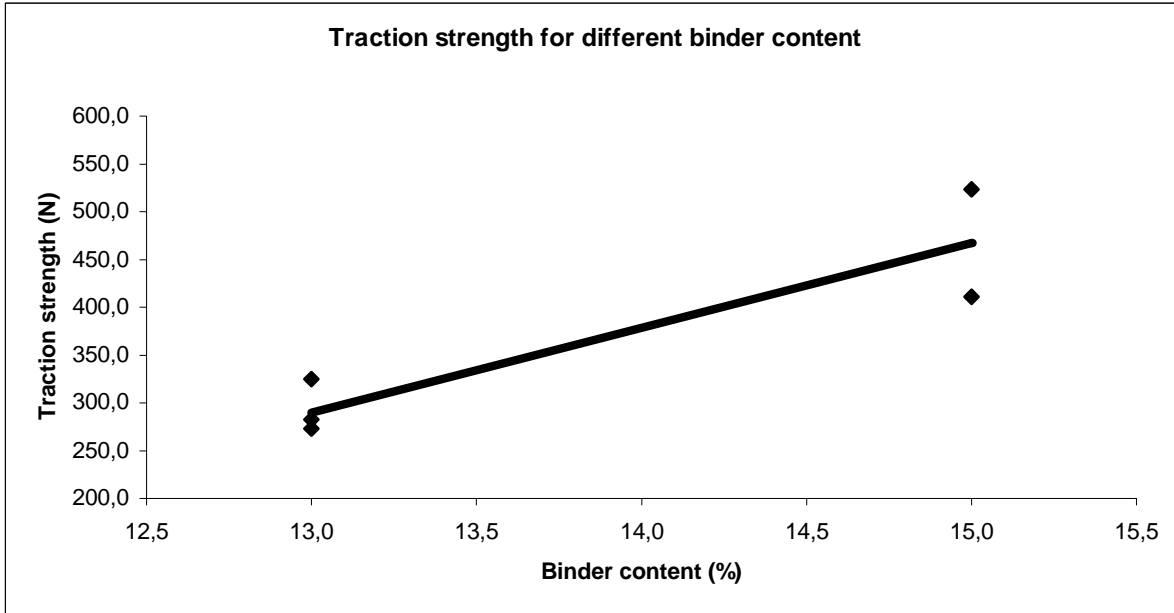


Figure 10 – Results of the tensile adhesion tests on mastic specimens with different binder contents

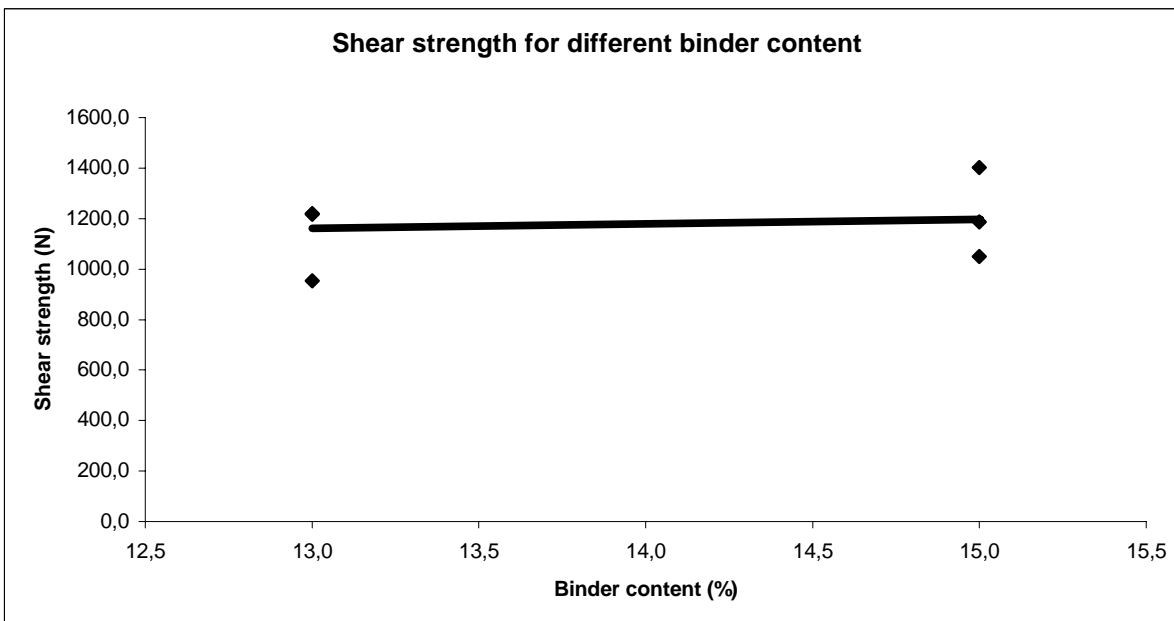


Figure 11 – Results of the shear adhesion tests at constant height on mastic specimens with different binder contents

## 6 ANALYSIS OF RESULTS

The adhesion tests, related to fatigue and permanent deformation, have been shown to be a new option in predicting the mechanical behaviour of a mix. The test reproducibility is definitely good, although, when the resistance increased, with higher binder content, the results (for each binder content) had a higher dispersion.

Some tests can not be taken into account because of their irregular rupture, but the simplicity and speed of production and test for a large number of specimens make these adhesion tests a good choice for evaluation and study of the mechanical behaviour of a mix.

The final results demonstrate that the adhesion performance related to fatigue and permanent deformation increases with the binder content. In fact,



higher binder contents should be analysed in order to find an optimum.

A relation should be established between the adhesion tests and other fundamental tests (4 point bending for fatigue, shear at constant height for permanent deformation), in order to validate the adhesion tests and allow their use for mix design.

In fact, once the optimum mastic binder content (achieved through the adhesion tests) is known, the optimum mixture can be established. In order to do this, the following procedure can be used:

Produce mastic with a grading curve equal to that of the fine aggregate (passing the n°4 sieve) of the mix to be designed;

Use the adhesion tests to obtain the optimum mastic binder content;

Calculate the optimum binder content of the mix through the following formula:

$$OBC_{MIX} = OBC_{MASTIC} \times \frac{W_T}{W_M} \quad (1)$$

$OBC_{MIX}$  – optimum binder content of the mix

$OBC_{MASTIC}$  – optimum binder content of the mastic

$W_T$  – total weight of the aggregate in the mix

$W_M$  – weight of the aggregate in the mix passing the n° 4 sieve

In relation to the mastic behaviour, only a few tests were made in order to confirm if the binder content (found in the composition analysis) was the one that would have an optimum behaviour.

The binder contents of 13% and 15% were used and the tests showed that the highest binder content was the one that revealed the best behaviour, particularly with regard to fatigue. A small increase in the shear resistance with the binder content was also noticed.

## 7 CONCLUSION

In conclusion, these adhesion tests could be used with good results for the characterization of bituminous mixtures, especially concerning the interaction forces between the coarse aggregate particles and the mastic.

It was observed that the adhesion performance increased with the binder content relating to both fatigue and permanent deformation.

Thus, this test can be used for the formulation and study of bituminous mixtures. However, some adjustments must be made to improve the method and the test.

Presently, the only studied variable is the binder content. This work will proceed with the analysis of other variables such as the chemical and physical composition of the aggregates, type of binder, use of additives, influence of water, moisture and dust.

Other types of loadings should also be experimented with during the tests.

The preparation of the specimens should be calibrated. The compaction must be more efficient and the placing of the coarse aggregate more controllable. These problems might explain the variation in the results.

Thus, in the future, the mixtures should be compacted as a slab and then sawn to form the specimens. A mechanism must be developed for the placement of the coarse aggregate particles in a systematic manner.

The tension test was adequate. The shear test at constant stress can be used, but the axial stress of confinement must be higher or substituted by a shear test at constant height, because of the sliding of the specimens.

Concerning the mechanical behaviour of the mastic, it was observed that both the resistance to fatigue (i.e. tension) and the resistance to permanent deformation (i.e. shear) increased with the binder content. If this was expected in relation to fatigue, this was not the case for permanent deformation. Higher binder contents should be tested to know to what extent the behaviour of the material would improve.

## 8 REFERENCES

- APORBET (1998). *Misturas Betuminosas. Contribuição para a Normalização do Fabrico e Aplicação*. Lisboa.
- Azevedo, M. (1997). "Improved design methodology for base-course bituminous mixtures" in Di Benedetto, H., Francken, L. (eds.). *Mechanical Tests for Bituminous Materials – Recent Improvements and Future Prospects*. Rotterdam.
- Belošovič, Š., Žideková, E. (1996). "Adhesion of bitumen binders on aggregates" in *Proceedings of the Eurasphalt & Eurobitume Congress*. Strasbourg.
- Curtis, C.W., Ensley, K., Epps, J. (1993). *Fundamental Properties of Asphalt-Aggregate Interactions Including Adhesion and Adsorption*. SHRP-A-341. Washington D.C.
- Lin, J.-D., Lin, D.-F., Yu, J. (1997). "A study of stripping resistance of asphalt concrete by artificial environment conditioning system" in Di Benedetto, H., Francken, L. (eds.). *Mechanical Tests for Bituminous Materials – Recent Improvements and Future Prospects*. Rotterdam.
- Luminari, M., Fidato, A. (1998). "State of the art report on mix design" in *RILEM Report 17. Bituminous Binders and Mixes. State of Art and Interlaboratory Tests on Mechanical Behaviour and Mix Design*. Francken, L. (ed.). London. E & FN Spon. Pp. 69-101.
- Nösler, I. (2000). "New possibilities for a quantitative test of the adhesion between mineral aggregate and bitumen" in *Proceedings of the 2nd Eurasphalt & Eurobitume Congress*. Barcelona.
- Nösler, I., Beckedahl, H. (2000). "Adhesion between aggregates and bitumen – performance testing of compacted asphalt specimens by means of the dynamic indirect tensile test" in *Proceedings of the 2nd Eurasphalt & Eurobitume Congress*. Barcelona.
- Pais, J., Pereira, P., Azevedo, M. (2000). "Ensaio mecânicos para caracterização de misturas betuminosas" in *Procedimentos do 1º Congresso Rodoviário Português – Estrada 2000*. Lisboa.

- Scholz, T.V. (1995). Durability of Bituminous Paving Mixtures. Thesis submitted to the University of Nottingham for the degree of Doctor of Philosophy. Nottingham.
- Scholz, T.V., Brown, S.F. (1996). "Rheological characteristics of bitumen in contact with mineral aggregate" in Asphalt Paving Technology. Vol.65.
- Silva, H., Pais, J, Thom, N. and Pereira, P. (2002). "Evaluation of mastic in bituminous mixtures", Paper presented in the 4th European Symposium of Performance of Bituminous and Hydraulic Materials in Pavements, Nottingham.
- Woodside, A.R., Woodward, W.D.H., Russell, T.E.I., Peden, R.A. (1996). "Measuring the adhesion of bitumen to aggregate" in Proceedings of the Eurasphalt & Eurobitume Congress. Strasbourg.
- Zolotarev, V.A., Ageeva, E.N., Zinchenko, V.N. (1997). "Quantitative evaluation of bitumen's adhesion by the method of its displacement by water" in Di Benedetto, H., Francken, L. (eds.). Mechanical Tests for Bituminous Materials – Recent Improvements and Future Prospects. Rotterdam.