

## DURABILITY OF A BITUMEN IN A HOT MIX ASPHALT: CONSEQUENCES OF OVER-HEATING AT THE MIXING PLANT

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### ABSTRACT

*A bitumen involved in hot mix manufacture undergoes two types of aging: first, short-term aging (building aging) during mixing step, simulated in laboratory by RTFOT, and then, long-term (in-situ) aging, simulated in laboratory by PAV.*

*During both steps, bitumen tends to harden, due to light fractions evaporation and chemical composition evolution.*

*In a first approach, the evolution in chemical composition, and hence in mechanical characteristics, is linked with the intrinsic bitumen sensitivity to aging. However, further analysis shows that bitumen durability depends not only on its sensitivity to aging, but also on initial aging undergone during mixing step.*

*A study has been carried out on a series of bitumens, belonging to the same grade but with distinct sensitivities to aging. The study included classical tests, and further analysis by chromatographic methods. For each bitumen, an overheating during mixing step was simulated by performing RTFOT at 230°C instead of 163°C. The characteristics obtained on overly RTFOT-aged bitumens were compared with those obtained after RTFOT at 163°C. Then, all the binders were aged by PAV (simulation of long-term aging: several years in situ).*

*For all bitumens analysed, aging due to RTFOT at 230°C (overheating in the mixing plant) proved to be stronger than aging due to RTFOT at 163°C + PAV (normal temperature in the mixing plant + several years in situ).*

*The consequences of overheating in mixing plant on bitumen durability, in relationship with bitumen sensitivity to aging, have been highlighted and explained by chemical analysis.*

**Keywords:** binder, chemical composition, aging, durability

## 1. INTRODUCTION

Road construction requires the use of bituminous binders in a variety of techniques, which include the laying of mixes that have been manufactured in a mixing plant. Although this way of using bitumen has been known and thoroughly mastered for several decades, it is the permanent subject of investigations in order to improve its implementation. Progress relates, in particular, to limiting its environmental impact.

In this context, investigations were undertaken regarding the phenomena which govern the manner in which bitumen changes during mix manufacture and laying but also throughout its service life. Improving knowledge in this area will help select the most appropriate bitumen for the intended use, specify the optimum conditions for its use, and identify the parameters for which particular caution is necessary.

This will enable to optimize the durability of bituminous mixes and thereby limit the amount of subsequent maintenance and reinstatement work.

These studies involve the use of various physicochemical methods for characterizing bitumens. Some frequently used methods are covered by normative specifications. But experience shows that just because a bitumen complies with the specifications does not mean that the manufactured mix will be durable. It is therefore necessary to extend the laboratory characterization beyond the standardized tests.

A study was conducted on three bitumens of different types. The aim was to monitor change in the chemical composition under normal and exceptional conditions of use. The impact of a change in conditions of use on the durability a bitumen will vary according to its sensitivity to ageing.

## 2. SOME GENERAL REMARKS ON BITUMENS

### 2.1. The causes of binder ageing

When a mix is manufactured, the bitumen undergoes mixing with the aggregate. The temperature selected for this is generally between 150°C and 180°C and depends on the binder, whose viscosity should be between  $0.17 \pm 0.02$  Pa.s.

When a continuously renewed very thin film of bitumen (of the order of a few  $\mu\text{m}$ ) is heated to high temperature, oxidation occurs. This is known as rapid ageing, or "construction" ageing.

When the road surfacing has been laid, bitumen continues to change throughout the service life of the mix. This so-called "in-service" ageing occurs relatively slowly over a number of years. When the mix's limits of durability have been reached, it is susceptible to various types of distress, for example cracking.

In the specific case of the bitumen, the end of the construction ageing stage marks the start of in-service ageing. In order to guarantee the mix's durability, it is therefore important to limit, as far as possible, the impact of construction ageing on the properties of the bitumen and therefore on its chemical composition.

### 2.2. Simulation of two types of bitumen ageing in a laboratory

Construction ageing, as it occurs in a mixing plant during the 20 seconds of so-called "moist" mixing (when the bitumen comes into contact with the aggregate), is simulated in a laboratory by the RTFOT (Rolling Thin Film Oven Test), as described in the standard EN 12607-1. In this test, the bitumen is maintained for 75 minutes in phials at 163°C, in a continuously renewed thin film, in a flow of compressed air (4000 ml/min).

The characteristics of bitumen with regard to penetration (EN 1426) and R&B softening point (EN 1427) after this treatment are covered by specifications described in the standards EN 12591 (specifications for paving grade bitumens) and EN 13924 (Specifications for hard paving grade bitumens). However, merely conforming to these specifications does not guarantee satisfactory resistance to ageing.

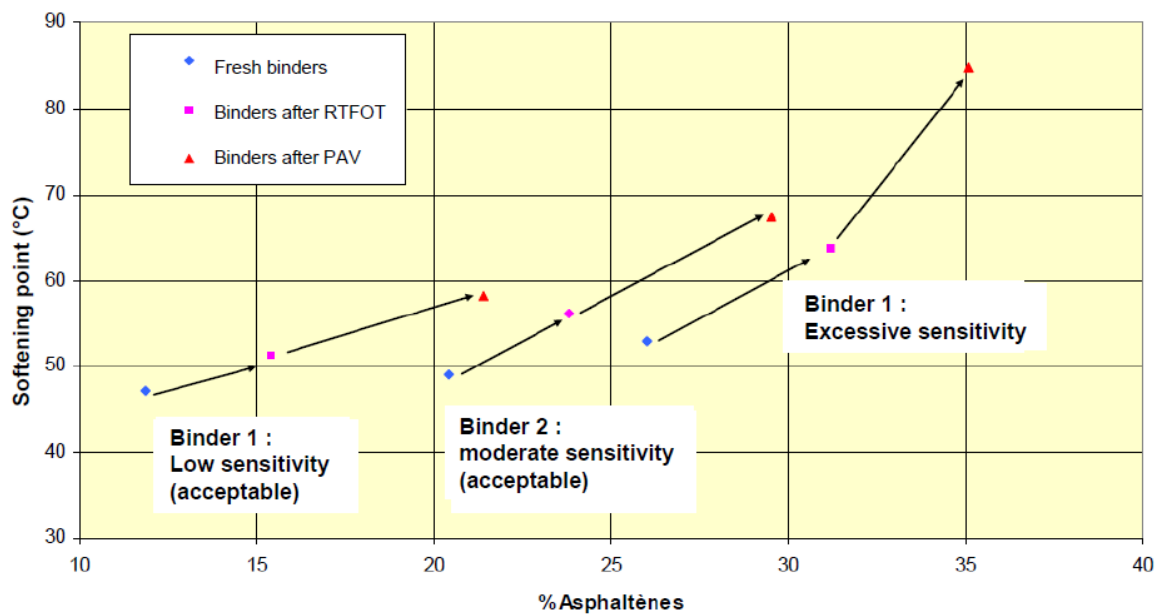
Moreover, in order to be able to predict how a bitumen will change in the years following laying of the mixture, it is necessary to simulate its ageing using an accelerated method. PAV ageing (Pressure Ageing Vessel) as described in EN 14769 simulates several years of in-situ ageing. The bitumen obtained after RTFOT ageing is placed in trays and kept at 100°C at a pressure of 2.1 MPa for 20 hours.

No normative specifications apply to the characteristics of the bitumens after PAV ageing. The sensitivity of the bitumen to ageing is therefore assessed by comparing these test results with those from previous studies.

This evaluation can be refined by combining the two ageing methods, i.e. by monitoring the change in the characteristics of the bitumen during the different stages of its use: in the fresh state, after RTFOT ageing and after RTFOT + PAV ageing.

In particular, it is possible to make an initial classification by determining the softening point as specified in EN 1427 and the asphaltene content (defined as the fraction of the bitumen which is insoluble in heptane).

Figure 1 shows examples of bitumens with acceptable sensitivity to ageing (binder 1 and 2) or excessive sensitivity to ageing (binder 3).



**Figure 1 : Examples of change in the characteristics of bitumens during ageing.**

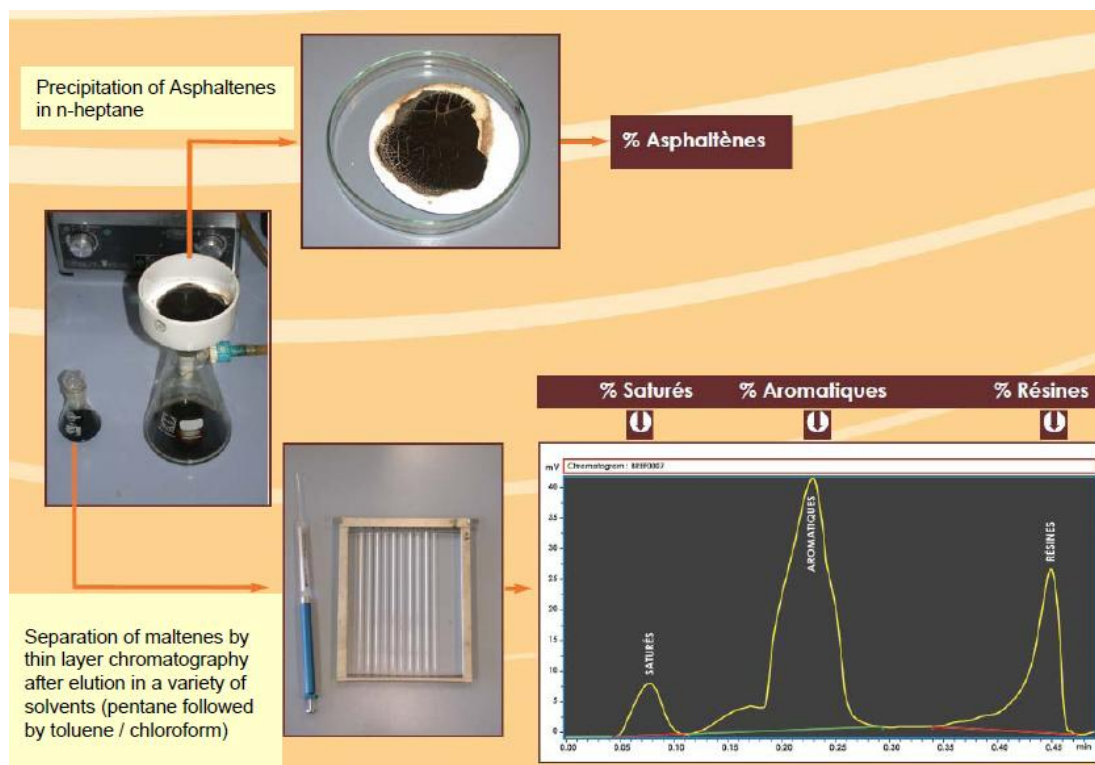
These results show that the sensitivity of bitumen to ageing is linked, amongst other things to the asphaltene content measured in the fresh binder.

But this criterion is not always sufficient: two bitumens with identical asphaltene contents may have very different ageing sensitivities. The chemical composition of bitumens must therefore be considered comprehensively.

The reason for this is that bitumens are made up of 4 main types of hydrocarbon: asphaltenes, resins, aromatics and saturates.

SARA analysis of a bitumen is determined in two stages as shown in Figure 2:

- First, the bitumen is dissolved in heptane. The insoluble fraction corresponds to the asphaltenes. The asphaltene content is determined by weighing the filter after it has been dried in an oven.
- Next, the heptane solution that contains the maltenes (aromatics + resins + saturates) is analyzed by Iatroscan chromatography in order to determine the proportions of the different constituents.

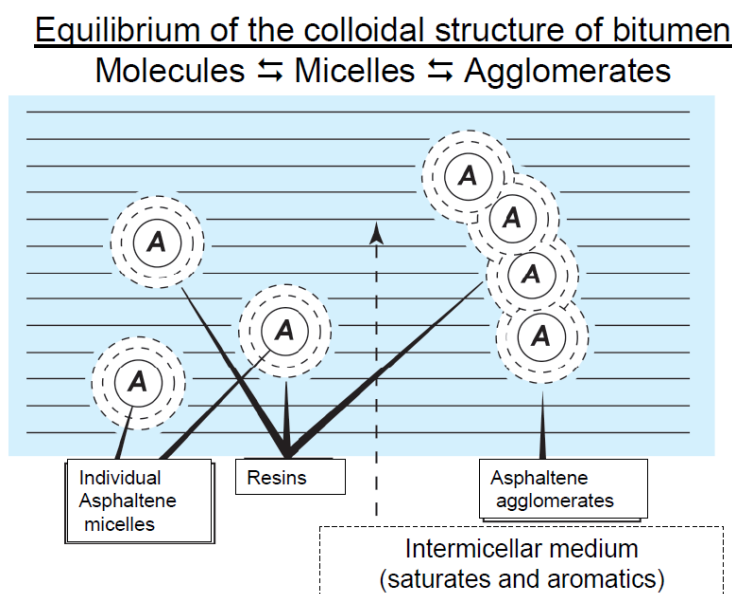


**Figure 2 : determination of the SARA composition of a bitumen by Iatroscan chromatography**

When the proportions of the 4 types of hydrocarbon have been determined, it is possible to calculate the colloidal index (C.I.) with the following formula:

$$C.I. = \frac{\% \text{Asphaltenes} + \% \text{Saturates}}{\% \text{Resins} + \% \text{Aromatics}}$$

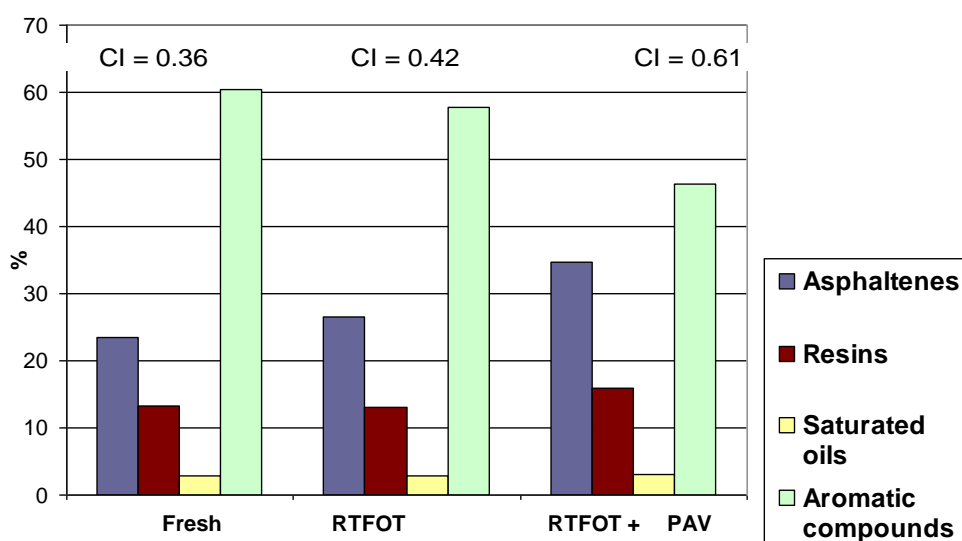
In broad terms, the structure of a bitumen is generally considered to consist of asphaltenes which are dispersed in aromatic and saturates, with resins acting as a “compatibility” agent. The suggested colloidal structure is presented in Figure 3.



**Figure 3: Colloidal structure of bitumen**

When bitumen ages, the aromatics are oxidized to resins which in turn oxidize to asphaltenes. So logically, when the chemical composition of a bitumen is monitored during ageing, there is therefore an increase in its asphaltene content and a reduction in its aromatic content. This change results, in particular, in an increase in the colloidal index.

Figure 4 presents an example of this type of change.



**Figure 4: Change in the chemical composition of bitumen during ageing**

The level of the change in the chemical composition depends on the sensitivity of the bitumen to ageing and the conditions to which it is subjected. This will affect the durability of the mixture.

### 3. CASE STUDY: COMPARISON BETWEEN DIFFERENT AGEING REGIMES.

The aim of the study was to evaluate the impact of overheating in the mixing plant on the state of ageing of the binder immediately after coating and the subsequent impact of this ageing on its durability.

This is important because although manufacturing temperatures in mixing plants are generally well controlled, they may occasionally be higher than the recommended temperatures. In this case, the bitumen undergoes higher levels of oxidation.

In order to investigate the effects of this situation, a study was conducted by carrying out RTFOT ageing at two different temperatures, 163°C, which is close to the temperature at which bitumen is normally used in hot mixes and 230°C, to simulate overheating.

Three 10/20 pen bitumens were considered. These exhibited different sensitivities to ageing, nevertheless all were classed as acceptable on the basis of the graph presented in Figure 1 (section II.2).

- bitumen A : low sensitivity
- bitumen B : moderate sensitivity
- bitumen C : high sensitivity

Each bitumen was characterized after being subjected to a variety of ageing regimes, each one simulating a given situation, as shown in the table below :

Fresh	Before coating
After RTFOT at 163°C	After coating – normal conditions
After RTFOT at 163°C + PAV	After coating (normal conditions) then several years of ageing in-situ
After RTFOT at 230°C	After coating - overheating
After RTFOT at 230°C + PAV.	After coating – overheating followed by several years of ageing in-situ

The following characteristics were determined after each of these ageing regimes: penetration according to EN 1426, softening point according to EN 1427 and Iatroscan chromatography SARA analysis as described above.

First of all, penetration values were used for the comparison of aging index for the 3 bitumens, calculated after each aging step as follows :

$$\text{Short Term Ageing Index} : \frac{\text{Pen (fresh)}}{\text{Pen (RTFOT)}}$$

$$\text{Long-Term Ageing Index} : \frac{\text{Pen (RTFOT)}}{\text{Pen (RTFOT + PAV)}}$$

Where Pen(fresh) = penetration at 25°C determined on fresh bitumen

Pen(RTFOT) = penetration at 25°C determined on bitumen after RTFOT ageing

Pen (RTFOT + PAV) = penetration at 25°C determined on bitumen after RTFOT + PAV ageing

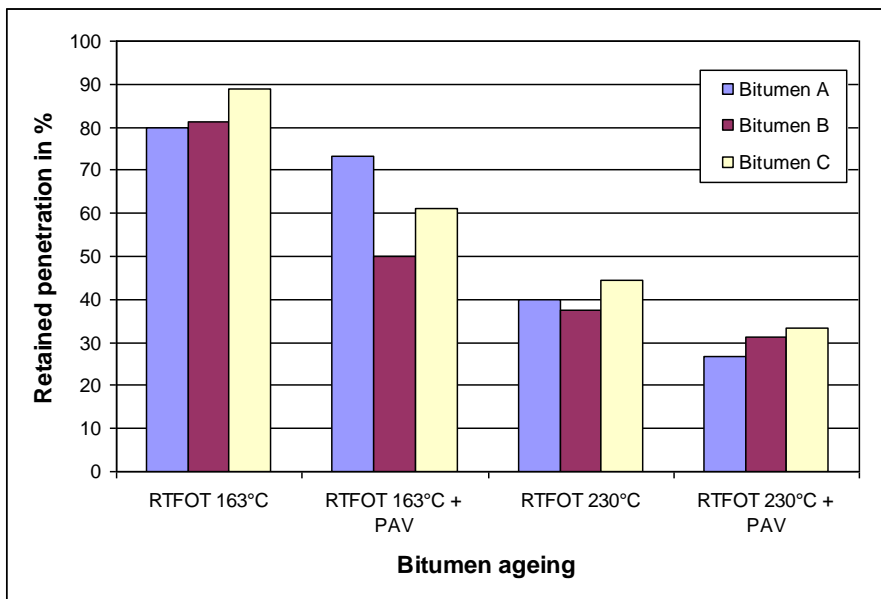
The results are gathered in the table below.

Aging index	Bitumen A	Bitumen B	Bitumen C
Short-term during mixing at 163°C	1,3	1,2	1,1
Long-term after mixing at 163°C	1,1	1,6	1,5
Short-term during mixing at 230°C	2,5	2,7	2,3
Long-term after mixing at 230°C	1,5	1,2	1,3

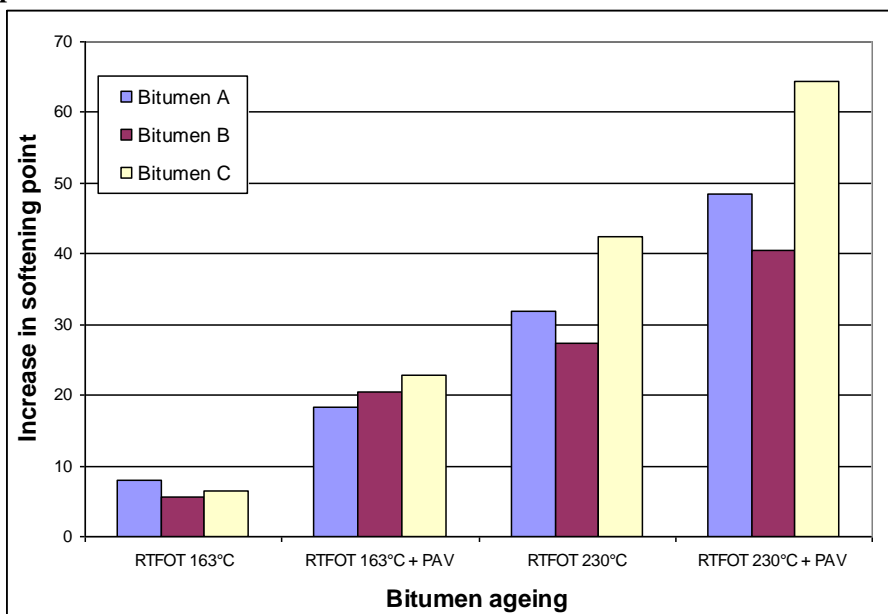
The influence of over-heating during mixing step (i.e during RTFOT ageing) is clearly shown, on a general point of view ; but these results do not reveal any strong differences between the three bitumens.

Therefore the results processing was carried on.

The graphs in Figures 5 and 6 show the change in the penetration (as a percent of initial value) and softening point (as an increase compared to initial value) for the different bitumens.



**Figure 5: Retained penetration (as a percentage of the penetration of the fresh binder), after the different ageing processes**



**Figure 6: increase in TR&B (compared with the TR&B of fresh binder), after the different ageing processes**

Irrespective of the sensitivity of the bitumen to ageing, the RTFOT alone, when conducted at 230°C, caused a greater change than the RTFOT at 163°C followed by PAV ageing. Transposed in the field, it means that the bitumen which is subjected to this type of overheating in the mixing plant is laid in a more advanced state of ageing than if it had been coated under normal conditions followed by several years of in-situ ageing.

However, the ageing process does not seem to be governed by the same mechanism in the case of these three bitumens. Overheating seems to have a more marked effect on the bitumen which was intrinsically sensitive to ageing.

In order to understand better the differences between these three bitumens, investigations were continued with Iatroscan chromatography SARA analysis of each bitumen at the different stages of ageing.

The graphs below present the change in the Iatroscan chemical composition and the colloidal index of each bitumen: bitumen A (Figure 7), bitumen B (Figure 8), and bitumen C (Figure 9).

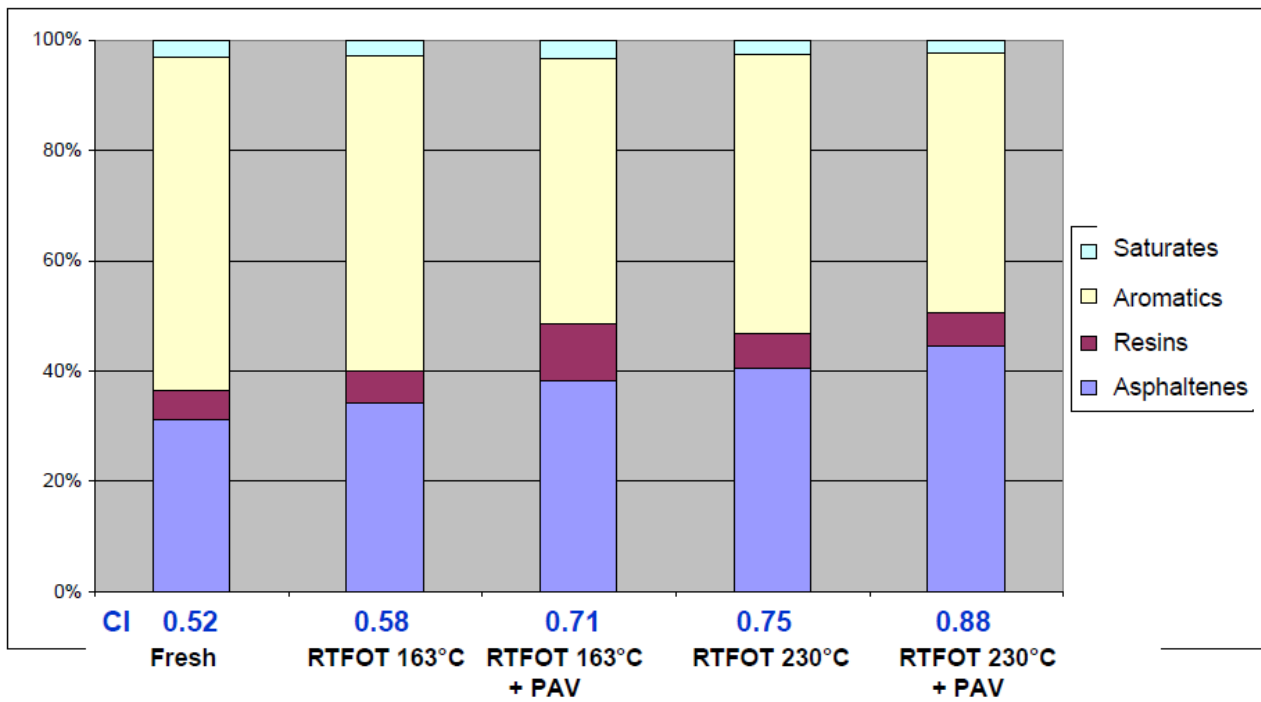


Figure 7 : Bitumen A

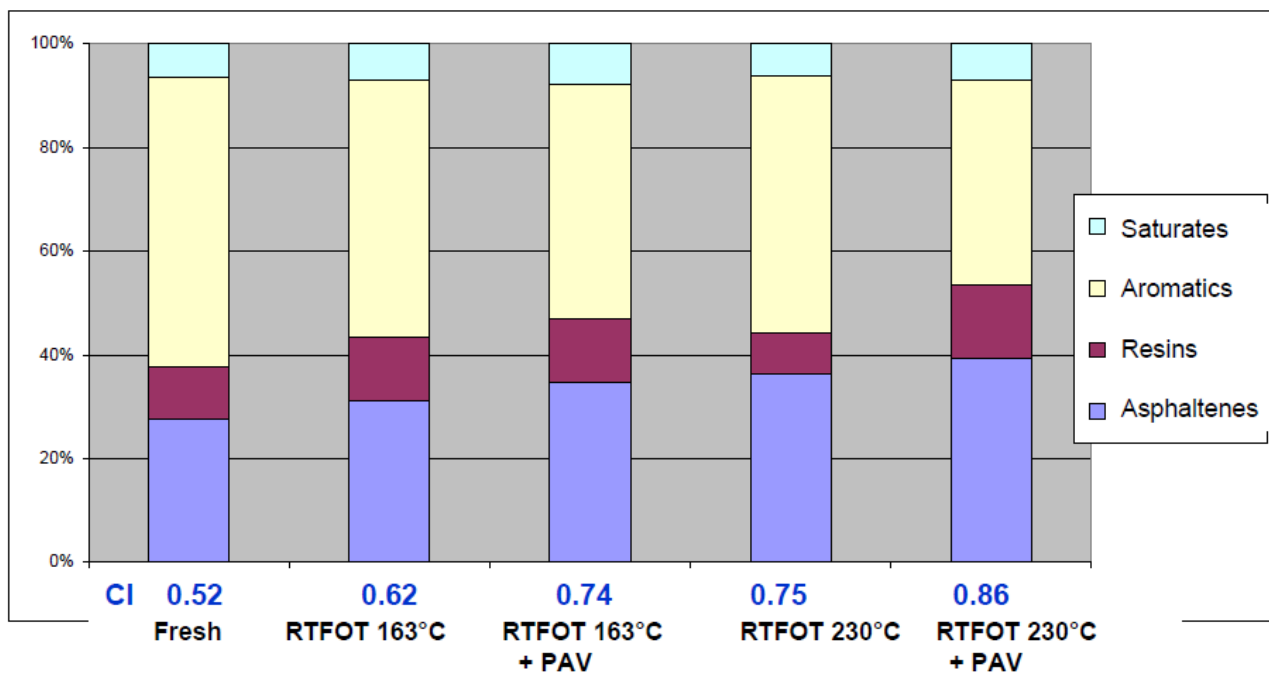
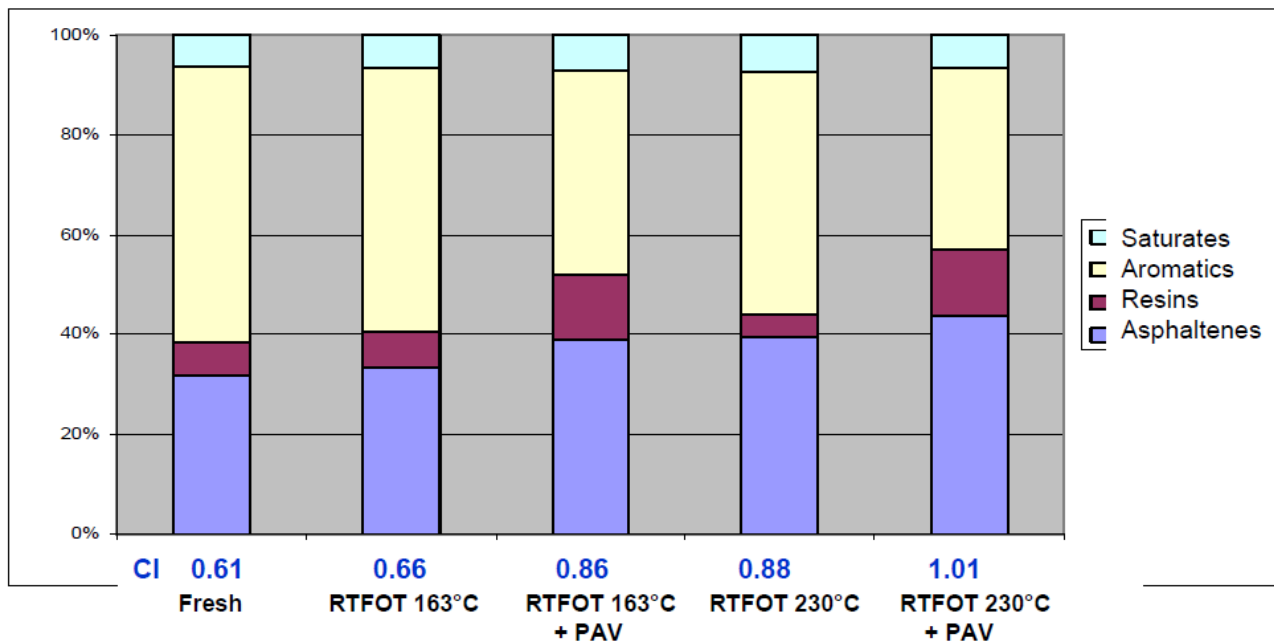


Figure n°8 : Bitumen B



**Figure n° 9 : Bitumen C**

These results confirmed the conclusions reached after the penetration and R&B softening point test: RTFOT ageing at 230°C leads to more pronounced ageing than RTFOT at 163°C followed by a PAV test. However, the change varies according to the bitumen.

As mentioned above, the ageing of bitumen leads to a change in its chemical composition  
aromatics → Resins → Asphaltenes.

As one would expect, the aromatic content diminishes and the asphaltene content increases. The change in resin content, cannot be easily predicted as this transitional product depends on the relative kinetic of both stages:

aromatics → resins  
resins → asphaltenes.

If we consider the changes that took place under the conditions of “normal” coating for the three bitumens, we can see that the percentage of resin tends to increase during ageing. The transformation of resins into asphaltenes is therefore slower than the transformation of aromatics into resins.

Under the conditions which represent overheating in the mixing plants (RTFOT at 230°C), differences are apparent between bitumen A and bitumens B and C.

In the case of bitumen A, the resin content increased during ageing, as under “normal” coating conditions: overheating during coating definitely increased the rate of ageing, but the same mechanisms nevertheless seem to apply.

For bitumens B and C, the resin content fell after RTFOT ageing at 230°C.

Thus, the transformation of resins into asphaltenes is faster than the transformation of aromatics into resins, which is the opposite to what was observed during RTFOT ageing at 163°C.

The considerable increase in temperature therefore seems to have modified the mechanism that governs the ageing of the bitumens, leading, in particular, to an acceleration in asphaltene formation.

Ultimately, the impact of overheating in a mixing plant was moderate in the case of bitumen A, which had low sensitivity to ageing, but was much more marked in the case of bitumens B and C whose intrinsic sensitivity to ageing was greater.

#### 4. CONCLUSION

It is important under all circumstances to comply with the recommended temperatures, but is particularly crucial in the case of bitumens which are intrinsically sensitive to ageing.

However sensitive a bitumen is to ageing, the RTFOT at 230°C leads to more pronounced ageing than the RTFOT at 163°C followed by a PAV test. Transposed in the field, it means that the bitumen which is subjected to this type of overheating in the mixing plant is laid in a more advanced state of ageing than if it had been coated under normal conditions followed by several years of in-situ ageing.



If the bitumen has a low intrinsic sensitivity to ageing, overheating during coating has a moderate effect on the durability of the mixture. The ageing of the bitumen after coating is greater, but ageing is still governed by the same mechanisms that were at work during coating under normal conditions.

However, in the case of a bitumen with a greater sensitivity to ageing, overheating during coating leads, in addition to more marked ageing, to a change in the mechanisms of ageing. The resulting acceleration in asphaltene formation may have an extremely adverse impact on the durability of the mixture.

This study was carried out on hard bitumens, mainly used for base courses ; the same study, carried out on softer bitumens, would be likely to lead to equivalent conclusions, as bitumen ageing mechanisms are likely to be the same.