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Titre : Development of emulsions for micro-surfacing

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ABSTRACT :

Micro-surfacing and slurry seal techniques have been developed as very thin surface courses for road construction and maintenance. They are a mixture of aggregates, sand and emulsion produced and laid in situ by a special machine. Until 1999, the bitumen used to produce the emulsion for micro-surfacing and slurry seal was a naphtenic type. Technical reasons were behind such choice (resistance of the mix to wear adhesion between the bitumen and the aggregates). Taking into account the development of these techniques in France and abroad, it became however necessary for Eurovia to look after new emulsion formulas based on other sources of bitumen.
The paper includes two parts:
1) the development and fine tuning of emulsion formulas in the laboratory (feasibility and mechanical performance).
2) industrial validation (production of the emulsion and application at job site).

KEYS WORDS : MICROSURFACING / EMULSIONS / PARAFENIC BITUMEN / NAPHTENIC BITUMEN

1. INTRODUCTION

Micro-surfacing is a bituminous material made from a mixture of aggregate, bitumen emulsion (modified or not), fiber, mineral additives (cement, etc.) and organic additives (dope).
This mixture is mixed and laid by a specially designed and adapted vehicle. After the laying and the breaking of the emulsion, this very thin micro-surfacing (10/13 mm) reaches its final consistency (rise in cohesion) very quickly.

The two essential parameters governing the formulation, mixing and laying of micro-surfacing are:

- The workability of the aggregate/emulsion mixture.
  Based on the reactivity of the aggregate (specific surface area, surface potential …), the designer seeks to optimize the various ingredients (water, additives, emulsion formula) to allow for a sufficient delay of workability.
- The kinetics of the "rise in cohesion".
  Within less than thirty minutes, the micro-surfacing must acquire sufficient cohesion to allow the re-opening to traffic.

For more than fifteen years now, EUROVIA has met these requirements using naphthenic bitumen-based emulsions.

In order to ensure supply security and to reduce micro-surfacing production costs, the company had however to develop new emulsion formulae to allow the use of other sources of road construction bitumen (paraffinic bitumens).

Two research approaches were available:
- Additivation of paraffinic bitumen. With this approach, it is not necessary to modify the formula of the aqueous phase of the emulsion and the nature of the mineral additive. These are important parameters to take into account because the emulsion production factories have limited storage capacity. This study began in 1998.
- Modification of the nature of the emulsifiers in parallel with the nature of the acid and/or additives to regulate the rise in pH of the system and the variation of the ionic power of the emulsifier as a function of the reactivity of the aggregate.

In this article we will develop only the formulae by additivation of bitumens.

2. BREAKING MECHANISM

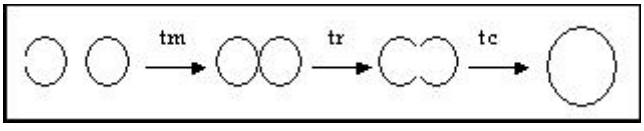
In general, in micro-surfacing, the emulsion/ aggregate breaking phenomenon can be summarized as follows:

Initially, the mixture of solids (aggregate, additive, fiber) in the presence of water will trigger a rise in the pH of the medium (surface hydrolysis). The use of a mineral additive (beyond a certain proportion) imposes a high pH and makes the system independent of the pH variation due to the aggregate.

During addition of the emulsion, various reactions come into competition:
1 The modification of the ionic power of the emulsifiers and of the polar components of the bitumen at the interface of the droplets, linked to the rise in pH and the modification of the ionic power of the medium (presence of Ca^{2+}, Na^{+} ions etc…).
2 The adsorption of free surfactant on the aggregates which modifies the difference of potential between the droplets of bitumen and the aggregate. This difference of potential will condition the adsorption of the bitumen globules on the aggregate and thus the quality of the coating and adhesion.
3 The coalescence among the droplets of bitumen. The kinetics of this reaction will condition the rapidity of the rise in cohesion of the micro-surfacing that may result in a sensitivity of the micro-surfacing to early aging conditions.

The kinetics of these reactions will depend on the emulsifier content of the emulsion, the additive content, the temperature and the moisture content.

All of these interactions lead to a more or less progressive modification, as a function of the aggregate and the water content, of the fluidity of the asphalt mix. The droplets of bitumen which are initially separated give the system fluidity and allow for easy laying using the specific micro-surfacing machines. The system is then viscous. The characteristic time during which this condition persists is called the workability time (tm).



The droplets of bitumen then coalesce and form a gel.
When all of the droplets of bitumen are grouped together, we consider that the emulsion has broken (breaking time: tr). The system is then viscoelastic. The system then tends to contract so as to reduce the area of contact between the water and the bitumen (cohesion time: tc).

This process involves kinetics which depend on the electrostatic repulsion between droplets and thus on the nature of the bitumen and the emulsifier.

The influence of the nature of the bitumen has been revealed through interfacial tension measurements (see bibliography B. Eckmann, JJ. Potti & B. Simaillaud). In basic media in particular we observe a significant difference linked to the ionization of polar compounds (carboxylic acids).

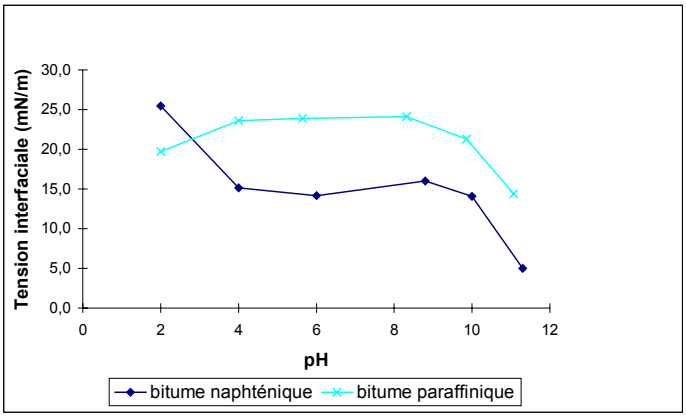


Figure 1 - Measurement of the interfacial tension of bitumen at various pH's for the Du Nouy ring method

3. LABORATORY CHARACTERIZATION TESTS

The laboratory tests used for the development of micro-surfacing formulae are:

- The hand mixing test, which is used to evaluate the compatibility between the aggregates, the emulsion and the breaking additives. This test can be used to determine the optimal proportions of the various ingredients so as to obtain a minimal mixing time.
- The breaking test which involves evaluating the time (tr) at the end of which the emulsion has completely broken on the aggregate. Blotting paper is applied to a slab of micro-surfacing over time with pressure of 0.2 MPa.
- The cohesion test (ASTM D-3910) done with a Benedict cohesivimeter. This test allows us to evaluate the speed of curing of micro-surfacing by monitoring the change in cohesion over time, with measurements made at 5, 10, 15, 20, 30 and 60 minutes. We thereby define the time (consolidation time) for which the couple measured becomes greater than 20 kg.cm. This time allows us to set a minimum time for opening the pavement to traffic without risk for the surface.
- The modified WTAT (Wet Track Abrasion Test). The modifications to the standard WTAT test (ASTM D-3910) concern essentially the conditions for curing of test pieces in the climatic chamber. The purpose of this test is to evaluate the influence of the

curing conditions (temperature, moisture content) on the mechanical properties at a young age of the micro-surfacing. This test can be done at three temperatures (10, 18 or 30°C) and at two different humidity levels (55 and 100%). Internal specifications correlated with the site allow us to differentiate and validate the formulae (4).

4. FORMULATION OF PARAFFINIC BITUMEN BASED MICROSURFACING

4.1. Ingredients

In developing the emulsion formula, we initially worked with the reference aggregate: Micro-diorite from the Kleber Moreau quarry.

The basic microsurfacing formula is:

Aggregate 0/6	100 %
Added water	9 pph
Emulsion (60 %)	12.4 pph
Mineral additive	0.5 pph

4.2. Influence of the nature of the bitumen

In order to see the influence of the bitumen on the properties of the micro-surfacing, we formulated a paraffinic bitumen-based emulsion made with the same aqueous phase as the reference emulsion (naphthenic bitumen).

Emulsion	reference "naphthenic" Emulsifier content: 9kg/t	"paraffinic" Emulsifier content: 9kg/t	**Internal Specifications**
Workability time (s)	105	> 120	**> 90 s**
Breaking time (min)	5	>120	**< 20 min**
Consolidation time (min)	15	>120	**< 30 min**
WTAT			
% loss (18°C, 55 %)	2.4	13	**< 5 %**
% loss (18°C, 100 %)	14.8	100	**< 25 %**

4.3. Addition of additive to bitumen/ adaptation of emulsifier content

During emulsification, the droplets of bitumen collect emulsifier molecules which creates electrostatic repulsion and stabilizes the emulsion. The surface of the droplets is thus composed of emulsifier and surfactant molecules from the bitumen. This distribution will depend on the nature of the bitumen and the ionic power of the droplets on the pH of the medium.
In order to simulate the anionic power of the components of the naphthenic bitumen at the pH of the micro-surfacing (imposed by the mineral additive) we studied the possibility of adding various molecules to the paraffinic bitumen. Taking into account the economic and safety constraints, we finally chose one particular additive. In order to evaluate the influence of this product on the properties of the micro-surfacing, a series of emulsions were made by simultaneously varying the emulsifier and additive contents of the bitumen. Measurement of the feasibility time (workability and breaking time) allowed us to evaluate the influence of the additive on the kinetics of the emulsion breaking (figures 2 and 3).

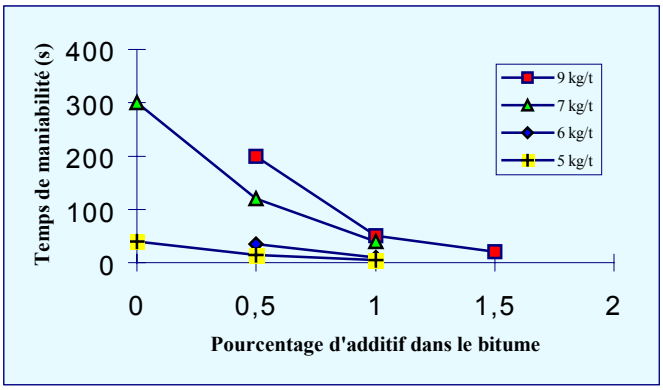


Figure 2 - Influence of additive content and emulsifier concentration on the workability time

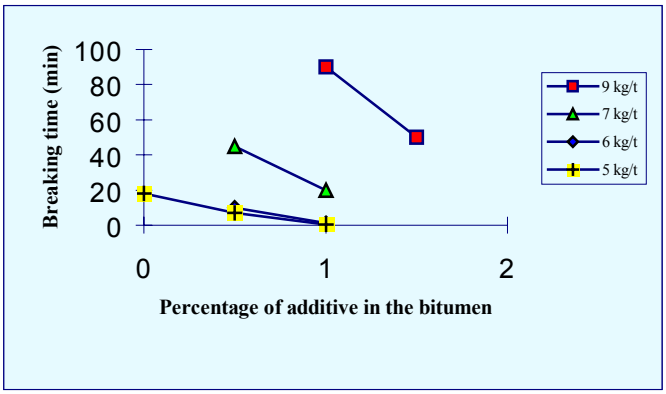


Figure 3 - Influence of additive content and emulsifier concentration on the breaking time

Formulae containing between 6 and 7 kg/t of emulsifier with 0.5 to 0.8 % additive in the bitumen give results in accordance with the specifications.

In order to evaluate the mechanical properties and to optimize the emulsion formula, WTAT tests (18°C, 100 % relative humidity) were done to evaluate the kinetics of the rise in cohesion and the sensitivity of the micro-surfacing at a young age (figure 4).

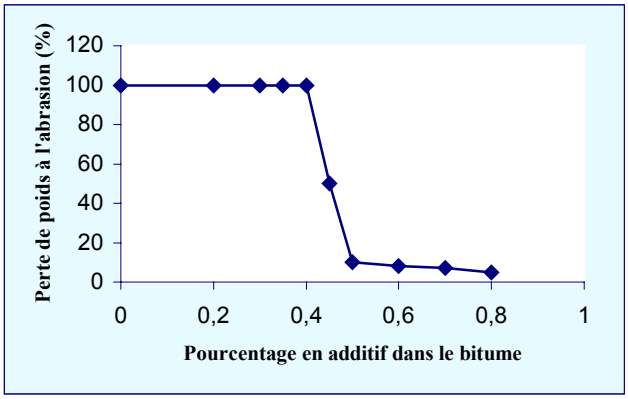


Figure 4 - Influence of additive content on the mechanical properties

Several emulsions containing 6.5 kg/t of emulsifier were made with different additive content levels (0.1 and 0.8%) in the bitumen.

Beyond 0.5 % additive (figure 4), the percentage loss is in accordance with specifications. A good technical/economic compromise seems possible with a formula containing 0.6 % additive in the bitumen and 6.5 kg/t of emulsifier.

This formula was validated with four paraffinic bitumens of various origins and with aggregates of various petrographic natures.

5. INDUSTRIAL VALIDATION

Based on the results obtained at the Dourdan Research Center, the industrial validation was done in various steps:

- making of the additive-containing binder
- making of the emulsion
- making and laying of the micro-surfacing

5.1. Making of the additivated binder

The reference bitumen commonly used in emulsions for micro-surfacing is a naphthenic bitumen modified with EVA made according to the industrial process illustrated by figure 5.

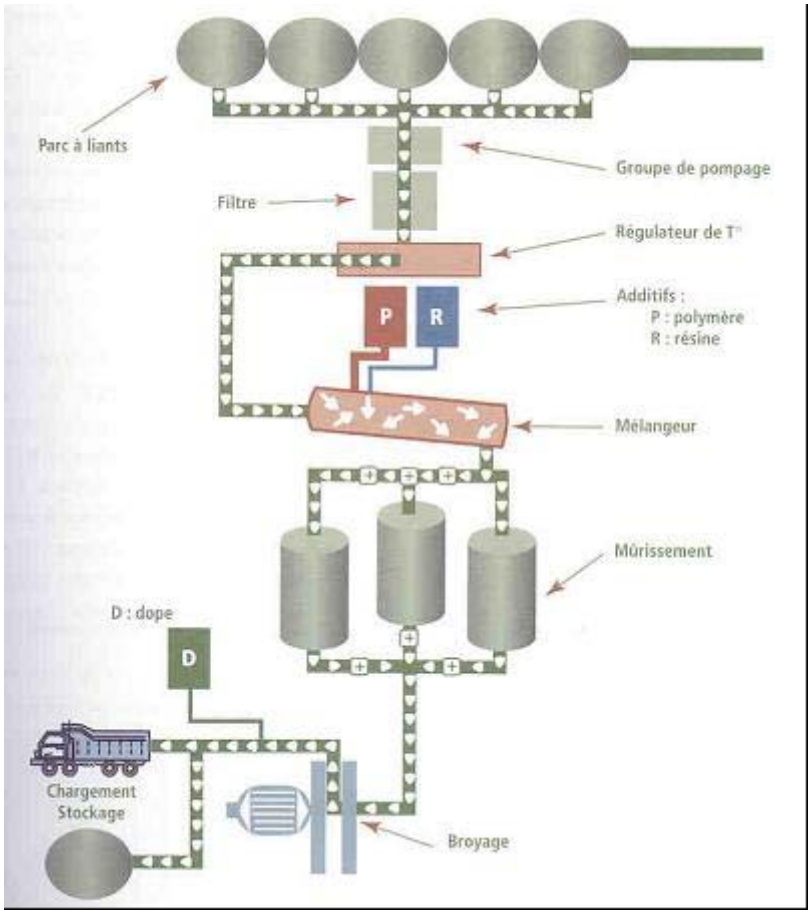


Figure 5 - diagram of the industrial process

The bitumen tested is thus paraffinic bitumen, commonly used by the road construction industry (grade 70/100, modified with EVA). During the process of making the modified binder, the additive is added to the maturation tank.

The quantity of additive is calculated very precisely with respect to the modified binder. The temperature of the additive-containing bitumen is 165°C. After 20 minutes of curing, the additive-containing bitumen goes through the grinder before being stored. Samples were taken and analyzed by the Research Center, proving the conformity of the product with the study.

5.2 - Making of the emulsion

The emulsion is made with the usual process and does not lead to any particular problem. Conformity analyses are done on the manufactured emulsion before it goes to the sites.

5.3 - Mixing and laying of micro-surfacing

More than 500,000 m² of micro-surfacing have been made from an emulsion with a paraffin-based bitumen modified with EVA and containing the additive. Several types of aggregate have been used.

The site observations, comparing with the micro-surfacing with naphthenic bitumen-based emulsion, were the following:

- the asphalt mixes made from paraffinic bitumen generally require less emulsifier for the workability times indicated,
- the appearance of this micro-surfacing at a young age is different and lighter,
- The breaking time of the paraffinic bitumen-based micro-surfacing is longer and the mixture is more sensitive to water,
- the consolidation time and the rise in cohesion depend on the aggregate and the temperature. For a temperature greater than or equal to 20°C, the time before reopening to traffic is less than one hour. Once it breaks, the micro-surfacing is well consolidated and no rejection of chippings is observed,
- for paraffinic bitumen based micro-surfacing, the use of a compactor is recommended to promote cohesion.

6. CONCLUSION

Under good laying conditions (temperature, humidity …), the additivation of paraffinic bitumen allows us to make micro-surfacing with different kinds of aggregates. These formulae will allow us to diversify the bitumen supply sources. Additional tests in late autumn (temperature < 15°C…) would seem to be necessary to adjust the formula in order to achieve more rapid cohesion.

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