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SUSTAINABLE FULLY RECYCLED ASPHALT CONCRETE

FINAL REPORT

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1. SUMMARY

The project aimed at producing asphalt concrete made of 100% Reclaimed Asphalt Pavement (RAP) duplicating the performance of mixtures fabricated according to the current Swiss standards. The project was supported by the Federal office for the environment, the companies BHZ and Ammann.

Reclaimed asphalt provided by the Swiss asphalt mixing plant from the company BHZ was used throughout the project. Three commercial bio-based rejuvenators were used: from natural seed oil, cashew nut shell oil and tall based oil. The rejuvenated binders and RAP were investigated environmentally, chemically and mechanically. Environmentally, it was found that for the sum of the 16 PAHs the rejuvenators do not mobilize PAHs bound in the RAP materials and therefore did not released or leached more easily. Values for individual PAHs could be higher than reference materials. Testing of particle abrasion from the asphalt slabs during loading with the traffic simulator MMLS3 demonstrated that 100% recycled asphalt emission factor is lower compared to traditional AC8N mixture.

Rheological tests showed how the rejuvenators may restore the mechanical properties of RAP binder. Considering the effect of aging is vital in identifying how rejuvenators affect the RAP binder chemically and mechanically long term.

Despite the addition of rejuvenators, physio-chemical oxidation did not reverse.

The project showed that in order to maximize the use of RAP, 100% RAP mixtures can be designed with the aim to perform similarly to AC 8N mixture. This mixture is used for low volume road surface courses in Switzerland. The mixtures were tested for fracture toughness using the Semi Circular Bend test, rutting using French wheel tracking test and complex modulus in cyclic indirect tension mode on cylindrical specimens (CIT-CY). The test results allow concluding the following:

- 100% RAP mixtures could not satisfy the conventional volumetric and Marshal test requirements. However, it is recognized that satisfying these requirements would not necessarily ensure the required pavement performance.
- Performance-based balanced mix design procedure using French Rutting test and Flexibility Index from semi-circular bend test was found practical and provided the expected trends as a result of changes in mix design.
- It was found by changing different mixture parameters that it is possible to ensure that 100% recycled surface layer asphalt mixture provides similar performance to

conventional asphalt which is designed for low volume roads with design traffic volume of up to 300 equivalent single axle loads.

- Testing with model mobile load simulator (MMLS3) demonstrated that the designed 100% recycled mixtures can sustain 2 times more wheel passes before fatigue failure.
- All recycled mixtures had higher fracture toughness at 0°C compared to the reference mixtures. However, fracture toughness test results, likely because of the test temperature (0°C), were not sensitive enough towards mix design parameters, including binder content, viscosity and coarseness of gradation. This suggests that fracture toughness should not be used as a mix design tool for the purpose of determining the effect of changes in these parameters. Since binder content and viscosity are important parameters of high RAP mixtures, the test method should not be used for optimizing high RAP mixture designs.
- The master curves showed that the stiffness of all mixtures is within the same range as the two reference mixtures over the entire frequency spectrum indicating that rejuvenator has been successfully used to reduce the stiffness of the aged recycled asphalt.
- It was not possible to design 100% recycled asphalt mixtures for highways because of the low quality of the reclaimed asphalt. It is therefore recommended to introduce RAP management procedures that would allow to increase its quality and homogeneity.

Possible rejuvenator addition locations in one type of asphalt plant were evaluated in order to provide guidelines to benefits and drawbacks for each of them.

- Rejuvenator addition on cold RAP on conveyor belt provided the same crack propagation resistance as rejuvenator addition on hot RAP in mixer despite 0.5% lower binder content.
- Rejuvenator addition on cold RAP conveyor belt provided higher fatigue resistance compared to addition in hot mixer (the fatigue test tests did not have enough repetitions to conform to standard).
- The samples for which rejuvenator was applied in mixer had lower complex modulus. This however, might be due to the 0.5% higher binder content of the samples.
- Rejuvenator addition resulted in no significant TOC emission increase, irrespective of 3% or 5% rejuvenator addition compared to 0% rejuvenator addition.
- It made no difference to TOC emissions whether rejuvenator was sprayed on the conveyor belt on cold RA or into the mixer on hot RA.
- Increase in RA rate also increased TOC emissions – a common and known effect.

2. ZUSAMMENFASSUNG

Ziel des Projektes war die Entwicklung einer Asphaltmischung, bestehend aus 100% rezykliertem Ausbauasphalt (RAP, Reclaimed Asphalt Pavement), mit ähnliche Leistungsfähigkeit des bisher nach Schweizer Standards hergestellten Recyclingasphalts. Das Projekt wurde von Bundesamt für Umwelt und die Firmen BHZ und Ammann unterstützt.

Dazu wurde in einem Asphaltwerk der Firma BHZ ein bestimmter Ausbauasphalt, mit drei handelsüblichen Verjüngungsmitteln behandelt: Pflanzenöl, Öl aus der Cashew Nussschale und Tallöl. Der verjüngte Recyclingasphalt und das Bindemittel wurden auf ihre Umweltverträglichkeit und ihre chemischen und mechanischen Eigenschaften untersucht.

Betreffend Umweltverträglichkeit stellte sich heraus, dass die 16 toxischen, polyzyklischen aromatischen Kohlehydrate (PAK), die im untersuchten Recyclingasphalt enthalten waren, nach der Beigabe des Verjüngungsmittels nicht freigesetzt oder leicht aus dem Belag ausgewaschen werden konnten. Tests mit dem Verkehrslastsimulator MMLS3 ermittelten einen geringeren Abrieb beim RAP Belag als bei einem üblichen AC 8 N Belag.

Rheologie Tests zeigten auf, wie die Verjüngungsmittel die mechanischen Eigenschaften von gealterten Bindemitteln im Ausbauasphalt wieder herstellen konnten. Ebenfalls ist der Alterungseffekt massgebend bei der Bestimmung der langjährigen chemischen und mechanischen Auswirkungen von Verjüngungsmitteln auf RAP.

Trotz der Zugabe von Verjüngungsmitteln bildete sich die physio-chemische Oxydation nicht zurück.

Das Projekt zeigte auf, dass 100% RAP Mischungen zusammengestellt werden können, die sich ähnlich wie AC 8 N Mischungen verhalten. Diese Belagsmischung wird in der Schweiz nur bei Strassen mit niedrigem Verkehrsaufkommen eingesetzt. Die Mischungen wurden auf ihre Bruchfestigkeit mittels Fracture Toughness Test (FTT) getestet. Für die Spurrinnenbildung wurde der Spurrinntest eingesetzt, und komplexe Modulwerte wurden mittels zyklische indirekte Zugversuche getestet (CIT-CY).

Die Untersuchungsergebnisse lassen folgende Schlüsse zu:

- Beläge mit 100% RAP konnten die Anforderungen, ermittelt durch den konventionellen volumetrischen Test sowie den Marshall Test, nicht erfüllen. Jedoch ist bekannt, dass das Erfüllen dieser Anforderungen nicht zwingend die gewünschte Leistungsfähigkeit des Belages garantiert.

- Performance orientiert Mix design mittels französischem Spurrinntest und Flexibilität Index (FI) wurde als praktischer Test eingestuft und hat die erwarteten Resultate geliefert.
- Es stellte sich heraus, dass es mit einer entsprechenden Veränderung der verschiedenen Mischungs-Kennwerte möglich ist, Deckschichten aus 100% Ausbauasphalt mit ähnlicher Performance zu entwickeln, wie konventionelle Beläge für niedrig befahrene Strassen bis zu 300 equivalent single axle loads (ESALs).
- Tests mit dem mobilen Verkehrslastsimulator (MMLS3) zeigten, dass ein Belag 100% rezykliertem Ausbauasphalt doppelt so vielen Überrollungen wie Herkömmlicher AC-Asphalt standhält, bis der Belag bricht.
- Alle Mischungen mit Recycling-Anteilen wiesen bei 0°C eine höhere Bruchfestigkeit aus als die Referenzmischungen. Die Untersuchungen legen aber den Schluss nahe, dass die Resultate des Bruchfestigkeitstests vor allem wegen der Testtemperatur von 0°C die Mischguteigenschaften wie Bindemittelgehalt, Viskosität und Abstufung der Körnung nicht aussagekräftig widerspiegeln. Es empfiehlt sich, den Fracture Toughness Test (FTT) nicht für die Bestimmung der Auswirkungen von Kennwertänderungen im Mischgut zu verwenden. Seit man festgestellt hat, dass der Bindemittelgehalt und die Viskosität wichtige Parameter bei Mischungen mit einem hohen Anteil an RAP sind, sollte der FTT nicht verwendet werden, um eine Optimierung von Mischungen mit hohem RAP Anteil zu entwickeln.
- Die Master-Kurven zeigen, dass die Steifigkeit von allen Mischungen über den ganzen Frequenzbereich ähnlich sind wie die Referenz. Deshalb hat die Verjüngungsmittel die Steifigkeit der Mischungen reduziert.
- Es war nicht möglich, ein Mischgut aus 100% RAP zu entwickeln, welches sich für den Belag auf Autobahnen eignete, da die Qualität des dazu verwendeten Ausbauasphalts sehr gering war. Deshalb empfiehlt es sich, RAP Management Prozesse einzuführen, die eine Verbesserung der Qualität und Homogenität von RAP Belägen bewirken.

Mögliche Orte für die Zugabe von Verjüngungsmittel an den Mischanlagen wurden evaluiert und deren Nutzen und Nachteile definiert.

- Eine Besprühung mit Verjüngungsmitteln des RAP Kaltmischguts während des Transportes auf dem Förderband ergab das gleiche Rissverhalten des daraus entstandenen Belages wie die Zugabe von Verjüngungsmitteln im RAP Heissmischgut während des Mischungsprozesses im Mixer.
- Hingegen zeigte bei gleichen Versuchsanordnungen für die Zugabe von Verjüngungsmitteln, dass der Belag aus RAP Kaltmischgut einen höheren Ermüdungswiderstand

aufwies als der Belag aus RAP Heissmischgut (allerdings konnten die Ermüdungstests nicht genügend wiederholt werden, um der Norm zu entsprechen).

- Die Prüfkörper aus dem im Mixer zugefügten Verjüngungsmitteln hergestellten Mischgut wiesen einen geringeres Komplex- Modul auf. Dies könnte sehr wahrscheinlich mit dem 0.5 % höheren Bindemittelgehalt in den Prüfkörpern zusammenhängen.
- Die Zugabe von Verjüngungsmitteln führte nicht zu einem erheblich erhöhten TOC-Gehalt im RAP, sei es bei einer Zugabe von 3% oder 5% verglichen mit 0%.
- Es konnte auch kein Unterschied betreffend des Einflusses auf die Art und Weise, wie das Verjüngungsmittel dem Mischgut zugeführt wird, festgestellt werden (aufgesprayt auf dem Förderband bei RAP Kaltmischgut oder Zugabe zum Mixprozess beim RAP Heissmischgut).
- Die Erhöhung des RAP Anteils im Mischgut hat auch eine Erhöhung der TOC Werte zur Folge, was eine bereits bekannte Auswirkung ist.

3. INTRODUCTION

When conventional materials are used, empirical relationships have been successfully used for multiple decades to design asphalt mixtures. However, when there is a significant deviation from the traditional practice, these relationships may not hold true anymore. The ambitious aim of designing 100% recycled hot mix asphalt certainly falls into this category. More proof of the performance is necessary than simple volumetric design.

Performance-based mix design principles can be applied in this case. A carefully chosen set of tests that demonstrate the mechanical performance can be used to compare the performance of an un-traditional mixture to conventional asphalt. Assuming that tests are chosen that are appropriate for the climate and truly reflect the expected field performance of the pavement, one can verify how the new material compares to and fulfils the standard requirements. In case of highly recycled asphalt it is most important to verify the cracking performance due to negative aging effect of the RAP binder. At the same time since rejuvenators are added, one must also verify if rutting does not exceed acceptable limits. Balancing of the cracking and rutting performance is possible with multiple means, most notably changing of gradation, and varying the binder content as it was done in this study.

A basic understanding of the effect of rejuvenator on the performance of the binder delivers fundamental knowledge that can be applied to the mixtures. Reclaimed asphalt pavement (RAP) is harder than virgin bitumen due to ageing and it needs to be rejuvenated. Generally, bitumen ages due to different processes such as, oxidation, changes in molecular organization, or loss of volatiles. For this reason, so called “rejuvenators” are used for restoring the properties of the RAP binder. To this end, in order to have a complete picture of the effect of rejuvenators, the viscosity reducing capacity of the rejuvenator should be considered, as well as its chemical composition. Fourier Transform infrared spectroscopy (FTIR) has been shown to be successful in analysing ageing as a result of oxidation [Marsac 2014]. Gel permeation chromatography (GPC) in combination with FTIR, were found to be useful for characterizing effects of ageing on the chemical structure of bitumen [Bowers 2014]. SARA (saturates, aromatics, resins and asphaltenes) fractioning has shown an increase of polar components due to ageing [Mousavi 2016]. As for mechanical performances, dynamic shear rheometer (DSR) is generally used for determining the time-temperature behaviour and the mechanical restoration that rejuvenators can cause [Yu 2014]. The objective of this study is to characterize and understand the effects of ageing of RAP modified with three rejuvenators. To this end, different techniques were used such as: FTIR for investigating oxidation, GPC for determining molecular size distribution,

SARA fractioning for detecting polar-nonpolar components, and DSR for analysing the mechanical time-temperature dependent behaviour.

4. OBJECTIVES

The objective of the research can then be summarized as follows:

- 1) Design a 100% recycled asphalt surface course and evaluate its cracking and rutting performance in comparison to a conventional reference mixture while verifying the potential to use the chosen test methods for design of mixtures.
- 2) In order to design the 100% RAP mixture evaluate the effect of various rejuvenators on RAP binder mechanical, chemical and microstructural development.
- 3) Evaluate the environmental effect of using such high RAP mixtures.

5. EXPERIMENTAL METHODS

5.1. BITUMEN TESTS

The pure bitumen, the bitumen extracted from RAP and rejuvenated RAP was investigated using a chemo-mechanical approach. The experimental methods used are as follows:

5.1.1. Dynamic shear rheometer

Dynamic mechanical properties of a bitumen may be expressed in terms of a complex shear modulus G^* that is composed of two components G' and G'' representing the dynamic storage and loss modulus respectively and are either time or frequency and temperature dependent. They can be determined through sinusoidal loading in a dynamic shear rheometer (DSR) from the phase angle δ between sinusoidal input load or deformation and material response together with the corresponding amplitudes of shear stress and shear strain.

G' gives a measure of the capability of the material to store energy and corresponds to the elastic part of a viscoelastic material whereas G'' refers to the viscous part and is related to the energy that the material loses due to the internal friction.

The reference temperature T_{ref} in this study was 20° C. In order to achieve the best fit between the measured values of the complex modulus and the values described by the sigmoidal model, the shifting algorithm was used. According to EN 14770 for the rheological measurements, 1.5 g of each material was placed in a disc shaped silicon mould with diameters of 8 mm and 25 mm and tested with the DSR Physica MCR at temperatures between -10° C to 80° C. According to EN 14770, the 8 mm plate-plate geometry with 2 mm gap was used in strain controlled mode for the temperature range -10° C to + 40° C while the 25 mm plate-plate geometry with 1 mm gap was used for the temperature range +40° C to + 80° C. Each measurement was repeated four times. Testing frequencies ranged from 0.1 to 20 Hz at each temperature.

5.1.2. Fracture Toughness Test

The fracture toughness test (FTT) is based on a European standard (European Committee for Standardization CEN TS15963, 2010). It is a three point bending test where the test sample is a beam with a thin notch in the middle. The samples are tested in a temperature controlled 99.5% ethanol bath in order to cool the sample from room temperature down to the one desired for testing (range -10° C/0° C). Samples were prepared by heating all binders at 110° C for 20 minutes to ensure the required viscosity for pouring the material in metal moulds without significant ageing. For each binder type, pre-notched samples were created. The pre-notch of 5

mm depth was introduced by inserting a 0.15 mm double film polyvinyl chloride paper in the middle of the specimen. The fracture toughness temperature (TFTT) of a bituminous binder is defined in the standard (CEN/TS 15963:20) as the temperature at which the deflection in the middle span at maximum force is equal to 0.3 mm. In order to calculate such temperature, the fracture toughness test was repeated at different temperatures until a deflection ≥ 0.3 mm was measured.

5.1.3. Attenuated total reflectance Fourier transform infrared spectroscopy

Attenuated total reflectance Fourier transform infrared (ATR-FTIR) is a method for determining chemical functional groups within a medium and was used to characterize chemical changes due to ageing and addition of rejuvenators in the material. ATR-FTIR measurements allow the identification of certain functional groups in bitumen. Infrared spectroscopy measures the infrared light absorbed by bonds in molecules when the infrared light has the same frequency as the vibration frequency of the bonds, enabling identification of chemical functionalities corresponding to the different bonds/functional groups, as a function of the wavenumber. The intensity of the peaks is a function of the concentration of the bonds/functional groups. In the case of aging in bitumen, such as in RAP binder, it is well established that changes in the intensity of the spectral peaks corresponding to carbonyl (peak around 1700cm^{-1}) and sulfoxide functional groups (peak around 1030cm^{-1}) are relevant as reported in the literature [Marsac 2014]. The ATR-FTIR measurements were performed at room temperature in a Tensor 27 from Bruker spectrometer using a diamond crystal. For the spectroscopy measurements, a small amount of bitumen (ca.10 mg) was placed directly on the diamond crystal with a metal spatula. The spectra were collected in the 4000 to 500 cm^{-1} wavenumber range with a resolution of 4 cm^{-1} and each spectrum represented an accumulation of 32 spectra. The data were collected with the OPUS ® software.

5.1.4. Gel permeation chromatography

Gel Permeation Chromatography (GPC) is an analytical technique that separates dissolved macromolecules by size, based on their elution from columns filled with a porous gel. The smaller molecules spend more time in the column and therefore will elute last. Conversely, larger molecules will spend less time in the pores and will be eluted earlier. For the gel permeation chromatography experiments, an HPLC-Pump (Agilent G1310B/1260 Iso Pump) was used with column oven (SFD 12590) at a temperature of 30°C . Two detectors were utilized: a variable wavelength detector (Agilent 1260 DEABB05519) with UV signal (wavelength equal to 215 nm)

and a refractive index (RI) detector (Agilent 1100/1200 G 1362A) in the range between 1 and 1.75. As eluent solvent, high purity tetrahydrofuran, at a flow rate of 1.0 ml/min, was used. The column was composed of Agilent PL 5M-mixed C, 5 μ m, 300 x 7.5 mm with a sampling rate of 9/s and volume of injection equal to 50 μ l.

5.1.5. Saturates Aromatics Resins Asphaltenes (SARA) fractioning

Saturates, aromatics, resins and asphaltenes (SARA) fractioning aims at finding to which extent polar and nonpolar components change due to ageing. Saturates include all hydrocarbon components with single-bonded carbon atoms. Aromatics include benzene and all their derivatives. Resins are components with a highly polar end group and long alkane tails. Asphaltenes are large highly polar components made up of condensed aromatic and naphthenic rings, which also contain heteroatoms. Pure asphaltenes are black, non-volatile powders. Saturates, aromatics and resins form the matrix, the so called maltene phase. It was observed that an increase of asphaltenes, which are polar components, is correlated with ageing and substantial decrease of aromatics. In this work, SARA fractioning was used to detect the chemical composition of each bitumen. To do so, high performance liquid chromatography was used. Asphaltenes were separated by precipitation in n-heptane. Afterwards, maltenes were split in saturates, aromatics and resins by silica gel NH₂ chromatography. Saturates were split thanks to refractive index chromatography while aromatics and resins thanks to UV chromatography. The procedure followed the requirements in the American standard D 4124 – 01.

5.1.6. Atomic Force Microscopy

Atomic force microscopy (AFM) is an imaging tool that delivers information on the topography and phase contrast of the sample surface and is particularly useful for multi-phase materials such as bitumen. The advantage of AFM is that it requires relatively simple sample preparation and operates under ambient conditions. Recently developed imaging techniques also allow the imaging of the dynamic interaction of the probe tip with the substrate material. In that case, in addition the probe is actuated and the damping of the vibrations of the probe while being in close contact with the substrate is registered and used as a feed-back signal. Also and especially in case of sticky substrates like bitumen, the difference in phase of the actuated vibration and of the registered response, the phase signal, can indicate areas which differ in stiffness and tackiness [Soenen et al 2014].

5.2. MIXTURE TESTS

Where possible, mixtures for mechanical testing were prepared using wheel compactor. It is considered that this compaction method replicates the field compaction conditions closer compared to gyratory and Marshall compaction.

5.2.1. Laboratory mixing

The materials, except rejuvenator which remained in room temperature, were heated in a laboratory oven to the mixing temperature of 170°C. Thereafter they were blended in an oil-heated laboratory mixer in the following sequence: RAP aggregates were pre-blended for 0.5 minutes after which rejuvenator was introduced at the required dosage and mixed for 1.5 minutes. Finally neat binder (if any) was introduced, followed by 3.5 minutes of mixing. It is considered that rejuvenators should be added directly to RAP, instead of pre-blending with fresh bitumen in order to allow direct contact with the RAP binder. This is expected to facilitate diffusion and activation of RAP binder.

5.2.2. Compaction

All samples that were tested for performance were prepared by French Roller Compactor. The loose mixture was short-term aged at a forced-draft oven at 150°C for 4 h followed by compaction using the roller compactor which was equipped with a steel wheel. The slab was compacted to dimensions of 100mm × 180mm × 500mm to a target density that equals that of Marshall specimens. Marshall samples were brought to compaction temperature of 145°C directly after mixing. This temperature corresponds to the requirements set in Switzerland National standard for the target penetration binder grade of 50/70. Compaction effort of 50 blows from each side was applied.

5.2.3. Wheel tracking test

Rutting resistance of the asphalt mixes was evaluated using French Rutting Tester (FRT). The FRT is run using a rubber pneumatic test wheel that has a pressure of 0.60±0.03 Mpa and a load of 500±5 kN is applied to the table and specimen as the wheel moves across the sample. A preconditioning load is applied at a room temperature for 1,000 cycles after which the sample is conditioned for about 16 hours at a temperature chamber that is set to 60°C. The test is run for 10,000 cycles and rut depth is measured using a gauge after 30, 100, 300, 1000, 3000, and 10,000 cycles at 15 pre-defined points across the length of the rut. Two parallel specimens are run and the mean rut depth at each number of cycles is reported.

Swiss technical specifications require rut resistance in terms of proportional rut depth of less than 10% for AC-S type mixes. However, this requirement applies to samples that are prepared using a pneumatic compactor (a steel wheel compactor was used in this research) so a comparison of rutting test results prepared using steel wheel and pneumatic wheel was made for the same virgin AC-8 S type asphalt mixture. The results demonstrated 5.9% proportional rut depth for steel wheel and 3.5% rut depth for the pneumatic wheel at 10,000 cycles. The bulk density of the samples can be considered similar at 4.0% for the steel wheel and 3.5% for the pneumatic wheel compactor. These results demonstrate that the same requirements can not be applied for the two types of compactors. Finally, it was decided to continue with the steel wheel compaction because of ease of operation and the fact that it produced more homogeneous densification through the specimen which is important when cracking test specimens were cored out from different locations within the slab.

5.2.4. Semi Circular Bend (SCB) test

To prepare a semi circular bend test sample a cylindrical sample is cored from asphalt slab, trimmed to the required height of 50 mm and cut in half. In this research slabs were prepared using steel compaction wheel (developed at EMPA) to a target density that equals that of Marshall samples, but use of gyratory compactor or field cores are also permitted according to standard. A notch is then cut into the half cylinders to control the crack initiation point. The specimen is positioned in a three point testing frame as can be seen in *Figure 1* and a load is applied at a monotonic rate along the vertical axes. Load and displacement are measured during the test.

This is an empirical test to compare mixes and screen the ones that are prone to cracking so the range of acceptable results will vary according to local environmental conditions, application of the mixture, nominal maximum aggregate size, bitumen type and grade, air voids, expected service life, etc. Therefore a local criteria should be established.



Figure 1 Semi Circular Bend (SCB) test frame

Two variations of Semi Circular Bend (SCB) test methods were considered. The test conditions as were used in this research for both test variants are summerized in *Table 1*.

AASHTO TP 124-16: Standard method of test for determining the fracture potential of asphalt mixture using semicircular bend geometry at intermediate temperature. From here on referred to as “American SCB”. There were three main deviations from the test method:

A test frame according to EN requirements was used. The main differences are that (1) the two base wheel bearings are 35mm and instead of 25mm in diameter and (2) the loading head is flat instead of concave.

The notch width was 3,5 instead of required 1,5mm.

The cilindrical samples were cored from asphalt slabs instead of compaction using gyratory compactor and the target density was that of Marshal samples instead of 7% as in standard.

EN 12697-44: Crack propagation by semi-circular bending test. From here on referred to as “European SCB”. There is one deviation from the standard:

Standard states that notch should be 0,35 mm in width, which is believed to be a mistake. A notch of 3,5 mm in width was actually cut.

Table 1 SCB test conditions for European and American test methods as used in study

<i>Parameter</i>	<i>European SCB</i>	<i>American SCB</i>
Parameter	European SCB	American SCB
Sample diameter	150 mm	150 mm
Sample thickness	50 mm	50 mm
Test temperature	0 °C	25 °C
Loading rate	5 mm/min	50 mm/min
Notch	10 mm deep and 3.5mm wide	15 mm deep and 3.5 mm wide
Sample preparation	Roller compactor, coring, cutting	Roller compactor, coring, cutting

The samples were compacted using steel wheel compactor to target density that equals density of respective Marshall samples. The result interpretation differs for each of the methods and is explained further.

5.2.5. SCB at 0°C (Fracture Toughness)

Result calculation

The parameters that are used for result expression are summarized in Figure 2 and explained briefly here:

Work of fracture (Wf) is calculated as the area under the load versus displacement curve.

Fracture energy (Gf) is calculated by dividing work of fracture by ligament area (the product of ligament length and the thickness of the specimen).

$$G_f = \frac{W_f}{Area_{lig}} \times 10^6 \quad \text{Equation 1}$$

where

Arealig = ligament length × t

t – specimen thickness, mm

Post-peak slope (m) is drawn at the inflection point of the post peak load-displacement curve.

Critical displacement (u1) is the intersection of the tangential post-peak slope with the displacement axis.

Flexibility index (FI) is calculated from fracture energy and post peak slope.

$$FI = \frac{G_f}{|m|} \times A$$

Equation 2

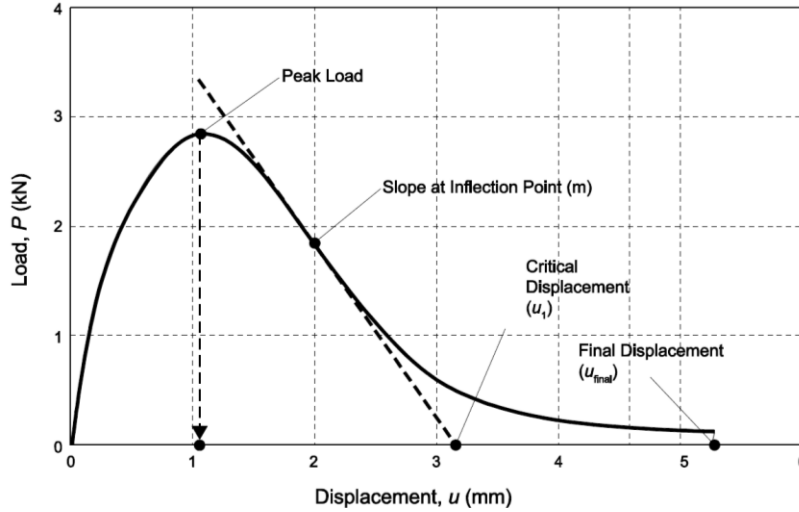


Figure 2 Typical test result for American SCB test

Result interpretation

According to the authors of the test method (Ozer, Al-Qadi et al. 2016) the two critical parameters are fracture energy (G_f) and Fracture index (FI). Larger fracture energy indicates asphalt mixtures that can withstand greater stresses with higher damage resistance. This parameter is derived from work-of-fracture (the area under load vs displacement curve) that was refined by RILEM (RILEM TC-50 FMC 1985). It was, however, further discussed by the authors, that the pattern of the load-displacement curve, especially the post-peak part is also important to discriminate cracking potential of mixtures. It was found that estimated crack propagation velocity correlates well with the post peak slope (m) which was therefore used in calculation of the FI. The FI

A correlation of FI with field performance was carried out using results from FHWA (Federal Highway Agency) test track. Here, seven different mixes with various RAP and RAS (reclaimed asphalt shingles) contents and different warm mix asphalt technologies were placed using equal structural design and tested for cycles to fatigue threshold. The results of this study correlate well with the results of FI. Based on the study results, FI thresholds for distinguishing between good

(FI>10), acceptable (FI>6) and bad (FI<2) performing mixes were proposed, with a note that these should be adjusted based on local circumstances (Ozer, Al-Qadi et al. 2016). Based on these results it was concluded that fracture index provides means to identify brittle mixes that are prone to premature cracking and the FI distinguishes between mixtures more clearly than fracture energy.

European SCB

Result calculation

Strain at maximum force ($\epsilon_{\max, i}$)

$$\epsilon_{(\max, i)} = W / \Delta W$$

W is the height of the specimen, mm

ΔW is the vertical displacement at maximum force of specimen

Maximum stress at failure (σ_{\max})

$$\sigma_{(\max, i)} = (4.263 \times F_{\max}) / (D \times t) \times 10^{-6}$$

D is the diameter of specimen, mm

t is the thickness of specimen, mm

$F_{\max, i}$ is the maximum force of specimen, N

Fracture toughness (K_{Ic})

$$K_{Ic} = \sigma_{\max} \cdot f(a/W)$$

K_{Ic} is fracture toughness, $N/mm^{3/2}$

a is notch depth of specimen, mm

W is height of specimen, mm

σ_{\max} is stress at failure of specimen, N/mm^2

$f(a/W)$ – geometric factor. For sample size used in this study it is 5,956.

Result interpretation

The two critical parameters of the test are tensile strength and fracture toughness of the specimen. These results can then be used to assess potential of a material to resist crack propagation. Ideally the results should be viewed in tandem with fatigue test results, which cover the crack initiation phase and therefore are complementary to this test. The standard recommends performing the test at 0°C, although other temperatures are permitted. Previous research results have demonstrated that fracture toughness was not dependent on specimen thickness in the range of 25-75 mm at temperatures below 15°C (Saha and Biligiri 2016), which is beneficial if field specimens are to be tested. It has also been reported that increase in air voids reduces fracture toughness and the results are sensitive to binder content and binder grade (Saha and Biligiri 2016). No reports on the test result calibration or correlation with field performance have been found, but laboratory research reports demonstrate sensitivity of the test results to type of aggregates, air void content, binder grade, and modifier type.

The test method has been included in the European Standards for allowing specification of AC (EN 13108-1) and SMA (EN 13108-5) type asphalt based on minimum fracture toughness. The range of categories provided in the standard is from 10 to 55 N/mm^{1,5}.

5.2.1. Stiffness

Stiffness of the asphalt mixtures was determined in indirect tension mode on cylindrical specimens (CIT-CY) according to EN 12697-26. For each type of mix four samples of 100 mm diameter were cored from the asphalt slabs and cut to 40 mm in height. The tests were performed by applying a sinusoidal load at frequencies of 0.1, 1 and 10 Hz. The load level was chosen to induce horizontal strains in the specimen in the range between 0.05 and 0.10 that is assumed to be in the linear viscoelastic range for the samples and no permanent damage is induced. Testing temperatures were chosen following a sensitivity study where the initial mix design was tested in 10°C increments from -10°C to +40°C with the aim to find a wide range of stiffness for later input in master curve calculations (explained lower). It was determined that using the three temperatures of -10, 20 and 30°C compared to any other group of three test temperatures ensured the smallest error in calculation of master curve as compared to testing at six temperatures.

Master curve uses the principle of time-temperature superposition to shift data at multiple temperatures and frequencies to a reference temperature so that the stiffness data can be viewed without temperature as a variable. This method of analysis allows for visual relative comparisons to be made between multiple mixes.

A sigmoidal model was used as proposed by Witzack and Fonseca (1996), with the shift factors calculated following the Williams-Landel-Ferry (1955) relation

Where a_T is a factor for shifting complex modulus at certain temperature T to a reference temperature T_{ref} (20°C in this study). $C1$ and $C2$ are material constants and a least squares regression was performed to obtain the parameters.

6. MATERIALS

6.1. RECLAIMED ASPHALT

The reclaimed asphalt used in this project was of one batch that was stored for the duration of the project. Both lab mixtures and plant mixtures used the same batch. RAP from a Swiss supplier (BHZ Baustoff Verwaltungs AG) was used. The toluene extracted binder content was 4.60% by weight of the mixture, having a penetration of 22 x 10⁻¹ mm at 25° C and a softening point equal to 65.7° C. 50/70 virgin binder showed a penetration of 62 x 10⁻¹ mm at 25° C and a softening point temperature of 48.75° C.

6.2. PLANT PRODUCED MIXTURE

The plant produced mixtures were transported from the plant in 1 tonne batches in carton boxes of 25kg each as shown in figure x. The process assures homogeneous distribution of the mixture in the boxes. The boxes were then heated, mixed and compacted in the laboratory.

Figure X: include photo

6.3. REJUVENATORS

Three commercial bio-based rejuvenators were used: rejuvenator defined as “A” from natural seed oil, rejuvenator designated as “B” from cashew nut shell oil and rejuvenator “C” from tall based oil. The rejuvenator dosage was set at 5% by mass of RAP binder in order to have a direct comparison of the effects of the three.

7. ENVIRONMENTAL EFFECTS

The content of this chapter is a summary of the following publication. For complete information the reader is referred to this publication or the authors.

Muñoz M., Zaumanis M., Poulikakos L.D, Cavalli, MC; Haag R., Heeb, N. Environmental impact of rejuvenators in high RAP mixtures, In preparation

Various oils have been used as the base of rejuvenators, including flux oils, slurry oils, lube stock, lube extracts, extender oils, bio-based oils, waste-engine oils, waste-vegetable oils, etc. [Zaumanis 2014]. They are also often commercial products with proprietary composition. Because of the multiple potential sources and un-known composition of the products, it is not obvious that all the rejuvenators are safe to the people and environment. The sustainability of a specific rejuvenator should therefore be established by determining if its usage has harmful effects during production and paving when it is exposed to elevated temperatures as well as during the service life when asphalt is exposed to the elements and traffic. Such evaluation is carried out in this re-search by testing rejuvenator composition, simulating aging at production temperature, determining potential leaching to groundwater from rejuvenated asphalt use as well as testing released particulate matter as a result of trafficking. Emissions due to use of rejuvenators from full scale production were also measured in the project and are reported elsewhere [Zaumanis et al., 2018].

7.1. EXPERIMENTAL METHODS

7.1.1. PAH in RAP binder

A preliminary PAH analysis was performed in order to know whether the PAH content in the binder was below the Swiss recommendation limit (5000 ppm). An aliquot of the extract from the binder extraction (explained above) was taken for PAH analysis. An aliquot of the solvent used (toluene) was also analyzed as a reference for background determination. PAH analysis was performed following a multistep cleanup procedure described before [Munoz 2016]. The final extract is analyzed by means of ultra-high gas chromatography (Fisons Instruments HRGC Mega 2, Rodano, Italy) on a 30 m x 0.25 μ m x 0.10 μ m capillary column (Restek, Belle-fonte, USA). Detection and identification of compounds were achieved by high resolution mass spectrometry (Thermo Finnigan MAT 95, Bremen, Germany) in electron-impact ionization mode (GC/EI-HRMS).

The internal standard method is used for PAH quantification and therefore prior to the analysis, samples are spiked with a known amount of a mix containing the deuterated 16 PAHs internal standards. Five concentrations containing deuterated compounds, 16 native PAHs (Supelco, Bellefonte, USA) were analyzed to determine respective calibration curves and response factors. A similar approach was used for PAH determination in the rejuvenators.

7.1.2. PAH and heavy metals in rejuvenators and raw asphalt samples

Before performing the leaching tests, an amount of 5 grams of each sample was Soxhlet extracted with di-chloromethane, followed by the same cleanup procedure explained above. The final extract was analyzed by means of the recent acquired equipment, HRGC-Ultra-HR-Orbitrap-MS (Thermo-Fisher Scientific, Germany). The samples studied here were: Virgin, RAP and RAP+rejuvenator C, all unaged and aged.

Heavy metals were qualitatively measured in the virgin asphalt sample, RAP, RAP+Rej and in the rejuvenator C alone. The metal content was determined following a standardized Empa procedure (SOP Nr.6000) by means of WD-XRF.

7.1.3. Leaching test

Leaching tests were performed on the same samples in triplicate (except for virgin, which was done in duplicate). According to the literature there are different procedures to evaluate leachates [Brantley 1999, Legret 2005]. One of the most commonly employed method is the batch Toxicity Characteristic Leaching Procedure (TCLP) [US EPA 1311]. We implemented this methodology in our laboratory. This test implies leaching a size-reduced sample (particle size < 9.5 mm) in an acidic solution for 18h at a liquid-to-solid ratio of 20 to 1.

Briefly, the initial alkalinity of the material is measured to determine the extraction fluid to be used. In our case, fluid 1, an acetic acid solution in ultrapure water with pH 4.93 was used. 100 g were placed in glass bottles and 2 L of the acidic solution were added. After that, the extraction fluid is filtrated through a 0.6-0.8 μm borosilicare.

Extraction of the PAHs was performed by means of ENVITM-18 DSK solid phase extraction disks according to the US EPA Method 525.1[30]. Identification of PAHs was performed by using HRGC-Ultra-HR-Orbitrap-MS (Thermo-Fisher Scientific, Germany).

Aliquots of 50 ml each from the filtered solution were used for heavy metal and mercury analysis in the leachates. Quantitative determination of Al, As, Cd, Cr, Cu, Ni, Pb, V and Zn was performed by means of ICP-MS (Agilent 8800 QQQ). For the quantitative determination of Hg

atomic absorption FIMS was used (FIMS-400, Perkin-Elmer). Finally, for the determination of Fe and Mn, ICP-OES (Agilent 5110) was used. For quality purposes, the analysis of certified standard material was also performed and results compared.

2.4.1. Determination of PAHs in leachates

Organic compounds need to be extracted from the filtered solution (2 L). Extraction was performed by means of ENVITM-18 DSK solid phase extraction disks according to the US EPA Method 525.1[30]. Identification of PAHs was performed by using HRGC-Ultra-HR-Orbitrap-MS (Thermo-Fisher Scientific, Germany).

7.1.4. Determination of heavy metals in leachates

Two aliquots of 50 ml each from the filtered solution were used for heavy metal and mercury analysis in the leachates. Quantitative determination of Al, As, Cd, Cr, Cu, Ni, Pb, V and Zn was performed by means of ICP-MS (Agilent 8800 QQQ). For the quantitative determination of Hg atomic absorption FIMS was used (FIMS-400, Perkin-Elmer). Finally, for the determination of Fe and Mn, ICP-OES (Agilent 5110) was used. For quality purposes, the analysis of certified standard material was also performed and results compared.

7.2. PAH IN INITIAL BINDER AND REJUVENATORS

The concentration of the 16 PAHs in the initial binder sample from RAP was found to be 4800 mg/kg which is below the Swiss limit of 5000 mg/kg [Bafu 2006]. Very low concentrations were found in the rejuvenators. These values agree with the literature (e.g.: pyrene concentrations in a sunflower oil was found to be 0.001-0.0027 mg/kg) [Ciecierska 2013, Mafra 2010]. The limit values for PAH binder content differ significantly between countries. Germany has established a value of 20 mg/kg of EPA-PAH in binder [RuVA-StB 2001; LAGA 1997; Umwelt 2016], which would be much below the values found here. The same levels apply to Austria and The Netherlands, which also have lower values.

7.3. PAH CONTENT IN RAW MIXTURES

Concentrations found in the raw samples, which were used for leaching tests are shown in Figure 3. PAH content (sum of 16 PAHs) in all samples was below the Swiss legislation limit (250 mg/kg asphalt).

These findings indicate that these asphalt sample can be reused in terms of PAH concentrations. Even though, PAH concentrations were below the limit in both, RAP and RAP + rejuvenator C, leaching tests need to be performed to evaluate the risk of pollutants that may be leached.

Results shown in Figure 3 illustrate that RAP containing rejuvenator C contains lower PAH concentrations (sum of 16 PAHs), 8 and 33 % in the unaged and aged samples respectively. It may happen that they are partially retained in the matrix and they are not fully extracted with the organic solvent (dichloromethane). On average, PAH content in RAP was 7-fold higher than in the virgin sample.

On the other hand, aging has no effect on PAH concentration on the RAP, whereas the concentration in the aged RAP+Rej C sample was 27% lower than in the unaged was found. However, these are the results for only one sample.

Even though, PAH concentrations were below the limit in both, RAP and RAP + rejuvenator C samples, leaching tests need to be performed to evaluate the risk of pollutants that may be leached. Threshold values or tolerable concentrations of these compounds in groundwater, soil and other types of water are much lower and in the level of few $\mu\text{g/L}$. Very low concentrations of PAHs leached to the water or soil are able to damage biota and ecosystems.

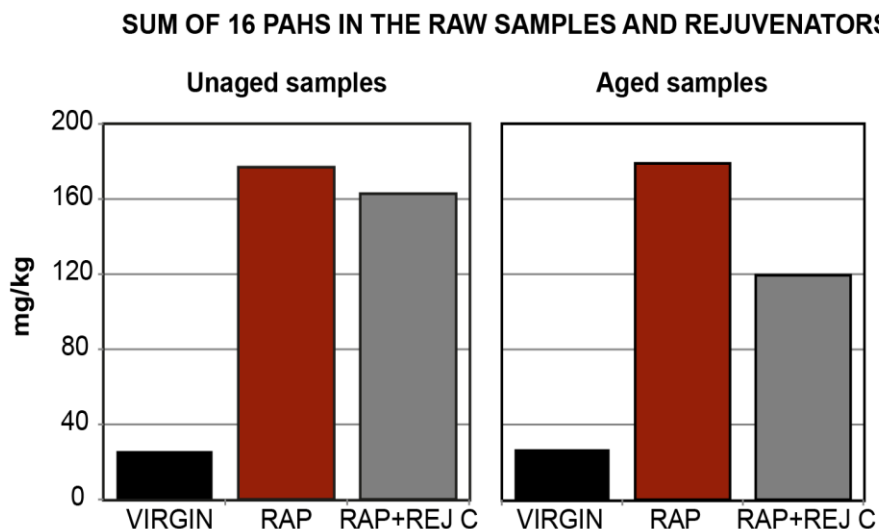


Figure 3. 16 PAH concentrations in the raw samples (unaged and aged) which were used for leaching

7.4. METAL CONTENT IN RAW MATERIAL AND REJUVENATORS

Most toxic heavy metals (Cd, Cr, Cu, Ni, Pb and Zn) were not found or found in very low concentrations in all samples tested. However, the low concentrations found could pose a risk if these compounds are leached and finally end in water.

Table 2. Heavy metal content in the initial binder (qualitative method).

Sample	Result and concentration range
	High concentrations: S (%)
RAP	Medium concentration: Si, Cl, K, Ca, V, Fe (< 1000 ppm) Very low concentration: Ni, Cu, Zn (trace, few ppm)
RAP + 5% rejuvenator C	High concentrations: S (%) Medium concentration: Si, Al, Mg, Cl, K, V, Fe (< 1000 ppm) Very low concentration: Ni, Cu, Zn (trace, few ppm)
Virgin (reference)	High concentrations: S (%) Medium concentration: Al, V (< 1000 ppm) Very low concentration: Ca, Fe, Ni, Zn (trace, few ppm)
Rejuvenator C	High concentrations: --- (%) Medium concentration: Si (< 1000 ppm) Very low concentration: Ca, P, S, Zn (trace, few ppm)

7.5. PAHS AND METAL CONTENT IN LEACHATES

A complete list with individual concentrations in leachates is shown in *Figure 4*. For comparison purposes, we made the assumption that all the PAHs from the raw samples (27 – 180 mg/kg) are leached. This would result in concentrations shown in *Figure 4* (upper right). These values are then compared with the concentrations obtained in the leachates (*Figure 4*, upper left).

It is found that less than 0.5% of the PAH content in the raw material is leached under the given conditions. Moreover, the trend observed above for the sum of the 16 PAHs, where lower concentrations were found in the RAP+Rejuvenator C sample, is also observed here. On the other hand, concentrations in leachates from the aged samples were found to be 2-fold lower than in the unaged samples. Relative standard deviations were in all cases below 10 %.

Individual PAH patterns are also displayed in *Figure 4*. Patterns are very similar in the unaged and aged samples. However, it can be deduced these data, that concentrations of higher molecular weight compound (fluoranthene, pyrene, chrysene...) are lower in aged samples compared to the unaged samples. Differences between patterns in RAP and RAP+Rej C are negligible, as shown in *Figure 4*.

Regarding the heavy metal content, concentrations in most samples were found to be below the detection limits. For comparison purposes the concentration limits in drinking water standard according to an EC Directive are 5 µg/L for Cd, 50 µg/L for Cr, 2000 µg/L for Cu, 10 µg/L for Pb, 1 µg/L for Hg, 20 µg/L for Ni and 10 µg/L for As (according to WHO). The concentrations found for heavy metals are also in agreement with literature [RuVA-StB 01.2001]. Fe content was found to be slightly higher than values given in the regulation.

However, taking a look to individual PAHs, it is observed that the aforementioned trend changes. A slight increase in PAH concentration is observed in those sample with rejuvenator, like for benzo(a)pyrene (Figure 4). Moreover, if leachate concentrations are compared to concentrations of selected PAHs in different environmental media like drinking water, groundwater or surface waters, it is found that for some PAHs those limits are exceeded. This is sometimes controversial in literature as it is not clear to which values they should be compared to. In *Table 3* concentrations of selected PAHs and sums of some PAHs and limit values in two different regulations are compared. However, how to interpret these results is not clear because, these values are given for the concentration of those pollutants in the water. Assuming that the leaching effluent ends up in, for example, the drinking water system, this would mean that benzo(a)pyrene concentration will be 1-3 times above the limit value.

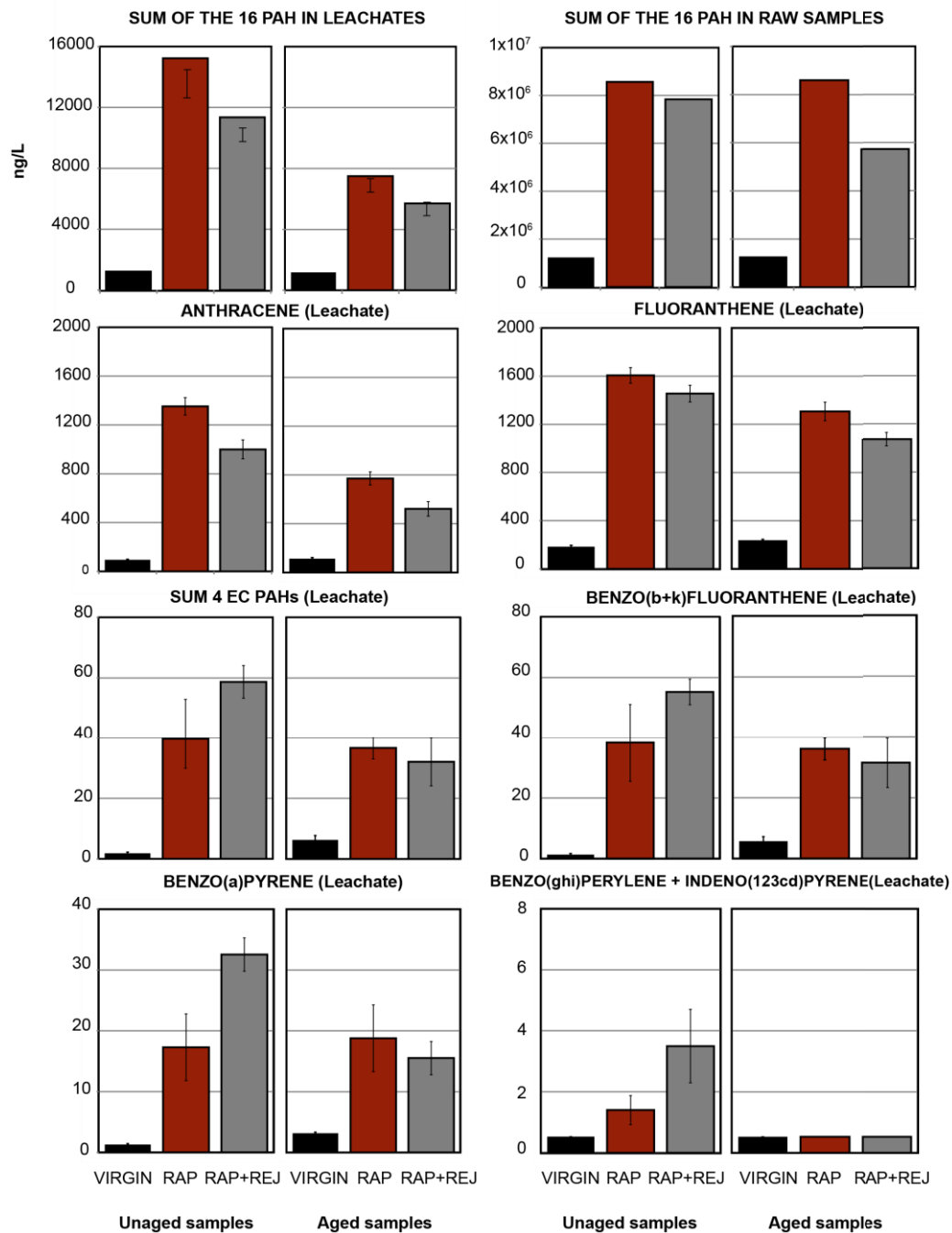


Figure 4. Concentration of 16 PAHs in the leachates of a 100g sample (S/L: 1/20, 2 L) and the theoretic concentration assuming that all PAHs contained in 100 g of the raw material would be dissolved in the same amount of liquid used in the leaching tests (2 L). Individual patterns are also shown (below). * Different units are used here in comparison to Figure 6 to make easier the comparison with the leachate samples

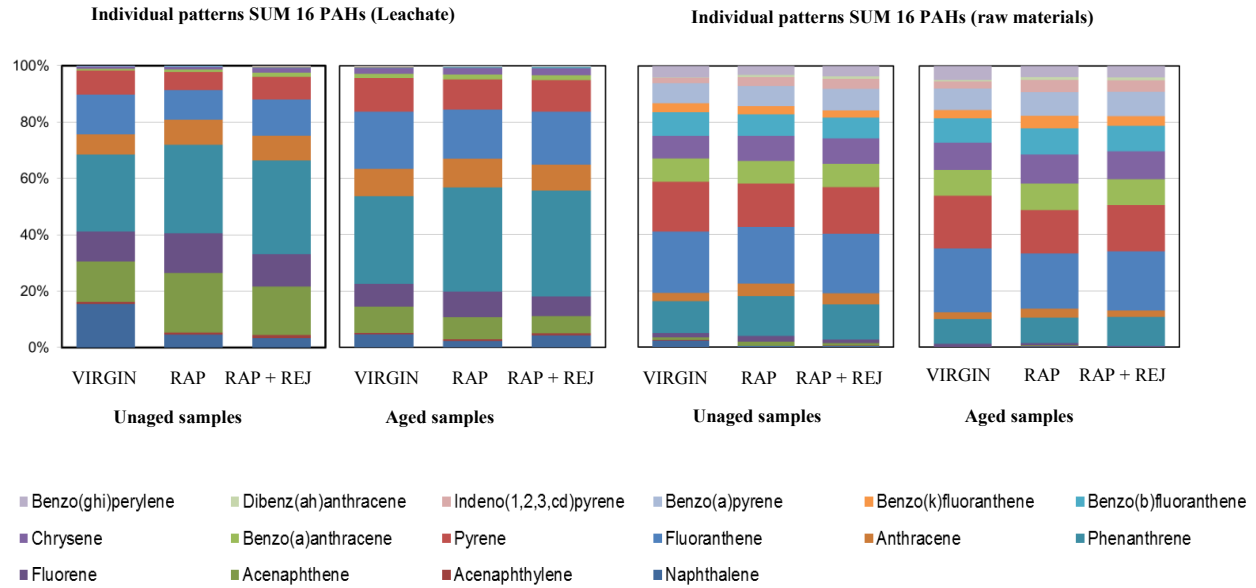


Figure 5. Individual patterns for the sum of 16 PAHs in leachates and raw material.

Table 3. Concentration of selected PAHs mentioned in different regulations and respective limit values. Concentrations at the detection limit are indicated in italics. Values in bold indicate that they are above the limit according to the regulation.

	LEACHATES (ng/L)						ng/L		
	UNAGED			AGED			Drinking water	EQS - Inland/Surface waters	
CHEMICAL	VIRGIN	RAP	RAP+ REJ C	VIRGIN	RAP	RAP+ REJ C	98/83/EC [15] Swiss regul. [26]	2008/105/EC [16]	x-fold (max)
Naphthalene	202	680	380	54	162	240		2400	
Anthracene	95	1350	1000	113	772	522		100	13.5
Fluoranthene	180	1610	1460	238	1310	1070		100	16.1
Benzo(a)pyrene	1.3	17	33	3.2	19	16	10	50	3.3
Sum Bghi+Ipyr	0.5	1.4	3.5	0.5	0.5	0.5		2	
sum B(b+k)F	1.3	38	55	5.7	36	32		30	1.8
sum 4 (EC regul)	1.8	40	60	6.3	37	32	100		
sum 16 PAHs	1310	15230	11360	1170	7490	5703			

7.6. PRODUCTION IN ASPHALT PLANT

During the mixture production process discussed in section 11, pollutants emissions in the plant were measured. The BHZ Birmensdorf asphalt mixing plant is equipped with a micro-processor plant control system "Ammann as1". The as1 plant control system records and stores the operating data of all actuators and sensors continuously to a database from where it can be downloaded, converted and processed for further evaluation. In the test evaluation, a sampling rate of 1 data points per 30 seconds was used. After raw data conversion, both the emission and plant data were merged into one common database using NI (National Instruments) DIAdem software. DIAdem is designed to quickly locate, inspect, analyze, and report on large volumes of measurement data.

The experimental production was carried out in six windows by ensuring a sufficient time to gather representative emissions measurements for each production case. In addition to the 100% RA mixtures that were used for testing of mixture properties, 60% RA mixture (None-0-60) was produced. The None-0-60 mixture was chosen as the reference because it is within permitted RAP rate locally and thus provides a reasonable baseline. The mixture composition and time of production windows according to the sequence of production are summarized in Figure 9. The average temperature during the testing day was 23°C with no precipitation ensuring favorable ambient conditions for the measurements.

The emission test results and production temperature are summarized in *Figure 6* according to the sequence of production. First of all, it is important to recognize that heating temperature is a significant contributor towards generating emissions. In order to minimize the variables, every effort was placed to achieve constant temperature throughout the six production windows. It can be seen in Figure 9 that this was achieved since RA material temperatures are in a narrow 160 °C to 167 °C range for all evaluation windows. For this reason RA material temperature influence on TOC emissions can be said to be negligible and will not be considered in further analysis.

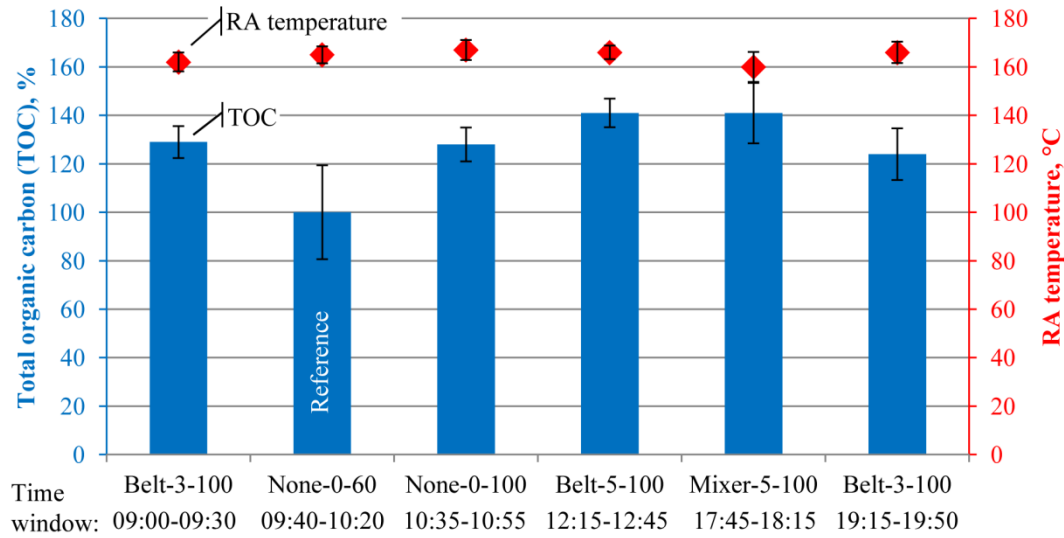


Figure 6. Total organic carbon (bars on primary axes) and RA temperature (rhombs on secondary axes) test results (average result of each measurement time window)

As mentioned earlier, the None-0-60 mixture (60% RA) is used as the TOC emission reference. All other TOC emission values in Figure 6 are normalized relative to this reference. The following findings can be drawn by analyzing the TOC emissions in the figure:

1. Two production windows of the Belt-3-100 in the morning and evening were used to evaluate consistency of the measurements. It can be seen in Figure 6 that the two production windows resulted in virtually identical TOC emission indicating that a good measurement consistency throughout the day was achieved.
2. The different windows with various contents of rejuvenator at 100% RA rate can be used to evaluate the effect of rejuvenator on TOC emissions. It can be seen that compared to the 0% rejuvenator window (None-0-100), the TOC emissions at 3% and 5% rejuvenator windows vary slightly but, considering the test variability, they can be considered similar. Therefore, based on the limited production data in this experiment, no clear correlation between TOC emissions and rejuvenator addition rate can be stated.
3. Comparison of the Belt-5-100 and the Mixer-5-100 windows allows evaluating the effect of rejuvenator addition location on TOC emissions. It can be seen in Figure 9 that the emissions in both cases are identical, indicating that there is no difference in TOC emissions whether 5% rejuvenator is sprayed on the cold RA on the belt or on the hot RA directly into the mixer.
4. Since there is no significant effect of either rejuvenator addition location or rejuvenator content, the 100% RA production windows can be compared to the reference 60% RA window (None-0-60). On the average 100% RA samples resulted in 33% higher TOC emissions

compared to the 60% RA window. Higher TOC emissions are a common and known effect when increasing the RA rate. Generally the main contributors for this are: fumes from heating RA bitumen, RA material grading, RA bitumen type and content, moisture content, fumes from fresh bitumen.

It must be noted that the above results and findings are based on and only valid for:

- The present one-day measurement campaign with only a limited number – a total of six production and evaluation windows – during the test.
- The particular plant type, process and design.
- The plant operating conditions during the test.
- The material properties – and in particular the RA material properties – during the test.
- The specific rejuvenator used during the test.

Results may vary significantly if one or more of the above mentioned parameters are changed.

7.7. ENVIRONMENTAL TESTING AND MONITORING

During the MMLS3 tests (refer Chapter 10) it was possible to monitor the Emission in form of dust due to pavement abrasion [Gehrig 2010].

Particle abrasion measurements were performed in an open environment and the setup is illustrated in Figure 6. TSI APS Model 3321 aerodynamic particle size spectrometer was used to count the abraded particles. An SF₆ tracer gas (Sulphurhexafluoride) was introduced next to the wheel at the direction of the movement and it was collected at the other end of the MMLS3 next to the point of sampling particles. This allowed to determine the air volume flow under the MMLS3 according to Equation 3 (Gehrig et al., 2010).

$$V = (f_{tr} \cdot c_{tr}) / c_{meas} \quad \text{Equation 3}$$

, where f_{tr} – flow of tracer gas injection, ml/min

c_{tr} – concentration of injected tracer gas, ppm

V – Air flow volume through the simulator, m³/min

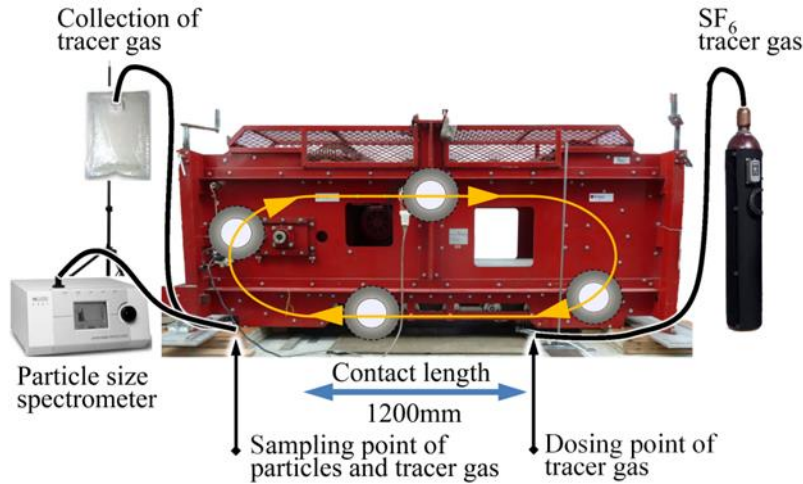


Figure 7 Setup of the particle abrasion test

From the measured particle concentrations, the driven wheel distance and the air flow volume below the MMLS3, an emission factor can be calculated according to the Equation below. Only particle size up to 10 μm was considered (PM10) and as previously reported by Gehrig et al. (Gehrig et al., 2010) it was assumed that the density of released particles is 1 g/cm³.

$$EF = ((c_{op} - c_{amb}) \cdot V) / d$$

, where EF – Emission factor, mg/km

c_{op} – Particle concentration during operation, mg/m³

c_{amb} – Particle concentration of ambient air, mg/m³

d – Driving distance of the simulator wheels on the pavement per minute, km/min (0.140)

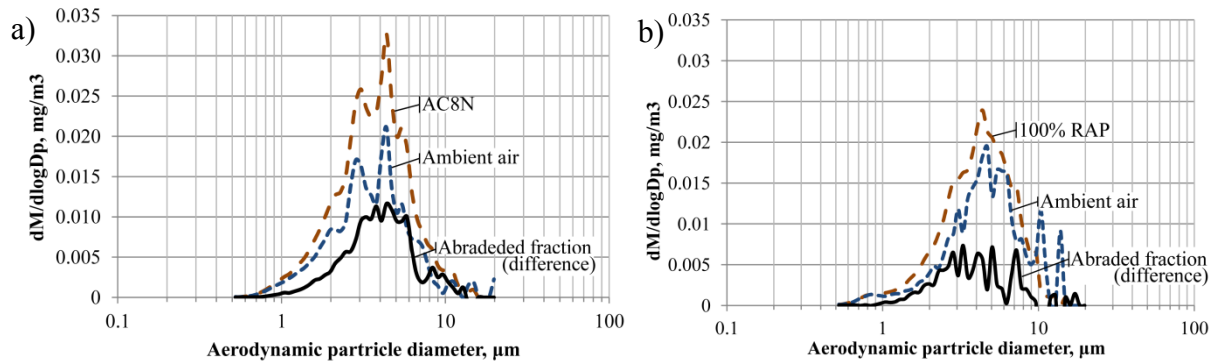


Figure 8 Average particle size distribution over 1h of test (a – AC8N and b – 100% RAP)

Figure 8 demonstrates the average particle size distribution during the MMLS3 loading period of 1h. Before starting the MMLS3 test, the particle distribution in ambient air (background) was collected and these results are also illustrated in the figure. The difference between these two distributions is the amount of abraded particles from asphalt as a result of the MMLS3 wheels passing over slab. It can be seen in Figure 8 that some particles are being abraded in the air from the asphalt slabs, but the amount is even lower than the amount of particles already in the ambient air. It can also be seen that the amount of particles in ambient air in both cases is similar despite the tests being performed a month apart. This shows that the results are relatively robust.

The calculated emission factors throughout the test period of 60 minutes are illustrated in Figure 9. The sampling was performed in 5 minute intervals for the 100% RAP and 10 minute intervals for the AC8N mixture. The average emission factor for each sampling period is illustrated in the figure. Only one measurement per asphalt type was performed and therefore no statistical evaluation is provided. It was previously reported by Gehrig et al. [Gehrig et al., 2010] that for this test a measurement uncertainty of 30 % can be assumed at 95% confidence interval. It can be seen that the AC8N throughout most of the test has higher emission factor compared to the AC8N. By average the emission factor of RAP is 0.94 mg/km and for the AC8N mixture it is 5.8 mg/km. The results of the AC8N mixture are similar to what has been reported previously and such emission factors can be generally considered low [Bukowiecki et al., 2010; Gehrig et al., 2010]. It can be hypothesized that the recycled asphalt is abrading fewer particles than the AC8N because the mineral aggregates have been encapsulated by the bitumen twice (originally and during recycling) and/or because it has lower density at 2.3 % compared to 2.8 % for the AC8N mixture.

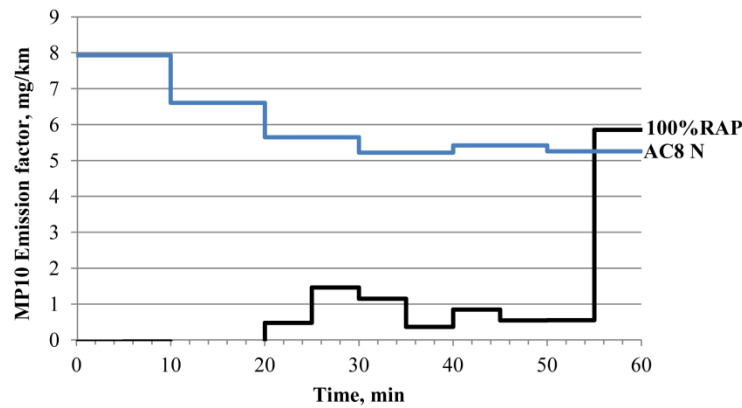


Figure 9 Emission factor as a function of time

7.8. SECTION CONSLUCIONS

The initial hypothesis that rejuvenators may mobilize PAHs bound in the RAP materials and therefore PAHs would be released or leached more easily from those materials was found to be not correct for the sum of the 16 PAHs. However, a more specific look to individual PAHs shows that indeed some PAHs are released at high levels from unaged RAP with rejuvenator. Other PAHs, especially low- to medium-sized PAHs are retained better in the material in the presence of rejuvenator C. Rejuvenators seem to stabilize the chemical structure of the asphalt samples, probably by intercalation or adsorption on surfaces in the matrix and therefore making it more difficult to be released or leached. Concentrations of 16 PAHs in leachates from RAP+Rej C were 25% and 24% lower than in the RAP for the unaged and aged samples respectively.

Aging is having an effect on PAH concentrations of the leachates, where lower concentrations of the 16 PAHs were found in comparison to the unaged samples. However, if we take a look to individual PAHs, this trend differs for higher molecular weight PAHs. Benzo(a)pyrene, the most carcinogenic PAH, has a 1.9-fold higher concentration in leachates of the RAP+REJ C (unaged) sample. Concentrations of higher molecular weight PAHs in the RAP+Rej C samples are in general higher than in the RAP in the unaged samples. Moreover, concentrations of some PAHs or group of PAHs exceed the limit values for drinking and/or inland/surface water regulations.

We conclude that for further research any kind of rejuvenator should be assessed for its enviromental impact. Rejuvenators are oil-based materials which can have very different origin and it cannot be assured that they are completely safe.

According to the literature review, there are different ways of performing the leaching tests. It would be interesting to compare results with some static leaching methods and also evaluate the results with the evolution in time.

Finally, it should be important to consider the different origins of the material and according to this extend the environmental analysis to other type of pollutants. Other types of additives or binder may also bring new pollutants to the mix which should be considered.

There is no significant effect of either rejuvenator addition location or rejuvenator content on plant emissions, the 100% RA production windows can be compared to the reference 60% RA window. On the average 100% RA samples resulted in 33% higher TOC emissions compared to the 60% RA window. Higher TOC emissions are a common and known effect when increasing the RA rate. Generally the main contributors for this are: fumes from heating RA bitumen, RA material grading, RA bitumen type and content, moisture content, fumes from fresh bitumen.

8. EFFECT OF REJUVENATORS ON BITUMEN PROPERTIES

The information provided in this chapter is a summary of two publications listed below and in the reference section. For more detail the reader is referred to these two publications:

Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Aging effect on rheology and cracking behaviour of reclaimed binder with bio-based rejuvenators Journal of Cleaner Production 189 (2018) 88e97

Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Effect of Aging on the Mechanical and Chemical Properties of RAP Binder Treated with Bio-Based Rejuvenators Composites Part B 141 (2018) 174–181

8.1. SARA FRACTIONING

Figure 10 shows the results of the SARA analysis on virgin binder, RAP binder and RAP binder with rejuvenators A, B and C. It was shown that virgin binder is less affected by aging than RAP in terms of the formation of asphaltenes (Cavalli et al., in preparation), thus, resulting in a so called “sol” type structure with majority of resins and maltenes and less asphaltenes. On the contrary, RAP binder is supposed to be stiffer than the virgin binder, having a “gel” structure with asphaltenes forming agglomerations between the resins and the maltenes, (Bonemazzi & Giavarini, 1999). It is supposed that because of the aging process, the content of asphaltenes, which are solid domains containing sulphur and oxygen, is higher in the RAP than the virgin binder. It is hypothesized that the addition of rejuvenator A, B and C could create a new network in the RAP binder and partially dispersing the asphaltenes as shown in *Figure 10*.

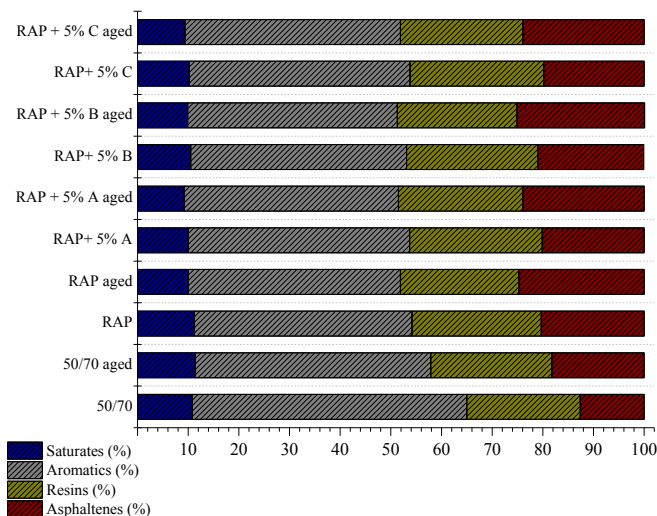


Figure 10 SARA results with the fractioning by mass of each components for the virgin binder, the RAP binder and the rejuvenated RAP binder before and after aging.

8.2. FTIR

Figure 11 shows the spectra for all the unaged materials showing the intensity of the spectral peaks, corresponding to the different bonds/functional groups, as a function of the wavenumber. Changes due to oxidative ageing correspond to the intensity of the carbonyl (peak centred around 1700 cm^{-1}) and sulfoxide functional groups (centred around 1030 cm^{-1}), and are relevant in the asphalt field [9]. As a result of ageing, functionalities are formed that increase the overall polarity of the bitumen, thus influencing bitumen rheology. From Figure 11 follows that the spectrum of the virgin binder diverges from the binders containing RAP at two locations. The majority of the peaks of the RAP binders, even after adding 5% of the three rejuvenators, remain the same. The peaks corresponding to the ageing of binders due to oxidation do not disappear although rejuvenators were added to the RAP binder belonging to the non-reversible type of ageing. Chemical structures of RAP + 5% C and RAP + 5% A were found to be similar, suggesting that the chemical effect of rejuvenator C and rejuvenator A on RAP binder is similar. This is expected as the spectra of rejuvenator A and C are very similar. For example, a peak centred at a wavenumber of 1750 cm^{-1} and to the RCOOR' ester group was found in both materials.

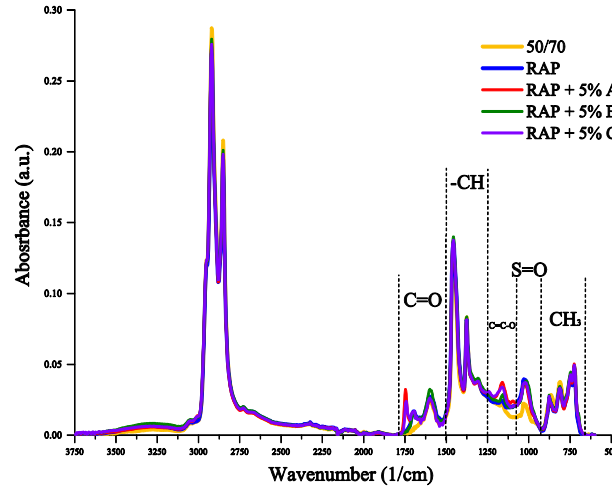


Figure 11 ATR-FTIR spectra of the virgin binder 50/70, the RAP binder and the RAP binders with rejuvenators A, B and C (5% by mass of RAP binder each).

Carbonyl and sulfoxide indexes before and after ageing

As suggested in [Marsac 2014], the carbonyl and the sulfoxide index are calculated by dividing the area of the carbonyl and sulfoxide functional groups through the area of peaks related to the asymmetric vibration of CH₂ and CH₃ (around 1455 cm⁻¹) and to the symmetric deformation vibration of CH₃ (around 1376 cm⁻¹) as the latter areas do not change significantly. Hence the following equation holds:

$$\text{Carbonyl index (CI)} = \frac{\text{Area}_{\text{C=O}}}{\text{Area}_{\text{CH}_2} + \text{Area}_{\text{CH}_3}}$$

$$\text{Sulfoxide index (SI)} = \frac{\text{Area}_{\text{S=O}}}{\text{Area}_{\text{CH}_2} + \text{Area}_{\text{CH}_3}}$$

The sum of both indices (CI + SI) was used to determine the chemical ageing index (CAI) before and after ageing:

$$\text{Chemical ageing index (CAI)} = \text{CI} + \text{SI}$$

An increase in carbonyl and sulfoxide index means rising degree of oxidation in the bituminous binder, causing stiffening. As shown in Figure 9, the unaged binders RAP + 5% A and RAP + 5% C produced a higher absorbance in the spectra at wavenumber corresponding to the carbonyl index than the plain RAP binder. This may be due to the fact that seed oil and tall oil themselves

contain carboxylic groups C=O. Therefore, it can be concluded that the addition of rejuvenators could soften the RAP binder but not breaking its chemical bonds at molecular level. As shown in the previous sections, rejuvenator alone were not affected by ageing thus, the increase of both C=O and S=O groups is a result of ageing of the RAP binder.

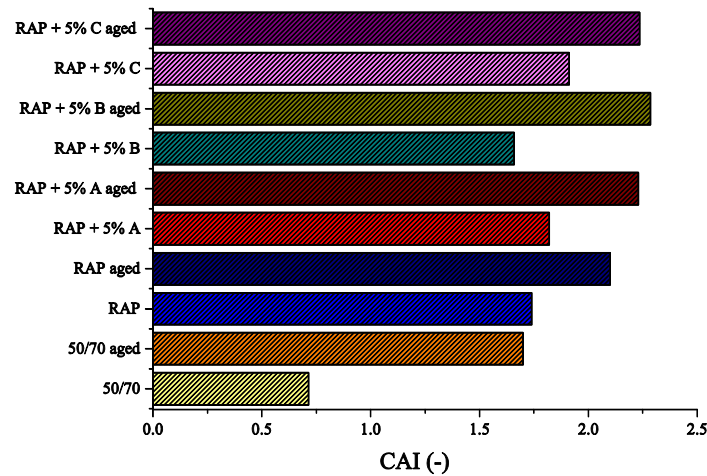


Figure 12 Comparison between the CAI of the virgin binder, RAP binder and RAP binder plus 5% by mass of rejuvenators before and after ageing

8.3. BLENDING OF RAP BITUMEN AND VIRGIN MATERIALS

After having the RAP binder extracted with toluene (EN 12697-1), it was placed in the oven at a temperature equal to the softening point temperature, plus 80 ° C for 45 minutes (Fehler! Verweisquelle konnte nicht ge-funden werden.). The amount of time was found to be the minimum amount required for the RAP binder to be-come liquid. The rejuvenators (which were at room temperature) were added to the hot binder and were placed in the Speed Mixer™ for one minute at 3500 rpm (total mass of this mixture was 100g). One minute was found suitable for homogenization that was verified visually.

8.4. AGING

The rejuvenated binders were laboratory aged using RTFOT (EN 12607-1) and subsequently PAV (EN 14769:2012). The combined aging procedures are expected to simulate both the early stage aging before placement and the in situ aging.

8.5. PERFORMANCE

Aim of this section, is to show how the addition of rejuvenator could influence the mechanical performances of RAP binder. Emphasis was on the effect of ageing on the overall mechanical performances.

8.5.1. Rheological analysis of the unaged materials

Figure 13 left, shows the master curves of complex moduli $|G^*|$ as function of frequency at the reference temperature of 20° C. It follows that the differences between the unmodified RAP binder and the modified RAP binder are significant within the whole frequency spectrum. In particular, RAP + 5% B or RAP + 5% C had lower moduli than the virgin bitumen 50/70. Hence, it was found that at unaged state, the RAP binder modified with 5% rejuvenators B and C were mechanically softer than virgin bitumen 50/70 while RAP binder with rejuvenator A was harder in the low frequency range. The trend of the phase angle (Figure 1, right) confirmed what was shown in the rheological measurements: rejuvenator could soften the RAP binder for all frequencies. In particular, at lower frequencies G'' was prominent for all the material (viscous portion of G^*). RAP + 5% C and RAP + 5% B could show higher values of G'' compared to the other two rejuvenated binders while virgin 50/70 and RAP showed similar value of G'' . On the contrary, at highest frequency it was found that RAP + 5% B and RAP + 5% C had higher phase angles in comparison to the other materials and therefore higher G'' than RAP + 5% A. All modified binders showed higher G'' than the virgin binder itself at high frequencies (low temperature) showing a higher viscous component of the complex modulus in comparison to RAP. This is advantageous for low temperature behaviour of the material.

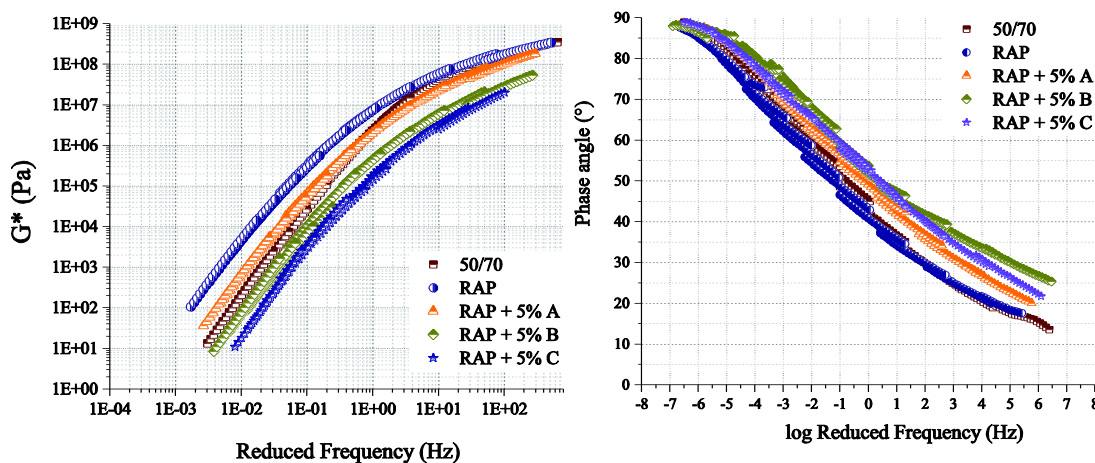


Figure 13 Dynamic shear moduli master curves for reference temperature of 20° C (left) and phase angle measurements (right) from frequency sweeps (0.1-20 Hz). The presented values are an average of four measured values.

It can be seen how RAP binder and 50/70 reached the same value of $\log G^*$ (equal to 8.5 Pa) at high values of reduced frequency (6.5 Hz) while having a gap of two orders of magnitude at low frequencies between -7 Hz and +1 Hz. Moreover, rejuvenators caused a general shift downwards for all the master curves at all frequency ranges showing how at low frequencies rejuvenators could soften the RAP binder (remaining close to the mastercurve of the virgin binder) while for higher frequencies range, rejuvenators could soften the RAP binder even more than the virgin binder.

8.5.2. Rheological analysis of the aged materials

After ageing, the overall complex moduli of each material tested increased in comparison to the unaged materials as shown in Figure 14 and in comparison to *Figure 13*. It was observed that over the most of the frequency range, RAP + 5% A and RAP + 5% C had similar complex moduli after ageing. On the contrary, RAP + 5% B was closer to the complex moduli of the aged RAP binder indicating no improvement in mechanical stiffness after ageing. The aged virgin binder 50/70 had similar values to the unaged RAP binder showing how the standard ageing procedure could simulate closely the ageing caused by service life. Figure 14 left, shows how, despite the addition of rejuvenators, after ageing there is in general an increase of both complex moduli and phase angle along the whole frequency range. Furthermore, the difference in the values of complex moduli at aged state is more prominent at low frequencies (higher temperatures, from +40° C to +80° C) than at high frequencies (low temperatures, from +30° C to -10° C) as the curves converge at high frequencies after ageing showing that rejuvenator's effect is more prominent at high temperatures. Furthermore it can be seen from the results that aging plays a significant role in materials rheological behaviour and should be considered when long term mechanical performance is sought. Figure 14 right shows that at low frequencies, the phase angle of the virgin bitumen is high indicating a dominance of the viscous portion of the complex modulus in comparison to the other materials. RAP + 5% A and RAP + 5% C resulted in a higher phase angle than the RAP binder, indicating that they could maintain the viscous portion of the complex modulus towards the values of the virgin binder. At highest frequencies (low temperature) a general tendency for all the material could be observed: the G' is dominant with a decrease of the phase angle towards to pure elastic regime. However the RAP + 5% A and RAP + 5% C had a higher phase angle than other materials and therefore more viscous in comparison at low temperatures which is a beneficial. A comparison of the results of unaged and aged binders clearly indicates how aging can affect the mechanical response of the rejuvenated materials.

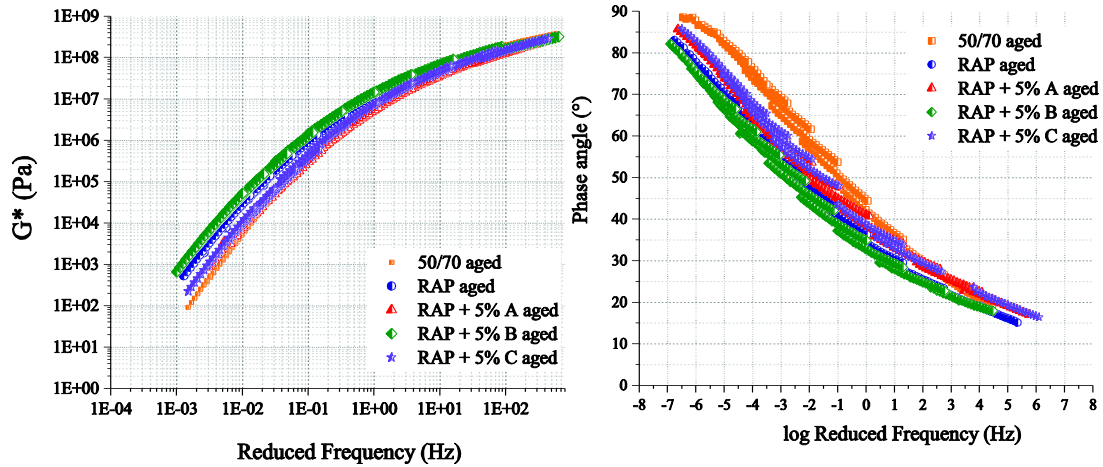


Figure 14 Dynamic shear moduli results of the aged materials measured at reference temperature of 20° C (left) and phase angle measurements (right) at frequency sweeps (0.1-20 Hz). The presented values are an average of four measurements.

8.5.3. Fracture Toughness

As can be seen in Figure 15, after ageing all the binders tested decreased their work up to fracture, thus becoming more susceptible to low temperature cracking before the unaged state. These results demonstrate how ageing has a significant effect on low temperature cracking behaviour. In addition, RAP binder performance at low temperature at both aged and unaged state show significant improvement after adding the rejuvenators. In particular, at unaged state, RAP + 5% A and RAP + 5% C could increase the RAP binders work of fracture up to 50% and RAP + 5% B up to about 40%. Furthermore, at low temperature (-10° C), RAP binder appeared more sensitive to ageing than the virgin binder, halving its work up to fracture. Although still higher than virgin and RAP binder, the work of fracture decreased for all modified binders. The results confirm that, despite the ageing procedure, rejuvenator could improve the crack resistance of RAP binder at -10° C. In addition, the type of rejuvenator had a significant effect on fracture behaviour of the rejuvenated binders after ageing and even better than virgin binder as discussed above and shown in Figure 15,.

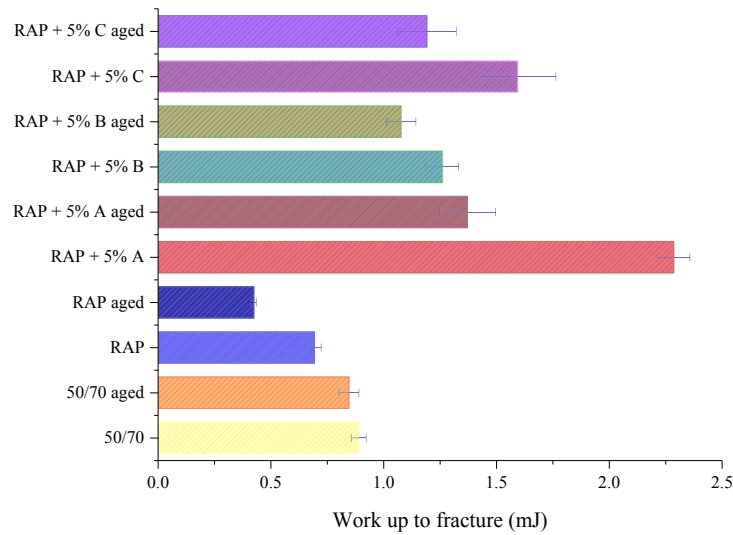


Figure 15 Work of fracture at crack initiation at -10°C before and after ageing.

8.5.4. Microstructure

As can be seen in Figure 12, the virgin binder images present a softer paraphase surrounding the “bees” structures. On the contrary, RAP binder images appear generally brighter with few softer spots. general observation can be made: RAP binder Young’s moduli at the surface are higher than the virgin binder both at unaged and aged state. As can be seen in Figure 16, after aging the Young’s moduli for both RAP binder and virgin binder increase. In particular, in Figure 16a) and c) it’s possible to observe how after aging, “bees” appear smaller and with higher elastic moduli. In Figure 12 b) and c), it’s shown how the morphology of RAP binder doesn’t change significantly after aging however, it’s elastic modulus increase of around 46%. The AFM investigations show that the RAP binder is a different material than the virgin binder although its mechanical properties can be restored.

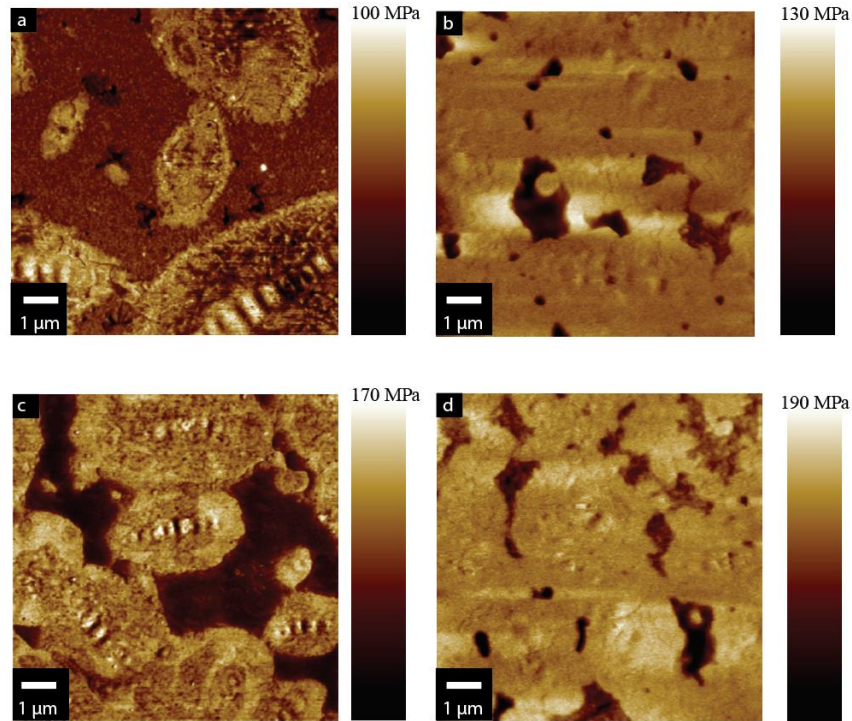


Figure 16 AFM QNM images ($10 \times 10 \mu\text{m}$). a) Virgin binder 50/70 unaged; b) RAP binder unaged; c) Virgin binder 50/70 aged; d) RAP binder aged.

8.6. SECTION CONCLUSIONS

Reclaimed asphalt pavement (RAP) is harder than virgin bitumen due to ageing and it needs to be rejuvenated. Three bio-based rejuvenators are used and the rejuvenating effects are evaluated. Rheological tests show how the rejuvenators may restore the mechanical properties of RAP binder. An ageing index is proposed to show that the rejuvenators are affected differently by ageing. Despite their addition, physio-chemical oxidation did not reverse. Mechanical changes were not caused by chemical changes at functional groups level but a rearrangement of polar/nonpolar components. The results show that considering the effect of aging is vital in identifying how rejuvenators affect the RAP binder chemically and mechanically long term. RAP binder and virgin binder are different materials apparent from their different microstructures, rejuvenators cannot restore the microstructure to virgin state. The binder evaluations showed that rejuvenator C had in some tests promising results and was therefore used for the mixture experiments.

9. BALANCED MIXTURE DESIGN

The contents of this chapter are a summary of the following publications. For further detail the reader is referred to these or the authors

Zaumanis, Martins, Cavalli, Maria Chiara, Poulikakos, Lily D. Design of 100% RAP Hot-Mix Asphalt to Balance Rutting and Cracking Performance, submitted

Zaumanis, Martins, Cavalli, Maria Chiara, Poulikakos, Lily D. Performance-based design of 100% RAP hot-mix asphalt and accelerated testing of mixture, in preparation

The traditional mix design procedures consider volumetric proportions (bitumen, content, gradation, porosity, etc.) and strength characteristics of mixtures, which have been derived from correlation of traditional mixtures with field performance through monitoring of pavement performance for many years. This approach historically provided the desired pavement performance, while minimizing the necessary testing time and expenses. However, in recent years, increasing number of pavements are failing well before their life expectancy. This can be related to many things, most importantly to increasing transport loads and reduction of bitumen quality due to modernization of technological production process in oil refineries. Another issue is use of untraditional asphalt production technologies and materials, including polymer modified asphalt, use of reclaimed asphalt, and warm mix asphalt.

The evaluation of such mixtures using traditional mix design methods is impossible in many cases because of unsuitable test methods and unknown correlation of the laboratory performance vs field performance.

This approach works fine while deviations from the original mixtures and technologies are relatively minor. However, when uncommon materials, new material combinations or modified production technologies are used, using the traditional design criteria may not ensure the expected pavement performance. A modified criteria is necessary. Introduction of new, performance based, test methods compared to relying only on volumetric criteria is advantageous because it allows to predict the pavement properties

Among others, distresses that are often associated with distresses that lead to pavement failure are rutting and cracking.

9.1. CHOICE OF TEST METHODS

Conventional volumetric mix design procedures cannot ensure the required asphalt pavement performance for high RAP mixtures. This is because of the many unconventional variables including rejuvenator use, blending and diffusion of binders, potential susceptibility to cracking, etc. This chapter presents a performance-based design of 100% recycled asphalt surface coarse by balancing cracking and rutting. French rut tester and semi-circular bend test were used for this purpose. Five iterations of different grading and binder combinations of 100% RAP mixture were tested before achieving the same performance as a traditional mixture.

The principles of designing asphalt mixture by mainly relying on performance-based test methods can be summarized in *Figure 17* as follows (Zaumanis, Poulikakos et al. 2018):

1. The requirements to constituent materials and mixture composition are kept to minimum to allow innovation. Instead, information is collected regarding constituent materials and volumetric properties to aid in optimization of mixture performance.
2. Aging is performed on samples to simulate field aging conditions.
3. The mixture is tested using the chosen performance-based test methods and verified against the specified criteria.

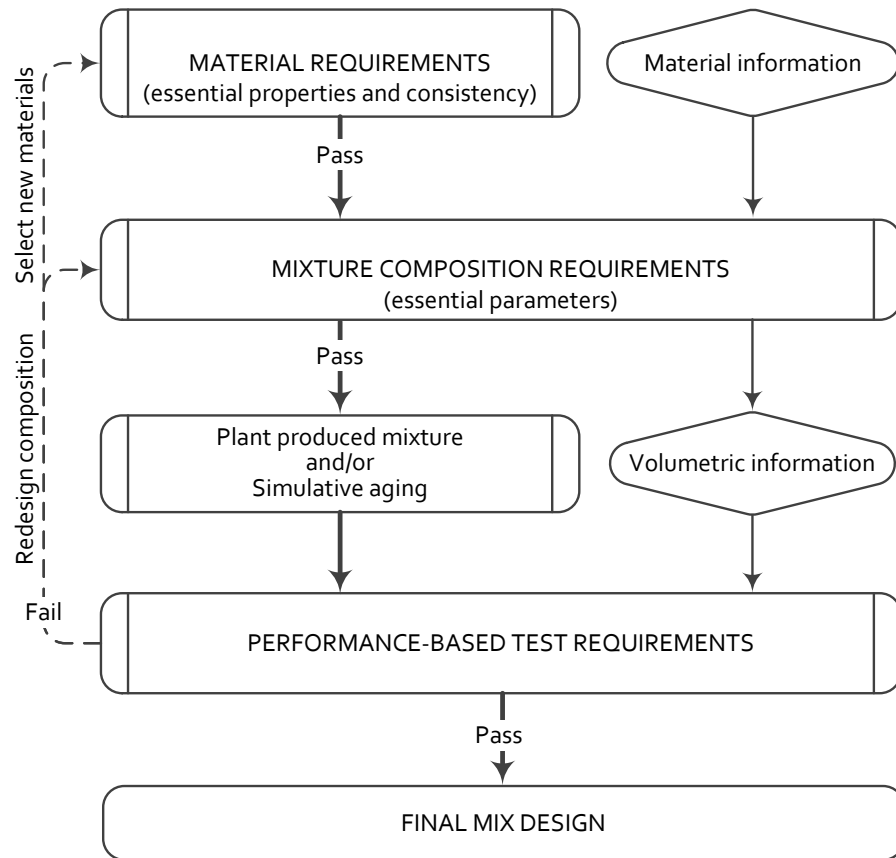


Figure 17 Performance mix design framework (Zaumanis, Poulikakos et al. 2018)

9.1.1. Sample manufacturing

Samples were mixed in laboratory using an oil heated mixer at a temperature of 170°C which corresponds to the mixing temperature for 50/70 penetration grade binder. Samples were mixed for five minutes. Marshal samples were made for each mix at 150°C and 50 blows from each side to determine the bulk density for the following performance tests.

The loose mix that was dedicated for performance testing was then aged for four hours at 150°C after which the mix was compacted using steel wheel roller compactor at dimensions of 100 mm × 180 mm × 500 mm to a target density that equals that of Marshall specimens. Cores were drilled from the slabs after which they were cut and sanded to the required dimensions.

9.2. MATERIALS USED

9.2.1. Mix type

Reclaimed asphalt was collected in Switzerland. Its exact source is unknown. It had been screened on an 11 mm sieve at the RAP processing facility and has a binder content of 5.6%. It can be seen in *Figure 18* that its gradation (“RAP fine”) is close to the upper limit of the Swiss requirements for AC 8 mixtures. Therefore it was decided to prepare a set of 100% recycled samples without modification of the gradation.

Another set of recycled samples was prepared by screening the RAP on 5.6mm sieve and changing the proportions to achieve grading curve as similar as possible to the reference plant produced AC 8N mixture. This recycled mixture gradation is denominated “RAP coarse”. Because of less binder-rich fine particles the binder content at 5.1% is lower than that of “RAP fine”.

AC 8N plant produced surface coarse mixture was chosen as a reference. It is intended for traffic volume of up to 300 ESAL. This choice was made to correspond to the existing nominal maximum aggregate size of the RAP and the relatively low traffic volume was chosen because of the unknown source of RAP. The AC 8N mixture has 5.9% binder content.

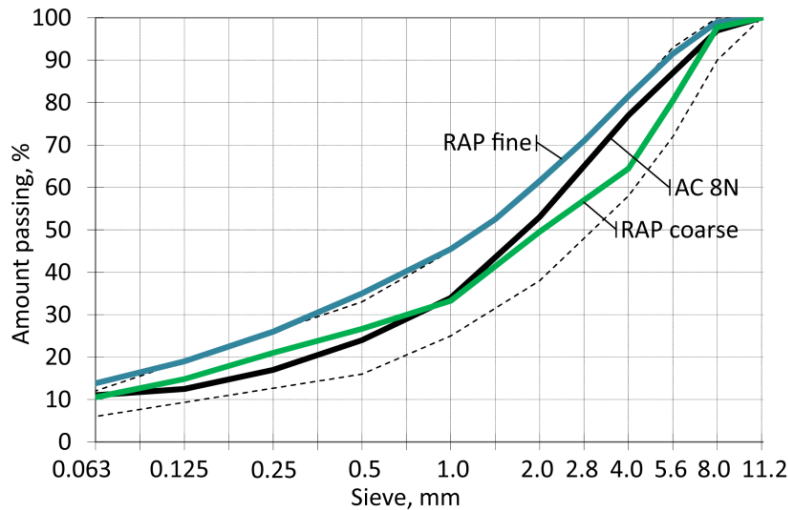


Figure 18 Gradation curves

9.3. CHOICE OF INITIAL REJUVENATOR DOSE

A commercially available rejuvenator based on distilled tall oil was used for the experiments. It is a by-product of Kraft manufacturing process. The rejuvenator was added at 7.3% of RAP

binder mass for all recycled samples. This amount was based on an earlier study where the required dose to reach penetration of 60 mm-1 was determined.

For each of the recycled mixtures, two binder contents were used: the original and the original +0.5% bitumen. Either 50/70 grade or highly Polymer Modified Bitumen (PMB) was added. The final binder+rejuvenator content of the “RAP fine” gradation samples was 6.0% and 6.5% while for the “RAP coarse” samples it was 5.5% and 6.0%.

9.4. OPTIMISATION OF MIX PERFORMANCE

As can be seen in the table below the flow coefficient of the two fractions <5.6 and >5.6 differ considerably from the virgin aggregates indicating that there recycled aggregates have more rounded characteristics as a result of in situ aging. Therefore it was decided as an optimisation step to separate the two fractions.

Table 4. Flow coefficient of fine particles

Sample	Flow coefficient, s
Virgin sand (AC 8S mix)	34
RAP <5.6mm	30
RAP >5.6mm	26

9.5. CRACK PROPAGATION

The two key parameters in SCB test are fracture energy (G_f) and Fracture index (FI) (Ozer, Al-Qadi et al. 2016) discussed in sections 5.2.4 and 5.2.5. Larger fracture energy indicates asphalt mixtures that can withstand greater stresses with higher damage resistance. This parameter is derived from work-of-fracture (the area under load vs displacement curve) that was refined by RILEM (RILEM TC-50 FMC 1985). It was, however, further discussed by the authors, that the pattern of the load-displacement curve, especially the post-peak part is also important to discriminate cracking potential of mixtures. It was found that estimated crack propagation velocity correlates well with the post peak slope (m) which was therefore used in calculation of the FI.

A correlation of FI with field performance was carried out using results from FHWA (Federal Highway Agency) test track. Here, seven different mixes with various RAP and RAS (reclaimed asphalt shingles) contents and different warm mix asphalt technologies were placed using equal structural design and tested for cycles to fatigue threshold. The results of this study correlate well

with the results of FI. Based on the study results, FI thresholds for distinguishing between good ($FI > 10$), acceptable ($FI > 6$) and bad ($FI < 2$) performing mixes were proposed, with a note that these should be adjusted based on local circumstances (Ozer, Al-Qadi et al. 2016). Based on these results it was concluded that fracture index provides means to identify brittle mixes that are prone to premature cracking and the FI distinguishes between mixtures more clearly than fracture energy.

Based on these findings FI is proposed in this study as a mixture design tool to compare mixes and screen the ones that are prone to cracking. The load displacement curves of all mixtures are demonstrated in *Figure 19* and the FI along with fracture energy in *Figure 20*. The following observations can be made:

- An increase in binder content for both RAP gradations lowers the maximum load but increases the post-peak slope. This results in an increased FI (see *Figure 20*) indicating higher cracking resistance. This is what is intuitively expected and somewhat confirms the validity of the test method.
- The reference AC 8 N has an FI of 5.5 which will be therefore considered as the threshold for pass/fail criteria for the recycled mixtures. Only the *RAP fine+0.5% bit.* mixture has statistically similar performance to the reference mix. As can be seen in *Figure 19* this is due to a much higher post-peak slope compared to the AC 8 N mixture.
- Another pair of mixtures that should be compared are *RAP fine* and the *RAP coarse+0.5% bit.* These mixtures have equal total binder content and the same binder penetration. The only difference is gradation. It can be observed that the coarser gradation has improved the FI considerably (from 0.5 to 4). As can be inferred from *Figure 19* and *Figure 20* this is due to both higher slope and higher fracture energy.
- It can be observed that the addition of 0.5% PMB instead of paving grade bitumen for the *RAP coarse* mixture has not resulted in statistically significant change of FI, likely due to the relatively small PMB content.
- Finally, it can be observed in *Figure 20* that fracture energy provides similar ranking of the mixtures compared to FI. The difference between the mixtures is smaller for the fracture energy (highest result is 2 times higher compared to the lowest result) than for the FI (highest result is 6.2 times higher compared to the lowest), but the variability of fracture energy is 2.3 times lower (12% compared to 28%).

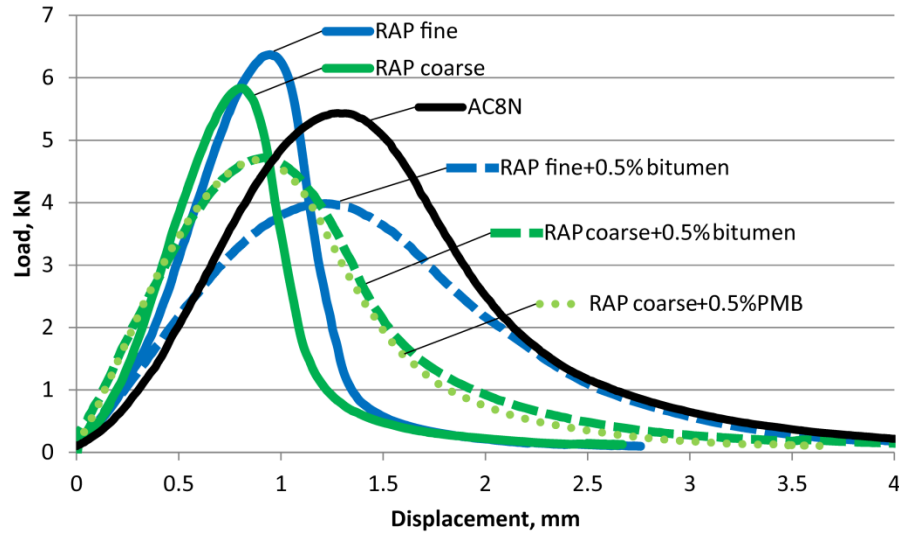


Figure 19. SCB test load displacement curves

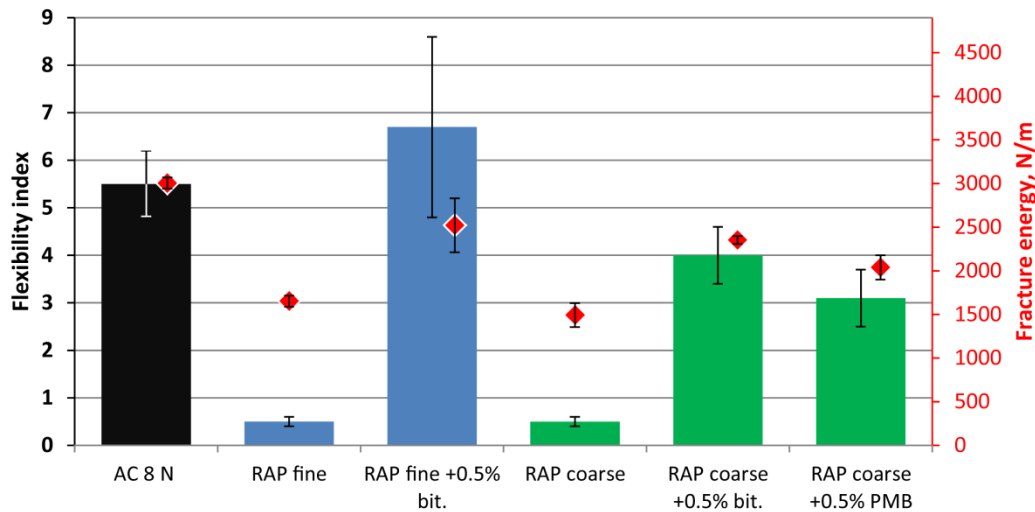


Figure 20. Flexibility index (bars) and fracture energy (rhombs)

9.6. RESULTS

The fracture toughness and rutting test results, along with the result range of each test sample and air void content (V_a) are summarized in Figure 21. V_a of 2-5% is required for AC 8N mixtures. In this figure the fracture toughness is illustrated on the vertical axis while the rut depth is placed on horizontal axis. The results of AC 8N reference mixture are used as a reference and the dotted lines that go through the test result to allow comparing the performance of other mixtures to it. All the mixtures that perform better in rutting compared to the AC 8N

will align to the left from the vertical dotted line. All the mixtures that perform better in terms of fracture toughness will align to the top from the horizontal dotted line. Thus any mixture performing better than the reference in both cracking and rutting will be situated in the upper left rectangle.

It can be seen in the figure that all of the RAP coarse mixtures and the “RAP fine” mixture are situated in the upper left rectangle meaning they perform better than the AC 8N mixture both in fracture toughness test and rutting test. The “RAP fine +0.5% 50/70” mixture performs better in terms of fracture toughness, but has unacceptable rutting performance.

The chart demonstrates that in absolute numbers the rutting resistance of the recycled mixtures is not very high, since often in specifications less than 10% is required. However, since the mixtures are intended for low volume roads, such result is acceptable. Even more so because these mixtures perform better than the AC 8N reference mix. The results also allow to conclude that re-grading of RAP has allowed to improve the rutting resistance, but has no statistical effect on cracking resistance. This is what normally should be expected for low temperature cracking tests. Use of PMB bitumen has not provided any notable improvement in neither rutting nor cracking compared to the paired mixture with the same gradation but 50/70 bitumen.

If the effects of binder content are isolated, one can see that as expected, increasing bitumen content by 0.5% has caused reduction in rutting resistance. At the same time there are no changes in cracking resistance. Although somewhat questionable, similar conclusions regarding binder content effect on low temperature performance have been formed also previously [5]. This allows concluding that fracture toughness should not be used for optimization of mixture design binder content due to the test method insensitivity.

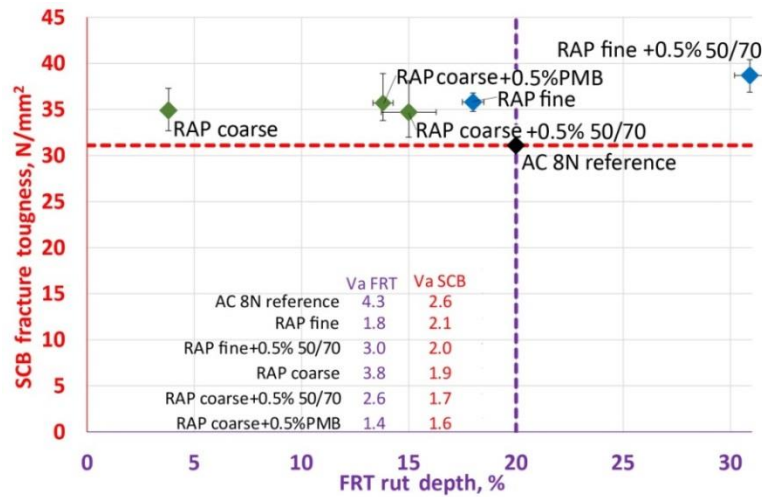


Figure 21 Balancing of cracking and rutting performance

9.7. COMPLEX MODULUS

Stiffness results with one statistical deviation from four test repetitions are illustrated in *Figure 22*. It can be seen that the reference AC 8N mixture has stiffness of 8,000 MPa and only the two RAP mixtures without any virgin bitumen have higher stiffness. Addition of 0.5% 50/70 bitumen to both *RAP coarse* and *RAP fine* gradations has resulted in reduction of stiffness by 30%. Exchanging of 50/70 bitumen with PMB resulted in further 10% stiffness reduction. If RAP mixtures having the same binder content are compared, it can be seen that *RAP fine* has higher stiffness compared to *RAP coarse+0.5% bit*. Both mixtures in have the same binder viscosity. It is then likely that this effect is a result of (1) thinner binder film as a result of higher fines content and thus larger aggregate surface area of the *RAP fine* and (2) lower active binder content of *RAP fine* as a result of no added virgin bitumen.

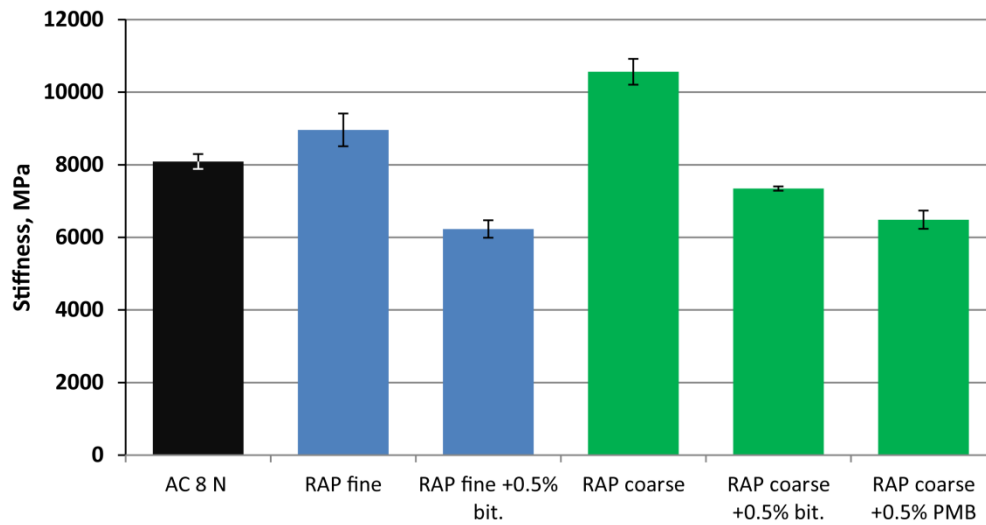


Figure 22. Stiffness at 20°C and 10Hz

9.8. BALANCED MIX DESIGN

The aim of performing the performance-based tests in this study was to develop a mixture design with balanced rutting and cracking performance. In the previous sections performance-based test results were demonstrated and the pass/fail criteria established. Here, balanced design approach to choose the optimum binder content is demonstrated.

Figure 23 (a) and (b) illustrates the results of *RAP fine* and *RAP coarse* mixtures with two different binder contents each (illustrated in the horizontal axis). Left vertical axis demonstrates rutting tests results while on the right vertical axis the FI results are demonstrated. Two dotted horizontal lines denote the rutting and FI pass/fail criteria based on the reference AC 8 N results as discussed earlier. Color coding indicates the axes that the results belong to.

It can be seen in the Figure 23a that when *RAP fine* gradation was used the ranges of acceptable rutting (<6.1%) and acceptable cracking (>6.35%) do not intersect. This means that an optimum binder content that would allow balancing cracking and rutting results to reach at least the performance to the virgin mixture does not exist. Other changes besides binder content in mixture design are necessary to improve either cracking or rutting performance. Such potential improvements include increased fine and coarse aggregate angularity, higher aggregate strength, larger nominal maximum aggregate size, use of PMB, etc. (Zaumanis, Poulikakos et al. 2018).

Unfortunately, unlike for virgin or low RAP mixtures, the potential for changes in mix design for high RAP mixtures is limited because of the necessity to work with the material in hand. Since binder content changes did not provide the necessary performance, the two other key parameters that could be changed are coarseness of grading curve and to certain extent the grade of the

binder (through changing the dosage of rejuvenator). Since the gradation did not fit into the grading curve of AC 8 mix type, it was decided to re-grade the RAP to make it coarser and the results are illustrated in *Figure 23b*. It can be seen that re-grading has allowed obtaining bitumen content (6.2%) where the rutting and FI requirements are passed simultaneously. This requires extrapolation of binder content by 0.2% which can be considered acceptably small and can be accepted. The overlapping area, however, is relatively narrow, especially considering the inherent RAP variability. For an increased confidence, the mixture should be further re-designed to widen the acceptable binder content range. Due to time constraint, however, this was not possible and the mixture design was used in the plant production as described in the next section.

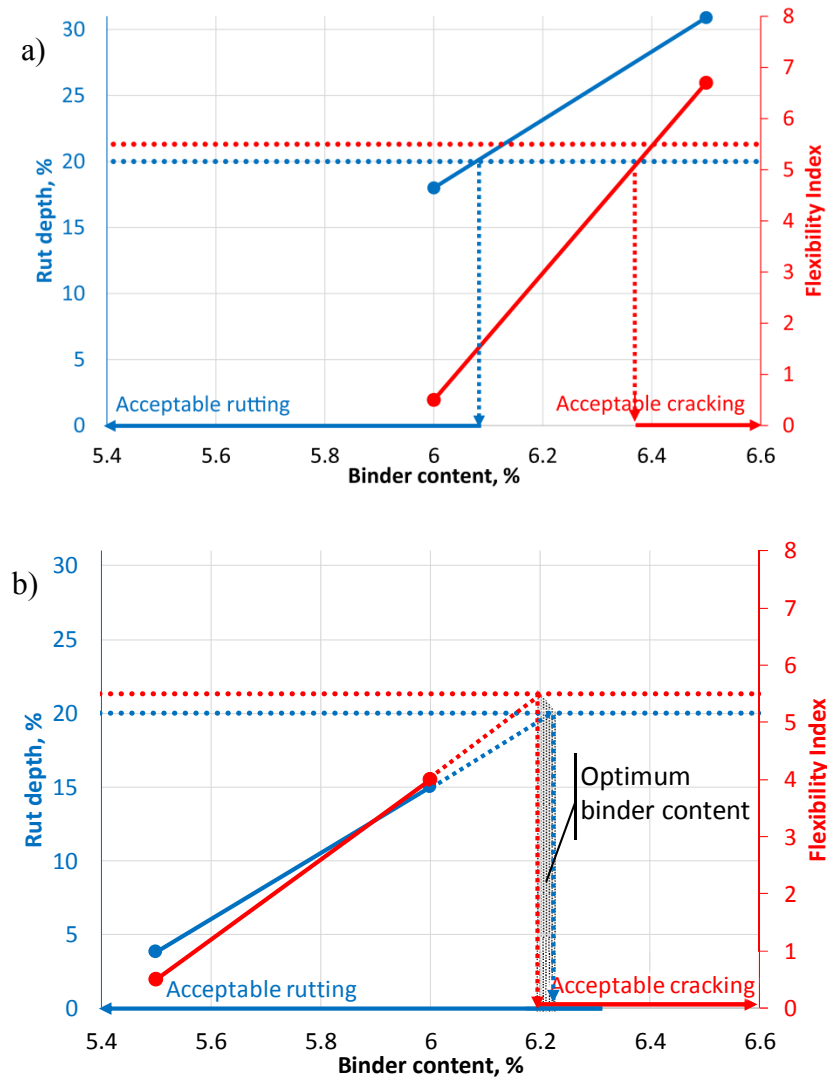


Figure 23. Balanced design of RAP fine (a) and RAP coarse (b) mixtures

9.9. SECTION CONSLUCIONS

Conventional volumetric mix design procedures cannot ensure the required asphalt pavement performance for mixtures with high recycled asphalt content. This is because of the many unconventional variables including rejuvenator use, blending and diffusion of binders, increased susceptibility to cracking, and others. In this study a 100% recycled mixture was designed by balancing two performance-based properties of asphalt: rutting using French wheel tester and flexibility index using semi-circular bend test. Two different RAP grading curves were used with two binder contents each. For one of the mixtures, a polymer-modified bitumen was also used. The design approach was then validated by testing the optimum mixture design using mobile movable load simulator (MMLS3). Finally the pavement surface was tested for skid resistance and particle abrasion due to moving wheel.

The following conclusions can be drawn from the research:

- 1) 100% RAP mixtures could not satisfy the conventional volumetric and Marshall test requirements. However, it is recognized that even correspondence to these requirements would not necessarily ensure the required pavement performance.
- 2) Performance-based balanced design procedure to balance French Rutting test and Flexibility Index from semi-circular bend test was found practical and provided the expected trends as a result of changes in mix design.
- 3) It was found by changing different mixture parameters that is possible to ensure that 100% recycled surface layer asphalt mixture provides similar performance to conventional asphalt intended for roads with design traffic volume of up to 300 equivalent single axle loads per day.
- 4) Testing with mobile movable load simulator (MMLS3) demonstrated that the optimum design of 100% recycled mixtures can sustain 2 times more wheel passes before fatigue failure.

- 5) Testing of particle abrasion from the asphalt slabs during loading with MMLS3 demonstrated that 100% recycled asphalt emission factor is lower compared to traditional AC8N mixture. Both emissions factors can be considered low.
- 6) Moisture resistance of the 100% recycled asphalt was excellent having 97% tensile strength ratio.
- 7) The skid resistance of 100% recycled and virgin mixtures was similar and sufficient for the intended traffic intensity.

Based on the positive experience of using performance-based mixture tests to balance cracking and rutting, it is recommended to consider such an approach as part of design procedure for mixtures containing high content of reclaimed asphalt. In this study French wheel tester was used because of its long-standing use in Switzerland. Semi-circular bend test flexibility index was found responsive to changes in mixture design, repeatable and simple enough for practical use.

10.100% RAP METER SCALE PERFORMANCE

10.1. LAB PRODUCED MIXTURES

The mix designs that fulfilled the balanced mix design procedure presented in the previous chapter were produced in a larger scale for the tests in this chapter.

10.2. MECHANICAL TESTING AND MONITORING OF TEST SECTIONS (M-SCALE)

Selected solutions developed in the previous sections were constructed in the laboratory in the form of large test sections (3mx1m) with a notch and tested until failure using the vehicle load simulator MMLS3 (Poulikakos et al., 2008) , resulting in pavement response from traffic loading. Using this type of simulator, monitoring of pavement performance in traffic and due to seasonal changes will be investigated. According to the number of load applications required to reach failure of the slab, a ranking containing the most durable materials will be obtained. The model mobile load simulator MMLS3 is a laboratory sized accelerated pavement testing (APT) machine for studying scaled pavement distress under repetitive rolling tires. The MMLS3 applies a downscaled load with four single pneumatic tires (*Figure 20*). They are smaller than standard truck tires, having a diameter of 0.3 m and a width of 0.11 m. The machine is 2.4m long by 0.6m wide and 1.2m high. Each tire loads the pavement width over a path length of 1 m with a load up to 2.1kN, induced through a spring suspension system. At a maximum speed of 9 km/h, the MMLS3 allows approximately 7200 load applications per hour, corresponding to nearly a 2 Hz loading frequency rate.



Figure 24 The Model Mobile Load Simulator MMLS3 used to obtain down size accelerated information about performance of pavements

10.3. ENVIRONMENTAL TESTING AND MONITORING

During the MMLS3 tests it was possible to monitor the Emission in form of dust due to pavement abrasion [Gehrig 2010]. These results are reported in chapter 7.

10.4. TEST RESULTS

Particle abrasion measurements were performed in an open environment and the setup is illustrated in *Figure 25*. TSI APS Model 3321 aerodynamic particle size spectrometer was used to count the abraded particles. An SF₆ tracer gas (Sulphurhexafluoride) was introduced next to the wheel at the direction of the movement and it was collected at the other end of the MMLS3 next to the point of sampling of the particles. This allowed to determine the air volume flow under the MMLS3 according to Equation 3 (Gehrig et al., 2010).

$$\dot{V} = \frac{f_{tr} \cdot c_{tr}}{c_{meas}} \quad \text{Equation 3}$$

, where f_{tr} – flow of tracer gas injection, ml/min

c_{tr} – concentration of injected tracer gas, ppm

\dot{V} – Air flow volume through the simulator, m³/min

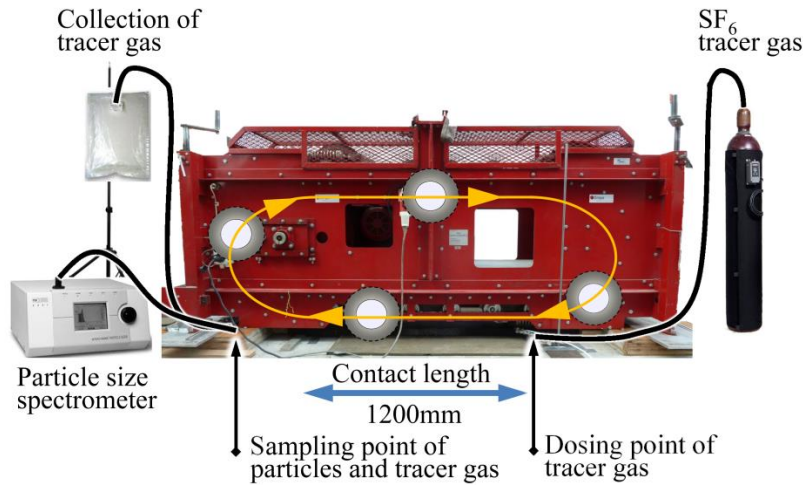


Figure 25. Setup of the particle abrasion test

From the sampling measured particle concentrations, the driven wheel distance and the air flow volume below the MMLS, an emission factor can be calculated according to Equation 4. Only

particle size up to 10 μm was considered (PM10) and as previously reported by Gehrig et al. (Gehrig et al., 2010) it was assumed that the density of released particles is 1 g/cm^3 .

$$EF = \frac{(c_{op} - c_{amb}) \cdot \dot{V}}{d} \quad \text{Equation 4}$$

, where EF – Emission factor, mg/km

c_{op} – Particle concentration during operation, mg/m^3

c_{amb} – Particle concentration of ambient air, mg/m^3

d – Driving distance of the simulator wheels on the pavement per minute, km/min (0.140)

10.1. RESULTS

Figure 26 demonstrates the average particle size distribution during the MMLS loading period of 1h. Before starting the MMLS3, the particle distribution in ambient air (background) was collected and these results are also illustrated in the figure. The difference between these two distributions is the amount of abraded particles from asphalt as a result of the MMLS wheels passing over slab. It can be seen in Figure 26 that there some particles are being abraded in the air from the asphalt slabs, but the amount is even lower than the amount of particles already in the ambient air. It can also be seen that the amount of particles in ambient air in both cases is similar despite the tests being performed a month apart. This shows that the results are relatively robust.

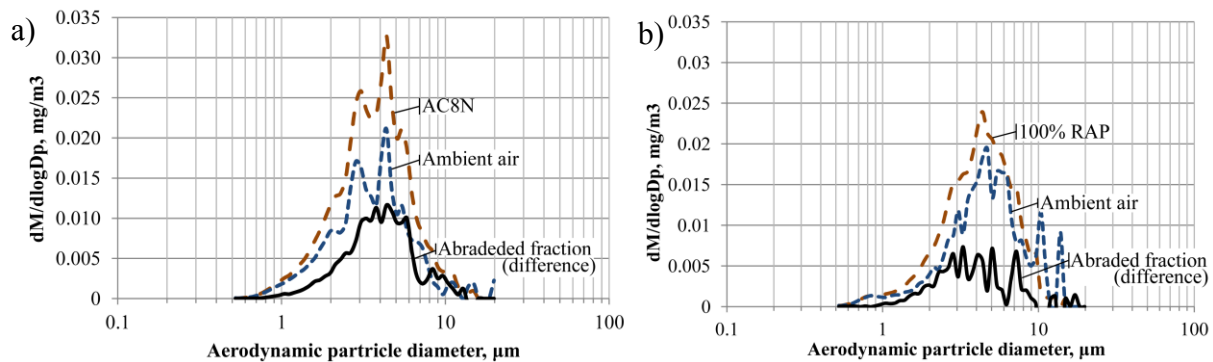


Figure 26. Average particle size distribution over 1h of test (a – AC8N and b – 100% RAP)

The calculated emission factors throughout the test period of 60 minutes are illustrated in Figure 27. The sampling was performed in 5 minute intervals for the 100% RAP and 10 minute intervals for the AC8N mixture and the average emission factor for each sampling period is illustrated. Only one measurement per asphalt type was performed and therefore no statistical evaluation is provided. However, it was previously reported by Gehrig et al. (Gehrig et al., 2010) that for this test a measurement uncertainty of 30 % can be assumed at 95% confidence interval. It can be

seen that the AC8N throughout most of the test has higher emission factor compared to the AC8N. By average the emission factor of RAP is 0.94 mg/km and for the AC8N mixture it is 5.8 mg/km. The results of the AC8N mixture are similar to what has been reported previously (Bukowiecki et al., 2010; Gehrig et al., 2010). It can be hypothesized that the recycled asphalt is abrading fewer particles because the mineral aggregates have been encapsulated by the bitumen twice (originally and during recycling) and/or because it has lower density at 2.3% compared to 2.8%.

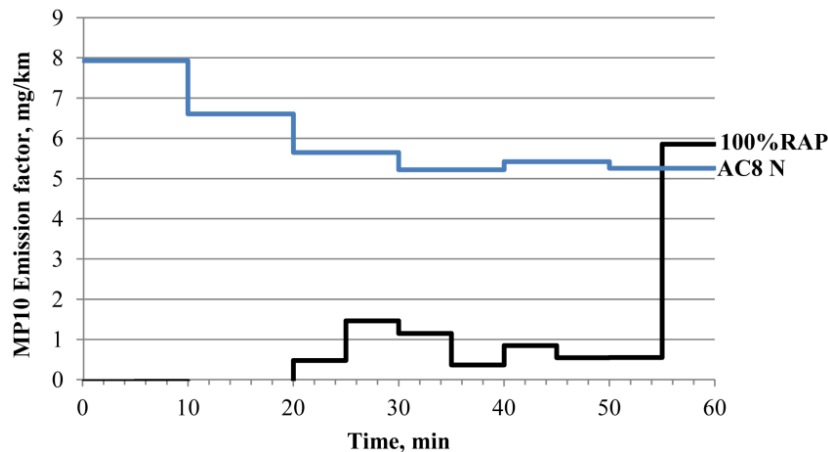


Figure 27. Emission factor as a function of time

10.2. SECTION CONCLUSIONS

Testing of particle abrasion from the asphalt slabs during loading with MMLS3 demonstrated that 100% recycled asphalt emission factor is lower compared to traditional AC8N mixture.

11. PLANT PRODUCTION CONSIDERATIONS

The information in this chapter is a summary of the following publication. For more details the reader is referred to the publication or the authors.

Zaumanis, M, Boesiger, L, Kunz, B, Cavalli, Maria Chiara, Poulikakos, L. D, Determining optimum rejuvenator addition location in asphalt production plant, 'Construction and Building Materials 198 (2019), 368-378 <https://doi.org/10.1016/j.conbuildmat.2018.11.239>'

11.1. RAP MANAGEMENT

In order to improve the quality of high RAP mixtures a well-defined RAP management policy is recommended. The following are recommended however it is clear that practical considerations can hinder the use of these techniques: Separation and storage based on layer and source, Additional fractionation and Keeping the RAP dry.

11.2. AMMANN RAH100 PLANT

The Ammann “RAH100” plant is located in the following sites in Switzerland: Birmensdorf (BHZ/BAB), Rubigen (Berag), Oberwil (BHZ), Walliswil (Marti), Sigrino (Comibit), Untervaz (Catram). The location in Birmensdorf was visited by the applicants and therefore commented on in detail here. BAB Belag AG in Birmensdorf operates the Ammann “Uniglobe 200” plant with “RAH100” reclaimed asphalt dryer drum. The plant utilizes a unique counter-flow dryer with two phase drum designed by Ammann to allow RAP heating without direct contact to flame. The material heating and drying phase of the drum rotates, while the combustion chamber is static as demonstrated in Figure 8. The RAP is heated with hot air and is discharged before getting in contact with the flame, reducing emissions and limiting RAP binder aging. The drum is installed on top of the tower to ensure gravity-driven handling of the hot RAP.

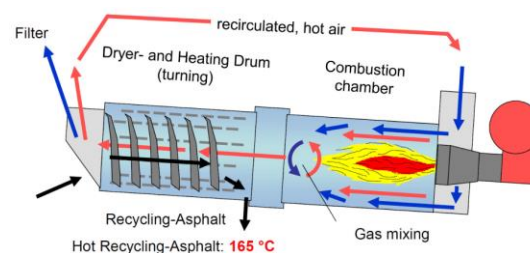


Figure 28. Heating drum

11.3. THEORETICAL ANALYSIS OF REJUVENATOR ADDITION SPOTS

The potential rejuvenator addition locations in asphalt plant can be categorized in three groups:

1. Upstream of RA dryer drum
2. Downstream of RA dryer drum
3. Bitumen

Within each of these groups there are several options resulting in at least ten theoretical locations for rejuvenation addition as summarized in Figure 5. The factors that have to be taken into account when choosing the optimum location include environmental aspects (emissions, leaching), operator and plant safety, pavement quality (even rejuvenator distribution, residence time, temperature, diffusion, loss of rejuvenator), technological (process stability, practicability) and economic reasons. Based on Boesiger et al. [2017] a summary of benefits and drawbacks for each addition location is provided in Table 3. The number of each addition location in Table 3 corresponds to the visualization in Figure 5. It must be noted that these are theoretical considerations based on the authors' experience and can change depending on plant type, setup and experience of operation.

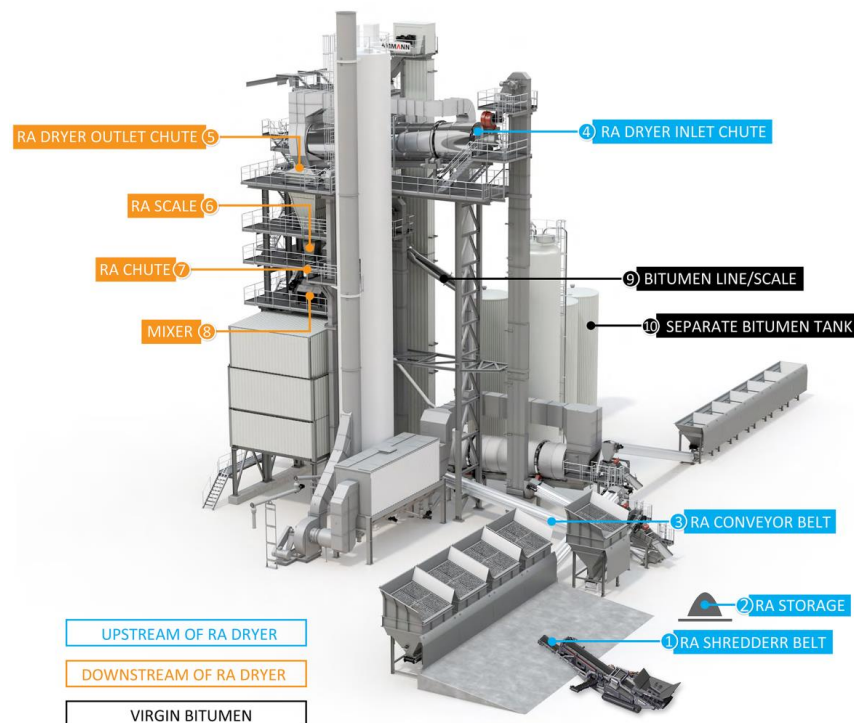


Figure 29. Rejuvenator addition locations (Source: Ammann)

Table 5. Benefits and drawbacks of each rejuvenator addition location

Addition location	Benefit	Drawback
Upstream of RA dryer		
1. RA shredder belt	Long rejuvenator exposure on RA binder Potentially lower RA heating temperature due to lower RA binder viscosity (reduced emissions, energy consumption, increased plant capacity)	Inhomogeneous rejuvenator distribution Rejuvenator drainage Stockpile stickiness Worker exposure to rejuvenator Rejuvenator evaporation in RA dryer Possibly increased exhaust gas emissions due to rejuvenator presence
2. RA storage	Long rejuvenator exposure on RA binder Potentially lower RA heating temperature due to lower RA binder viscosity (reduced emissions, energy consumption, increased plant capacity)	Inhomogeneous rejuvenator distribution Rejuvenator drainage Stockpile stickiness Worker exposure to rejuvenator Rejuvenator evaporation in RA dryer Possibly increased exhaust gas emissions due to rejuvenator presence
3. RA conveyor belt	Long rejuvenator exposure on RA binder Potentially lower RA heating temperature due to lower RA binder viscosity (reduced emissions, energy consumption, increased plant capacity) Precise dosage based on RA feed rate	Possibly inhomogeneous rejuvenator distribution Worker exposure to rejuvenator Rejuvenator evaporation in RA dryer Possibly increased exhaust gas emissions due to rejuvenator presence
4. RA dryer inlet chute	Long rejuvenator exposure on RA binder Potentially lower RA heating temperature (reduced emissions, energy consumption, increased plant capacity) Precise dosage based on RA feed rate	Possibly inhomogeneous rejuvenator distribution Worker exposure to rejuvenator Rejuvenator evaporation in RA dryer Very short interaction of rejuvenator and binder before heating likely resulting in high volatilization of rejuvenator and increased exhaust gas emissions due to rejuvenator presence
Downstream of RA dryer		
5. RA dryer outlet chute	Precise dosage based on RA feed rate Rejuvenator not exposed to RA dryer (potentially lower emissions, safety)	Shorter exposure time compared to “upstream of RA dryer” options Possibly inhomogeneous rejuvenator distribution
6. RA scale	Rejuvenator not exposed to RA dryer (potentially lower emissions, safety)	Shorter exposure time to RA binder compared to “upstream of RA dryer” options Inhomogeneous rejuvenator distribution
7. RA chute	Rejuvenator not exposed to RA dryer (potentially lower emissions, safety)	Shorter exposure time to RA binder compared to “upstream of RA dryer” options Inhomogeneous rejuvenator distribution
8. Mixer	Rejuvenator not exposed to RA dryer (potentially lower emissions, safety) Most experience Simple plant integration Most precise rejuvenator dosing	Shorter exposure time compared to “upstream of RA dryer” options Shortest rejuvenator exposure time to RA binder
Fresh bitumen		
9. Bitumen line/scale	Precise rejuvenator dosing Simple plant integration	No direct contact of rejuvenator with RA binder
10. Separate bitumen tank	Precise rejuvenator dosing	No direct contact of rejuvenator with RA binder No flexibility to change rejuvenators/dosages depending on RA rate and properties Separate bitumen tank necessary

11.4. EXPERIMENT OF REJUVENATOR ADDITION TO COLD AND HOT RAP IN PLANT

Asphalt pavements age during the service life causing formation of pavement distresses that eventually lead to replacement of pavement. Rejuvenators are aimed at restoring the aged asphalt binder to a state where Reclaimed Asphalt (RA) can be re-used and would sustain another pavement service period. Control of RA homogeneity, optimum rejuvenator dosage, homogeneous distribution of rejuvenator, good diffusion of rejuvenator into the RA binder film, and good blending of virgin with RA binder are the key parameters for ensuring the expected pavement performance when using rejuvenators. These parameters depend on the processes in production plant more so than practices in mix design laboratory. It is, therefore, of key importance to determine the best approaches for rejuvenator addition in asphalt plant. This research theoretically compares ten different options for rejuvenator addition in asphalt plant by analyzing the effects on pavement performance, plant operation, and environmental safety. For two most promising rejuvenator addition locations, RA conveyor belt and in mixer, a controlled experimental production in a full scale asphalt plant was performed. The produced samples were analyzed by comparing their compactability, stiffness, fatigue, and crack propagation. The test results indicate that addition of rejuvenator onto cold RA might be beneficial for improving fatigue and crack propagation resistance. During production total organic carbon emissions were tested demonstrating that there is no difference between rejuvenator addition on belt versus addition in mixer. A summary table ranking the ten different rejuvenator addition locations based on environment, plant operation and mixture quality considerations is provided.

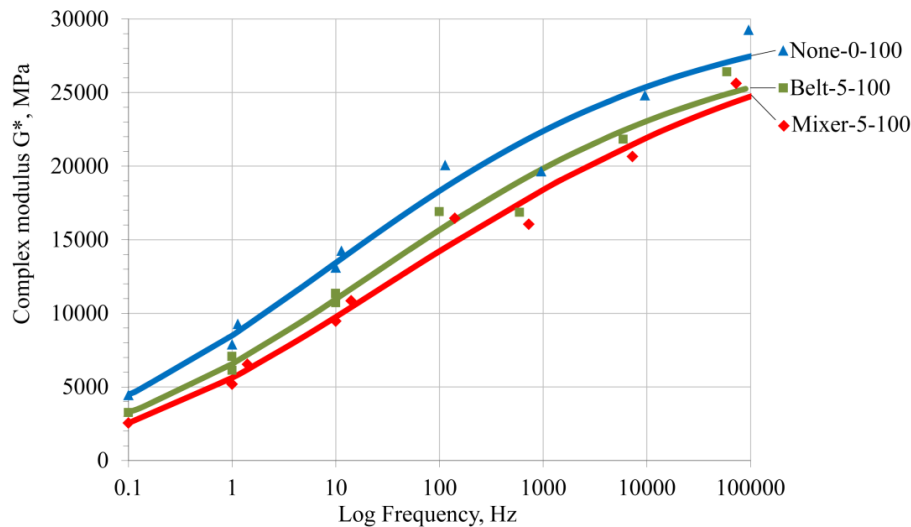


Figure 30. Complex modulus master curves (lines) and data points (markers) shifted to 20°C

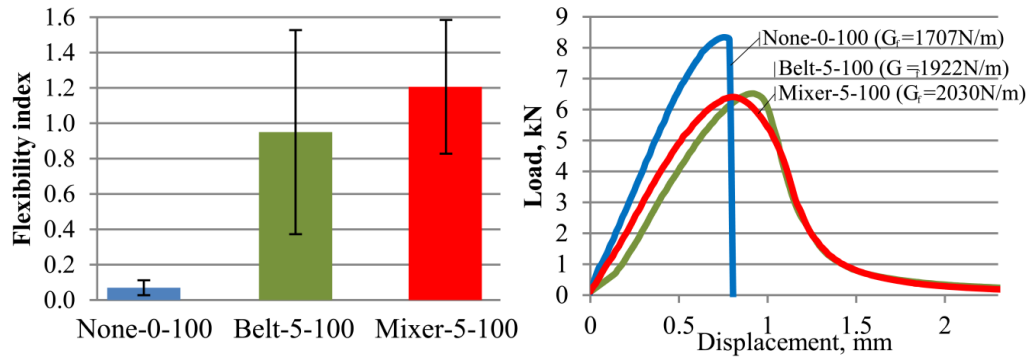


Figure 31. Flexibility index (a) and load-displacement curves (b)

Emission test results

The experimental production was carried out in six windows by ensuring a sufficient time to gather representative emissions measurements for each production case. In addition to the 100% RA mixtures that were used for testing of mixture properties, 60% RA mixture (*None-0-60*) was produced. The *None-0-60* mixture was chosen as the reference because it is within permitted RAP rate locally and thus provides a reasonable baseline. The mixture composition and time of production windows according to the sequence of production are summarized in Figure 32. The average temperature during the testing day was 23°C with no precipitation ensuring favorable ambient conditions for the measurements.

The emission test results and production temperature are summarized in Figure 32 according to the sequence of production. First of all, it is important to recognize that heating temperature is a significant contributor towards generating emissions. In order to minimize the variables, every effort was placed to achieve constant temperature throughout the six production windows. It can be seen in Figure 32 that this was achieved since RA material temperatures are in a narrow 160 °C to 167 °C range for all evaluation windows. For this reason RA material temperature influence on TOC emissions can be said to be negligible and will not be considered in further analysis.

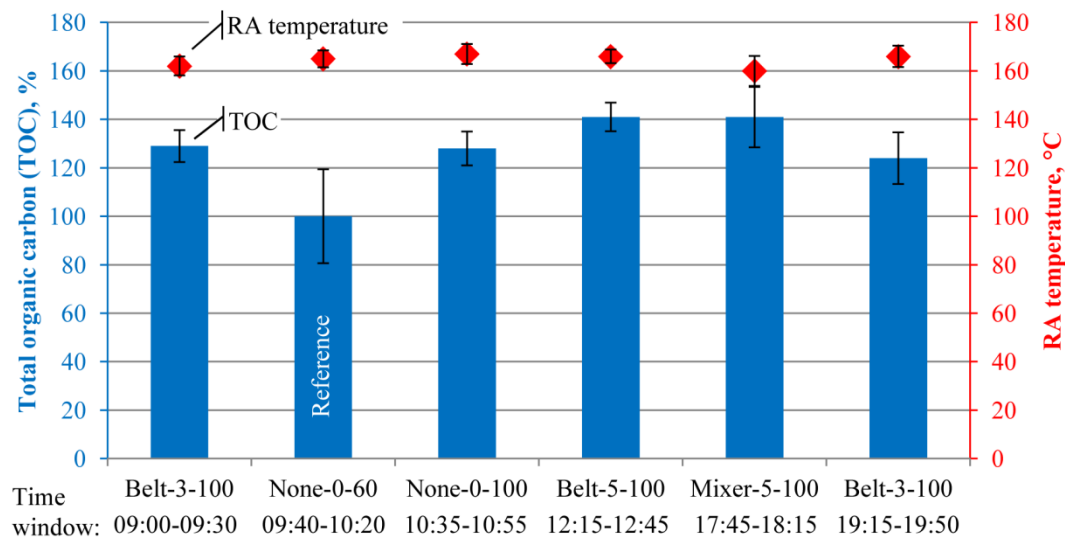


Figure 32. Total organic carbon (bars on primary axes) and RA temperature (rhombs on secondary axes) test results (average result of each measurement time window)

11.5. SECTION CONCLUSION

With ever increasing asphalt recycling rates, rejuvenator use is becoming inevitable for ensuring the required pavement performance. This research evaluated the possible rejuvenator addition locations in one type of asphalt plant in order to provide guidelines to benefits and drawbacks for each of them. Although the work is done for one type of plant, the rationale behind the analysis can be applied to any type of asphalt mixing plant. In total ten locations were identified and analyzed theoretically and two most promising were chosen for further investigation. The results were compared in a practical experiment as summarized in a Video: <https://youtu.be/a9FoeErImM> (Online Resource 1). RA was heated to 160°C in Ammann RAH100 counter flow heating RA dryer and a rejuvenator was applied to cold RA on feed belt and to hot RA in mixer. Three asphalt mixture samples were gathered to evaluate the effect of rejuvenator addition location on binder properties and asphalt mixture mechanic performance. Emissions were measured during the production to determine the environmental effects of rejuvenator addition location and high RA use in general. Based on the testing results the following conclusions can be made:

- Rejuvenator addition on cold RAP on conveyor belt provided the same crack propagation resistance as rejuvenator addition on hot RAP in mixer despite 0.5% lower binder content.

- Rejuvenator addition on cold RAP conveyor belt provided higher fatigue resistance compared to addition in hot mixer (the fatigue test tests did not have enough repetitions to conform to standard).
- The samples for which rejuvenator was applied in mixer had lower complex modulus. This however, might be due to the 0.5% higher binder content of the samples.
- Rejuvenator addition resulted in no significant TOC emission increase, irrespective of 3% or 5% rejuvenator addition compared to 0% rejuvenator addition.
- It made no difference to TOC emissions whether rejuvenator was sprayed on the conveyor belt on cold RA or into the mixer on hot RA.
- Increase in RA rate also increased TOC emissions – a common and known effect.

12.DISSEMINATION

12.1. INTERNATIONAL PUBLICATIONS

The following publications resulted from this project.

- Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Effect of Aging on the Mechanical and Chemical Properties of RAP Binder Treated with Bio-Based Rejuvenators. *Composites Part B* 141 pp174–181, DOI: <https://doi.org/10.1016/j.compositesb.2017.12.060>, (2018)
- Zaumanis M., Poulikakos L.D., Partl, M.N.: Performance-based Design of Asphalt Mixtures and Review of Key Parameters, *Materials and Design* 141, pp 185–201, DOI:10.1016/j.matdes.2017.12.035 (2018)
- Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Aging Effect on Rheology and Cracking Behaviour of Reclaimed Binder with Bio-based Rejuvenators. *J of Cleaner production*. Vol 189. pp 88-97 (print 10Jul18) DOI: 10.1016/j.jclepro.2018.03.305
- Zaumanis M., Cavalli M.C., Poulikakos L.D., Effect of rejuvenator addition location in plant on mechanical and chemical properties of RAP binder, DOI: 10.1080/10298436.2018.1492133

12.2. INTERNATIONAL PRESENTATIONS

- Poulikakos, LD Invited key note: Multiscale characterization of recycled asphalt concrete 3rd International Conference on Transportation Infrastructure and Materials (ICTIM) June 1-4, 2018, Tianjin, China
- Cavalli, M. C.; Zaumanis, M.; Poulikakos, L.D.. Multi-scale investigation on bio-modified reclaimed asphalt binder. 93rd Annual Meeting and Technical Sessions of the Association of Asphalt Pavement Technologists (AAPT). (2018). Jacksonville, United States of America. Poster Presentation.
- Zaumanis, M., Cavalli, M-C., Poulikakos, L. : Design of 100% RAP Hot-Mix Asphalt to Balance Rutting and Cracking Performance. Nr 190, ISAP 2018. Fortaleza, Brazil
- Boesiger, L., Zaumanis, M., Poulikakos, L., Cavalli, M.C., Fierz, R., Kunz, B. Assessment of Rejuvenator Addition in Asphalt Plants, EATA 2017, Zurich. Poster presentation (2017)

- Cavalli M. C., Zaumanis M., Partl M.N., Poulikakos L. D.: Evaluation of reclaimed asphalt binder treated with different bio-based rejuvenators. EATA 2017 Dübendorf. Poster presentation (2017)
- Zaumanis, M., Cavalli, M.C., Poulikakos, L.: Effect of Rejuvenator Addition Location in Asphalt Plant on Binder Properties. 29th international Baltic road conference, August 28-30, Tallin, Estonia, electronic proceedings (2017)
- Cavalli, M.C., Partl, M.N., Poulikakos, L.D.: Effect of Aging on the Microstructure of Recalimed Asphalt Binder with Biobased rejuvenators. 4th ISAP APE Symposium, , Tokyo. Electronic proceedings (2017)
- Cavalli, M C, Griffo, Partl, M N, Poulikakos, L D: Digital Sieving as Tool for Designing Mixtures with High RAP Content, ISAP Symposium. Wyoming, USA. Electronic Proceedings (2016).
- Zaumanis M., 100% Recycled asphalt, 2nd International Road Federation Asia Regional Congress, 16-20 October, Kuala Lumpur, Malaysia (2016)
- Cavalli, M. C.; Griffo, M.; Partl, M. N.; Poulikakos, L.D. (2016). Imaging and image analysis of the effect of mixing temperature in asphalt concrete. 4th Imaging Analysis International Symposium. Dübendorf, Switzerland. Oral presentation.

12.3. WEBSITE

Information about the project can be found on the Empa web site:

<https://www.empa.ch/web/s301/fully-recycled-asphalt>

12.4. ONLINE PRESENTATION/VIDEO

The project web site includes information about the goals of the project and findings:

<https://www.empa.ch/web/s301/fully-recycled-asphalt>

Two videos were made: a video demonstrating the basic principles of performance-based mixture design and is available in the link above. The second video summarizes the principles of asphalt recycling and it is available at the link: https://youtu.be/YWOjfJ23H_M. The videos are designed to increase awareness of asphalt recycling and guide the viewer to read the full publications, which contain explanation of the principles demonstrated in the video.

12.5. PRESS

The project was publisized by Empa in August 2018 and a very positive article appeared in the Zurich newspaper Tages Anzeiger on 17.08.2018.

(<https://www.tagesanzeiger.ch/wissen/technik/der-perfekte-recyclingbelag/story/26063416>)

13.RECOMMENDATIONS

- The traditional mixture design method as specified by the Switzerland Standards is not sufficient to ensure the performance of asphalt mixture containing high content of recycled asphalt to levels similar to that of conventional asphalt mixtures.
- In designing unconventional mixtures, performance-based mix design method can be applied using carefully chosen set of tests that demonstrate the expected mechanical performance of asphalt.
- Depending on the type and amount of rejuvenation, high RAP mixtures are prone to cracking or rutting. Therefore for such mixtures both these aspects need to be satisfied (balanced) to ensure performance.
- Flexibility Index obtained from the semi-circular bend test is a good candidate to include in performance-based mixture design specifications for determining asphalt cracking resistance, because it is sensitive to changes in mixture design and it is simple and quick to perform. In combination with French Rutting Test, the semi-circular bend test can be used to balance the rutting and cracking performance of asphalt mixtures to determine the optimum mixture design.
- Flexibility Index obtained from the Semi-circular bend test should be validated for various mixture types and correlated with field performance to develop acceptance criteria.
- Semi-circular bend fracture toughness results, likely because of the test temperature (0°C), were not sensitive enough towards mix design parameters, including binder content, viscosity and coarseness of gradation. This suggests that fracture toughness should not be used as a mix design tool for the purpose of determining the effect of changes in these parameters. Since binder content and viscosity are important parameters of high RAP mixtures, the test method should not be used for optimizing high RAP mixture designs.
- Rejuvenators can be used to restore the mechanical properties of RAP. Their dosage may be selected by testing at least three rejuvenator dosages and fitting an exponential function to the penetration results.
- In selection of rejuvenator, aging of rejuvenated binder should be used to assess long term effect of the rejuvenators. Development of a routine rejuvenator approval procedure is recommended.

- Although in this project environmental concerns regarding sum of 16 PAHs was not a concern, it is recommended that tests be done on RAP to quantify these environmental hazards.
- Rejuvenator addition location in asphalt plant should be carefully chosen to maximize its efficiency. In this study two addition locations were recommended: addition in mixer or addition to cold RAP on the conveyor belt. Other locations may also be suitable, depending on the plant setup.
- It is recommended to introduce RAP management procedures that would allow to use the RAP for higher traffic intensity roads. For example, homogenization of the RAP, testing of the RAP aggregate angularity and other parameters, separate stockpiles for different qualities of RAP.
- It is recommended to introduce methods for more frequent RAP testing and ranking of stockpiles to ensure homogeneity of the material and subsequent validity of the mixture design.
- Any kind of rejuvenator should be assessed for its environmental impact. Rejuvenators are oil-based materials which can have very different origin and it cannot be assured that they are completely safe.
- According to the literature review, there are different ways of performing the leaching tests. It would be interesting to compare results with some static leaching methods and also to evaluate the results with the evolution in time.
- Assuming mechanical and environmental performance is equal using rejuvenators is more practical than softer binders. As different grades of binder need to be stored in the plant, whereas rejuvenator dosage can be easily calculated.

14.ACKNOWLEDGEMENTS

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15. REFERENCES

1. Forschungsgesellschaft für Strassen- und Verkehrswesen, Arbeitsgruppe Asphaltstrassen, Richtlinien für die umweltverträgliche Verwertung von Ausbaustoffen mit teer/pechtypischen Bestandteilen sowie für die Verwertung von Ausbauasphalt im Strassenbau RuVA-StB 01. 2001, Senatsverwaltung für Umwelt, Verkehr und Klimaschutz. San Diego. p. 489-498
2. BAFU, Richtlinie für die Verwertung mineralischer Bauabfälle. . 2006, Bundesamt für Umwelt, Bern.
3. Boesiger L, Zaumanis M, Poulikakos LD, et al (2017) Assessment of Rejuvenator Addition in Asphalt Plants. In: Poster presented at the EATA 2017 conference. Zurich, Switzerland
4. Bowers BF, Moore J, Huang B, Shu X. Blending efficiency of reclaimed asphalt pavement: An approach utilizing rheological properties and molecular weight distributions. *Fuel* 2014;135:63–8. doi:10.1016/j.fuel.2014.05.059
5. Brantley, A.S. and T.G. Townsend, Leaching of Pollutants from Reclaimed Asphalt Pavement. *Environmental Engineering Science*, 1999. 16(2): p. 105-116.
6. Bukowiecki, N., Lienemann, P., Hill, M., Furger, M., Richard, A., Amato, F., Prévôt, A.S.H., Baltensperger, U., Buchmann, B., Gehrig, R., 2010. PM10 emission factors for non-exhaust particles generated by road traffic in an urban street canyon and along a freeway in Switzerland. *Atmos. Environ.* 44, 2330–2340. <https://doi.org/10.1016/J.ATMOSENV.2010.03.039>
7. Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Aging effect on rheology and cracking behaviour of reclaimed binder with bio-based rejuvenators *Journal of Cleaner Production* 189 (2018) 88e97
8. Cavalli M. C., Zaumanis M., Mazza E., Partl M.N., Poulikakos L. D.: Effect of Aging on the Mechanical and Chemical Properties of RAP Binder Treated with Bio-Based Rejuvenators *Composites Part B* 141 (2018) 174–181
9. Ciecierska, M. and M.W. Obiedziński, Polycyclic aromatic hydrocarbons in vegetable oils from unconventional sources. *Food Control*, 2013. 30(2): p. 556-562.
10. Die Bundesversammlung der Schweizerischen Eidgenossenschaft (2018). Verordnung des EDI über Trinkwasser sowie Wasser in öffentlich zugänglichen Bädern und Duschanlagen 817.022.11. Bundesrat, .

11. Gehrig, R., Zeyer, K., Bukowiecki, N., Lienemann, P., Poulikakos, L. D., Furger, M., Buchmann, B. Mobile Load Simulators – a tool to distinguish between the emissions due to abrasion and resuspension of PM10 from road surfaces. *Atmospheric Environment*, Vol 44 No 38, pp 4937...4943 December (2010)
12. Mafrá, I., J.S. Amaral, and M.B.P.P. Oliveira, Chapter 54 - Polycyclic Aromatic Hydrocarbons (PAH) in Olive Oils and Other Vegetable Oils; Potential for Carcinogenesis A2 - Preedy, Victor R, in *Olive and Olive Oil in Health and Disease Prevention*, R.R. Watson, Editor. 2010, Academic Press:
13. Marsac P, Piérard N, Porot L, Van den bergh W, Grenfell J, Mouillet V, et al. Potential and limits of FTIR methods for reclaimed asphalt characterisation. *Mater Struct* 2014;47:1–14. doi:doi:10.1617/s11527-014-0248-0.
14. Mousavi M, Pahlavan F, Oldham D, Hosseinnézhad S, Fini EH. Multiscale Investigation of Oxidative Aging in Biomodified Asphalt Binder. *J Phys Chem* 2016
15. Muñoz, M., et al. (2016). "Bioethanol Blending Reduces Nanoparticle, PAH, and Alkyl- and Nitro-PAH Emissions and the Genotoxic Potential of Exhaust from a Gasoline Direct Injection Flex-Fuel Vehicle." *Environmental Science & Technology* 50(21): 11853-11861
16. Ozer, H., et al. (2016). "Development of the fracture-based flexibility index for asphalt concrete cracking potential using modified semi-circular bending test parameters." *Construction and Building Materials* 115: 390-401.
17. Ozer, H., et al. (2016). Fracture characterisation of asphalt mixtures with RAP and RAS using the Illinois semi-circular bending test method and flexibility index. 95th Annual Meeting of the Transportation Research Board, Washington, D.C., Transportation Research Board.
18. RILEM TC-50 FMC (1985). "Determination of fracture energy of mortar and concrete by means of three point bend tests on notched beams." *Materials and Structures* 18(106): 285-290..
19. Soenen H, Besamusca J, Fischer HR, Poulikakos LD, Planche J-P, Das PK, et al. Laboratory investigation of bitumen based on round robin DSC and AFM tests. *Mater Struct* 2013;47:1205–20. doi:10.1617/s11527-013-0123-4.
20. US EPA, Method 1311. Toxicity Characteristic Leaching Procedure, in SW-846. 1992, U.S. Environmental Protection Agency: Washington, D.C
21. Williams ML, Landel RF, Ferry JD. The Temperature Dependence of Relaxation Mechanisms in Amorphous Polymers and Other Glass-forming Liquids. *J Am Chem Soc* 1955;77:3701–7. doi:10.1021/ja01619a008

22. Witczak, M. W., and O. A. Fonseca. 1996. "Revised Predictive Model for Dynamic (Complex) Modulus of Asphalt Mixtures." *Transportation Research Record* 1540 (1): 15–23. doi:10.3141/1540-03
23. Yu X, Zaumanis M, Dos Santos S, Poulikakos LD. Rheological, microscopic, and chemical characterization of the rejuvenating effect on asphalt binders. *Fuel* 2014;135:162–71
24. Zaumanis M., Poulikakos L.D., Partl, M.N.: Performance-based Design of Asphalt Mixtures and Review of Key Parameters, *Materials and Design* 141 (2018) 185–201
25. Zaumanis, M, Boesiger, L, Kunz, B, Cavalli, Maria Chiara, Poulikakos, L. D, Determining optimum rejuvenator addition location in asphalt production plant, submitted
26. Zaumanis, M, Mallick, R.B.; Poulikakos, L. D., Frank, R. Performance Properties of RAP Binder and 100 % Mixture Treated with Six Rejuvenators *Construction and Building Materials*, 71 (2014) 538–550
27. Zaumanis, Martins, Cavalli, Maria Chiara, Poulikakos, Lily D. Design of 100% RAP Hot-Mix Asphalt to Balance Rutting and Cracking Performance, submitted