



HAL
open science

Advanced characterisation of multi-recycled warm asphalt pavement (MRWAP) with high content of recycled asphalt pavement

S. Pouget, P. Marsac, A. Pedraza, C. Sauzéat, H. Di Benedetto, V. Gaudefroy,
L. Boulangé, A. Pévère, V. Mouillet

► To cite this version:

S. Pouget, P. Marsac, A. Pedraza, C. Sauzéat, H. Di Benedetto, et al.. Advanced characterisation of multi-recycled warm asphalt pavement (MRWAP) with high content of recycled asphalt pavement. Road Materials and Pavement Design, 2023, 24 (2), pp.388-409. 10.1080/14680629.2021.2018352 . hal-04088132

HAL Id: hal-04088132

<https://hal.science/hal-04088132>

Submitted on 4 Oct 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Advanced characterisation of multi-recycled warm asphalt pavement (MRWAP) with high content of recycled asphalt pavement

S. Pouget^a, P.Marsac^b, A.Pedraza^c, C. Sauzéat^c, H. Di Benedetto^c, V.Gaudefroy^b, L. Boulangé^a, A. Pévère^d and V. Mouillet^d

^aEiffage Infrastructures, Vélizy-Villacoublay, France

^bUni. Gustave Eiffel, MAST/MIT, Bouguenais Cedex, France

^cUni. of Lyon-ENTPE, LTDS (UMR 5513), Vaulx-en-Velin, France

^dCerema Méditerranée, Équipe de recherche DIMA, Pôle d'activité Les Milles, Aix-en-Provence Cedex 3, France

ABSTRACT

The main results of the French collaborative project Improvmure, funded by the National Research Agency are presented. A very wide study was performed on bituminous mixtures produced on experimental sites or in laboratory at different scales (semi-industrial process or lab process). Objectives were to evaluate the influence of the recycling rate (0%, 40% and 70%), the number of successive recycling cycles (from 1 to 3 cycles), and the manufacturing process (hot mix or warm mix using additives or foamed bitumen). The impact was studied in laboratory on the emitted fumes during the production process, on the properties used for mix design (compactability, water sensitivity, complex modulus at 15°C/10 Hz, fatigue), on advanced thermomechanical behaviour (linear viscoelasticity, crack behaviour at low temperature, crack propagation) and on surface properties (skid resistance). The paper gathers many valuable results, showing the possibility to recycle bituminous mixtures multiple times, at high rates and with warm manufacturing process.

Introduction

The French road network (about 980,000 km) potentially represents a deposit of several billion tonnes of recycled asphalt pavements (RAP) resulting from the deconstruction of old pavements. These RAP have potential technical properties that it is important to develop. Their reuse in the manufacture of new asphalt mixes makes it possible to reduce the quantity of product sent to landfill sites while preserving natural resources (bitumen and quarry aggregates). However, this recovery must remain subject to compliance with technological, mechanical, environmental and public health requirements.

To limit environmental pollution, the road industry has developed new manufacturing processes at reduced temperatures (Guide Technique, 2015). These 'warm' techniques make it possible to reduce fuel consumption and gas emissions during manufacture while limiting the exposure of workers during implementation. In addition, the reduction in bitumen aging associated with lower asphalt mix temperatures allows for higher aggregate incorporation rates. These new techniques, therefore,

represent an opportunity to reconcile circular economy and the reduction of environmental and health nuisances.

The two complementary projects PN MURE and IMPROVMURE were initiated in 2013 and 2014 (Poirier et al., 2014) for a 5–6 years term, to answer these questions of temperature reduction and high recycling level. Based on collaborative research between project owners, companies and research institutes, the objective of the MURE National technical Project is to validate knowledge by carrying out 'full-scale' experimental road sections. The IMPROVMURE project (acronym for 'Innovation en Matériaux et PROCédés pour la Valorisation du Multi-Recyclage des Enrobés', in French) that can be translated as 'Innovations in Materials and Processes for MULTI-Recycling of Asphalt Mixes', aims to answer the technical questions raised by the MURE project, using a rigorous laboratory-based scientific study approach (Pouget et al., 2019). French National Research Agency (ANR) certified the project and funded it. The partners involved in the IMPROVMURE project are University Gustave Eiffel, University of Lyon/ENTPE, CEREMA (Centre for Studies and Expertise on Risks, the Environment, Mobility and Urban Planning), EIFFAGE company, IREX (Institute for applied research and experimentation in civil engineering) and Routes de France. The financial support for the project was over 2.5 million Euros.

The main objective of the IMPROVMURE project was to determine, over the life cycle of pavement materials, the impact of successive recycling at different recycling rates and for different manufacturing techniques (hot and warm processes). Thermo-mechanical, chemical and environmental properties were studied.

For this purpose, several methods and tests have been developed. The results presented in the next sections focus on the:

- Evaluation of the environmental impact of recycling by leaching measurements and analysis of fumes emitted during the manufacture of asphalt mixes
- Characterisation of the classical properties of multi-recycled asphalt mixes
- Characterisation of the advanced thermomechanical properties of multi-recycled asphalt mixes
- Characterisation of the surface properties of asphalt mixes.

Materials

A formula for a surface course layer of the type 'Semi-Graded Bituminous Concrete' BBSG of class 3 (BBSG 3 0/10), according to the EN, 13108-1 European standard, was considered. 12 variants of this formula were tested. In addition, six samples of BBSG taken from three road sites carried out within the framework of PN MURE were investigated. These samples were taken by coring on the hard shoulder (BAU) just before the pavement was put into service.

The experimental design is shown in Figure 1. A 100% RAP variant was also considered (Pedraza, 2018). It is not presented in this paper due to limited space.

The asphalt mixes were manufactured trying to keep the same skeleton grading curve. The targeted voids content (e) is 8% and the binder content (BC) is 5.4% of total weight. It is important to notice that the same penetration class (Pen 35/50, European standard EN, 1426, 2015) was also assured for the mix binder, according to the classical law of mixtures (Equation 1).

$$\log P_{\text{mix}} = a \times \log P_1 + b \times \log P_2 \quad (1)$$

where P_1 and P_2 are the penetrability values of the two bitumens (from RAP and fresh added) and a and b their proportion.

Other design parameters, which were part of the study, are listed below and presented in Figure 1:

- Manufacturing Condition: Laboratory (L) or In-Situ/field (S)
- Manufacturing method: hot (H), warm with foamed bitumen (WF) and warm with additives (WA)

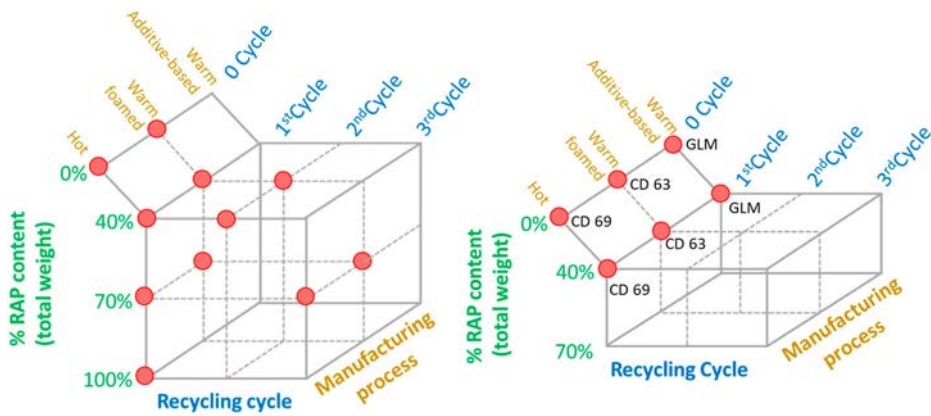


Figure 1. Experimental plan, red dots are tested specimens made: in the laboratory (left); taken on site (right).

- Percentage of RAP: 0%, 40% and 70%
- Recycling cycles: 0, 1, 2 and 3.

Asphalt mixes are named according to these design parameters (e.g. LH40-1, defines asphalt mixes manufactured in the laboratory (L), hot mix (H), with 40% RAP, from a single recycling cycle, i.e. first time they are used as RAP).

The filler aggregates used to manufacture laboratory mixes come from the Lafarge Granulats Cusset Quarry (CLGC). Société Lyonnaise d'Enrobés (SLE) supplied the RAP, which are used for the first recycling cycle in the laboratory and for construction sites at 40% RAP (the materials obtained are then used for subsequent recycling cycles). The fresh binders come from the TOTAL refinery in Feyzin:

- 35/50 Pen binder (4.1 mm penetrability) for asphalt mixes without RAP
- 50/70 Pen binder (6 mm penetrability) for asphalt mixes with 40% RAP
- 160/220 Pen binder (18 mm penetrability) for asphalt mixes with 70% RAP.

The manufacturing processes for laboratory mixes are described in Figure 2.

The penetrability and the ring & ball softening temperature (RBT) of the binders from RAP were determined after each recycling cycle. The binder's extraction and recovery of bituminous samples have been carried out according to a French testing method (Mouillet et al., 2011) that specifies and complements the European standard NF EN 12697-3 (2013) by adding additional experimental verifications which guarantee the quality of the extraction. The principle is to dissolve the bituminous mixture in a chlorinated solvent, dichloromethane, at room temperature. The solution is then filtered and centrifuged to remove any mineral particles present, then vacuum distilled to remove the solvent. The binder is then directly recovered for subsequent characterisation, making sure beforehand that it no longer contains fine elements and residual solvent. The results are reported in Table 1.

The penetrability of the binders extracted from each mixture was compared to the one obtained with the law of mixtures (Equation 1). The results (Figure 3) show a small difference between the estimated values, which are always higher than the measured values. The estimation considers the perfect blend of bitumen before producing mixtures, while the measurement was made on extracted bitumen after producing mixture. The observed difference could be partly explained by the ageing during the manufacturing process of the mixes.

It should be noted that the use of suitable fresh binders allowed the penetrability of the RAP binder to be corrected, regardless of the RAP content, so that the penetrability of the binders of produced

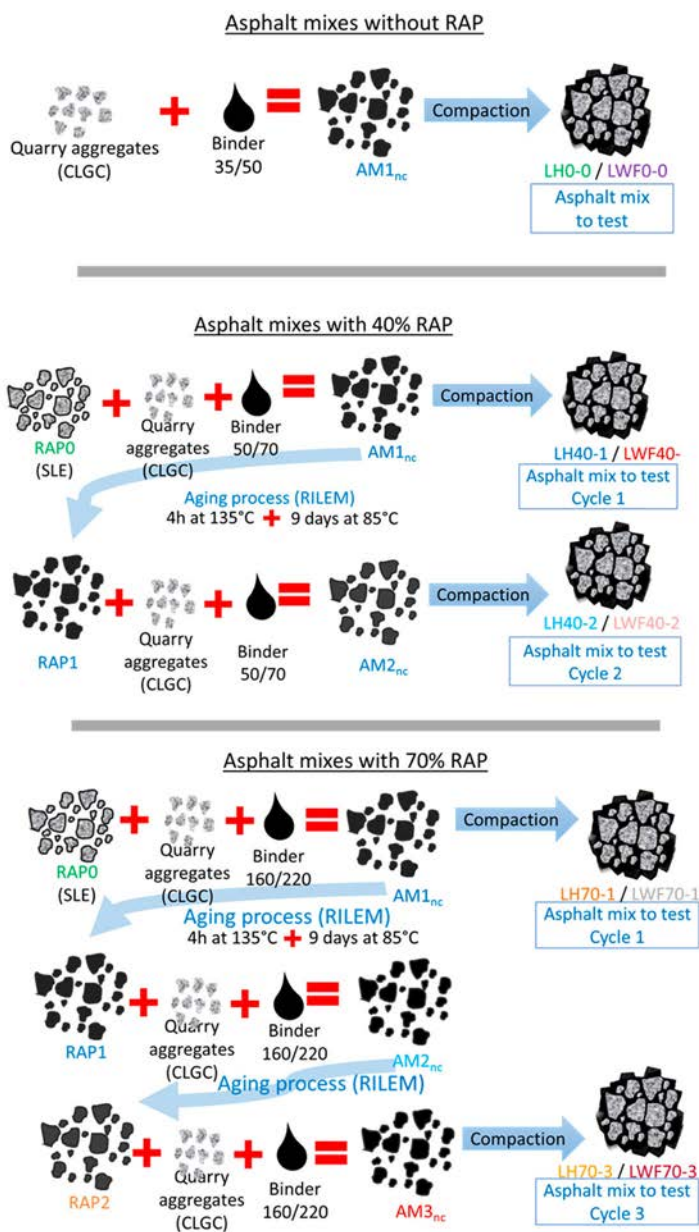


Figure 2. Manufacturing processes for asphalt mixes containing 0% RAP, 40% RAP with 1 and 2 recycling cycles and 70% RAP with one and three recycling cycles.

mixtures could approach the value of the reference asphalt mixes LH0-0 for HMA and LWF0-0 for warm mix asphalt (WMA) (Figure 3).

Comparative analysis of asphalt emissions

From an environmental perspective, multiple recycling of reclaimed asphalt pavements (RAP) could modify the nature and the concentration of substances emitted during the manufacturing or the life-time of the asphalt mixtures. It then becomes essential to assess the impact of multi-recycling and

Table 1. Penetrability and ring & ball softening temperature (RBT) of binders extracted from RAP used in the manufacture of asphalt mixes.

RAP	Specimens including this RAP	Penetrability (1/10 mm)	RBT (°C)
RAP0	LH40-1; LWF40-1; LH70-1; LWF70-1	10	76.2
RAP1	LH40-2	14	72.8
RAP1	LWF40-2	15	70.0
RAP2	LH70-3	18	67.8
RAP2	LWF70-3	18	57.9

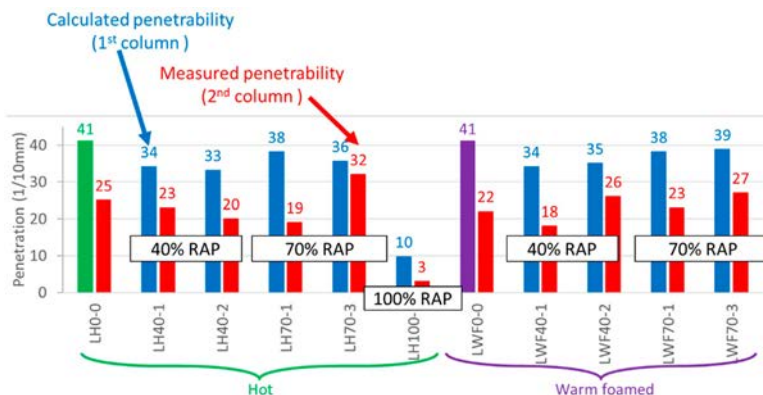


Figure 3. Measured (after mixture production and extraction) and estimated penetrability (from Equation 1) of the binders of the tested asphalt mixes.

manufacturing process on the composition of the emitted fumes. To answer this question, Uni. Gustave Eiffel conducted a study with EIFFAGE Infrastructures and CEREMA Méditerranée as part of the IMPROVMURE project. The analysis of the fume composition was carried out from a global point of view by the quantification of total organic compounds (TOCs). The results obtained in the laboratory on hot and warm processes incorporating multi-recycling confirm that a lower manufacturing temperature leads to a lower emission of total organic compounds, regardless of the aggregate recycling cycle. Lower coating temperatures also promote lower organic compounds concentrations in the fumes.

The Construction Products Regulation (CPR 305/2011/EU) focuses on the life cycle analysis and sustainability while stressing the importance of assessing emissions of particulate matter, toxic gases and volatile organic compounds into outdoor air and the work environment (ANSES, 2013). The testing methods (Brandt et al., 1985; Gaudefroy et al., 2010) developed to measure these emissions during the life cycle of the asphalt mixtures (Enell et al., 2012; Mouillet et al., 2012) can bring a complementary evidence of the interest of WMA.

The fumes of the asphalt mixtures were analysed using a system that efficiently and reproducibly collects total organic compounds (TOC).

Experimental approach

The production of asphalt mixes in a plant generates fumes from the combustion of fuel used to heat the aggregates and from the mix itself. At the scale of the asphalt plant, it is difficult to identify the contribution of each origin (combustion and material). This is why Uni. Gustave Eiffel developed several years ago a laboratory methodology to identify the specific influence of the material on the produced emissions. The organic phase of the fumes generated during the manufacture of asphalt mixes comes from the bitumen and contains aerosols as well as volatile and semi-volatile organic compounds. Uni. Gustave Eiffel's laboratory fume generation system was used as a predictive test to predict the quantities and nature of fumes generated by bituminous mixes for different emission scenarios. To achieve

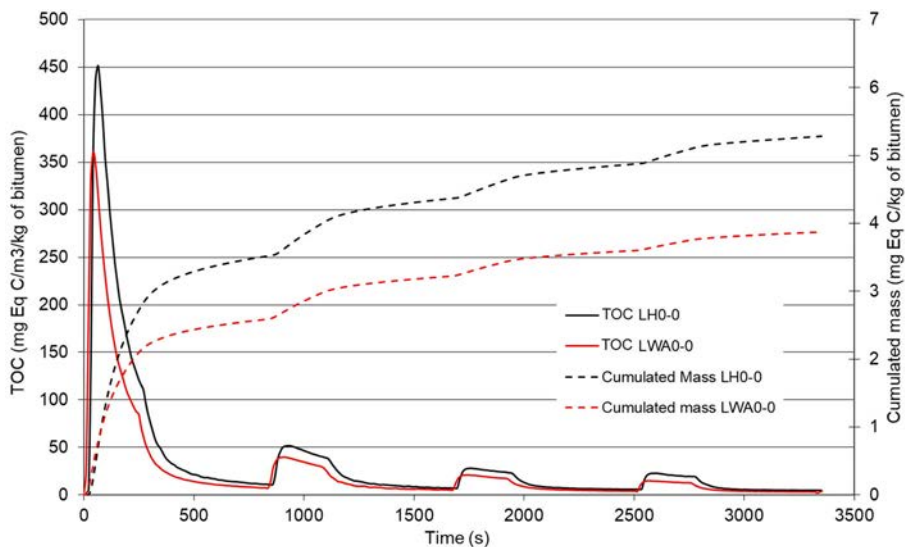


Figure 4. TOC concentration and cumulative mass as a function of time for hot (LH) and warm (LWA) asphalt mixes (same formula without RAP).

this objective, the experimental principle is to simplify the production line by considering the formulation and the effect of temperature. The compounds monitored for this study are the total organic compounds. For each mixture, 4 periods of 14 min were applied in a specifically designed mixer where the fumes can be analysed. Each period is composed of 4 min of mixing and 10 min of rest. This corresponds to four steps in a mixture life: production in plant, transfer into a truck, transfer into a finisher and mixing into the finisher. This protocol does not intend to reproduce the real fumes emission but aims at detecting fumes emission. In the presented Figures 4–6, the instantaneous TOC concentration and cumulative mass (calculated from instantaneous TOC concentration data) are followed during these four periods.

The study without and with RAP was performed with 35/50 and 50/70 penetration grade bitumens. The same asphalt mixture formula was manufactured using two processes (laboratory hot ‘LH’ at 160°C and laboratory warm with additive ‘LWA’ at 140°C) at a constant temperature during the mixing period. In addition to hot and warm mixes with virgin aggregates a 35/50 bitumen asphalt mix incorporating 40% RAP with a 50/70 bitumen have been studied. The RAP0, RAP1 and RAP2 are from the first, second and third recycling cycles as explained previously.

Influence of manufacturing temperature

The results show different levels of emissions depending on the manufacturing temperature. In particular, for mixes without RAP, Figure 4 shows a more emissive behaviour for the material at 160°C. The WMA, which is produced at 140°C with the same 35/50 grade of bitumen, generates less TOC (30% reduction). The results of mixes incorporating recycled material (40%) from a first, second or third recycling cycle for both processes (hot and warm) in the laboratory with the same bitumen (50/70 grade) confirm the trend observed for formulae without RAP presented in Figure 4: a lower production temperature leads to lower TOC emissions regardless of the aggregate recycling cycle.

Influence of the recycling cycle

The results show a decrease in the level of TOC emissions with the increase in the recycling cycle, whether for laboratory manufacturing at 160°C (LH, Figure 5) or at 140°C (LWA, Figure 6). This decrease is primarily related to the decrease of the amount of added 50/70 grade bitumen because the mixture

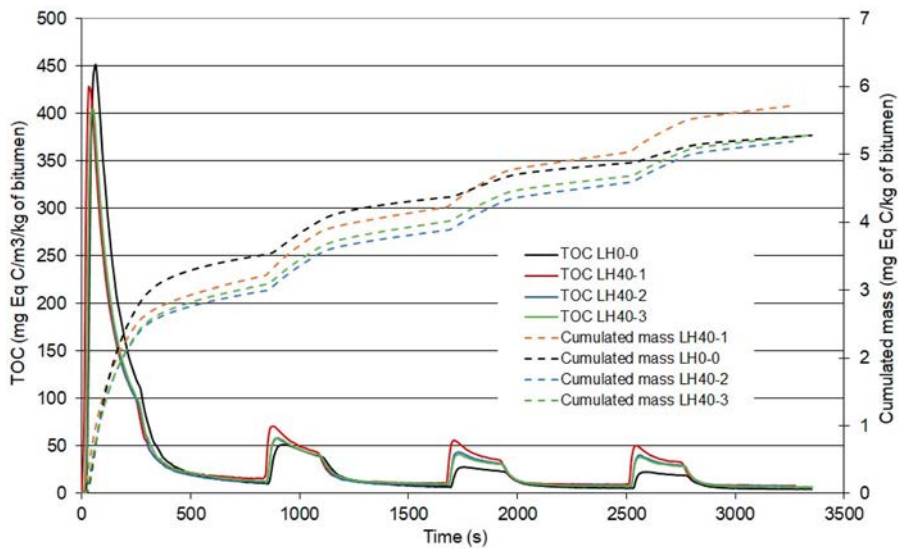


Figure 5. TOC concentration and cumulative mass as a function of time for hot (LH) asphalt mixes having 40% of RAP after 0, 1, 2 and 3 recycling cycles.

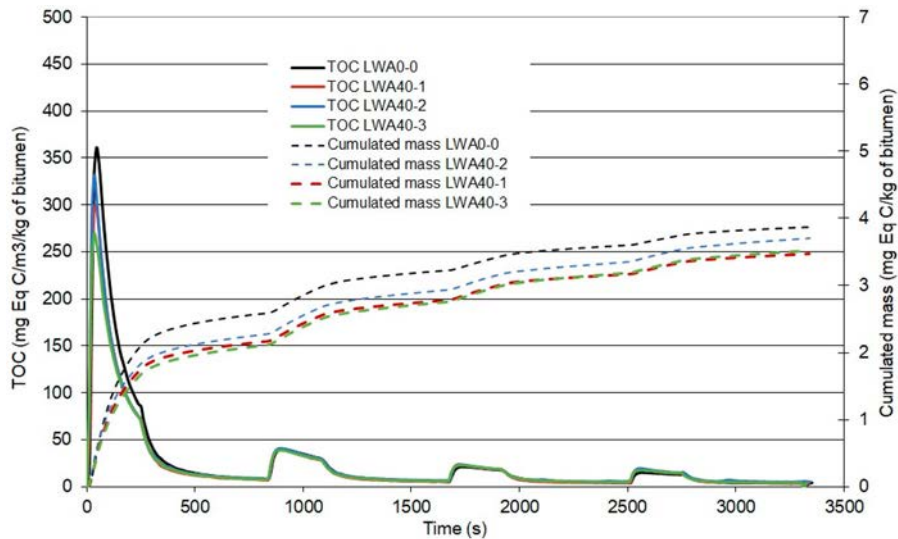


Figure 6. TOC concentration and cumulative mass as a function of time for warm (WMA) asphalt mixes having 40% of RAP after 0, 1, 2 and 3 recycling cycles.

is enriched by the aged binder at each recycling cycle. It could be related to the gradual depletion of the potentially emissive organic phase of the RAP.

During the IMPROVMURE project, only the emissions generated by road materials under channelised conditions directly at the source were considered. It excludes the site or construction site environmental factors that could add additional emissions (e.g. from construction equipment) or dilution (working in an open environment).

Main features in emission measurements

The results obtained, with the materials used in this study, highlight that:

- warm mixes with additives (WMA) generate fewer organic compounds in the bitumen fumes than hot mixes,
- TOC concentrations decrease as the number of recycling cycle increases,
- compound emissions were mainly from the RAP.

More results are available in the deliverable of the Task 2.2 of the IMPROVMURE project (Gaudefroy, 2018).

Properties used for design

The French asphalt mix design method and in particular the interpretation of the results of the associated laboratory tests is based almost exclusively on experience limited to hot mix asphalt that does not incorporate RAP. New manufacturing processes combining high recycling rates and warm processes are significantly different from the classical HMA processes allowing validation of this design method. In this section, the objective is to specify the combined influence of the new processes and multi recycling on the following design properties: compactability, workability, water sensitivity, modulus of rigidity and fatigue resistance of asphalt mixes.

Twenty-one BBSG of type 3 (grading range from 0 to 10 mm) were tested. They correspond to a combination of three recycling cycles for three manufacturing processes (hot control, warm additive-based and warm foamed bitumen-based) and two recycling rates (40% and 70%), supplemented by a non-recycling cycle for the three processes. To better simulate the real production processes, the materials were produced under semi-industrial conditions with, in particular, a discontinuous plant, an industrial bitumen foaming process (MARINI-ERMONT AQUABlack[®]) and aggregate production by milling (Figure 7). For this semi-industrial production, the ageing process differed slightly from the one used in the laboratory. The first step of ageing (4 h at 135°C of the RILEM procedure) was not applied. This step is supposed to reproduce the short term ageing occurring at the early age of pavement production (transportation from plant to building site and paving). As the produced asphalt mix was compacted to produce small scale pavement, ageing during this step would have been replicated.

Table 2 presents the measured properties.

Obtained results

The 21 asphalt mixes comply with the requirements of the French foreword of the product standard EN, 13108-1 for (a) water sensitivity ($i/C \geq 70$), (b) modulus at 15°C 10 Hz ($S_{min} \geq 7000$ MPa) and (c) fatigue resistance ($\epsilon_6 \geq 100 \cdot 10^{-6}$ m/m).

18 of the 21 asphalt mixes do not comply all the standard requirements because their void contents at 60 gyrations are higher than the requirement $V_{max} = 10\%$. It includes the hot mix reference without recycling. Therefore, combinations of warm processes, high recycling rates and multi-recycling did not induce very significant variations in the properties of the mixes.

A Principal Component Analysis (PCA) was conducted on the data to detect possible trends. The principle of PCA is to consider the data as a cloud of points in the six-dimensional space of the properties and to project it onto the plane that best explains the dispersion of the data (principal component plane). The representation of the variables in this plane gives physical meaning to the principal component axes. The best-represented variables are F_{GC} , V_{GC} , IC and ϵ_6 (Table 2). In the resulting plane, a positive translation on the horizontal axis represents a decrease in compactability and workability and an increase in water sensitivity. A positive translation on the vertical axis represents an increase in fatigue resistance.

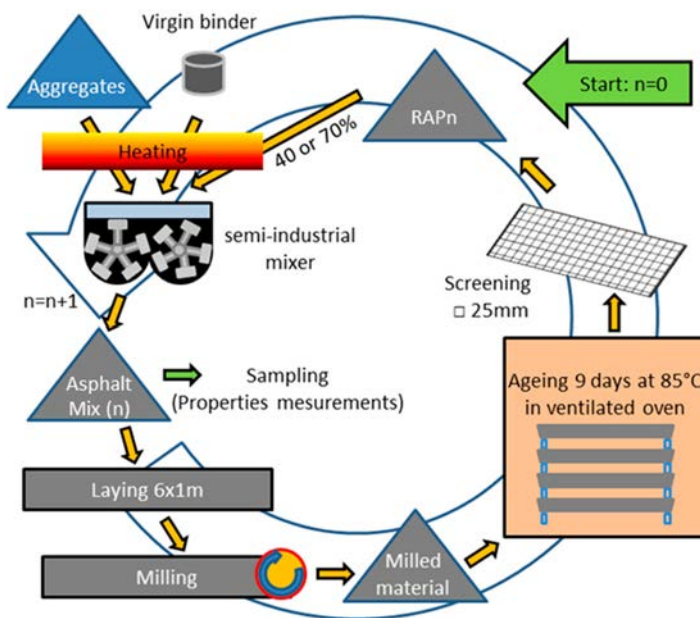


Figure 7. Diagram of the multi-recycling procedure set up.

Table 2. Measured properties for the 21 mixes.

Symbol	Description	European Standard
V_GC	Compactibility: void content @ 60 rounds with French Gyratory compactor	EN, 12697-31
F_GC	Maniability: Shear load @ 60 rounds with French Gyratory compactor	EN, 12697-31
IC	Water sensitivity: Ratio i/C with Duriez test	EN, 12697-12
E_Mod	2 point bending stiffness modulus @ 15°C & 10Hz	EN, 12697-26
ϵ_6	Strain value giving a failure at 10^6 cycles during 2 point bending fatigue test @ 10°C & 25 Hz	EN, 12697-24
P_Fat	Slope of Wöhler fatigue curve obtained with 2 point bending fatigue test @ 10°C & 25 Hz	EN, 12697-24

Figures 8–10 present the data in the principal component plane. Coloured zones correspond to area of 95% confidence intervals for the following groups according to the process (Figure 8), recycling rate (Figure 9) and number of recycling cycles (Figure 10). The main significant detected effect is a decrease in compactability and workability and an increase in water sensitivity with the introduction of RAP and multi-recycling.

Main features in properties used for design

The studied combinations of warm processes, high recycling rates and multi-recycling did not induce very significant variations in the properties used for the design of the mixes. However, a slight decrease in compactability and workability and a slight increase in water sensitivity with the introduction of RAP and multi-recycling were observed.

More results are available in the deliverable of Task 2.1 of the IMPROVMURE project (Marsac, 2018).

Advanced thermo-mechanical properties

Subtask 3.2 of the IMPROVMURE project dealt with the thermomechanical properties of asphalt mixes manufactured using two different processes (HMA and Foam WMA) and having different recycling rates and number of cycles (Sauzéat et al., 2018). It was carried out within the framework of the thesis of A. Pedraza (Pedraza, 2018), which received financial support from ANR and allowed a collaboration

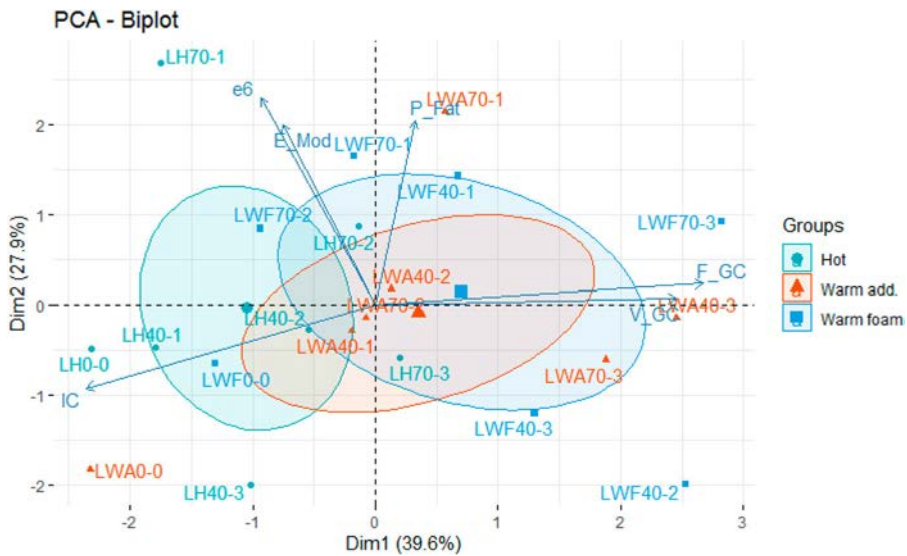


Figure 8. Data in the principal component plane, coloured areas indicate the 95% confidence intervals for HMA, WMA additive and foam WM.

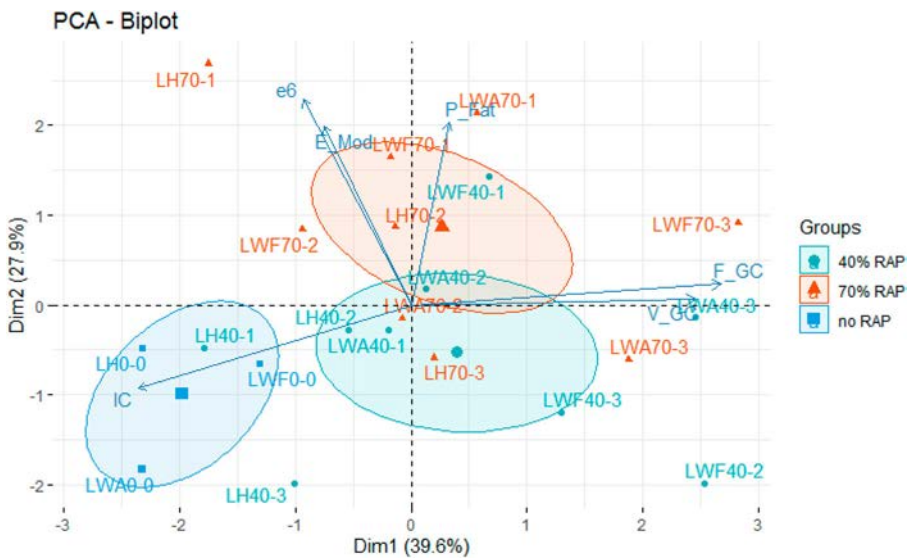


Figure 9. Data in the principal component plane, coloured areas indicate the 95% confidence intervals for asphalt mixes without RAP, with 40% RAP and 70% RAP.

between the company EIFFAGE and the Laboratory of Tribology and Systems Dynamics (LTDS) of the University of Lyon/ENTPE.

The following thermomechanical properties of asphalt mixes were studied: (i) Linear Visco-Elastic Behaviour (LVE), (ii) thermomechanical coupling at low temperature and (iii) crack propagation behaviour.

To obtain the properties in the LVE behaviour domain, complex modulus tests in tension-compression were performed over a wide range of temperatures and frequencies on cylindrical specimens (Carret et al., 2018; Graziani et al., 2017;; Gudmarsson et al., 2014, 2019 Mahmoudi et al.,

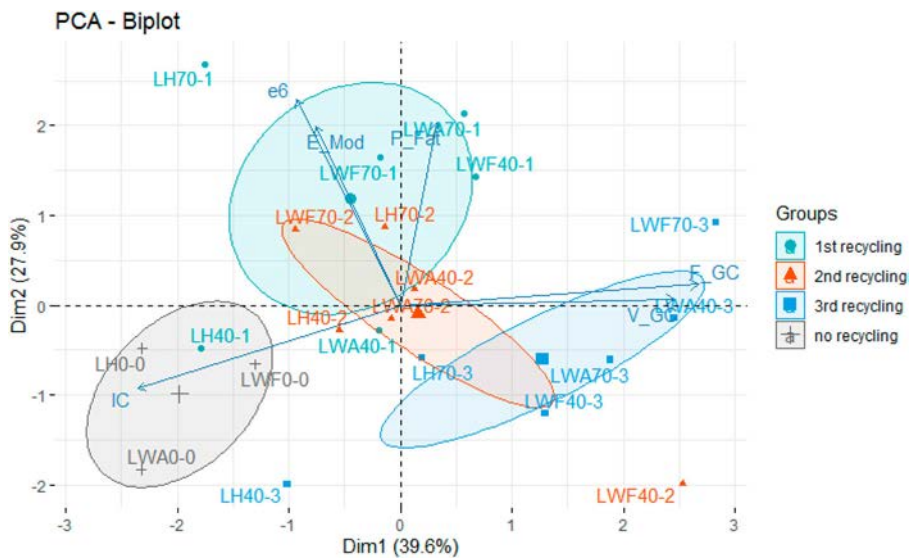


Figure 10. Data in the principal component plane, coloured areas indicate the 95% confidence intervals for HMA, WMA additive and Foam WM.

2020 Nguyen et al., 2015; Pedraza et al., 2017; Pedraza et al., 2019a; Perraton et al., 2016). This test developed at University of Lyon/ENTPE was also used to characterise nonlinear domain (Babadopulos et al., 2019 Nguyen et al., 2019). The results were modelled using the 2S2P1D (for: 2 Spring, 2 Parabolic creep elements & 1 Dashpot) rheological model, which was developed at University of Lyon/ENTPE (Di Benedetto et al., 2004; Di Benedetto et al., 2007; Olard & Di Benedetto, 2003).

The behaviour at low temperature was characterised using the Thermal Stress Restrained Specimen Test (TSRST), which couples thermal and mechanical stresses (Tapsoba et al., 2016).

Finally, crack propagation tests were carried out with a 4-point bending test on pre-notched beam (Nguyen et al., 2013, 2016). The crack height could be monitored during the test using a Digital Image Correlation (DIC) method.

Linear viscoelastic behaviour

Complex module tests carried out at University of Lyon/ENTPE have determined complex modulus and complex Poisson's ratios over a wide range of frequencies and temperatures (Pedraza, 2018 Pedraza et al., 2017; Sauzéat et al., 2018). The 2S2P1D rheological model was then calibrated for each of the materials (Pedraza, 2018; Pedraza et al., 2019a).

The complex modulus values at 15°C/10 Hz were the subject of a particular study, since they correspond to the modulus values classically used in French pavement design method. The influence of void content on the modulus values being established, a correction of the measured values could be applied, using a regression line. The modulus values presented in Figure 11 are extrapolated at a void content of 5% (which is a value close to the middle of the air void content range of all tested samples). The results in Figure 11 show very close modulus values for all tested mixes when the influence of the void content is corrected. Asphalt mixes containing RAP after three recycling cycles have the lowest values. This result can be related to the higher values of measured penetrability for the extracted binders. The average value for all asphalt mixes is 11,500 MPa with a coefficient of variation (CV) of 5.6%. Once the asphalt mixes containing RAP with three recycling cycles are excluded, the average value is 11,700 MPa with a CV of 3.5%.

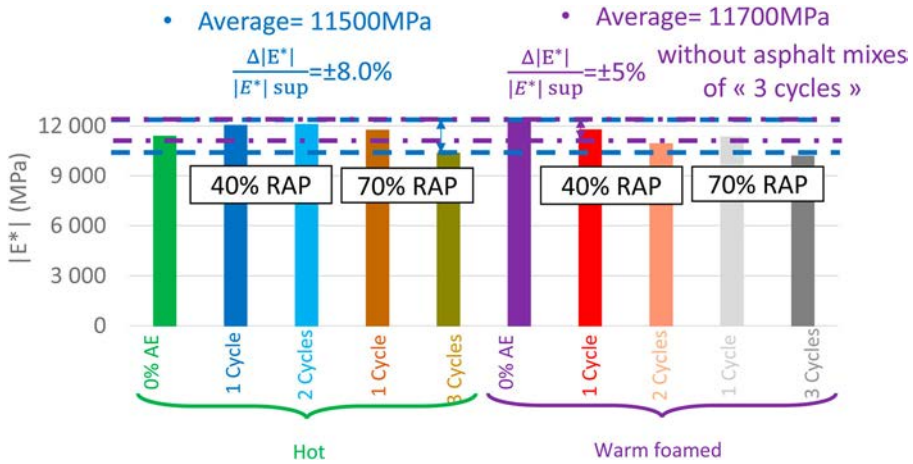


Figure 11. Norm of the complex modulus values at 15°C, 10 Hz at 5% voids content (obtained by extrapolation from measured data).

The master curves of the complex modulus (norm and phase angle) and complex Poisson's ratios could be plotted and modelled using the 2S2P1D model. The asymptotic values of the complex modulus that are real numbers, E_0 (for very high frequencies/low temperature) and E_{00} (for very low frequencies/high temperature) are plotted in Figure 12. In this figure, results obtained for all the specimens (two replicates by material) are considered for the values of E_0 , E_{00} and air void content. The linear evolution of E_0 as a function of the void content appears clearly, while E_{00} can be considered as a constant (the observed differences are not significant given the accuracy of the test). To get rid of these differences and to study the evolution of the complex modulus (or complex Poisson's ratio) as a function of temperature and frequency without the effects of these two constants, complex module values were normalised (Pedraza et al., 2019a). The normalised complex module E^*_{norm} is given in Equation 2. The two parameters E_0 and E_{00} are also two constants used by the 2S2P1D model.

$$E^*_{norm} = \frac{E^* - E_{00}}{E_0 - E_{00}} \quad (2)$$

Figure 13 shows the evolution of the complex normalised modulus E^*_{norm} in the Cole–Cole plane, as well as the simulated curves of the 2S2P1D model. The superimposition of the curves shows that the materials have a very similar linear viscoelastic behaviour (this observation is also valid for the Poisson's ratio not presented in this paper, Pedraza et al., 2019a). In this plane, only four constants of the model are used (k , h , δ and β) to obtain the simulated curve. Then, as a first approximation, it is possible to obtain a single set of these four parameters, whatever the RAP content, the number of recycling cycles and the manufacturing process (Figure 13a). A closer analysis makes possible to differentiate the mixes following the number of recycling cycles (Figure 13b).

TSRST tests

TSRST tests simulate a coupling between a stress created by thermal and mechanical effects. When decreasing temperature, the asphalt mix tends to contract. As this contraction is not allowed, a tensile stress is generated and increases until the failure of the material.

Two parameters were considered for comparing materials, the temperature and the stress at failure. During the tests carried out at University of Lyon/ENTPE, radial deformation was also measured, but the results are not presented here. The influence of air void content for all specimens was analysed but no clear tendency could be found on the studied parameters. The average values and standard deviations

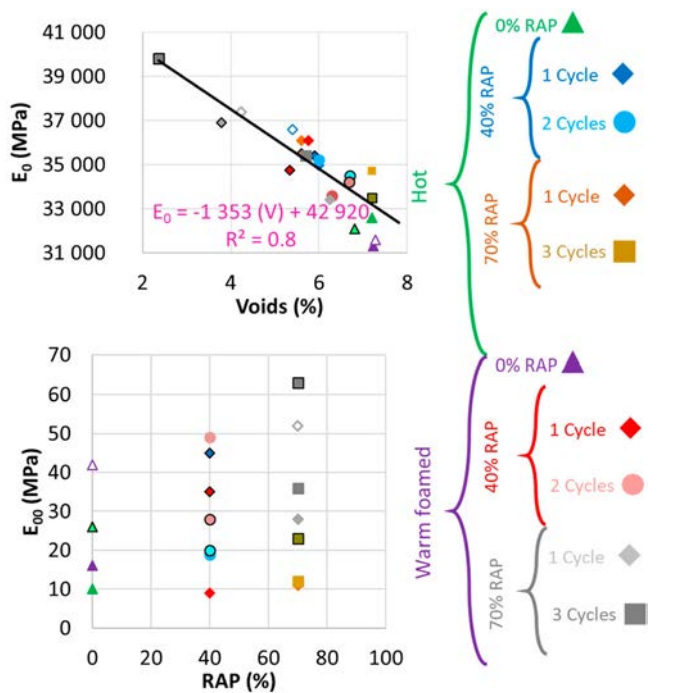


Figure 12. Asymptotic values of the complex modulus, E_0 (for very high frequencies/low temperatures) as a function of void content and E_{00} (for very low frequencies/high temperatures) as a function of RAP content.

(3 repeated tests) of temperature and stress at failure are shown in Figure 14. Small differences exist for the average values, but do not appear to be significant given the CV. The addition of RAP seems involving a small increase of the failure stress compared to reference asphalt mixtures that do not contain RAP (regardless of the manufacturing process or the number of recycling cycles). Asphalt mixes with 70% of RAP or 3 recycling cycles have lower temperatures at failure compared to other asphalt mixes, giving better performance. This result is in agreement with the higher penetrability measured for the extracted bitumen mix. All other asphalt mixes have comparable temperatures.

Crack propagation tests

The crack propagation tests were carried out with a 4-point bending test on pre-notched beam (Nguyen, 2009; Nguyen et al., 2013, 2016). To be able to analyse correctly the crack propagation, it is necessary to be able to measure the height (depth) of cracking throughout the test. A digital image correlation measuring device was used during Pedraza's thesis (2018). This device, coupled with an original method of image analysis, made it possible to obtain the height of the crack during its propagation (Pedraza et al., 2019b). A thermal chamber adapted for these measurements made it possible to carry out tests at the controlled temperatures of -5°C and -15°C (Figure 15).

In addition to the maximum measured force, two parameters were studied to characterise the cracking resistance properties of asphalt mixes, the strain energy release rate and the stress intensity factor, which are two parameters defined within the framework of the linear fracture mechanics.

The intrinsic strain energy release rate (G_c), corresponds to the energy supplied per unit area to crack the specimen. It can be obtained from the area below the load-displacement of the actuator curve. G_c and maximum force values are close together for all tested mixtures. For these two parameters, no clear link could be established with the composition, number of recycling cycles or type of manufacturing process.

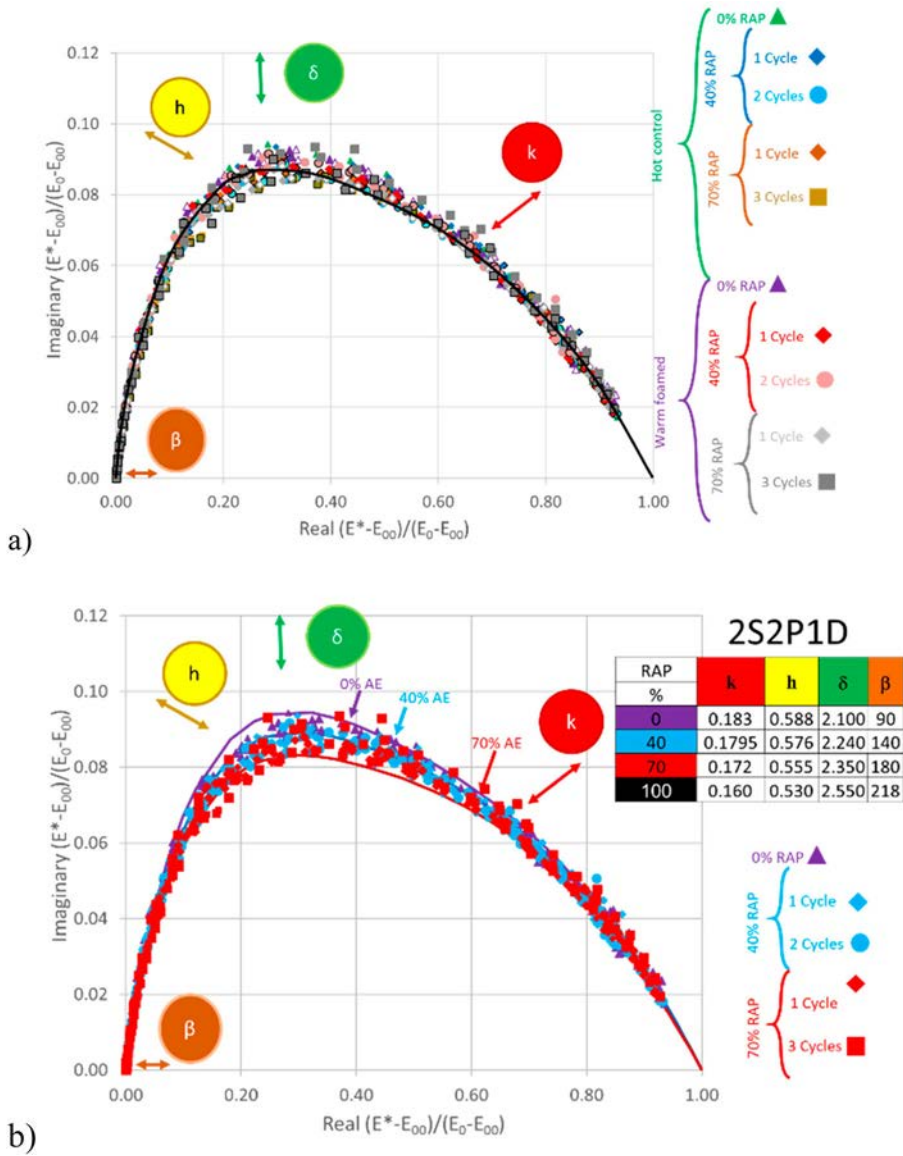


Figure 13. Normalised complex modulus (E^*_{norm}) plotted in the Cole–Cole plane: (a) experimental values for each material and averaged 2S2P1D model simulated curve, (b) experimental values grouped according to RAP content and 2S2P1D model simulated curve for each group.

The critical stress intensity factor or fracture toughness K_{IC} , characterises the stress intensity at the crack tip when the crack is propagating. The determination of this parameter during a test requires knowledge of the evolution of the applied load and also of the crack height. Figure 16 shows the evolution of the stress intensity factor as a function of the crack height for tests at -5°C and -15°C , for all materials (Figure 16a) and for materials manufactured warm with foam (Figure 16b). Warm mixes seem to be more resistant to crack propagation with an increase in RAP content. When comparing all the results, it was not possible to make a difference between the two processes and the RAP content. Air void content (measured on the entire sample) did not appear to show a clear influence.

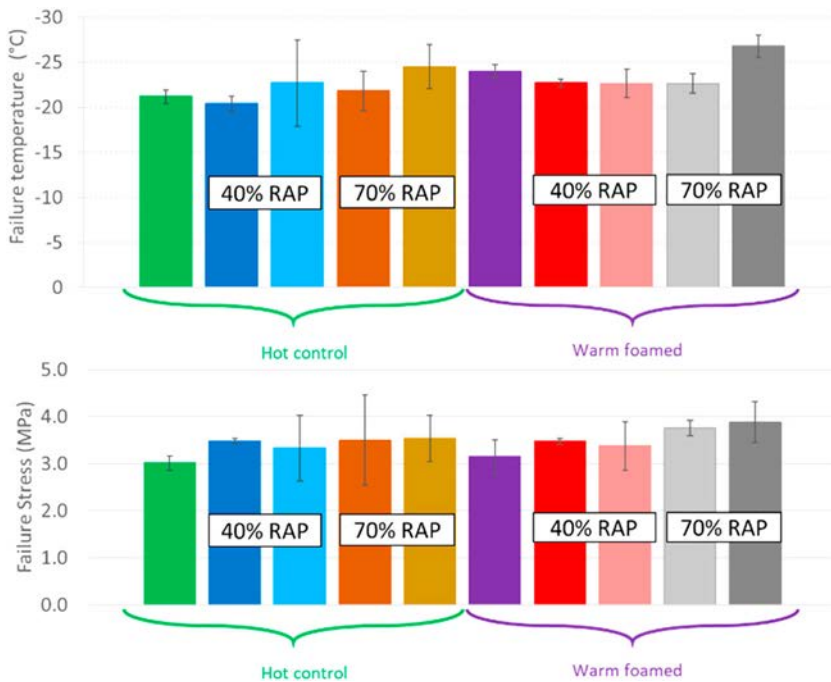


Figure 14. TSRS Test results (TSRST), temperature and stress at failure (average of three specimens and bar of standard deviations).

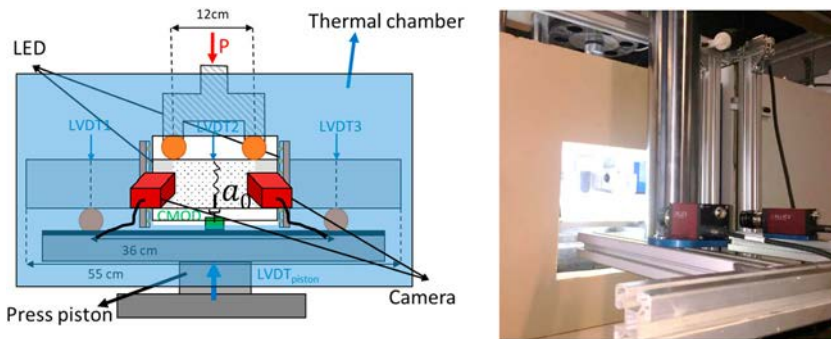


Figure 15. 4-point bending test on pre-notched beam, schematic presentation (left); Picture of the cameras and of the thermal chamber window allowing the measurements by digital image correlation (DIC) (right).

Main features for advanced thermo-mechanical properties

This work focused on linear viscoelastic behaviour, crack behaviour at low temperature and cracking resistance of mixes. The obtained results showed that recycling and multi-recycling do not seem to introduce any loss of thermomechanical performance for the manufactured mixes with respect to these properties. Concerning the studied foam warm manufacturing process, it does not seem either that the use of RAP degrades the properties of the final mix. During the manufacture of the asphalt mixes, the objective was to correct the consistency of the RAP binder to obtain mixes whose binder, once mixed, would have penetrability very close to the one of the reference mix using Equation (1).

It is important to underline that the selection of added fresh bitumen consistency giving the same penetrability of the global mix binder (Equation 1), resulted in similar behaviour between all asphalt mixes.

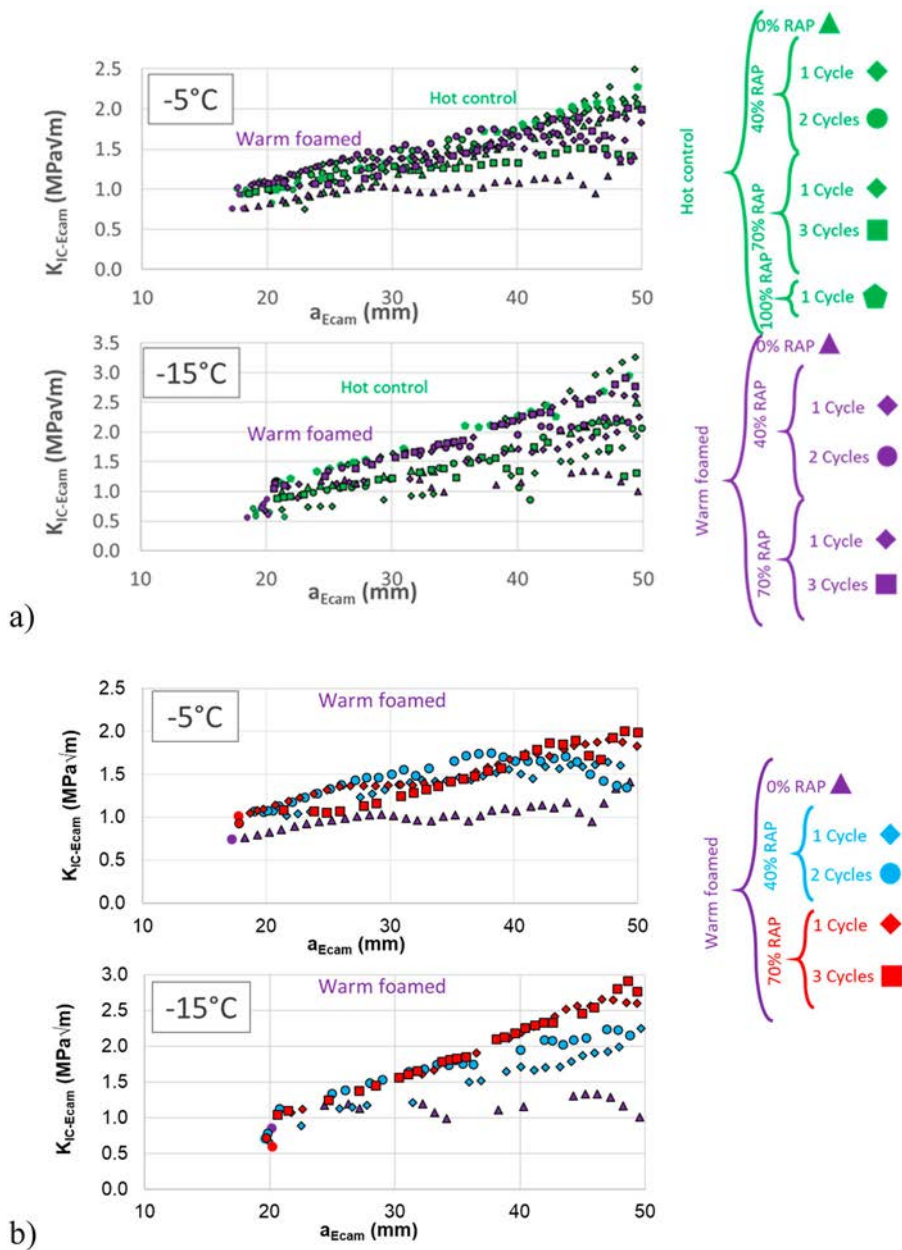


Figure 16. Fracture toughness K_{Ic} as a function of crack height: (a) all tested asphalt mixes and (b) warm foamed asphalt mixes.

Surface properties

The general objective of the work presented in this section is to characterise the skid resistance properties of asphalt mixes for surface layer using RAP (Reclaimed Asphalt Pavement) from successive recycling.

The evaluation of the performance of the tested asphalt with respect to skid resistance was carried out with the Wehner/Schulze machine test method (EN 12697-49). This machine allows the calculation of the coefficient of friction of asphalt mixes after different polishing cycles (Figure 17).



Figure 17. Wehner/Schulze test device.

Friction after polishing (FAP) test

The purpose of FAP tests is to measure the skid resistance capacity of a wearing course, which can be expressed as a coefficient of friction. For pavements, this coefficient has to be representative of their grip capacity in current service, that is to say, having been subjected to the effects of traffic and the environment. The tests, therefore, simulate polishing by the action of traffic and make it possible to measure the coefficient of friction in hydroplaning conditions (under water). They take place in two stages: a first phase of accelerated polishing and a second phase of measuring the skid resistance of the polished surface in the presence of water.

For this purpose, the Wehner/Schulze machine includes two heads: one for polishing the surface to be tested, and another for measuring the coefficient of friction.

The test pieces are 22.5 cm diameter disks. The test can be done on bituminous mixes (cored samples) or on aggregate mosaics glued to an epoxy base.

To reproduce the pavement condition as closely as possible, the binder on the surface of the specimens was not stripped before the polishing phase, as established in the standard. Indeed, this stripping does not take place on a real new roadway.

The coefficient of friction μ can therefore be calculated and plotted as a function of the speed. The value which serves as a μ FAP reference is that calculated for a speed of 60 km/h. For this study, the curves of friction coefficients at 60 km/h for different polishing cycles (0, 1000, 2000, 5000, 10,000, 15,000, 20,000, 50,000, 90,000 and 180,000 cycles) are analysed.

Results

The FAP tests made it possible to determine the influence of different parameters on the coefficient of friction of the asphalt tested. Two tests were carried out per configuration.

Influence of the RAP content

Figure 18 shows the change in the coefficient of friction at 60 km/h as a function of the number of polishing cycles for the BBSG mix at different RAP contents (0%, 40% and 70%). The figure only shows the results of tests on specimens with RAP from only one recycling cycle. The values of the coefficient of friction presented are calculated as the average of the two tests carried out for each variant of the BBSG mix.

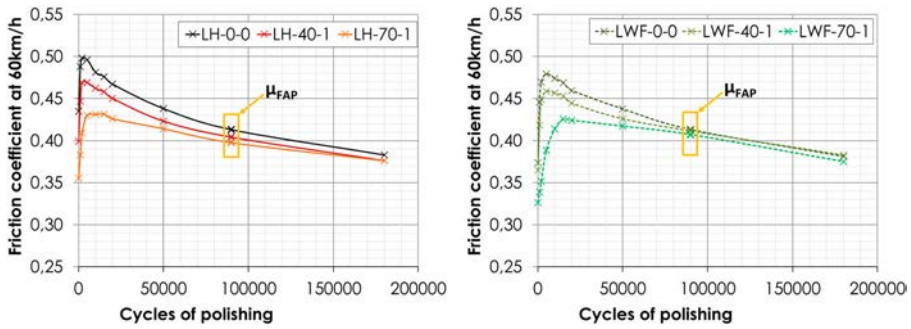


Figure 18. Influence of the RAP content: on the left the hot mixes, on the right the warm mixes with foam.

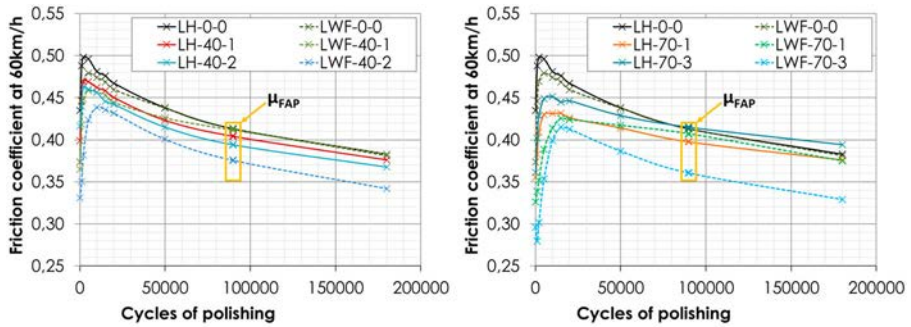


Figure 19. Influence of the asphalt manufacturing process: on the left the mixtures with 40% RAP, on the right the mixtures with 70% RAP.

By analysing the friction curve in its entirety, it is possible to distinguish different behaviours depending on the RAP content.

A decrease in the maximum friction value is observed with the increase in the RAP content, which could be attributed either to a lower angularity of the aggregates from the RAP, or to a different pickling behaviour. Also, the stripping time increases with the RAP content.

Compared to the normalised μ_{FAP} value, framed in Figure 18, for the two manufacturing processes, this value does not vary significantly (less than 5%) with the increase in the RAP content.

Influence of the asphalt production process and the number of recycling operations

Figure 19 compares the results obtained for hot mixes and warm mixes with foamed bitumen for equal RAP content.

For the configurations without RAP, no remarkable difference exists between hot mixes and warm mixes. On the other hand, for mixes manufactured with RAP resulting from multiple recycling, differences in the measurements are starting to appear. In particular:

- Warm mixes have μ_{max} values slightly lower than those of hot mixes.
- The stripping time of warm mixes (5000 cycles) is slightly longer than that of hot mixes (2000 cycles).

For the configuration made with RAP0, the normalised μ_{FAP} values of warm mixes are 0.01 higher (which is equivalent to 2% on average) than those of hot mixes. However, these differences are negligible. The manufacturing process does not significantly affect the classification of asphalt mixes with RAP0 in terms of skid resistance, even at RAP content of 70%.

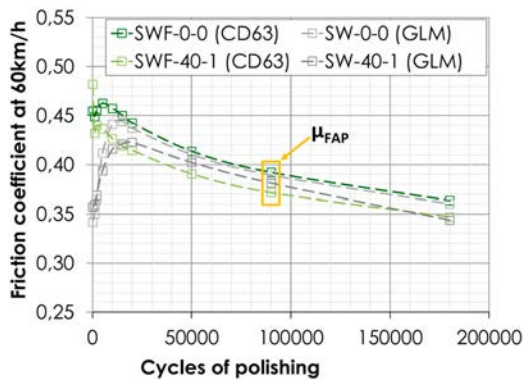


Figure 20. Influence of the warm mix-manufacturing process: mixes manufactured on site with bitumen additives or with foamed bitumen.

For the configurations in the second and third recycling cycles, warm mixes have lower skid resistance properties, both in μ_{max} and in μ_{FAP} , than hot mixes. This is more evident for the 3rd cycle recycling configurations. The stripping of the binder at surface corresponding to the first part of the curve (where the friction coefficient increases), requires also longer time. This might be due to the presence of aged bitumen at the RAP aggregates surface (not completely blended with fresh bitumen in the warm process) more difficult to strip. This would explain the lower parameters obtained in the second phase of the test. As this is not observed with the RAP0 used at the first recycling cycle, this might also be due to the process used in the laboratory to obtain RAP for following recycling cycles. This requires further investigation before a definitive conclusion.

The hot manufacturing process is therefore suitable for multiple recycling (up to three cycles).

The manufacturing method with warm bitumen foam does not present major drawbacks for the first cycle of recycling. It should be optimised to allow the use of more recycling cycles.

Figure 20 compares asphalt produced on site with two different warm processes. The asphalt mixes from the CD63 site were manufactured with bitumen foam and those from the GLM ('Grand Lyon Métropole', Lyon urban area) site were manufactured with additive in bitumen.

The results show a decrease in the maximum value of the coefficient of friction with the addition of RAP in asphalt mixes manufactured on site, as observed on the test specimens manufactured in the laboratory, but to a lesser extent.

Asphalt specimens without RAP exhibit the same behaviour regardless of the manufacturing process (foam or bitumen with additives).

There is no significant difference in μ_{FAP} between the two warm manufacturing processes with 40% RAP. The two asphalt variants have the same classification in terms of skid resistance.

The use of RAP even at a rate of 70% does not affect skid resistance, as measured by the coefficient of friction. The number of recycling cycles reduces this skid resistance level very little, in the order of 10% for warm asphalt and 5% for hot asphalt.

The hot, warm manufacturing processes do not change the classification of the asphalt in terms of skid resistance. However, the foam process could be optimised in the case of the incorporation of 70% RAP. Other results are available in the deliverable of task 3.3 of the IMPROVMURE project (Ramirez Cardona & Pouget, 2018).

Conclusion

The project made it possible to address the issue of multi-recycling in pavements through a multi-scale and multidisciplinary approach. The methods developed within this project may also find applications in other fields.

The IMPROVMURE research program has given the opportunity to study RAP recycling as well as warm production process for different aspects. A lot of results were collected and they constitute a large database for the studied materials. From the data presented in this paper, the following results were found:

- Fume emission during manufacturing is reduced with warm processes and when the recycling cycles number increases.
- A slight decrease in compactability and workability and a slight increase in water sensitivity were noted with RAP addition and multiple recycling cycles.
- The studied thermo-mechanical properties were not significantly affected by the RAP addition, multiple recycling cycles or warm manufacturing process.
- The increasing number of recycling cycles seems to affect the surface properties (maximum 10% decrease). This point requires further investigation to be concluded.

From a general point of view, the project has demonstrated that warm mixes with up to 70% RAP manufactured in the laboratory have properties very close to those of hot mix asphalt without RAP. From an industrial point of view, the conclusions of the project confirm that the French road industry has the laboratory tools and knowledge to formulate sustainable multi-recycled asphalt mixes.

However, the project partners agree that the results and conclusions obtained on a limited sample of asphalt mix designs need to be validated with other aggregates and other sources of RAP before being generalised. The impact of the use of polymer-modified binders could also be the subject of development.

Acknowledgments

The partners of the project would like to thank the French national agency ANR for the financial support and the French IREX association for the administrative support.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by Agence Nationale de la Recherche [grant number ANR-13-RMNP-0008].

References

- ANSES. (2013). Évaluation des risques sanitaires liés à l'utilisation professionnelle des produits bitumineux et de leurs additifs, Rapport d'expertise collective, saisine n° 2008-SA-0410, 2013 [in French]
- Babadopulos, L., Orozco, G., Mangiafico, S., Sauzéat, C., & Di Benedetto, H. (2019). Influence of loading amplitude on viscoelastic properties of bituminous materials. *Road Materials and Pavement Design*, 20(sup2), S780–S796. <https://doi.org/10.1080/14680629.2019.1628428>
- Brandt, H., De Groot, P., Molyneux, M., & Tindle, P. (1985). Sampling and analysis of bitumen fumes. *The Annals of Occupational Hygiene*, 29(1), 27–80. <https://doi.org/10.1093/annhyg/29.1.27>
- Carret, J. C., Pedraza, A., Di Benedetto, H., & Sauzeat, C. (2018). Comparison of the 3-Dim linear viscoelastic behavior of asphalt mixes determined with tension-compression and dynamic tests. *Construction & Building Materials*, 174, 529–536. <https://doi.org/10.1016/j.conbuildmat.2018.04.156>
- CPR 305/2011/EU. (n.d.). Regulation no 305/2011 of the European Parliament and of the Council of 9 March 2011 laying down harmonised conditions for the marketing of construction products and repealing Council Directive 89/106/EEC. *Official Journal of the European Union*, <https://eur-lex.europa.eu/eli/reg/2011/305/oj>
- Di Benedetto, H., Neifar, M., Sauzéat, C., & Olard, F. (2007). Three-dimensional thermo-viscoplastic behaviour of bituminous materials: The DBN model. *Road Materials and Pavement Design*, 8(2), 285–316. <https://doi.org/10.1080/14680629.2007.9690076>

- Di Benedetto, H., Olard, F., Sauzéat, C., & Delaporte, B. (2004). Linear viscoelastic behavior of bituminous materials: From binders to mixes. *Road Materials and Pavement Design*, 5(sup1), 163–202. <https://doi.org/10.1080/14680629.2004.9689992>
- EN 12697-12. (2018). European standard, Bituminous mixtures. Test methods. Part 12. Determination of the water sensitivity of bituminous specimens.
- EN 12697-24. (2018). European standard, Bituminous mixtures. Test methods. Part 24: Resistance to fatigue.
- EN 12697-26. (2018). European standard, Bituminous mixtures. Test methods. Part 26. Stiffness.
- EN 12697-3. (2013). European standard, Bituminous mixtures. Test methods. Part 4: Bitumen recovery: Rotary evaporator.
- EN 12697-31. (2019). European standard, Bituminous mixtures. Test methods. Part 31: Specimen preparation by gyratory compactor.
- EN 13108-1. (2016). European standard, Bituminous mixtures. Material specifications. Asphalt concrete.
- EN 1426. (2015). European standard, Bitumen and bituminous binders - Determination of needle penetration.
- Enell, A., Quinn, A., McNally, C., Huzlik, J., Wik, O., Licbinsky, R., Gaudefroy, V., & Mouillet, V. (2012). Deliverable 1.6. Re-Road. <http://re-road.fehrl.org/>
- Gaudefroy, V. (2018). Analyse comparative des émissions selon les procédés de fabrication. livrable tâche 2.2 projet ANR IMPROVMURE, 19p. [in French].
- Gaudefroy, V., Viranaiken, V., Paranhos, R., Jullien, A., & de La Roche, C. (2010). Laboratory assessment of fumes generated by bituminous mixtures and bitumen. *Road Materials & Pavement Design*, 11(1), 83–100. <https://doi.org/10.1080/14680629.2010.9690261>
- Graziani, A., Di Benedetto, H., Perraton, D., Sauzéat, C., Hofko, B., Poulidakos, L. D., & Pouget, S. (2017). Recommendation of RILEM TC 237-SIB on complex Poisson's ratio characterization of bituminous mixtures. *Materials and Structures*, 50(2), Article number 142. <https://doi.org/10.1617/s11527-017-1008-8>
- Gudmarsson, A., Carret, J. C., Pouget, S., Nilsson, R., Ahmede, A., Di Benedetto, H., & Sauzéat, C. (2019). Precision of modal analysis to characterize the complex modulus of asphalt concrete. *Road Materials and Pavement Design*, 20(S1), S217–S232. <https://doi.org/10.1080/14680629.2019.1590224>
- Gudmarsson, A., Ryden, N., Di Benedetto, H., Sauzéat, C., Tapsoba, N., & Birgisson, B. (2014). Comparing linear viscoelastic properties of asphalt concrete measured by laboratory seismic and tension-compression tests. *Jl of Nondestructive Evaluation*, 33(4), 571–582. <https://doi.org/10.1007/s1007-014-0253-9>
- Guide technique IDRRIM. (2015). Abaissement de température des mélanges bitumineux: Etat de l'art et recommandations, Edition CEREMA, 60p, Octobre 2015 [in French].
- Mahmoudi, Y., Mangiafico, S., Sauzéat, C., Di Benedetto, H., Pouget, S., & Faure, J. P. (2020). Tridimensional linear viscoelastic properties of bituminous mixtures produced with crumb rubber added by dry process. *Road Materials and Pavement Design*(SI rubber), 11. <https://doi.org/10.1080/14680629.2020.1737566>
- Marsac, P. (2018). Formulation, fabrication en laboratoire et propriétés d'usage des enrobés tièdes multi-recyclés, livrable tâche 2.1 projet ANR IMPROVMURE, 84 p. [in French].
- Mouillet, V., Dumas, P., Molinengo, J., Chailleux, E., & Porquet, T. (2011). Extraction des liants d'un enrobé bitumineux pour caractérisation du liant récupéré. *Méthode D'essai*, 83. <https://hal.archives-ouvertes.fr/hal-00907318>. LPC [in French].
- Mouillet, V., Gabet, T., Wayman, M., Piéard, N., Vanelstraete, A., Mollenhauer, K., Enell, A., Licbinsky, R., McNally, C., Carlsson, C., Gaudefroy, V., & Adame, V. (2012). Deliverable 1.1. Re-Road. <http://re-road.fehrl.org/>
- Nguyen, M. L. (2009). Etude de la fissuration et de la fatigue des enrobés bitumineux. PhD Thesis, INSA de Lyon/ENTPE [in French].
- Nguyen, M. L., Di Benedetto, H., & Sauzéat, C. (2016). Crack propagation characterisation of bituminous mixtures using a four-point bending notched specimen test. *Road Materials and Pavement Design*, 17(1), 70–86. <https://doi.org/10.1080/14680629.2015.1063535>
- Nguyen, M. L., Sauzéat, C., Di Benedetto, H., & Tapsoba, N. (2013). Validation of the time-temperature superposition principle for crack propagation in bituminous mixtures. *Materials and Structures*, 46(7), 1075–1087. <https://doi.org/10.1617/s11527-012-9954-7>
- Nguyen, Q. T., Di Benedetto, H., & Sauzeat, C. (2015). Linear and nonlinear viscoelastic behaviour of bituminous mixtures. *Materials and Structures*, 48(7), 2339–2351. <https://doi.org/10.1617/s11527-014-0316-5>
- Nguyen, Q. T., Nguyen, M. L., Di Benedetto, H., Sauzéat, C., & Chailleux, E. (2019). Thi thanh nhan hoang, nonlinearity of bituminous materials for small amplitude cyclic loadings. *Road Materials and Pavement Design*, 20(7), 1571–1585. <https://doi.org/10.1080/14680629.2018.1465452>
- Olard, F., & Di Benedetto, H. (2003). General "2S2P1D" model and relation between the linear viscoelastic behaviours of bituminous binders and mixes. *Road Materials and Pavement Design*, 4(2), 185–224. <https://doi.org/10.1080/14680629.2003.9689946>
- Pedraza, A. (2018). Propriétés thermomécaniques d'enrobés multi-recyclés, PhD Thesis. University of Lyon/ENTPE, 2018 [in French].
- Pedraza, A., Di Benedetto, H., Sauzéat, C., & Pouget, S. (2019a). 3D linear viscoelastic behaviour of bituminous mixtures containing high content of multi-recycled RAP. *Road Materials and Pavement Design*, 20(7), 1709–1721. <https://doi.org/10.1080/14680629.2019.1594054>

- Pedraza, A., Di Benedetto, H., Sauzéat, C., Torres, S., Pouget, S., & Olard, F. (2017). Projet improvmure: Comportement viscoélastique linéaire des enrobés multi-recyclés. *Revue Générale des Routes et des Aéroports*, 949, 35–39. [in French].
- Pedraza, A., Di Benedetto, H., Sauzéat, C., & Pouget, S. (2019b). Fracture properties of multi-recycled asphalt mixes from four point bending test using digital image correlation and back calculation. *Journal of Testing and Evaluation*, 47(5), 3275–3288. <https://doi.org/10.1520/JTE20180524>
- Perraton, D., Di Benedetto, H., Sauzéat, C., Hofko, B., Graziani, A., Nguyen, Q. T., Pouget, S., Poulidakos, L. D., Tapsoba, N., & Grenfell, J. (2016). 3D experimental investigation of linear viscoelastic properties of bituminous mixtures. *Materials and Structures*, 49(11), 4813–4829. <https://doi.org/10.1617/s11527-016-0827-3>
- Poirier, J.-E., Pouget, S., & Ch, L. (2014). MURE et IMPROVMURE dans les starting-blocks: promouvoir la pratique conjointe du tiède et du recyclage multiple des enrobés. RGRA N°919 [in French].
- Pouget, S., Marsac, P., Pedraza, A., Sauzéat, C., Di Benedetto, H., Gaudefroy, V., Boulangé, L., & Mouillet, V. (2019). Caractérisation avancée des enrobés multi-recyclés. *Revue Générale des Routes et de L'Aménagement*, 968, 32–43. [in French].
- Ramirez Cardona, D., & Pouget, S. (2018). Propriétés de surface des chaussées en enrobés multi-recyclés », livrable tâche 3.3 projet ANR IMPROVMURE, 25p. [in French].
- Sauzéat, C., Di Benedetto, H., & Pedraza, A. (2018). Propriétés thermomécaniques des enrobés multi-recyclés, livrable tâche 3.2 projet ANR IMPROVMURE, 26p. [in French].
- Tapsoba, N., Baaj, H., Sauzéat, C., Di Benedetto, H., & Ech, M. (2016). 3D analysis and modelling of Thermal Stress Restrained Specimen test (TSRST) on asphalt mixes with RAP and roofing shingles. *Construction and Building Materials*, 120, 393–402. <https://doi.org/10.1016/j.conbuildmat.2016.05.092>