Tailoring a new laboratory methodology for stripping resistance evaluation of asphalt materials

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ABSTRACT

Road maintenance is an important part of civil engineering works because of pavement materials damage which are due to traffic load and climatic conditions. Water appears to be one of the most significant factors in performance degradation of pavements because it leads to the loss of bond between bitumen and aggregate. Stripping phenomenon and mechanisms which occur when road materials are immersed in water are not fully understood but getting a better comprehension about it would enable to predict and improve pavements durability. This study realized in IFSTTAR Nantes in partnership with EUROVIA and INGEVITY aims at determining the influence of different parameters such as material nature, initial coating conditions and immersion parameters on stripping kinetic and then loss of adhesion in presence of water using a multiscale approach. The first part of the study focus on adhesion in presence of water using binder droplets and polished aggregates. Then, coated surface quantity of loose asphalt-aggregate mixtures after water immersion is evaluated. Stripping's kinetic and coated surface enable to rank different aggregate/bitumen couple in function of their resistance to water. Classification of each part will be compared and discussed regarding aggregates/binder properties and wetting/debonding conditions.

Keywords: Bitumen, stripping resistance, coated surface, asphalt mixture

1. INTRODUCTION

Individually or in combination, the mechanical, thermal, and oxidative stresses caused by traffic, climate, and age may adversely alter the strength and viscoelastic properties of asphalt pavements, leading to deterioration in overall performance and durability. The breaking of the adhesive bond between bitumen and aggregate in water is known as stripping and is a form of passive adhesion, in opposition to active adhesion during coating of aggregate surface by bitumen. Thus, the strength and durability of the adhesive bond (passive and active) between bitumen and an aggregate surface depends on numerous production and construction variables.

There are numerous standardized laboratory test methods to measure moisture damage in use today. Though large in number and varied in procedural details, today's moisture sensitivity tests can generally be divided into two broad categories: tests wherein thoroughly coated bituminous mixtures are evaluated for the percent of retained surface coating after immersion, estimated visually as well as by instrumental methods like image analysis [1-2] and tests wherein mechanical properties of compacted bituminous mixtures (moisture conditioned or not) are measured. Another approach to evaluating moisture susceptibility consists of evaluating stripping directly from the properties of the interface between aggregate and bitumen. In the presence of water, bitumen-aggregate bonds break and contact angles of the bitumen drop on the aggregate surface increase with time of immersion. Basu shows this phenomenon: the film of bitumen in contact with water becomes distorted and adopts a spherical shape to minimize interfacial energy [3].

This study aims at evaluating the influence of aggregate mineralogy on moisture susceptibility as well as the influence of conditions under which the bitumen-aggregate interface is exposed to water. In this work, standardized tests for moisture susceptibility in loose materials (Method of Scale 2 – MS2) are compared to a new experimental protocol of studies of the stripping of bitumen drops deposited on polished aggregate surfaces (Method of Scale 1 – MS1).

2.0 MATERIALS AND METHODS

2.1 Materials characterization

2.1.1 Minerals characterisation

For this study, 7 different aggregates are used. Well-established optical image analysis procedures have been applied to the identification and quantitation of mineral crystals in thin, polished aggregate cross-sections (around 30µm of thickness):

- Quartzite, gneiss and diorite seems to be siliceous aggregates, with a minimum of 60% quartz content. According to literature, they are acid aggregates [4]
- Limestone 1, 2 and 3 are very similar with very high content of calcite. They are basic aggregates. Basalt seems to be more complex, with bigger minerals scattered into a matrix of smaller minerals. In literature, it is considered as a basic aggregate

2.1.2 Surface energy of aggregates

The surface energy of a material is defined as the work done by a force to create a new unit of surface area. Indeed, creation of the new surface involves the movement of molecules from the bulk of the material to the new interface, which creates an energy deficit. The work (W) needed to achieve this expanded surface area (S) may be defined as:

$$dW = \gamma \, dS \tag{1}$$

γ is the surface energy of the materials. For each aggregate, slides were grinded by 6 different disks until use of ¼ µm aqueous solution of polycristallized diamond. Surface energy is measured 7 days after grinding, depositing drops of several reference liquids (1 µL) with a drop tensiometer (DSA 100, Krüss GmbH) [5]. Results are shown in Figure 1. Dispersion component are higher than polar component for all aggregates except for both limestone. Those results can be explain by high polarity of Si-O bond, highly present in quartz. Low polarity and low surface energy for limestone can be explain by the presence of basic component.
A Peltier temperature chamber was set up at 25°C to maintain the multidosing cell at 25°C. All contact angles were measured after stabilisation of the contact line, using DSA software (Krüss GmbH), following axisymmetric drop shape analysis (ADSA).

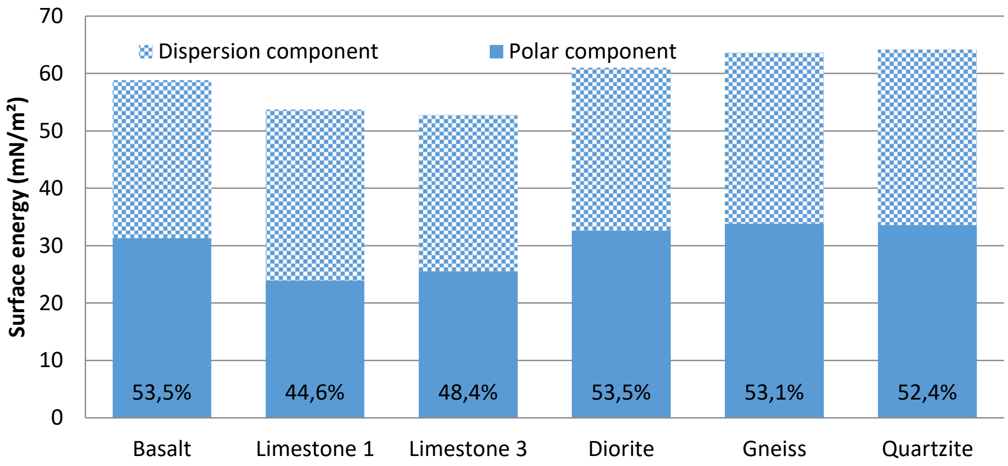

FIGURE 1 : Polar and dispersion components of aggregates and ratio of polarity

2.2 Evaluation of drop stripping – Method of Phase 1 (MS1)

A new method of stripping evaluation studies the dynamic in the shape (area) of a bitumen drop deposited on a polished aggregate while the sample is immersed in water at a fixed temperature (Figure 2). The first day of preparation is noted as D0 and the others steps are referred to it (example: D+1 is one day after D0).

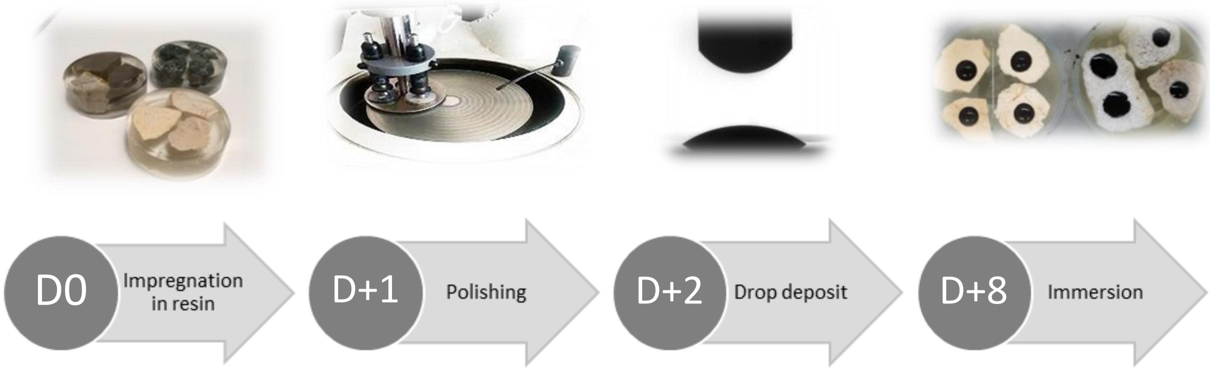

FIGURE 2 : Sample preparation protocol

- Preparation of aggregates: The same polishing method described before is used.

- Drop deposit: The same tensiometer is used with a thermal cell for precise temperature control during bitumen drop deposit (between 6 and 10 µL). All the results presented in this work were obtained from sample prepared with bitumen temperature T_b = 150°C and aggregates temperature T_a = 110°C.

- Sample immersion: One day (24 h) after the bitumen drops were deposit, samples are immersed in hot distilled water and photographs were taken.

- Data analysis: Image J software is used to detect all drops on the photographs. The initial area (at time, t = 0) of the bitumen droplet was defined as S(t=0) and the area of the bitumen droplet at some time t was defined as S(t). Eq.(2) is a theoretical description of the dynamic evolution of the relative change in droplet surface area with time of immersion. The term A stands for the equilibrium at long time value and τ represents a time constant.

$$\frac{S(t)}{S(t=0)} = A - (A - 100)\, e^{-\frac{t}{\tau}} \qquad (2)$$

2.3 Test for moisture sensitivity on loose materials – Method of Phase 2 (MS2)

Two adhesion tests on loose bituminous mixtures were conducted in this study (Table 1). For both, mixtures were formulated with 6/10 aggregate, which had been water washed and dried prior to treatment with 3% bitumen. Aggregates and bitumen are heated to 150°C. Compared to the classic method of optical evaluation of the percent of retained bitumen coating, an automatic treatment with the software ImageJ is used.

TABLE 1 : Immersion conditions of adhesion tests on loose materials

Standardized test reference	Immersion conditions
Static immersion XP T66-043-2 [6]	Cold coated loose materials are immersed in distilled water heated to 60°C during 24 hours.
Boiling test ASTM D3625 [7]	Coated loose materials at 85°C are immersed in boiled distilled water during 10 minutes.

3.0 RESULTS AND DISCUSSIONS

The focus of this study is on the influence of the mineralogy of the aggregate on stripping under during exposure to water at varying temperatures.

3.1 The influence of the mineralogy

The influence of mineralogy on stripping resistance was studied at two different levels: 1) on model materials, studying the stripping of a bitumen drop on a polished aggregate (MS1) and 2) on loose materials using standardized test methods for moisture resistance (MS2).

3.1.1 Experimental results

From MS1, the change in bitumen drop area (on a polished aggregate surface) was measured as a function of time of immersion in heated distilled water as a means characterize passive stripping. Table 2 shows the equilibrium retained surface area (i.e., S(t)/S(t=0)) for one bitumen on the seven different polished aggregate surfaces after immersion in water at 65°C. A high percent of retained droplet surface area indicates a good resistance to water.

The lowest equilibrium residual drop surface area at 65°C was measured with quartzite and gneiss (under 50%), whereas all the different limestone showed a high percent of residual surface (more than 90%). Final contact angle at the triple point in water can be approximated from the surface of the drop at the interface, if one regards the drop as a sphere. Values are presented in Table 2.

From the equilibrium value, it is possible to classify the bitumen-aggregate pairs according to their stripping resistance from the best to the worst: Limestone 1, 2 and 3 - Basalt - Diorite - Gneiss - Quartzite. This results are in accordance with research results from other scientists using other evaluation methods [1][8].

TABLE 2 : Results of drop stripping after 15 hours of static immersion in water at 65°C

Aggregate	Quartzite	Gneiss	Diorite	Limestone 1	Limestone 2	Limestone 3	Basalt
A (%)	24 ± 2	43 ± 1	61 ± 3	96 ± 1	96 ± 1	94 ± 1	82 ± 2
Tau	12.4	31.7	22.8	1.8	4.7	0.5	16.8
Angle in water (°)	86.6	77.1	58.4	42.8	43.5	40.0	50.8

From MS2, percent of aggregate surface coated with bitumen after immersion at 60°C are gathered in Table 3. Contrary to the previous experiment, which used polished aggregate, the roughness and macro texture of the aggregate in the static immersion test is a potential variable in stripping resistance in this experiment.

TABLE 3 : Results of static immersion at 60°C on loose materials

	Residual coated surface after immersion (%)						
	Quartzite	Gneiss	Diorite	Basalt	Limestone 1	Limestone 2	Limestone 3
Static Immersion	46	47	59	61	69	78	62

Quartzite and gneiss again displayed the lowest percent of retained coated surface after immersion whereas limestones 1 and 2 displayed the highest percentage of retained aggregate surface coating. Limestone 3 and the basalt showed close results and those from diorite are included between gneiss and basalt.

Again, the higher the percent of residual coated surface after 24 h at 60°C in the static immersion, the better the resistance of the bitumen-aggregate pair to stripping. The classification from the best stripping resistance to the worst is: Limestone 2 - Limestone 1 - Limestone 3 and Basalt - Diorite - Gneiss and Quartzite.

With this test, it is possible to discriminate one of the limestone, which seems to be similar at a drop scale, without taking into account the aggregate texture.

3.1.2 Comparison between MS1 and MS2 and discussion on the influence of mineralogy

Aggregates in the static immersion test are not polished. The static immersion test was conducted at 60°C whereas the drop test at 65°C. Despite these and other differences, there was a common trend in overall ranking of stripping resistance. It worth pointing out too that the values of percent retained coating in the drop test (MS1) is lower than those values obtained in the static immersion test (MS2) for acid aggregates (Quartzite and gneiss) due to aggregate smoothness difference.

Influence of mineralogy can be explained by chemistry affinity between aggregates and bitumen and by physico-chemical properties. Indeed, the composition can explain the difference of behaviour when the bitumen-aggregate interface is exposed to water [9-10]. From physicochemical property measurements, it can be shown that polarity of surface energy is lower for basic aggregates (Limestone) than for acid aggregates (see Figure 1). This higher polarity is due to the high quartz presence in quartzite, gneiss and diorite.

3.2 Influence of water temperature

3.2.1 Experimental results

The retained surface area of bitumen drops on polished aggregate was measured after reaching equilibrium in water at four different temperatures: 58, 65, 70 and 80°C (MS1). Results are shown in Figure 3 for quartzite and limestone 1. For quartzite, the higher the water temperature the lower the final percent of the retained surface area (i.e., S(t)/S(t=0)). In contrast, water temperature did not impact stripping of bitumen from limestone 1. The percent retained surface area at equilibrium is still higher than 90%, even at 80°C.

The effect of water temperature was examined in adhesion tests with unpolished aggregate (MS2). The static immersion test was conducted at 60°C, and the boiling water test was conducted around100°C. Percentages of retained coated surface after conducting these three tests are presented in Figure 4.

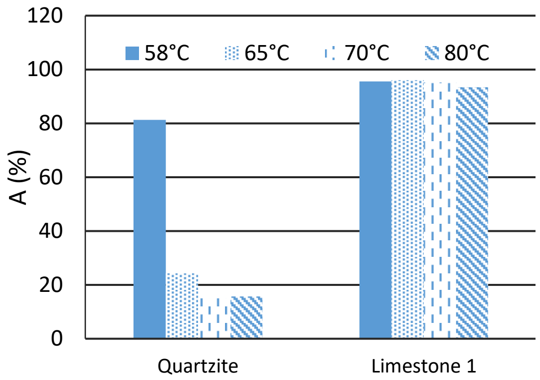

FIGURE 3 : Impact of water temperature on quartzite and limestone 1 (MS1)

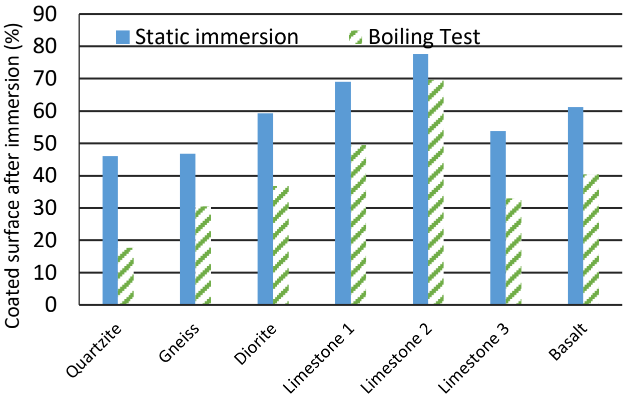

FIGURE 4 : Percent of coated surface after immersion for adhesion tests with 3 ppc bitumen content (MS2)

Static immersion for 24 hours at 60°C led to higher retained percent surface coating than the 10-minute, boiling water test (Figure 4). Increasing water temperature by 40°C decreased the percentage of retained surface coating even if the time of immersion is shorter.

3.2.2 Discussion : Influence of water temperature

Bitumen viscosity is temperature dependant. Increasing water temperature makes bitumen more fluid and less dense and make surface debonding more energetically favorable, From the rheology analyses of bitumen, it is possible to correlate A values to bitumen viscosity (Figure 5).

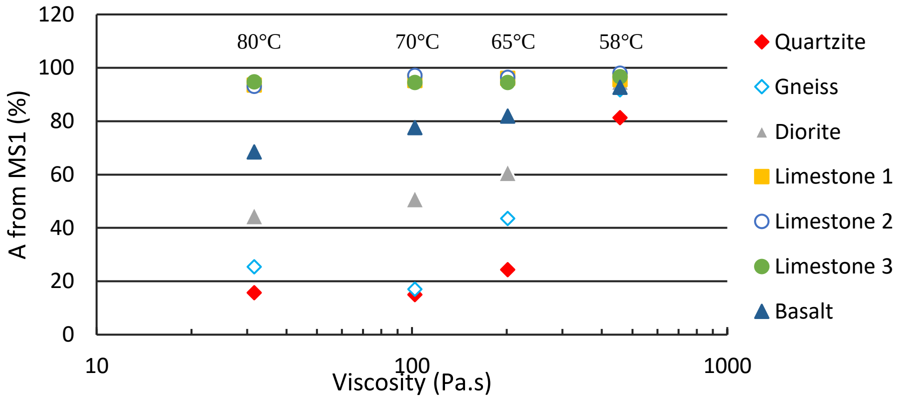

FIGURE 5: Evolution of A value as a function of bitumen viscosity

For all types of aggregates, higher is the viscosity, higher is the retained percent surface coating, meaning an improvement of stripping resistance. The gap between the higher percentage (around 500 Pa.s) and the lower (around 30 Pa.s) is bigger in case of acid aggregates. Increasing water temperature decreases bitumen viscosity. When water starts to deteriorate bitumen/aggregate interface, if viscosity is low enough, bitumen is able to move from the substrate due to water action.

4.0 CONCLUSION

The aim of this work was to evaluate stripping phenomenon at different scales and determine the influence of several parameters on water impact at aggregate/bitumen interface.

This study introduced a new small-scale test method for moisture sensitivity. The method uses optical image analysis to quantify the strength of bitumen film adhesion on polished aggregate exposed to water at varying temperatures. A small volume (6-10 μL) of hot bitumen is deposited as a drop on a polished and heated aggregate surface. The area of the droplet was then measured over time after immersion in water at a range of temperatures via optical image analysis. The ratio of the droplet surface area after immersion time t (expressed as S(t)) to the initial surface area of the droplet (S(t=0)) was a reliable measure of the resistance of the bitumen film to moisture-induced stripping.

This new proposed method allowed to minimize the effect of surface aggregate morphology and focus on the loose of chemical adhesion between bitumen and substrate in presence of water. Compared to existing method of moisture sensitivity tests on compacted material, there is no more pore pressure and water has a good access to the interface, so dewetting phenomenon is much easier visible and quantified with the A value. Then, a lot of bitumen/aggregate pair can be tested in the same time.

Moreover, by carefully characterizing the interfacial tension of the bitumen and by quantifying both the petrographic characteristics of the polished aggregate surface and its surface energy, the retained surface area ratio (S(t)/S(t=0)) could be correlated with physicochemical properties of the bitumen and aggregate interfaces, such as percent surface polarity, global surface energy, and contact angle. Results highlighted different families of mineralogy: basic aggregates such as limestone show a good resistance to water whereas basic aggregates, with high silica content, are very susceptible to water.

5.0 References

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