Realistic laboratory curing of bituminous mixtures

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ABSTRACT. *In order to perform some specific testing, road laboratories apply a preliminary thermal treatment that impacts the asphalt mixture mechanical and intrinsic properties. The current standardisation in Switzerland and in Europe does not sufficiently describe the procedures of thermal ageing and this leads to great variability in different test results. The present study allowed analysing the impact of thermal ageing and to establish, verify and calibrate a new reheating methodology.*

KEYWORDS: *thermal ageing, laboratory test, reheating methodology, asphalt mixture*

1. Introduction

Road laboratories perform many tests to characterise and evaluate the performances of bituminous mixtures produced in laboratory or taken in situ. For some cases, specimen preparation requires a reheating treatment in order to obtain predefined thermal conditions. Thermal ageing induces bitumen oxidation which, depending on bitumen generic composition and asphalt richness modulus, affects the mechanical and intrinsic properties. When asphalt pavements are submitted to various thermal conditioning, one can note, in some cases, that their properties are significantly different.

Some interlaboratory tests carried out in Switzerland by more than 30 laboratories (ROBIN Association) have shown that, the variations in thermal treatment conditions significantly affect the results and a large dispersion in the reproducibility has been measured. Reheating procedures for bituminous mixtures tests in laboratories are not exhaustively described in Swiss and European standards. Therefore, it is necessary to harmonise tests procedures in order to improve laboratory tests repeatability and reproducibility.

The traffic Facilities Laboratory (Lavoc) from the Swiss Federal Institute of Technology (EPFL) has been dealing with thermal ageing issues for more than a decade. A preliminary internal study highlighted the various parameters affecting the thermal ageing process and permitted to set the frameworks conditions for laboratory testing (laboratory accreditation and research purpose).

Based on these preliminary results, a research mandate financed by the Swiss Federal Roads Office (FEDRO, Switzerland) and led by Lavoc, allowed developing a new thermal reheating methodology that has been calibrated and checked. The aim being also to reach a better match between characteristics of bituminous mixtures produced in laboratories and those obtained in bituminous mixing plant.

The findings of this study, discussed within this paper, have been applied satisfactorily in road laboratories and are expected to give inputs for existing standards and/or for the establishment of new standardisation.

2. Previous experience with laboratory curing

The effects of the use of the microwave oven in order to dry aggregates have been highlighted by Lavoc in a previous study about Los Angeles test[1]. This study demonstrated that the uses of microwave oven have no contraindications in case of non-porous aggregates and non-ferrous materials.

Based on this, the new methodology proposed in the present study dealing with thermal ageing of bituminous mixtures will consequently focus on the use of microwave oven and the thermoregulated enclosure (traditional oven).

3. Exploratory study

The parameters involved in the evolution of thermal ageing as well as boundary conditions were described in a preliminary study that is based on several experiments.

3.1. *Mix design*

The selected asphalt mixture for the set of experiments is an AB 11S (comparable to AC 11S, max. aggregate size 11 mm) with a grading curve located in the middle of the envelope. The binder content is 5.30%/mass and Marshall voids 4%.

The aggregates used for the various mixtures come from Gasperini quarry (Altdorf, Switzerland), are sand-limestone in nature with blunt edges formed by the tertiary gyratory crusher. This increased the importance of the bituminous binder on the mechanical behaviour. It should be pointed out that in-depth study, calibration, verification and validation have been realised with aggregates with sharp edges whose behaviour on bituminous mixture generates a less marked effect of the bituminous binder.

3.2. Experimentation with different reheating rates

Three different experimentations have been performed to compare bituminous mixtures ageing rates based on the analysis of bituminous binder characteristics and mechanical Marshall properties.

[1] VSS research report number 180, 9/86. “Actualisation de l’essai Los Angeles”.

In total, 7 non-modified bituminous binders and polymer-modified bitumen (PmB) were selected for an analysis at different ageing time (at origin, RTFOT[2] aged, with different reheating times). The retained bituminous binders are:

– B80/100 straight-run bitumen (Feyzin, France)
– B55/70 straight-run bitumen (Feyzin, France)
– B80/100 semi-blown bitumen (Cressier, Switzerland)
– B55/70 semi-blown bitumen (Cressier, Switzerland)
– PmB B80/100 SBS grafted
– PmB B80/100 SBS two-phase
– PmB grafted (Styrelf 13/80)

A first experiment compares reheating impact on two batches for a bitumen non-modified B80/100: one reheated in the microwave oven, the other one in a traditional oven.

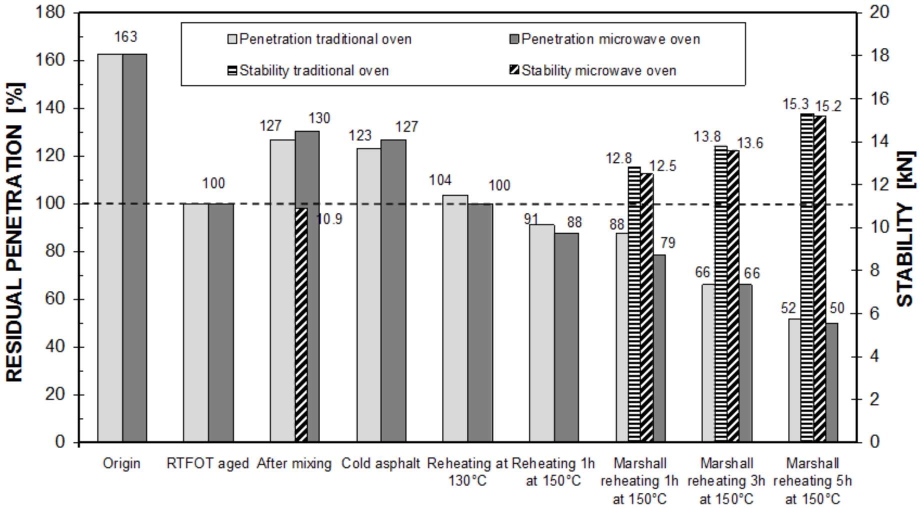

Figure 1. *Reheating impact. AB 11S, B80/100 Cressier. Microwave oven/Traditional oven. Collected bituminous binder, residual penetration at 25°C relative to the RTFOT penetration.*

After mixing and immediate binder recovery (on cold asphalt), the thermal ageing rate is low (Residual penetration: ~127%, ring-and-ball temperature: ~51°C). RTFOT penetration grades are obtained only after a thermal reheating at 130°C.

[2] SN 670516a, EN12607-1: 2007: Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air – Part 1: RTFOT method

After 3 to 5 hours thermal reheating at 150°C, particularly for the batch reheated in traditional oven, one can note significant changes in the bituminous binder ageing with a substantial increase of bituminous hardness (penetration grade). As Marshall stability increases constantly, the evolution of the flow is not significant.

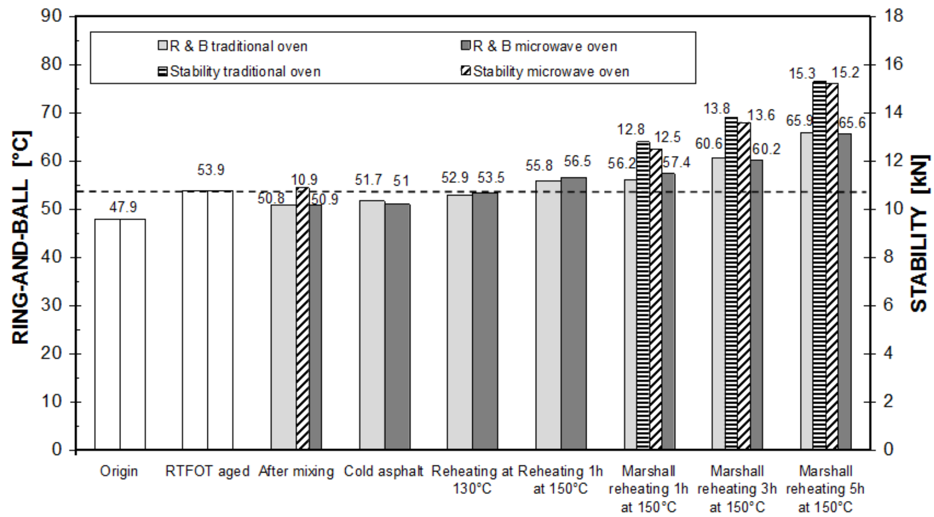

Figure 2. *Reheating impact. AB 11S, B80/100 Cressier. Microwave oven/Traditional oven. Collected bituminous binder, variation of ring-and-ball temperature.*

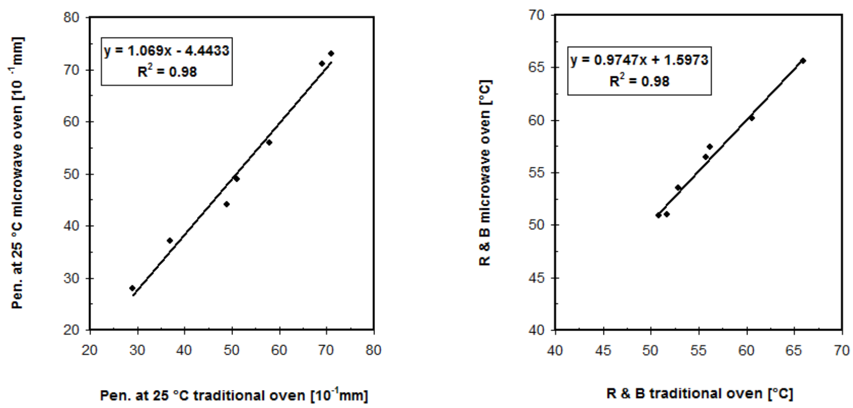

Figure 3. *Correlation for penetration at 25°C and ring-and-ball temperature. Microwave oven/Traditional oven.*

Considering the results obtained in this first experiment (Figures 1 and 2), one can note that the RTFOT ageing rate can be reached only after a thermal treatment (~130°C) on cold asphalt mixture. On the other hand, the negative impact of prolonged reheating, in particular in temperature-controlled oven, has been highlighted by the laboratory testing results: Marshall stability increases up to 50% of “after mixing value” and penetration decreases up to 50% of “RTFOT aged value” after 5 hours conditioning at 150°C. The various characteristics present a high concordance between microwave oven use and traditional oven use (Figure 3).

The other two experiments performed aimed at comparing different mixtures with various ageing time.

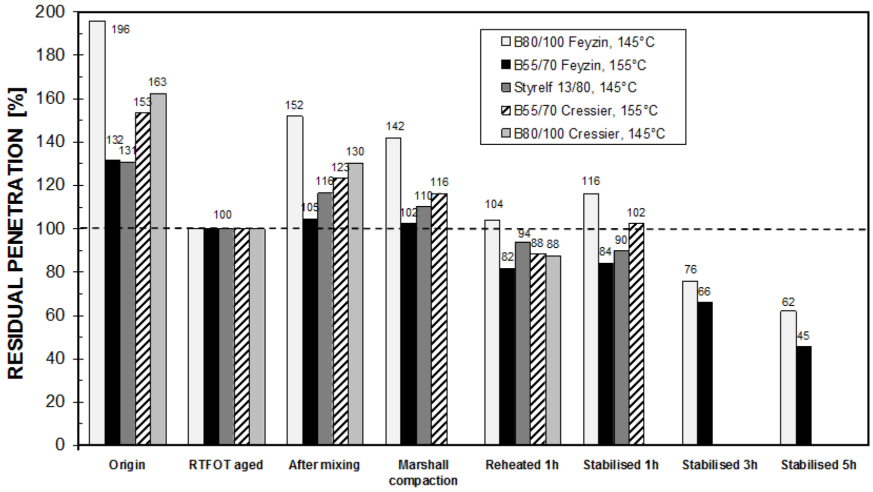

Figure 4. *Reheating impact. Collected bituminous binders. Residual penetration at 25°C relative to the RTFOT penetration. 2nd experiment.*

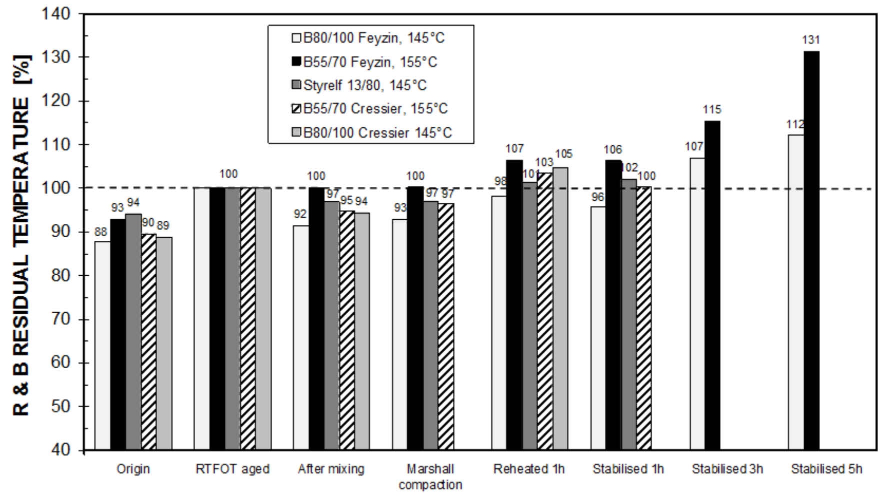

Figure 5. *Reheating impact. Collected bituminous binders. Residual ring-and-ball temperature relative to RTFOT ring-and-ball temperature. 2nd experiment.*

Except for the B55/70 straight-run bitumen, the RTFOT ageing rate has not been reached after mixing or immediate Marshall compaction. On the other hand, reheated bituminous mixtures or stabilised bituminous asphalt behaviour are quite similar. The ideal curing time seems to be located just below 60 minutes.

Differences between bituminous mixtures behaviour are likely due to the bitumen distinct generic composition and to the richness modulus of the asphalt mixture. Once again, the negative impact of prolonged reheating has been highlighted.

For the third experiment, a reheating time of 40-60 minutes has been analysed; this time gap corresponds to the necessary time interval to achieve Marshall compaction.

3.3. *FTIR study (Fourier transform infrared spectroscopy)*

The FTIR analysis provides the chemical composition of bituminous mixtures specimens. This infrared spectroscopy technology describes the evolutionary history of oxidized functions and allows following bituminous mixtures ageing. This study shows that only the oxidation index, sulphoxide index and the ratio sulphoxide index/oxidation index provide significant indications about asphalt thermal ageing. The lower this ratio is, the less ageing occur in bitumen.

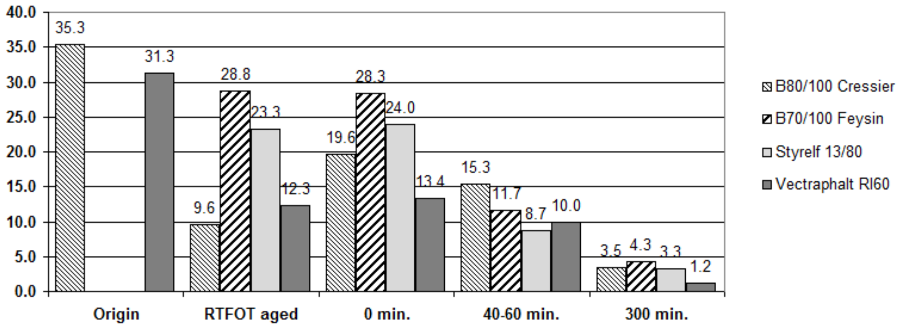

Figure 6. *Ratio sulphoxide index/oxidation index.*

This FTIR analysis demonstrated that the thermal treatment causes a bituminous binder oxidation which becomes very strong and detrimental under a prolonged reheating. On the other hand the results indicated that sulphoxide index has variations only in the very first evolution stages.

3.4. Rigidity modulus analysis

A complex modulus test campaign has been done on trapezoidal samples in order to compare following material:

– Samples prepared without asphalt mixtures reheating (compacted just after mixing).

– Samples fabricated with cooled asphalt mixture reheated with microwave oven and then stabilised in thermoregulated enclosure during 50 minutes.

The results indicate that the reheating method may have a minor impact in low temperature domain (high frequency), a moderate impact in median temperature and an important impact in higher temperature (i.e. low frequency). (Figure 7)

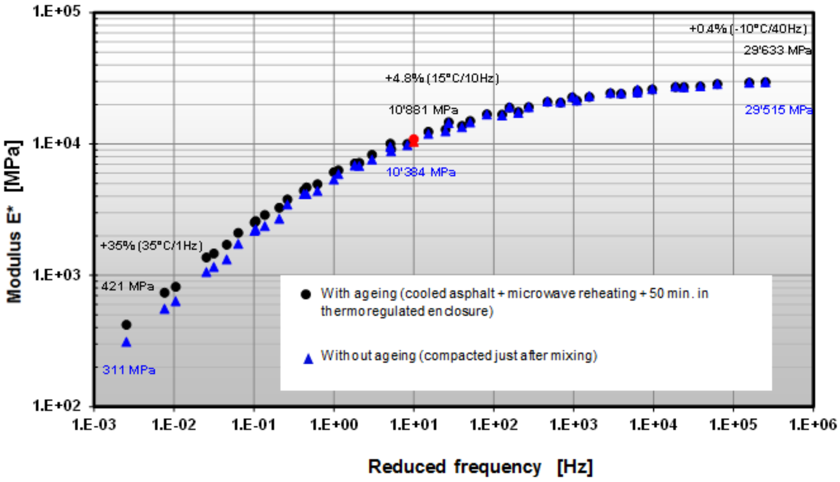

Figure 7. *Modulus E* master curve - Reference 15°C. AB 11S with 70/100 bituminous binder (5.31%/E – RM[3] 3.35 – HM[4] 3.5%).*

Another rigidity modulus test performed on a specimen taken in situ did not show the same trends. Indeed, the results showed a moderate impact (approximately 10% at low frequency) given to compaction conditions and various thermal conditions.

[3] RM: Richness modulus
[4] HM: Marshall voids content

4. Investigation and calibration

This study targets three different reheating procedures to compare their effects on different mix design. Following equipments have been used:

– Thermoregulated enclosure without fresh air, minimum ventilation

– Thermoregulated enclosure with nitrogen (2 liters/min.)

– Microwave oven (3300 Watts) with temperature measurement after each sequence (1 min.). Increasing temperature: ~25°C/min. for 5 kilograms and ~10°C/min. for 18 kilograms.

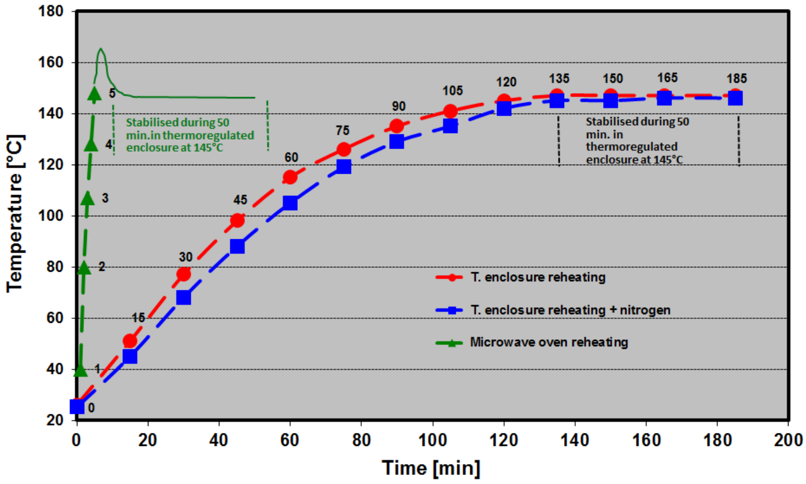

Figure 8. *Reheating procedures.*

The reheating procedure recommended by the standard EN 12697-30 that consists in a reheating at 130°C, followed by a second rapid reheating phase at compaction temperature appears not to be precise enough and was not advocate to use microwave oven. Besides it should be pointed out that the EN procedure concerns a bituminous mixture quantity of 5 kilograms (approx. 4 Marshall samples). Thus, in the case of higher quantity, the procedure requires adaptations.

This study shows that asphalt ageing under nitrogen atmosphere is slightly reduced and that microwave oven uses may be recommended. Besides, one can mention that reheating procedures with stabilisation during 50 minutes leads similar results to RTFOT values.

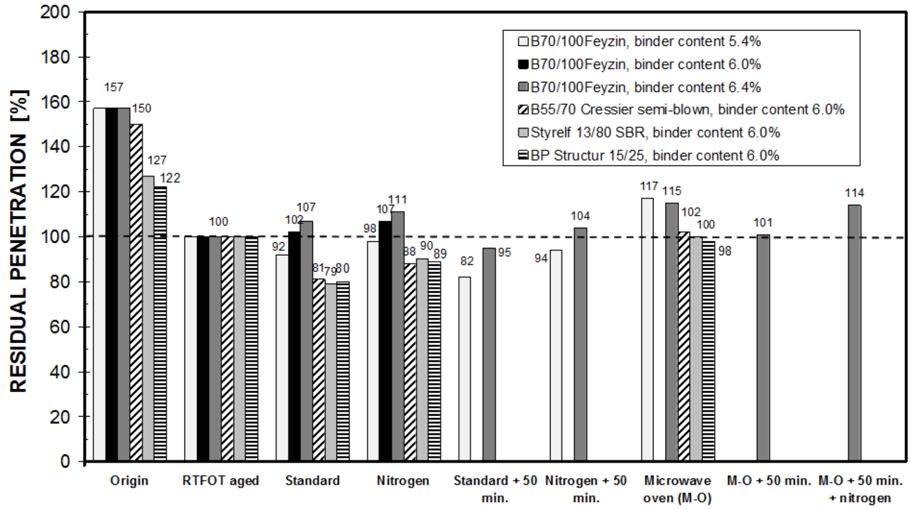

Figure 9. *Reheating procedures. 6 mix design. Residual penetration relative to RTFOT penetration.*

5. Proposal of a new bituminous mixtures reheating methodology

Based on the results and findings of these various analyses, a new bituminous mixture reheating methodology has been developed. This methodology implies the uses of a microwave oven (Gigatherm) and a thermoregulated enclosure (Heraeus). The proposed methodology is following:

1° Identify bitumen in the mix.

2° Define the mix design compaction temperature with standard SN 670428, EN 12697-28 "Bituminous mixtures – Test methods for hot mix asphalt – Part 28: Preparation of samples for determining binder content, water content and grading", see figure 10. With polymer-modified bitumen or bitumen with addition, refer to the manufacturer's prescriptions or customer's requirements.

Nominal grade of binder in sample	Maximum temperature of oven [°C][5]
> 330 penetration at 25°C	105
Above 60 up to 330 penetration at 25°C	120
25 to 60 penetration at 25°C	135
Less than 25 penetration at 25°C	150

Figure 10. *Temperatures of the oven for reheating laboratory samples prior to sample reduction. SN 670428 Tab. 1, EN 12697-28.*

3° For small specimens (cores …), start with a 30 seconds sequence.

[5] Only for traditional oven in the current standardisation

4° For large specimens, for example 18 kilograms packaging, reheat specimen with microwave oven (maximum power of 3300 W) up to about the quarter separation temperature according to the following procedure:

- Reheat bituminous mixture up to about 100°C (1st sequence, 4 minutes). At maximum power (3300 W), temperature increases about 10 to 15°C per minute, depending on treated materials.
- Reheat minute by minute, with temperature control after each sequence.
- If necessary, rehomogenise the material and change the packaging position in the oven, to prevent any risk of local overheating.

5° Quarter separation for tests preparation.

6° If temperature decrease is very important after quarter separation, continue microwave oven reheating up to the compaction temperature.

7° Maintain bituminous mixture, placed in a receptacle with a cover, in a thermoregulated enclosure during 40 minutes (for the 1st Marshall sample), 50 minutes for the other samples (thermal segregation, fatigue test, gyratory shear compactor press test, …) at compaction temperature with a setpoint temperature higher than the compaction temperature (+5°C).

8° By test or by series of tests, process with a maximum of 3 packaging (from 15 to 18 kilograms), to guarantee an adequate level of ageing homogenisation.

6. Verification and validation

The new proposed reheating methodology, based on the previous presented studies, has been verified and validated by different tests on asphalt mixture (Marshall test, moisture susceptibility ITSR, indirect tensile test ITS, rutting test, gyratory shear press test).

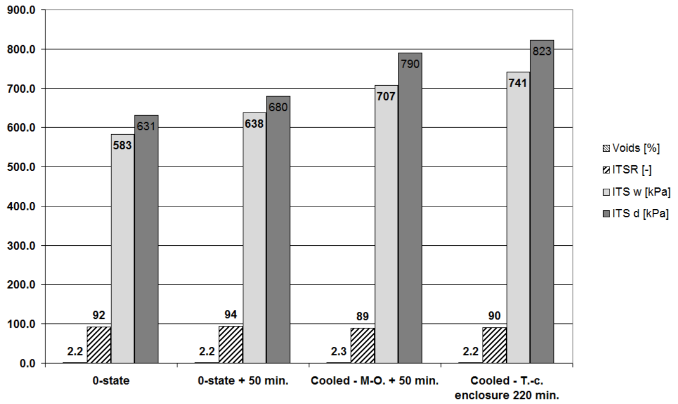

Figure 11. *Reheating impact. AC 11S (Bituminous binder content 6.4%). Voids content, ITSR, ITS w, ITS d. Test at 22°C.*

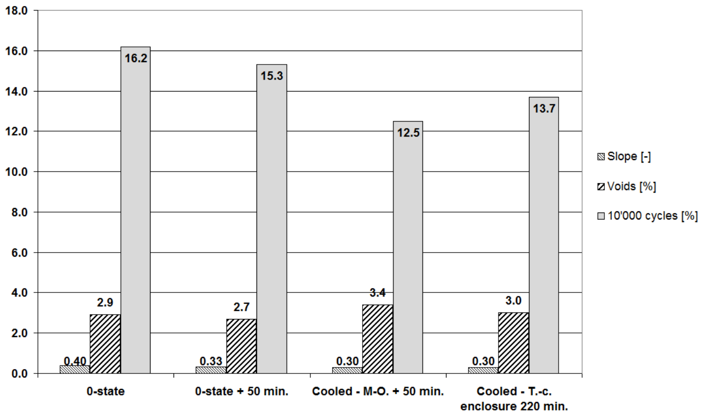

Figure 12. *Reheating impact. AC 11S (Bituminous binder content 6.4%). Rutting test at 60°C.*

For one mix design with higher binder content (6.4%. Figures 11 and 12), the different reheating procedures did not show any significant impact on the voids content and the moisture susceptibility. However, an important effect has been recognised for the indirect tensile test stress results.

Concerning the rutting test, one can note that voids content variation is not significant enough and the calculated slope is decreasing because of the reheating procedure. At 10'000 cycles, under thermal reheating, we perceive an important rutting decrease.

In this verification and validation study, realised with aggregates with sharp edges, contrary to the exploratory study, similar trends can be discerned but they are less pronounced due to the important internal angle of friction depending on sharp edges.

7. Conclusion

The various conducted experiments revealed the risks regarding the thermal history of preparation of bituminous samples. The thermal effect of reheating is resulting in oxidising the bitumen and in modifying the rheological and mechanical properties of the material, leading to performance degradation, particularly in case of prolonged reheating.

Since the beginning of the study, it was observed that a more precise definition of the rules that occur during thermal heating of samples deemed necessary, in order to provide a better fit between the recipes developed and optimised in the laboratory that are often used as reference, and the materials collected in the coating plant or in situ and controlled in the laboratory. The consistency of results between tests should also be guaranteed, along with the reproducibility between laboratories.

Provided that the granular is not porous and does not contain ferrous materials, it has been shown through verification tests for the performance of the materials that the controlled use of a microwave oven may be recommended. Thus, the heating time is considerably reduced with this equipment.

By comparing the obtained results for various heat treatment procedures, a new reheating methodology for bituminous mixtures using industrial microwave oven (3000 Watts power) and thermoregulated enclosures (650 litres volume, temperature controlled between 20°C and 200°C) has been developed.

The parameters that may influence, often significantly, the mechanical and rheological properties of the reheated material, were revealed. These (material, mass, time, temperature and reheating power properties) were then precisely defined for each step of the new methodology.

This proposed reheating procedure was forwarded to the experts involved in standardisation and enabled to influence standards development, although there are still gaps in the field. Adopted by several specialised laboratories, it has also provided the harmonisation of the procedures for sample processing and thus the reduction of differences between the results of standard tests.

This reheating methodology is currently being studied with samples containing reclaimed asphalt (RAP). These tests that have been conducted, as well as a wider dissemination of this procedure, would allow setting the repeatability and reproducibility values between laboratories and the limits of the method.

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