RECYPMA

State of the Art – Recycling Polymer Modified Asphalt

Deliverable No 2.1 & 2.2 (combined)

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Possibilities for high quality RECYling of Polymer Modified Asphalt

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– State of the Art – Recycling Polymer Modified Asphalt

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Executive summary

Throughout Europe polymer modified asphalt (PMA) is used extensively in the past decades for high trafficked roads and premium pavements; especially for surface layers. The addition of polymers contributes to improve performance of standard paving grade bitumens which will enable the binders to cover a broader range of application; especially for pavements with high traffic intensity or demanding load conditions. These pavements are now more and more reaching their end of life. Therefore the road sector is facing a rapidly increasing source of reclaimed asphalt (RA) that contains polymer modified bitumen (PMB). Such RA offers a potential source of bituminous binder with improved performance. It is the challenge to the road sector to ensure that the “RA containing PMB” will be recycled at its highest practical potential. The goal in recycling is not to achieve the highest possible recycling percentage, but a sufficient level to benefit from the improved properties and to **avoid downgrading of RA containing a potential valuable asset**.

The aim of the RECYPMA project is to investigate the possibilities for recycling polymer modified asphalt from surface layers into new high quality surface layers using hot mix recycling. The project should give answers to the following questions:

- What is the potential of using RPMA in new asphalt?
- What is the benefit?
- What (do we think, regarding the results of the project) should be done to get this implemented (technically) based on laboratory test results

The result of the RECYPMA project will be described in four reports:

1. Report on state of the art
2. Report on the properties of aged polymer modified binders
3. Report on asphalt mixtures using RA containing polymer modified binder
4. Report on the benefits of asphalt using polymer modified RA

Additionally a paper and a presentation to summarize the results of the project will be delivered.

This report concerns “State of the Art – Recycling Polymer Modified Asphalt”. This report is focused on the recycling of the polymers present in the RA. For this reason the state of the art only embraces documentation especially high lighting this issue “the added value/impact of polymers” and not recycling technology in general.

The report is also limited to the following three challenges of the project:

1. An updated state of the art on practical laboratory extraction methods that will allow characterisation of the bulk properties of an aged polymer modified bitumen (PMB). (The objective is to ensure that EN 12697-2 and -3 still seem to be the potential best extraction method for practical use)

2. An updated state of the art will be performed on conditions for laboratory mixing as opposed to full scale handling of materials in asphalt plants

3. From a framework on the main asphalt plant configurations for recycling hot mix asphalt a state of the art will be focused on the experience with utilisation of RA containing PMB

The items cover a great number of details which are difficult to mention in this executive summary and the reader is referred to the concluding paragraphs instead. The following statements high light the overall outline of the findings under each item.

Extraction and recovery:

The RECYPMA project focuses on the recycling of reclaimed asphalt that contain SBS-polymer with is the by far most generally applied polymer-type in hot mix asphalt production. Through literature review and a dedicated questionnaire sent out to leading laboratories and research institutions in Europe the conclusion is that

- for practical purposes the European standards EN 12697-1 and -3 can be used to recover a representative bituminous binder irrespectively of the solvent used and
- the RECYPMA project will utilise dichloromethane (methylene chloride) as solvent for the extraction and recovery of SBS-polymer containing bituminous binder from the reclaimed asphalts.

Laboratory mixing:

Based on the literature review a laboratory mixing procedure consisting of several steps is proposed to be used in the RECYPMA project. The procedure is aimed to produce a representative asphalt mix containing recycled polymer modified asphalt but will not try to mimic full scale production, since there is a huge difference in mixing efficiency and conditions in the asphalt plant and in the laboratory mixer. The mixing procedure mentions the order of adding the components and the considerations for determining the mixing temperature (depending on the properties of both the reclaimed binder and the virgin bitumen and its type (whether or not it also is polymer modified).

The mixing order contains an optional choice regarding the introduction of the fines in the mixing process based on local experience with the actual laboratory mixer in question. This is either as “all in” with the virgin aggregates or later after the virgin bitumen has been introduced. In the RECYPMA project the last option will be utilised, since that is in line with the experience at UNIZA which will produce all the asphalt mixes to be used in the project.

Experience with utilisation of RA containing PMB in full scale production:

In the development of polymer modified bitumens it was soon recognised that the polymer to bitumen ratio had to be above a certain level in order to show the changed performance. Typically the effect starts at polymer content in the bituminous binder of approx. 3 %. The era of introducing polymer modified bitumen in hot mix asphalt on a major scale started only a few decades ago. This means that recycling of these pavements has more or less just in the recent years reached a level where the optimal use of this potential asset has been considered. Up until now this special type of RA has not been utilised with the polymer potential in mind due to the small available amounts. In order to reach an effective polymer level when recycling “RA containing PMB” in production of new asphalt a selective recycling (harvesting) and RA management has become necessary to obtain economic feasible conditions. This rather new focus has also an impact on the amount of well documented experience to be found in the literature. The report discusses the available experience in three paragraphs:

- Recognition and availability of reclaimed polymer modified asphalt
- Limitation due to grading curve
- Transport of hot reclaimed asphalt inside the asphalt plant

List of Tables

List of Figures

List of abbreviations

DCM	Dichloromethane (solvent used for extraction)
DSR	Dynamic Shear Rheometer – equipment for rheological measurements on bituminous binders
EVA	Ethylene Vinyl Acetate (thermo plastic polymer)
GPC	Gel Permeation Chromatography – molecular size distribution analysis
HSE	Health, Safety and Environment
IR	Infra-Red as used in IR spectre and IR spectrometers
nPB	Normal propyl bromide ($CH_3CH_2CH_2Br$)
PCE	Perchloroethylene (solvent used for extraction)
PMB, PmB	Polymer Modified Bitumen
PMA	Polymer Modified Asphalt – asphalt produced by using a preblended polymer modified binder (like EN 14023) or by adding the polymer directly into the mixer of the asphalt plant which means no initial binder to test.
RA	Reclaimed Asphalt (US version: RAP - Reclaimed Asphalt Pavement)
RPMA	Reclaimed Polymer Modified Asphalt
SARA	Saturates, Aromatics, Resins and Asphaltenes – bitumen component analysis
SBS	Styrene – Butadiene – Styrene co-block polymer, elastic polymer
TCE	Trichloroethylene (solvent used for extraction)
VMA	Voids in mineral aggregate
WMA	Warm Mix Asphalt (asphalt technology that offers the possibility to produce and pave asphalt at reduced temperatures)

Table of content

1 Introduction

Throughout Europe polymer modified asphalt (PMA) is used predominantly in the past decades for high trafficked roads and premium pavements; especially for surface layers. The addition of polymers contributes to the durability and functionality of these premium pavements, such as preventing binder drainage in porous asphalt (the Netherlands) or rutting resisting pavements (Denmark and Slovakia). These pavements are increasingly approaching their end of life. Therefore the road sector is facing a rapidly increasing source of reclaimed asphalt (RA) that contains polymer modified bitumen (PMB), which offers a potential premium binder contribution if no or limited degradation of the polymer has occurred. Depending on the hardened state of the combined binder and the deterioration of the aggregate gradation during milling and further pre-processing, it is the challenge to the road sector to ensure – as far as possible – that the RA containing PMB is recycled at its highest practical potential. The goal in recycling is not to achieve the highest possible recycling percentage, but **to avoid downgrading of RA containing a potential valuable asset**.

The RECYPMA (RECYcling of Polymer Modified Asphalt) project is part of the program “ENR2 Design: Rapid and Durable Maintenance Methods and Techniques”. The "ERA-NET ROAD II - Coordination and Implementation of Road Research in Europe” (ENR2) program is a Coordination Action funded by the 7th Framework Programme of the EU.

1.1 Aim of RECYPMA project

The aim of the RECYPMA project is to investigate the possibilities for recycling polymer modified asphalt from surface layers into new high quality surface layers using hot mix recycling. The project should give answers to the following questions:

- What is the potential of using RPMA in new asphalt?
- What is the benefit?
- What (do we think, regarding the results of the project) should be done to get this implemented (technically) based on laboratory test results

To ensure a diversity that is relevant for Europe, a broad approach is taken through the state-of-the-art review (first project report), followed by a particular focus on three major types of RA asphalt mixtures (one per participating country), one type of polymer modification and one type of pavement application (surface layer) for determining the binder properties (second project report). Based on the results three asphalt mixtures will be designed with the reclaimed asphalt in order to determine the asphalt properties and quality (third project report). At the end, based on the results of WP 2, 3 & 4 a specific road case is used to estimate the environmental and economic benefits (fourth project report). Additionally a paper and a presentation to summarize the results of the project will be delivered. The four reports as well of the paper and presentation are all focussed on the overall aim of the project.

1.2 Aim of “state of the art” report

This report concerns the “State of the Art – Recycling Polymer Modified Asphalt”. This report is focused on the specific effects and benefits of the recycling of the polymer modified binder present in the RA. For this reason the state of the art only embraces documentation especially high lighting this issue “the added value/impact of polymers” and not recycling technology in general.

The items of the state of the art are also limited to the following three challenges that are identified as the key points in the objective of the project:

- An updated state of the art on practical laboratory extraction methods that will allow characterisation of the bulk properties of an aged polymer modified bitumen. (The objective is to ensure that EN 12697-2 and -3 still seem to be the potential best extraction method for practical use) [Task 2.2]. This step is very important in the identification and assessment of the properties of the bituminous binder in the reclaimed asphalt where the polymer plays a vital part. This assessment is eminent for the utilisation of RA containing PMB in mix design and in documentation of the new produced mix.
- An updated state of the art will be performed on conditions for laboratory mixing as opposed to full scale handling of materials in asphalt plants [Task 2.3]
- From a framework on the main asphalt plant configurations for recycling hot mix asphalt a state of the art will be performed with respect to experience with specific utilisation of RA containing PMB [Task 2.1].

The three items will be addressed in individual chapter of the report.

2 Extraction of polymer modified binders from reclaimed asphalt

2.1 Process description

It is necessary to isolate the binder from the asphalt material, when the properties of a bituminous binder shall be assessed. The purpose can either be as quality assurance control on freshly produced asphalt mix or as assessment of ageing/hardening of reclaimed asphalt. The latter case is part of the procedure for characterising the reclaimed asphalt in accordance with EN 13108-8. This is normally done in a two-step process: dissolution and recovery. This can be performed in many different ways but the principle is described below.

2.2 Dissolution (principle)

The extraction involves normally asphalt materials from either a sample of loose mix from the asphalt plant or cores taken from the produced pavement. The sample is softened by gently heating and granulated or broken apart (typically by hand) to ensure a larger surface area of the materials. The loose cooled material is then exposed to solvents with sufficient solubility power to solubilise the bituminous binders completely. Often the part of the process is performed at ambient temperature in a fuming cupboard in the laboratory. In order to increase the ability of the solvent to solubilise the binder heating can be applied. In one type of extraction (Soxhlet) the asphalt material is “washed” with condensed solvent which is close to the boiling point of the solvent. The extraction can also be performed in various types of partly/totally automated extraction equipment.

The bituminous solution is then separated from the aggregates and the filler by decanting or sieving followed by a centrifuge treatment to remove the finest aggregate particles. The extraction can either be performed qualitative or quantitative. In the first case you must be sure that the part of the binder in the solution is representative for the bulk properties of the binder in the asphalt material. In the second case it is important that all (or almost all) binder is extracted since the determination focuses on amount of binder being present.

The end product of the extraction is a representative solution of the bituminous binder.

Figure 1 Rotary evaporator for recovery of binder according to EN 12697-3

2.3 Recovery (principle)

The recovery process has the function to remove the solvent and to "re-establish" the structure of the binder to the same state as it was assumed to be present in the binder in the asphalt material. This is performed through evaporation/distillation of the solvent which leaves the bituminous binder as a residue or remanence. The recovery process is often performed in various steps due the huge change in properties of the solution going from thin solution to the bituminous binder. The first step is to evaporate the major bulk of the solvent and is relatively easy to perform because the solution has a low viscosity. In the following steps a combination of heat and vacuum (reduced pressure) is introduced in order to remove the smallest traces of solvents without endangering the properties of the bituminous binder due to excessive heat. An example of a test method for this purpose is shown in Figure 1.

Paragraph 2.4 highlights the background on several topics with impact of recovery of polymer modified binders. Paragraph 2.5 contains the documentation from professional contacts and reviewed literature and paragraph 2.6 contains the recommendation of RECYPMA for the recovery of PMB.

2.4 Considerations

2.4.1 Introduction to extraction of polymer modified binder from reclaimed asphalt

A literature search focused on a combination of terms like

- "polymer modified bitumen" or "polymer modified binder"
- "recovery method" or "procedure"
- "extraction

and similar terms have been used in Google and Google Scholar as an add-on to a recently performed literature search in the Re-Road project on the same issues.

In order to improve the literature search and to speed up the input of valuable information concerning the issues of polymer modified bitumen (especially SBS modified bitumen) and extraction an enquiry for documentation and personal experience has been sent out through the network and personal contacts of the partners of RECYPMA. The enquiry has been aimed at "high level" professionals in leading laboratories, research institutions and major contracting players in the road sector. This process has provided valuable feedback for the project. The received information has been most valuable because some input provided a retrospect conclusion of activity in this field and highlighting drawbacks and limitations observed over many years that might not have been documented in focused reports. The enquiry also asked the professionals for their references to literature on the subject which could contribute to the project

The following paragraphs are intended to set the scene for the review in the next sub-chapter of the documentation and literature that have been found for this state of the art report. The paragraphs high light some of the points or issues that are of concern when extraction of binders (straight run and polymer modified) is performed. In the review some reference will be given back to these issues to limit redundant mentioning of the back ground of discussion. Therefore the review will focus on the conclusions of each documentation and its contribution to the decision of whether or not EN 12697-3 and -4 [17, 18] are suitable. Can the methods be used for the purpose of RECYPMA in its quest for the properties of SBS polymer modified bitumen in either reclaimed or freshly produced asphalt?

The documentation of characterisation of bituminous binders in reclaimed asphalt are typically based on "before and after" studies like bitumen before and after its use in hot mixed asphalt or like bitumen before and after being dissolved and recovered. It is a recognised fact that especially the first but even the second isolated step alter the characteristics to some extent of the binder examined. This influences the researchers' ability to draw up very precise conclusions when different recovery methods are investigated and evaluated.

2.4.2 Solvents

The existing extraction methods and procedures have evolved from analysis of traditional unmodified asphalt materials using a straight run paving grade bituminous binder. Over many decades experience with extraction of these kinds of materials have been acceptable. Over the years the procedures have been optimised in order to comply with increasing demand from health, safety and environmental aspects of the analysis. Several procedures are described and standardised in European test methods (like EN 12697-3 and -4) where they are presumed to be equivalent in their ability for a "simple" description of the recovered bituminous binder.

The word "simple" covers the fact that the tests performed traditionally on the recovered binders were penetration and softening point ring & ball. These consistency tests have been sufficient for traditional binders with Newtonian behaviour. In the last decades advanced methods have been introduced on bituminous binders which have the ability to demonstrate engineering properties (like Dynamic Shear Rheometers) and minute differences in chemical nature (like Infrared spectrometers).

The solubilisation of the bituminous binder depends on the power of the used solvent and/or under the conditions it is used (temperature, pressure etc.). For extraction of traditional binders the solvents are normally selected as pure solvents from two classes: the chlorinated hydrocarbons and aromatic hydrocarbons. Examples of the most commonly used ones are given in Table 1. With increasing boiling point the solvents become increasingly difficult to evaporate sufficiently from the recovered binder without exposing it to excessive heat which can cause ageing/hardening of the bitumen irrespectively of the vacuum used.

Table 1 Solvent for extraction of bituminous binders from asphalt materials [17]

Solvent type	Name	Boiling point	Chemical formula
Chlorinated hydrocarbons	Dichloromethane (Methylene chloride)	40,0 ℃	CH_2Cl_2
Chlorinated hydrocarbons	Trichloroethylene	87,0 ℃	C_2HCl_3
Chlorinated hydrocarbons	Tetrachloroethylene	121 ℃	C_2Cl_4
Chlorinated hydrocarbons	1,1,1-Trichloroethane	74,1 ℃	CH_3Cl_3
Aromatic hydrocarbon	Benzene	80,1 ℃	C_6H_6
Aromatic hydrocarbon	Toluene	110,6 ℃	C_7H_8
Aromatic hydrocarbon	Xylene	~140 ℃	C_8H_{10} three isomers

Table 1 indicates that the standardised extraction methods use neat pure solvents, but the use of blends of solvents is introduced in some research work for non-standardised methods [8, 13]. The reason to introduce blends is normally to improve the extraction capability of the used solution towards materials that show difficulties (special additives or very aged components of bitumen). The use of blends of solvents can be a good solution in research work in well-equipped laboratories but there are several draw-backs which presumably are the reasons why neat pure solvents are mentioned exclusively in standardised test methods for intended use in quality control.

When a blend of solvents is chosen then the exact composition of that blend becomes highly important. The blend determines the “solvent strength” of the combined solution, and even minute changes in the proportion can alter the properties of that blend. Some solvents can form an azeotropic blend which means that when the solvents are mixed, one of the solvents (in a binary blend) will evaporate faster than the other until the stable, azeotropic mix proportion is reached, Hereafter the solvents will evaporate further in that relative composition.

It is possible to use blends of solvents for extraction that do not form azeoptropic blends. Here it can be problematic to verify that the correct proportion of solvents is present.. The extraction is often performed in the ordinary road sector laboratory with no access to advanced chemical analytical test equipment for that purpose. During the recovery process (distillation) the composition of these blends will changes, which might have an impact on how the structure of the bitumen and polymers reconstitute themselves. Three examples of blends of solvent for extraction are mentioned in the literature of this state of the art report:

[8] dichloromethane (78 %) and methanol (22 %)

[13] toluene (90%) and ethanol (10%)

[13] trichloroethylene (90%) and ethanol (10%)

None of these three blends are at the azeotropic mix composition. From chemical handbook literature the following are found:

- Dichloromethane and methanol do not seem to form an azeotropic blend.
- Toluene and ethanol form an azeotropic blend (32,0 % , 68,0 % respectively by volume)
- Trichloroethylene and ethanol form an azeotropic blend (73,0 % , 27,0 % respectively by volume)

In the last case an additional point is apparent. Extraction with chlorinated hydrocarbons imposes no fire hazard in the laboratory work, but introducing flammable liquids like alcohols with low flash point means that risk assessment must be performed for this activity in the laboratory.

Handling blends of solvents as opposed to neat pure solvent imposes also two other issues concerning Health, Safety and Environment (HSE). You can normally find protective gloves and clothing that can deal with one type of solvent, but it can be difficult to obtain protective measures for blends that gives the same level of protection (time before penetration of protective material). The other issue is handling of distilled solvents. When you use a neat pure solvent the distilled liquid can immediately be reused in laboratory. If blends of solvents are used the distilled liquids must - due to the formation of azeotropic blends – be shipped to professional recovery installation in order to recover the neat solvents.

The efficiency of these blends of solvents compared to neat solvents is questionable. In [13] the report concludes that no benefit is detected on the recovered binder. In [8] it is claimed that this blend of dichloromethane and methanol is necessary to extract the aged bitumen from the filler, but is must have been a special case since this procedure is not necessary to the general situation described in the European recovery methods [17, 18].

For the time being there are no rumours that the supercritical extraction with CO_2 for bituminous binder extraction from asphalt paving materials has reached a practical and feasible level, yet.

2.4.3 Polymers

Polymers in polymer modified bitumens that are pre-blended prior to use in the asphalt plant are dispersed and constitute a kind of 3-dimensional network in the matrix of the binder. The polymer must have a solubility parameter comparable to the range covered by the components of the bitumen in order to mix well and create an intimate blend. This would give as a first assumption that the solvents capable of dissolving bitumen would also dissolve the polymer. This is only partly true as many factors influence the situation (e.g. solvent strength, composition of the bitumen (relative proportions of saturates, aromatics, resins and asphalthenes) and the ability of polymer to “mix” with bitumen like described by the Hildebrand solubility parameter [33]). Straight run bituminous binders can normally be extracted by trichloroethylene at ambient temperature, but if the binder is modified with the polymer EVA then hot extraction is necessary with the same solvent. Usually it is the experience in the road sector that dichloromethane at ambient temperature is capable of extraction of SBS containing binders (See 6.2.2).

2.4.4 Aged binder and binder affinity towards aggregate

The solvent extraction methods have primarily been developed for quality control purposes of freshly produced asphalt. It is more or less assumed by experience that bituminous binders from even very old reclaimed traditional (unmodified) asphalt can be extracted by the standard procedures. The experience is that a quantitative extraction will become more and more difficult with time, as part of the bitumen change solubility parameter to an extent that it react increasingly as “inert material” from an active binder perspective, typically due to the increased amount of asphalthenes during ageing [19]. Ageing of bituminous binders may introduce functional groups in the dominating hydrocarbon nature of the bituminous binders. This can contribute to a higher affinity of the binder to the aggregate surfaces of the asphalt mix rendering it more difficult to achieve an extraction of a homogeneous binder [8]. Perhaps not detectable of simple consistency tests like penetration and softening point ring & ball. This situation will very likely be dependent of the chemical nature of the aggregate.

2.4.5 Polymer affinity towards aggregate

Like the situation for binder where some components may have a higher affinity for the aggregate surface than the rest polymers might also show this tendency. This can be seen by microscopy analysis of asphalt with different blends of polymers and aggregates [34]. The influence of such an affinity will probably also be dependent of the aggregate source and perhaps be dependent on the process of addition of the polymers. If the addition of polymer was not done through a pre-blended polymer modified bitumen but through production of polymer modified asphalt (PMA) (polymers added directly in the mixer together with hot aggregate and straight run paving grade bitumen) the effect could have a possible impact. If the virgin aggregate was superheated – in order to accommodate introduction of reclaimed asphalt by cold feed in to the mixer – this effect could be even more pronounced. The production of PMA without using a pre-blended PMB is becoming more and more used in countries in Europe. For several years the majority of PMA produced in Denmark has been without a pre-blended PMB. The impact has not been studied so far, but concerns have been flagged on the durability of thin surface layers with “in-situ” mixed as opposed to preblended polymer modified binders [35].

2.4.6 Polymer structure inside the bituminous binder

Since the introduction of polymer modified bitumen a lot of discussion has taken place whether or not the 3-dimensional polymer structure inside the recovered bituminous binder will resemble the structure it had inside the asphalt. This means that the analysis performed on the recovered binder can at best represent a model for the state it has (or had) inside the asphalt. Microscopy analysis of the polymer dispersion can show differences between the original polymer modified bitumen and the recovered binder [34]. Since the polymer modified reclaimed binder will not be "extracted" when the material is recycled/reused, the important point will be the properties of the produced new mix and not whatever properties can be measured on an extracted binder of the new mix. The binder in the new mix will – after extraction and recovered – have a mix of virgin bitumen and old binder in a manner that is not likely to be as intimate in the new produced asphalt [36].

No recent documentation of the polymer structure in reclaimed asphalt has been found, that flags a reservation of the changes to structure during the recovery process.

2.4.7 Degradation of the polymer

Like the organic bituminous binder the different polymers can exhibit different degradation behaviour during production and life time of the pavement prior to and during recycling. The main effects are thermal exposure (e.g. to high production temperature in the mixer at initial production and again when recycled) and oxidation and UV-light exposure during normal lifetime. These effects can contribute to some degradation of the polymer molecules. In general the polymer molecules are not more sensitive to chemical action than the binder, but the polymers usually consist of linear or branched co-block chains of polymers (e.g. SBS). To a certain extent these long chains can be broken.

At research facilities SEC (Size Exclusion Chromatography) or GPC (Gel Permeation Chromatography) can be used to give an indication of such a degradation of molecular size, but these techniques are not very sensitive in linking a potential small degradation of the polymer to an impact on the mechanical behaviour of the new asphalt mix containing polymer modified reclaimed asphalt. A determination of a potential degradation of the polymer will also demand some knowledge of the polymer from its virgin states, and this is normally not obtainable 15 – 20 years old polymer modified reclaimed asphalt of unknown source is received at the asphalt processing unit. The advanced SEC and GPC techniques are for these reasons not in wide spread use in general among contractors' laboratories. This means that the degradation of size is perhaps not known, but the presence can be detected by microscopy and the potential influence of remaining polymer can be assessed on the properties of the recovered binder by the traditional tests.

2.4.8 Future constituents in reclaimed polymer modified asphalt

A lot of development and innovation has taken place in the last decade concerning tailor-made additions to asphalt. Recent developments in Warm Mix Asphalt (WMA) technology have introduced a large variety of different chemicals which now have the potential to be found in future RA and RPMA as well. You can add adhesion improvers and waxes and even specialities like PPA (Poly Phosphorous Acid) to the range of addition that future recycling will have to cope with. For the moment the objective of RECYPMA is to concentrate on plain polymer addition and especially addition of SBS as the predominant polymer in the road sector. But there is a challenge for future recycling activities just a few decades ahead.

2.5 Review of documentation

2.5.1 Review No 1

Table 2 Collection of several responses on present opinions, practise and experience

References	Method/solvent	Experience and interpretation
Contractors		
Mona Teigen NCC Roads Norway	Solvent: dichloro-methane	Only experience with extraction of standard paving grade binders. In a complaint case with PMB extraction is hopefully avoided due to difficulties with interpretation of results (anxiety flagged if polymer structure „survive“ extraction) (See 2.4.6)
Erik Olesen, Vejdirektoratet, Denmark (former Tarco Vej A/S)	Automatic equip-ment (Strassentest) followed by EN 12697-3 using di-chloromethane	In his experience SBS polymer modified binders could be extracted by methylene chloride in a cold process, but successive washings/eluations would improve the recovery percentages of polymer, but you would never get the entire polymer content out of the asphalt.
Kees Plug OOMS The Netherlands	Solvent: dichloromethane Special product: hot toluene	The company indicates that extraction of specific binders (Sealoflex®) can be – apart from special types – be extracted with the use of dichloromethane). In some cases especially for normal bitumen that has interacted with the filler and in cured samples a secondary extraction can be necessary using a blend of solvents (78 % dichloromethane and 22 % methanol) [8].
Konrad Mollenhauer TU Braunschweig Germany	Automatic hot equipment according to EN 12697-1	For the recovery the German standard method using Trichloroethylene in automatic hot extraction devices according to EN 12697-1 has proven suitable for aged asphalt containing polymer modified binders.
Torbjørn Jørgensen Statens Vegvesen Norway	Extraction in an ap-paratus such as the “Infratest Asphalt Analyser”, using dichloromethane as solvent (EN 12697-1) followed by re-covery from extract using a rotary evaporator (EN 12697-3).	The recommended procedure is mentioned to the left and estimated to be used by 3-5 laboratories in Norway. The distillation conditions are fine-tuned by dissolving a 100 g sample of bitumen (or PMB) in dichloromethane, and after 1-2 hours do the recovery of binder from the extract. Penetration (+ softening point, elastic recovery etc.) of the original PMB compared to the recovered PMB should be within the reproducibility limit of the test method.

Table 3 Continuation of Table 2

FEHRL members		
Aleksander Ipavec ZAG Slovenia	Automatic asphalt analysator (Infratest) procedure followed by EN 12697-3 (using trichloroethylene, distillation conditions according Table 1).	Procedure presumed to deliver a representative sample.
Nathalie Pierard BRRC Belgium	For RPMA: 2 hours dissolution in toluene and manual continuous flow centrifuge followed by EN 12697-3	

2.5.2 Review No 2

TRL has participated in recycling polymer-modified asphalt, with a different polymer in the virgin binder than in the reclaimed asphalt, and has monitored it. This is described in [3], which was prepared for the Highway Agency.

In [3] chapter 4 describes the Residual binder and its properties. Neither extraction method nor the used solvent is explicitly stated in the beginning of the chapter. It can however – due to comparison with recovery data from 1993/94 and onwards – be deducted from the discussion in paragraph 4.2.3 that the used extraction method is BS 598-102 which was described to be replaced by EN 12697-1.

The investigations were performed on different materials. Reclaimed porous asphalt was originally produced with BP Olexibit 100, and the trials used three binders as new virgin binder (BP Olexibit 100, Shell Cariphalte Aroflex and Shell Mexphalte 40/60 pen grade). The test programme for mixed binders focuses to a large extent on the effect of binder drainage.

The main report about its construction and its early life was in Chapter 7 of TRL645. This document can also be relevant for other parts of the state of the art report.

According to Shell Bitumen Handbook [19] both trichloroethylene and methylene chloride (dichloromethane) are allowed/used options when the “rolling bottle” version of BSI 598-102 is used. The report [3] mentions neither of the two solvents explicitly. The discussion in the report high lights that “additional 1 % of Binder” could be extracted if the sample and solvent was left to stand for 90 minutes in the metal container prior to rolling.

2.5.3 Review No 3

I. Nösler et al. present a very thorough documentation in [1]. The objective of the paper is to evaluate the effect of recovery method on polymer modified bitumens with high content of SBS polymer. Different recovery methods were used (Soxhlet and centrifuge method) including solvents as dichloromethane, trichloroethylene and toluene.

Dissolving and recovering the highly modified PMB (without presence of aggregates) has only slight impact on molecular size, but the same polymers degraded heavily when the PMBs were mixed with hot aggregate. Laboratory ageing through RTFOT does not seem to simulate the degradation of the polymer observed when mixed with aggregate.

The paper concludes that the different recovery methods can give comparable result, but care must be taken in order to avoid remaining traces of solvents that can have an impact on the binder properties (like decrease in softening point and increase in penetration). One of the key messages from the paper is the importance to minimize the traces of solvent, since 0,9 % solvent can result in 6 ℃ decrease in soften ing point ring and ball.

2.5.4 Review No 4

X. Lu et al. compare in a study concerning ageing of bituminous binders [2] artificial short and long term ageing in the laboratory (RTFOT, PAV and RCAT) with field data. The recovery method used was a national standard analogous to EN 12697-3 and dichloromethane was used as solvent. One of the field sections was a SBS modified binders. The paper mentions no reservation for using recovered data from this procedure to compare with laboratory aged samples.

2.5.5 Review No 5

R. Roos et al. [4] describe the relations between elastic recovery of recovered PMBs and the properties of the asphalt mix. PBMs of German designation PMB 45 A and PMB 25 A from four different suppliers were used in two mixes: mastic asphalt produced at 250 ℃ and asphalt binder course material produced at 180 ℃. Standard recovery procedure were used but was not the main focus of the study. Elastic recovery confirms that the polymer was extracted using the procedure EN 12697-3, but gel permeation analysis (GPC) reveals that the molecular size of the polymers in the recovered binders were smaller than in the original binders. Especially the binders recovered from the mastic asphalt produced at very high temperature showed that effect. This confirms the degradation of the polymer found in [1] when PMBs are introduced in mixes with hot aggregates.

2.5.6 Review no 6

Willem Vonk, Kraton Polymers, gives through a personal communication documented in paragraph 6.2.2 his experience from the development and application of SBS polymers for the use in hot mix asphalt at Shell, KSLA in Amsterdam.

They made use of the support of the KSLA asphalt technical facilities and many binder recoveries were executed as Shell was heavily investing in gaining knowledge on every aspect of bitumen/SBS performance. A lot of comparative data viz-a-viz the unmodified base bitumens were generated. W. Vonk has no recollection of any anomalies reported from the technicians during the experiments with SBS, when they performed the binder extraction/ recovery of the PMB compared to the unmodified bitumen. Also in-house methods for ageing non-compacted mix for a certain period of time at high temperatures were used, after which they recovered the binder. Even in these experiments, no anomalies were detected. In those days the extractions were done with dichloromethane, mainly because of low temperatures needed to release the solvent from the dissolved binders.

There have been reports of issues when other polymers than SBS were tested. Polymers like polyethylene, scrap polyolefins and crumb rubber, as they were not or not sufficiently dissolved in the solvent. W. Vonk can also imagine that such things could in theory happen when blends are reacted with sulphur (cross-linking), as this reaction can potentially lead to some gel formation, which will not be fully dissolved and thus filtered out.

W. Vonk supports the choice of SBS polymer modified bitumen for the study of RECYPMA, as it is much more commonly used. He gives some reflection to the potential problems with selection of solvents and polymer structure inside the bituminous binder, which is mentioned in more details in 2.4.2 and 2.4.6. Still, he thinks that there should be no issue: SBS has no functional groups and does as such not form strong bonds with the aggregate surface. The fact that it has such high compatibility with bitumen is also indicative for the rather similar solution parameters with which one would expect also the same dissolving characteristics for both components in a certain solvent. However, with some of the other polymers being used in the industry (certainly for crumb rubber) one can be absolutely sure that recovery will not yield the same binder as being present in the asphalt mixture.

W. Vonk high lights in his communication that he has also made a quick scan in the readily accessible literature on binder recovery and he concludes that there were hardly any that addressed the potential problem/reservation that the solvent/recovery method would change the structure of the polymer in the recovered binder.

2.5.7 Review No 7

Nathalie Pierard reports from research projects at BRRC in Belgium, where they recently finished a research project on the extraction and recovery of PMB from bituminous mixes (EN 12697-1 and 3). The findings are documented in [9, 10, 11, 12]. The objectives were to evaluate in the case of bituminous mixtures with PMB (SBS, EVA) the effect of:

- Extraction on the determination of the binder content (EN 12697-1, part B2 : extraction with manual continuous flow centrifuge)
- Extraction and recovery on the properties of PMB (EN 12697-3: recovery with rotary evaporator)

The materials were freshly produced – either in laboratory or at asphalt plant. In the last case the samples were taken at the paver on the jobsite.

Some of the conclusions for SBS containing asphalt mixes are:

- For a quantitative determination of binder content toluene seems to be better than dichloromethane but both solvents gave results within the precision of the test method.
- No impact on dissolution method (EN 12697-1) for laboratory mixes but there is indication for small systematic differences between dissolution by “rolling bottle” and “mild agitation” on field mixes.
- On road mixes an impact of aggregate type on properties of recovered binder was found but generally within the repeatability of the recovery procedure.
- For binder recovery: no influence of used solvent (dichloromethane, trichloroethylene and toluene) was found on the binder properties.

2.5.8 Review No 8

E.A.M. Kuppens performed in 1998 a study of recovery of bitumen from porous asphalt produced in the laboratory [13] with the following set-up and conclusions:

Two methods are researched:

- cold extraction according to the SHRP method
- warm extraction with the Soxhlet method

Three solvents are used:

- dichloromethane
- toluene (90%) and ethanol (10%)
- trichloroethylene (90%) and ethanol (10%)

The following tests are performed to look at material behaviour of the extracted product.

- Penetration and Softening point ring & ball
- Dynamic Shear Rheometer from -10 ℃ to +60 ℃
- Gel Permeation Chromatography (GPC)

- Infrared analysis
- SARA analysis (bitumen component analysis)

There is not much difference found in the properties of the extracted binders. SARA, GPC and Infrared do not show much difference. It is noted that this doesn't coincide with the assumption that dichloromethane ages bitumen. This holds even for warm dichloromethane compared to cold toluene. With respect to the chemical behaviour it is noted that when cold extraction is used with toluene, solvent can stay in the sample resulting in very soft behaviour. This study recommends using hot dichloromethane for extractions.

The findings of this research in 1998 coincide with the opinion of W. Vonk in 2.5.6 and others that the “old” reservation for chlorinated hydrocarbons to harden binder during recovery is not valid.

2.5.9 Review No 9

K. Mollenhauer and Prof. Renken give information from a German research project by J. Gröninger et al. [15] that is going to be published soon, in which the extraction procedure is applied on several reclaimed asphalts originating from aged porous asphalt courses. Some points are mentioned.

The use of trichloroethylene has important disadvantages for health and safety. In Germany its use is only allowed in “closed automatic devices”, which are usually installed in fume hoods.

The alternative solvent to use is toluene in manual cold and hot extraction. Though, the experience is, that usually not all polymers can be extracted totally or the recovery according EN 12697-3 doesn’t work properly. One reason for this could be that the distillation conditions (temperature/pressure for recovery) according to the table No. 1 of the European standard EN 12697-3 does not meet the requirements for this solvent (Toluene). This observation was not analysed in detail, but appears to be a general experience among German laboratories.

2.5.10 Review No 10

Torbjørn Jørgensen, Statens Vegvesen (NRA), gives through a personal communication documented in paragraph 6.2.5 his experience as researcher in bituminous materials at the Norwegian Road Administration. The recommended procedure in Norway is

- Extraction in an apparatus such as the “Infratest Asphalt Analyser”, using dichloromethane as solvent (EN 12697-1)
- Recovery from extract using a rotary evaporator (EN 12697-3).

The distillation conditions are fine-tuned by dissolving a 100 g sample of bitumen (or PMB) in dichloromethane, and after 1-2 hours the recovery of binder from the extract is performed. Penetration (+ softening point, elastic recovery etc.) of the original PMB compared to the recovered PMB should be within the reproducibility limit of the test method.

Although there is no Norwegian systematic investigation of the accuracy of the recovery test, it is believed to give reasonable reliable results on recovered SBS-modified PMBs compared with the original binders.

Torbjørn Jørgensen comments also on the issue from paragraph 2.4.8 concerning additional additives in connection to PMB. The last 5 years wax-addition to PMBs is used in several applications (e.g. mastic asphalt) in Norway. Wax may be difficult to extract, and the Norwegians are not sure if recovered PMB with wax is representative of the binder in the asphalt pavement. If the use of Warm Mix Asphalt (WMA) adding wax to the PMB increases, difficulties in binder control and judging binder aging in the asphalt pavement is foreseen. The interpretation is that the recovered PMB may only represent the extractable binder.

Lastly he points to an innovative technique that in the future could be checked for the purpose of project like RECYPMA - Supercritical extraction with CO_2. He had it demonstrated in 1995, and succeeded to extract a few mL of bitumen. There was a limit on the sample size (and hence price pr. analysis), so the method was never developed. He states, that he has not followed up on the technique since that time and does not know if developments have made it more applicable to the road sector and binder extraction.

2.5.11 Review No 11

De Jonhge et al. [14] have compared the extraction and recovery procedure based on either separation of bitumen solution from the aggregate by centrifuge or by Soxhlet for three different solvents:

- Dichloromethane
- Toluene and
- Trichloroethylene

The five bituminous binders used for the asphalt to be evaluated have been produced with standard pen grade bitumen and two polymer modified binders:

- Paving grade bitumen
- EVA modified binder in two different percentages (3 % and 5 %).
- SBS modified binder in two different percentages (3 % and 5 %).

The recovered binders are analysed for penetration, softening point Ring & Ball, Bending Beam Rheometer (BBR, critical temperature) and Dynamic Shear Rheometer (DSR @ 25 °C and 0,4 Hz). The centrifuge method has not been used with

- toluene as solvent due to explosion hazard and
- trichloroethylene due to assessment of the environmental impact.

The authors conclude that all recovered procedures influence to some extent the properties of the recovered binders but the centrifuge method with dichloromethane is the preferred variant. The reasons for the conclusion are:

- Quicker method and less temperature exposure of the bitumen solution with extraction using the centrifuge method with dichloromethane.
- Extraction with Soxhlet exposed the binder solution for a longer period and at higher temperature (especially when trichloroethylene and toluene were used)
- Extraction with Soxhlet gave numerous practical problems due to bump boiling (very frequent for polymer contents of 5 %).

2.5.12 Review No 12

The Ministry of Defence in UK deals with recovery of polymer modified binder from bituminous materials in Specification 49 for Stone Mastic asphalt for airfields [20]. Among the routine tests on compacted cores from the airstrip for control purposes penetration on recovered binder shall be determined using EN 12697-3 or -4 and BS 2000-49. A footnote under Table 8.7 on page 28 states: "*Guidance is required from the binder supplier on the appropriate recovery method if a polymer modified binder is used.*" As the Ministry of Defence will be open to offers of a variety of different polymer modified binders without being limited in use of an inappropriate method for control purposes the same route is followed as laid down in the European standard EN 12594 "Sample preparation" where general guidelines are given for handling bituminous binders with the addition of: "*For modified bitumen use the same procedure, if no other guidance is provided by the supplier.* " This reference shows that the EN 12697-3 is accepted by the Ministry of Defence on quality control on airstrip which normally is exposed to very stringent requirement (more than for normal road paving applications).

2.5.13 Review No 13

J. E. Michael mentions in [21] several solvents that can be used to extract the binder from reclaimed asphalt

- Chlorinated solvents
- Non-chlorinated solvents and
- Biodegradable solvents

But she underlines that the choice is more limited when the recovered binder shall be used for measurements of properties afterwards. Trichloroethylene is one of the most used solvents according to a reference in 2000, but it is seen as a high risk solvent, but not carcinogenic. D-limonene (a solvent extracted from citrus rinds) has been proposed as a non-chlorinated, biodegradable substitution for trichloroethylene, but the bituminous binder cannot be recovered from the d-limonene solution, so the solvent is only applicable for binder content determination by extraction.

Another alternative solvent is n-Propyl Bromide (nPB) has also been suggested to replace chlorinated hydrocarbons and other ozone depleting chemicals. J. E. Michael gives reference to NCAT [22] for a comparison between trichloroethylene and four different grades commercial available of normal propyl bromide. Even though the conclusion in [22] is that nPB can be used as a replacement for trichloroethylene there is a potential incompatibility between one of the grades of nPB towards polymer modified binder. This reference shows the quest for alternative solvents for binder extraction which may provide better environmental performance compared to the locally used solvent (in this case trichloroethylene) but also that it is very difficult to find universal versatile candidates.

2.5.14 Review No 14

One of the most recent works documented in this field is V. L. Mouillet et al. [23] reports from the EU supported research project Re-Road. Re-Road project can also provide more detailed information on characterisation of RA containing polymers, but with respect to binder recovery [23] can be seen as the main reference. At the Eurasphalt & Eurobitume Congress 2012 in Istanbul the findings of a round robin into extraction and recovery methods on polymer modified reclaimed asphalt (RA) are reported.

Apart from soluble binder content using different normalized extraction methods and solvents according to EN 12697-1 the round robin aims for the characteristics of recovered PMB (namely penetration, softening point, oxidation degree) using different normalized recovery methods and solvents according to EN 12697-3 (rotary evaporator). The solvents are trichloroethylene (TCE), perchloroethylene (PCE), dichloromethane (DCM) and toluene.

Six laboratories participated in the round robin test, in which three different bituminous materials have been analysed:

- Stone Mastic Asphalt including 15% of reclaimed asphalt and
- two samples of RA with physical and cross-linked elastomer modified bitumens.

The round robin test has contributed to the evaluation of present methods and their (dis)advantages for studying RA containing Polymer modified Bitumen (PMB), the ultimate aims being to improve the characterization of RA containing PMB.

Figure 2 and Figure 3 show the various combinations of extraction and recovery methods used during the round robin and the obtained result for penetration and softening point Ring & Ball.

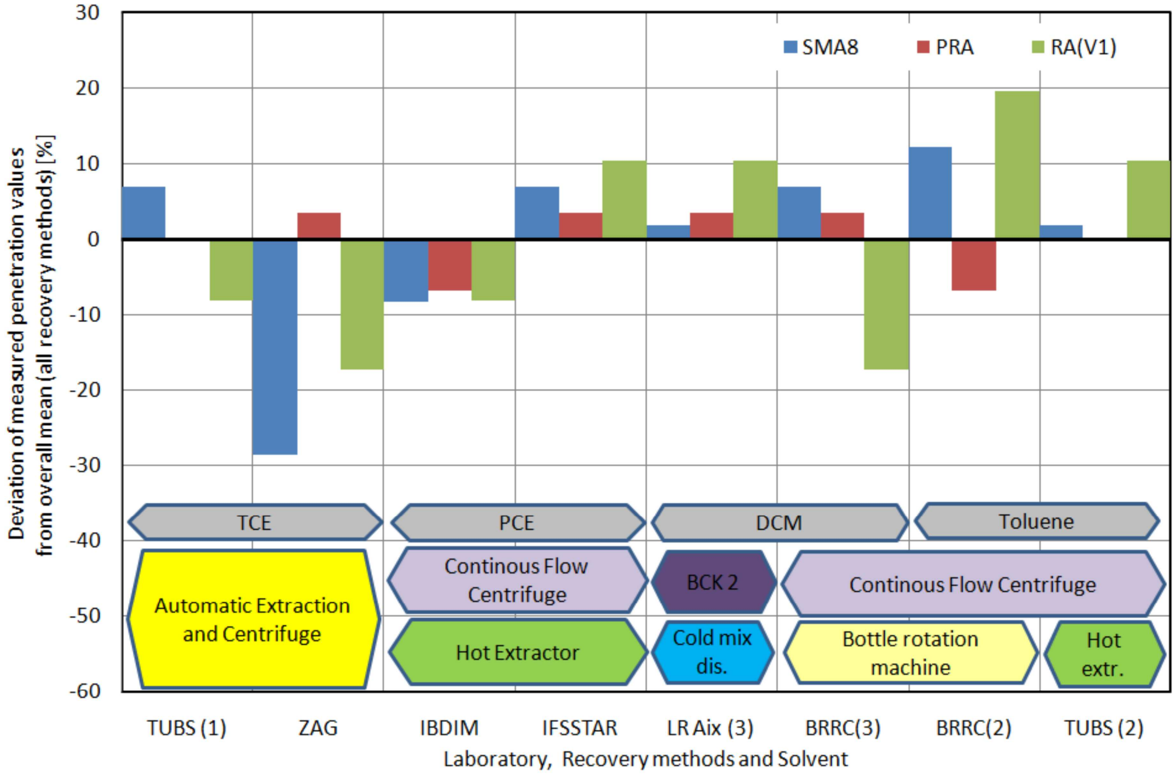

Figure 2 Deviation of measured penetration values from the overall mean

Based on the findings the paper concludes:

"For a new asphalt mixture with PMB and including a low content of RA, no impact of the choice of the method and the solvent was observed. But in the case of reclaimed asphalt, the choice of the couple testing method/solvent has clearly an impact on the measured binder content. The results obtained for all different testing methods/solvents combinations are very scattered. This can be due to the difficulty to extract the binder completely as the stage of ageing of the bitumen in the reclaimed asphalt is very advanced, combined with the presence of polymers which also leads to a more difficult extraction."

"Low content of RA" is in the study refers to 15 % RA in the new Stone Mastic Asphalt. The other two reclaimed asphalts tested was both 10 years old Porous Asphalt with two different kinds of SBS. First type was physical SBS polymer modified binder (Penetration 11 x 0.1 mm; Softening Point Ring and Ball 72.7 ℃, while th e second was a cross-linked SBS modified bitumen (Penetration 11 x 0.1 mm; Softening Point ring and Ball 82.5 ℃). Especially the binder content of the latter showed larger variations between the different combinations of methods and solvents. An interpretation of the conclusions is that the different variants of EN 12697-1 and -3 are applicable for binder recovery and binder content, but depending on age and nature (type of polymer) a quantitative extraction cannot be expected. Cross-linked SBS can difficult. Some scatter exists in the characterization data of the recovered binder but no combination of procedure and solvent proved outlier in this multi-laboratory exercise.

For the purposes of RECYPMA the impact can be marginal of the solvent and the extraction procedure since SBS polymer modified binders are chosen, but an impact can very well be expected if unknown type of polymers and very hardening binder are present in the reclaimed material.

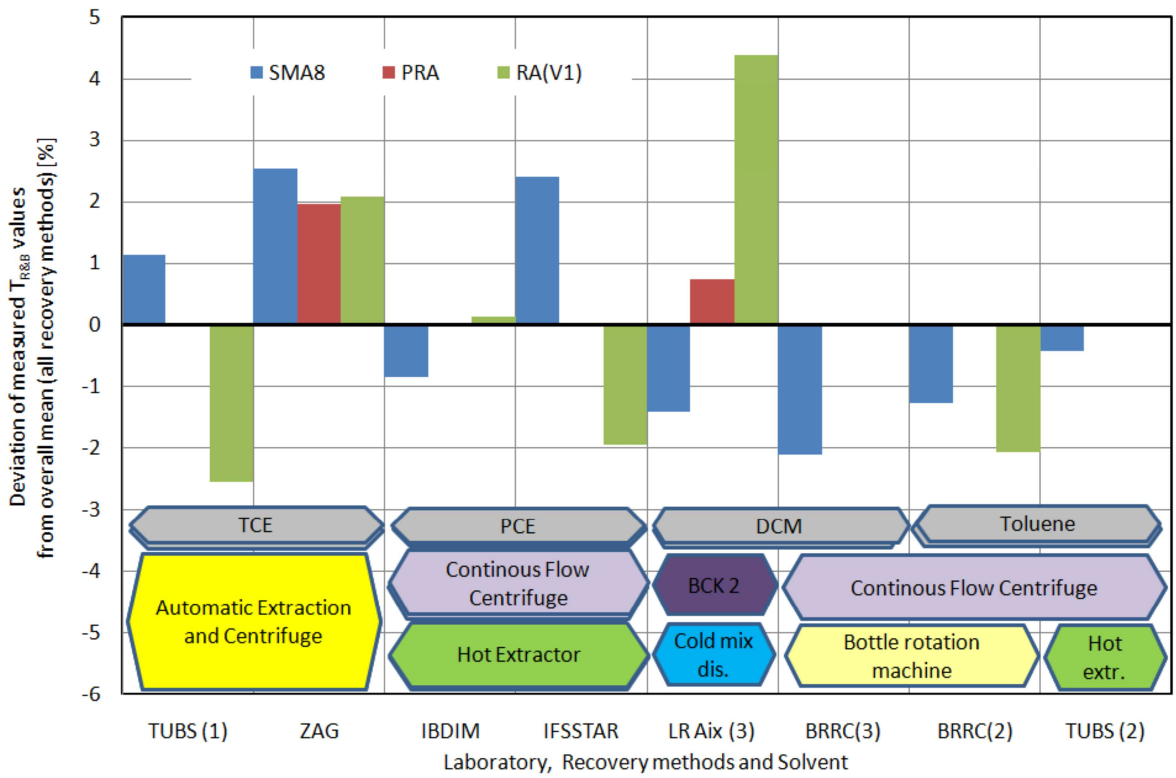

Figure 3 Deviation of measured softening point Ring & Ball values from the overall mean

2.6 Conclusions of extraction procedures in RECYPMA

The state of the art of extraction procedures is collected from a literature review and through an enquiry for documentation and personal experience to the network and professional contacts of the partners of RECYPMA among leading laboratories, research institutions and major contracting players in the road sector.

The stage of ageing and the type of polymer in the reclaimed asphalt can impose difficulties for a total extraction of a representative bituminous binder. Choice of solvent is not only influenced by issues for the technical extraction of binder (solvent strength and procedure) but also by several aspects of health, safety and environment. SBS (Styrene-Butadiene-Styrene) is recognised as the by far most dominant polymer used in the road sector.

The overall conclusion with respect to extraction procedure and solvent for extraction and recovery of SBS polymer containing reclaimed asphalt for the purpose of the ERANET Road project RECYPMA:

- The techniques covered by EN 12697-1 and -3 will be produce representative bituminous recovered binders irrespectively of the used solvent.
- RECYPMA decides to use Dichloromethane as solvent for extraction and recovery
- The extraction will follow EN 12697-1 for the extraction and the use of rotary evaporator in accordance with EN 12697-3 for the recovery of binder.

3 Conditions for laboratory mixing with RPMA

3.1 Introduction to laboratory mixing

Laboratory mixing is normally a part of the mix design in order to examine the properties of asphalt materials of the different combinations of virgin materials. A huge amount of literature has been documented through the years and is available on this issue. For the purpose of RECYPMA it has been interesting to survey more specifically experience and documentation for laboratory mixing when reclaimed asphalt and particular reclaimed polymer modified asphalt are added to the mix. The issue at this point is

- Handling RA and RPMA prior to laboratory mixing
- Influence on conditions of mixing
    - o order of mixing,
    - o temperature and
    - o duration for “dry” and “wet” mixing

Depending on the tradition and national background laboratory mixing as part of the mix design can be a rather restricted exercise following a specific standard or it can be voluntary how you have achieved the specification for the asphalt material as long as the properties of the mix are within the specified limit. This division can in the new common European set up of product specifications reflect whether or not you base your initial type testing on laboratory produced asphalt mix or directly from the asphalt plant during running production of the material. For this reason the references in this part of the state of the art report incorporate information from articles and papers as well as result of questionnaires into common practises.

3.1.1 Review No 16

The European project Re-Road has been mentioned before [23], but Work Package 4 of that project has performed – due to their contacts to industry – a survey of practises in laboratory mixing combining the use of mix design and utilisation of reclaimed asphalt. The report [25] is predominantly the result of more than 25 voluntary responses The objective of the survey has been to provide Work Package 2 of a quick sample of common practises and not a full covering Pan-European survey for input on the present situation in a selection of countries and among different companies operating under common conditions. The countries are:

- Belgium: 1 response (covering a general industry review)
- Denmark: 7 responses
- Germany: 2 responses
- Slovenia: 1 response
- Sweden: 7 responses and
- United Kingdom: 5 responses

At a first glance the responses seem to be unevenly distributed but a response can represent a national organization or major asphalt producer in that country or region with several asphalt plants following the same company policy or local tradition. For instance the Belgian response originates from an organization and the 6 responses from Denmark cover all asphalt producers with in total more than 50 asphalt plants of various configurations.

For this reason the contributions in the tables can be seen as raw data for a more thorough consideration by the partners in Work Package 2.

All countries covered by this survey are acting under the common set of European asphalt product specifications in the EN 13108-x series where a large range of test methods (predominantly the EN 12697-xx series) are offered as shared background common for type testing and quality control. Nevertheless, large differences are seen in the collected input which may correspond to either country or company tradition or policy. Regarding the present utilisation of RA national conditions can influence both percentage of RA in the produced mix and which layers in the pavement structure where addition of RA (in practise) are acceptable.

The following 12 bullet points are quoted from [25] as they concern important information extracted from the survey with respect to mixing conditions either in the laboratory, at the asphalt plant or concerns a possible relationship between those two mixing scenarios. :

Some items are highlighted below but it must be kept in mind that the responses may reflect variations across Europe due to local conditions..

1. *Generally a cross section on the responses on percentage of RA indicated that 15-20 % often is the maximum unless parallel drum or similar device for preheating the RA is available. Several points are mentioned as reason to stay at this level:*
    a. *Concern whether or not investment in production equipment for reaching higher percentages with the present and future level of availability of RA will have a reasonable payback period.*
    b. *European products specification provide more lenient quality control for the RA if the asphalt producer stays below 10 % RA in surface layers and 20 % RA in bituminous base courses.*
    c. *Technical limitations of the RA (like aggregate gradation and binder properties) enforced restriction in what is possible in normal practise.*
2. *Marshall mix design is in general still the most used guide for development of new mix recipes.*
3. *A wide selection of different sizes and manufacturers of lab mixing equipment exists.*
4. *If technical limitations are mentioned almost all responses point at maximum aggregate size.*
5. *Dry mixing with aggregate seems to be the predominant cleaning procedure of the mixing equipment.*
6. *Even though a European standard exists for laboratory mixing (EN 12697-35) it is only used by half of the companies responding.*
7. *A few responses mention a special sequence of addition of the constituents while the predominant part use an initial dry mixing of "all in" (e.g. all aggregate premixed before addition of binder).*
8. *Introduction of RA in laboratory mixing is predominantly performed with heated RA irrespectively of the situation at the asphalt plant (cold feed versus preheated by parallel drum or otherwise).*
9. *Some asphalt producers don't use laboratory mixing at all, but use full scale asphalt production facilities for their development of mix recipes. One response even claims it is cheaper.*
10. *In general mixing temperature are selected by viscosity of the binder or preselected depending on the grade of binder which may result in comparable temperatures used.*
11. *Only very few responses claim that their laboratory mixing procedure is linked closely to the conditions of the local asphalt plant.*
12. *In general the laboratory mixing procedure is used to establish the optimum mix with*

respect to homogeneity and binder coverage, so mixing times (especially wet-mixing times) are prolonged relative to actual asphalt plant conditions.

13. *If RA, PMB or cellulose fibres are added some responses mention extended mixing times.*

The report reveals that a great variety exists in the utilisation of reclaimed asphalt, percentage used and how laboratory mixing is performed (if used at all).

3.1.2 Review No 17

X. Carbonneau et al examine in [24] the European standard EN 12697-35 Laboratory mixing with respect to optimisation of addition of reclaimed asphalt. There is a good discussion part where the authors reflect on differences between the situation in the asphalt plant and the guidelines given in the European standard for laboratory mixing which according to the authors create unnecessary variability.

The arguments are then shown experimentally where a 0/14 mm base course mix with 35/50 bitumen (a standard grade in France) and 40 % reclaimed asphalt is produced in three situations:

- The reclaimed asphalt is dried at 60 °C and heated to 160 °C prior to mixing
- The reclaimed asphalt is dried at 60 °C and heated to 110 °C for 2 hours prior to mixing and the virgin aggregate is superheated to 190 °C
- The moist reclaimed asphalt is added at ambient temperature and the virgin aggregate is superheated to 230 °C

After examining the results on different test (gyratory compaction, Duriez, Wheel Tracking Test and modulus) the authors have the following recommendations for handled reclaimed asphalt in the laboratory:

- The reclaimed asphalt shall be dried at a moderate temperature (60 ± 10) °C after removal of lumps in the material and spreading it in a thin layer not more than 5 cm thick
- The dried reclaimed asphalt shall then be conditioned at approx. 110 °C for (2,5 ± 0,5) hours which implies the use of moderate superheated virgin aggregate to achieve the desired mix temperature. (Reclaimed asphalt must only once be subjected to this conditioning)
- The dry mixing time – including reclaimed asphalt if present – shall be approximately 30 seconds to resemble full scale production.

This procedure is aimed at producing a mix in the laboratory that resembles the mix obtained at their particular asphalt plant with respect to handling reclaimed asphalt.

3.1.3 Review No 18

J.R. Bukowski gives in [26] guidelines for laboratory mixing on how to handle the design of Superpave mixtures containing reclaimed asphalt pavement. From the general guidelines (irrespectively of the intended percentage of RA in the mix) the importance of knowing the moisture content of the RAP is highlighted. But from the different bullet points mentioned in the guidelines:

- Both the virgin and reclaimed materials shall be heated to the intended mixing temperature.
- The reclaimed materials must not be held at the mixing temperature for more than 1 hour.

It can be deduced that the reclaimed asphalt in practise in the laboratory is added dry when mixing takes place.

3.1.4 Review No 19

M. Meijas-Santiago et al. performed in [27] a study of the moisture damage potential for Warm Mix Asphalt containing reclaimed asphalt. Different mixes were produced in laboratory to demonstrate various Warm Mix additives or technologies. In the mixes where reclaimed asphalt pavement was used in 25 % or 50 % the RA materials were treated equal to the virgin aggregates and heated for two hours at mixing temperature prior to mixing. This implies that the reclaimed asphalt was added as dry material.

3.1.5 Review No 20

Viet Hung Nguyen has studied the effects of laboratory mixing methods and RA materials on performance of hot recycled asphalt mixtures in his thesis [28]. Even though the title of the thesis sounds promising the experimental design has serious draw-backs at least from the point of view of RECYPMA.

For the purpose of the investigations the author needs to be in total control of the composition and the properties of the reclaimed asphalt. For this reason it is artificially produced in the laboratory. A Dense Bituminous Macadam is produced with a 40/60 binder (Penetration 50.6 x 0.1 mm; Softening Point 56 ℃) and compacted into slab specimens (305 mm x 305 mm x 40 mm, 8 % voids) which are conditioned in a force draft oven at 85 ℃ for 120 hours. Unfortunately recovery of the binder reveals that hardly any ageing has occurred, apart from what could be expected at the mixing of the materials (Penetration 31 x 0.1 mm; Softening Point 58 ℃).

Later the slabs are softened for 1 hour at 100 ℃ and "granulated" by hand into approx. 40 mm lumps (for large size RA) and further with a jaw crusher to approx. 15-20 mm (for small size RA). These RA granulates are then later added at room temperature to superheated virgin aggregate to a new mix of the same type using 40 % RA and a coloured 160/220 virgin binder to produce a target binder of 70/100. Even though the laboratory mixing times is varied from 1 to 8 minutes huge inhomogeneity is found in cored sample from compacted slabs.

The author of the thesis might overestimate the efficiency of using small portions of superheated virgin aggregate in smaller laboratory mixers and underestimate the efficiency of industrial scale asphalt plants in his conclusions. The pictures of plane sections of produced compacted specimens after different mixing times show the total inefficiency of mixing 40 mm cold RA lumps in 40 % content together with superheated aggregates in a laboratory mixer. For this reason the only safe conclusion from this thesis for RECYPMA with respect to addition of real reclaimed asphalt in laboratory mixing is to avoid addition of lumps of RA at room temperature with small amount of superheated aggregates into the laboratory mixer.

3.1.6 Review No 21

Donatas Cygas et al. have in [29] studied the dependence of the recycled asphalt mixture's physical and mechanical properties on the grade and amount of rejuvenating bitumen. This binder mixing study concerns ordinary reclaimed asphalt (not containing polymer), but with respect to mixing conditions the reclaimed asphalt was placed in trays at 150 ℃ prior to mixing which indicates the use of dried reclaimed asphalt heated to the mixing temperature

3.1.7 Review No 22

Vincent Dubois et al. presented at the E&E Congress in Copenhagen in 2008 a paper on mix design considerations on bituminous materials including reclaimed asphalt where the main objectives were

- to estimate the heterogeneity of the reclaimed asphalt and
- to optimize the mix design procedure to better reproduce the process at a drum mixer facility with these materials.

The study involved recycling two different porous asphalts (placed on a motorway in 1990 and 1991 respectively) into a new high modulus mixture with a recycling rate of 50 % and the use of a 10/20 hard paving grade bitumen as virgin binder. Samples were taken from the site at milling to replicate the full scale production of a drum mixer facility in the laboratory through different approaches when mixing. The reclaimed binder from the two batches of approximately of 16 years porous pavements had aged to similar penetration levels as virgin 10/20 grade bitumen from their original values, but with a small difference in softening point over time. (Lot A: Penetration 8 x 0.1 mm; Softening Point Ring and Ball 77.6 °C and Lot B: Penetration 10 x 0.1 mm; Softening Point Ring and Ball 82,0 °C). IR spectrum reveals that one of the stretches contained SBS polymer, but for the laboratory study the two samples were homogenized into one sample.

Five mixes were produced through the different mixing approaches. The mixes were later compared on their workability during gyratory compaction. The different mixing approaches are here quoted from [30]:

- *"Mixture 1. The loose bituminous mix sampled on the building site is heated in an oven at 100 °C during 12 h then at 180 °C during 4 h. Mixing is carried out with a thermo regulated mixer during 2 minutes. This procedure only aims at homogenising the material.*
- *Mixture 2. All the components (RAP, virgin aggregates, and bitumen) of the high modulus asphalt are preheated in a drying oven at 180 °C. Mixing is carried out during 2 minutes.*
- *Mixture 3. Same preparation as mixture 2 but with a mixing duration of 5 minutes.*
- *Mixture 4. The virgin aggregates are preheated at 180 °C then, before mixing, heated at 380 °C during 15 min. Then, they are mixed with the cold RAP, in the thermo regulated mixer, in order to obtain a homogeneous granular mixture at 180 °C after mixing during 30 seconds. The virgin binder and the fillers, preheated at 180 °C, are finally added. The total mixing time is 5 minutes.*
- *Mixture 5. The binder was recovered from the RAP. The final mix is made by reincorporating the reclaimed aggregates and the reclaimed binder in the adequate proportions of the mix design, with the virgin aggregates, the virgin binder and the fillers. All these elements are preheated at 180 °C. Supplementary part of fillers is added in order to compensate the loss of 0/0.063 mm particles from RA caused by loss during the extraction process. Mixing time is 5 minutes."*

Further information concerning the mix conditions: Mix 1 is the reference mix. The order of components added for Mix 2 to Mix 5 is: the first mixing is carried out with the virgin aggregates and the reclaimed asphalt (raw or without binder) during 30 seconds. The virgin binder and the reclaimed binder alongside the additional filler in case of mixture 5 are added lastly.

The first four mixes have similar evolution of compaction level during gyration while Mix 5 has a deviating workability curve. Comparison between Mix 2 and Mix 3 shows that prolonging the mixing time from two to five minutes improves compaction by reduction of the void content with 1 %. Mix 4 with superheated aggregates gave the highest compaction level. The authors of [30] propose that this can be explained by a better mobilization of the old reclaimed binder which had a penetration value below 10 x 0.1 mm. Test with gamma densitometer showed a higher level of homogeneity of voids versus height of the cylindrical specimen.

3.1.8 Review No 23

The last reference in this part of the state of the art report is again a study from the Re-Road project where Joëlle de Visscher [31] on behalf of Work Package 2 reports on a guide for a laboratory procedure for simulating the mix conditions of asphalt. This comparison study used a mix of Stone Mastic Asphalt with a nominal maximum aggregates size 8 mm which was produced from virgin aggregates (including steel slag) and adding 15 % reclaimed asphalt from porous asphalt layer containing polymer (SBS). The virgin bitumen was also a SBS polymer modified bitumen 25/55-55 in accordance with EN 14023.

Five laboratories participated in producing gyratory compacted asphalt specimen for further assessment after using four different designs of laboratory mixer which all can comply with the European standard on laboratory mixing, EN 12697-35. Since the mix design was chosen to comply with a specific jobsite mix design which also provided the virgin and reclaimed materials, it was anticipated that the mixing time was one of the most important parameters within EN 12697-35 for the following reasons:

- *"Mixing time has an important impact on short term ageing, probably even more when the mix contains RA*
- *Mixing time has an effect on the aggregate shape and grading, due to grinding and crushing (especially the dry mixing time)*
- *Mixing time is a parameter that is not precisely specified in the Europe norm (because it depends on the type of mixer)*

For these reasons, it was decided to consider, in addition to the type of mixer, also the mixing time as a variable mixing condition: every lab was asked to prepare one mix with "normal" mixing times (= the usual mixing times) and a second mix with longer (double) mixing times."

Based on a literature review it was concluded

- There is a great variety of mixing devices used in practise
- Degradation of aggregates was found to be an issue which was handled in different ways – depending among others on the quality of aggregates used and whether or not type testing was performed on laboratory mixed material or full scale asphalt plant produced material
- Different laboratory mixing devices require varying mixing times for reaching a homogeneous asphalt mix and usually longer times than applied in batch plants. Research results indicate that with no fresh air supply to the mixing bowl longer mixing times do not lead to inadmissible aging.

Due to the properties of the materials and mix design the following specific temperatures were chosen:

- Reclaimed asphalt heated to (110 ± 5) °C for (2.5 ± 0.5) hours
- Virgin aggregate heated to (170 ± 5) °C for at least 8 hours (170 °C was chosen to compensate for the lower temperature of the reclaimed asphalt.)
- Polymer modified bitumen heated to (165 ± 5) °C initially for 3 hours but not more than 4 hours
- Laboratory mixer heated to (165 ± 5) °C (equal to asphalt plant mixing temperature)

As a recommendation the following sequence and mixing times were given as a suggestion because it was recognised that local experience with a particular mixer type should overrule the recommendation if it conflicted with achieving an optimal mix:

- Pour the dry aggregates (including fibres and fillers!) in the laboratory mixer and "dry-mix" for 30 s.
- Add reclaimed asphalt and mix for another 30 s.
- Add new binder and mix for another 90 s.

The research continued and has been reported in [37]. The laboratories were asked to produce two mixes: one called "normal mixing" and another where all used mixing times according to the sequence stated above should be doubled. The experimental program considered 10 different laboratory mixing procedures in 5 different laboratories. The performance testing consisted of compactability, stiffness, water sensitivity and permanent deformation. A mix type SMA 8 with 15 % of RA containing PMB from a German asphalt plant was used as test case. The main conclusions of this study are the following:

- Considering the 10 different laboratory mixing procedures, the mixing times are not as critical as expected since they don't have a significant impact on the test results. There was however a weak trend observed that the longer mixing times lead to higher densities caused by increased compactibility. Several possible explanations were given: a better coating of the aggregates and/or more grinding of the aggregates. This latter explanation could play a particular role in this mix because of the steel slag which contains internal pores that may become accessible when the stones are subjected to grinding. The visual techniques (X-ray CT scans and Optical Image Analysis) also gave some qualitative indications that the longer mixing times slightly improve the homogeneity of the mixes (this was seen in the distribution of the steel slag aggregate) and improve the aggregate coating. The differences between the 5 laboratories were larger than the variations due to mixing times. This was expected, since the laboratories used different types of mixers and different gyratory compactors to compact the specimens. These differences should be accounted for when imposing specifications on the test results.
- For the SMA 8 mix considered in the experimental program, there was no significant difference seen between the average results of the laboratory prepared mixtures on one hand and the plant mix on the other hand in any of the performance tests. This shows that it is valid to do an initial type testing study with laboratory prepared mixes, when the correct procedure for laboratory mixing is followed.

3.2 Conclusions of asphalt mixing procedures and for RECYPMA in particular

It is not possible to make a universally applicable description for laboratory mixing of asphalt materials because the intended use of the asphalt can have a large impact on the mixing procedure chosen to be optimal for the specific purpose. This is also reflected in the European standard for laboratory mixing where many parameters are mentioned but without too strict target values (apart from mixing temperature which is linked to binder rheology).

The reason for this allowed variability can be that laboratory mixing can serve different purposes. One use of laboratory mixing could be to find the asphalt properties under optimal conditions. Another purpose could be to investigate the moisture conditions in the virgin materials stored outside irrespectively whether this includes polymer modified reclaimed asphalt.

Another important reason is that laboratory mixing has to include a great variety of equipment of very different sizes with or without additional heating capability. For this reason general principles need to be fine-tuned to local experience with the equipment, conditions and materials at hand.

From the literature quoted it is found that laboratory mixing time at the chosen mix temperature is an important parameter, especially because the laboratory mixing is definitely not as efficient as full scale asphalt plant mixing. In the laboratory it becomes a balance or trade-off between improving mix homogeneity by prolonging the mixing time against increased hardening of the bituminous binder. And again balance is valid irrespectively whether or not polymers (virgin or reclaimed) are present.

The most common purpose for laboratory mixing is to obtain the optimal properties of the asphalt material to be mixed. Later the properties can be examined either on the loose mix or on the compacted specimens. The purpose of the RECYPMA project falls within this category. But as stated earlier this must be adjusted due to the local conditions – in this case the experience at UNIZA with the equipment at their facility – a laboratory mixer with a volume of 30 litres.

The conclusions for laboratory mixing incorporating reclaimed asphalt (and including reclaimed polymer modified asphalt) are presented as statements.

- The reclaimed asphalt material is prior to mixing granulated to the desired size of agglomerates and dried thoroughly at a temperature well below the intended mixing temperature.
- Superheating of virgin aggregate in order to compensate for administering the reclaimed polymer modified asphalt at a lower temperature than the mixing temperature is one option, but superheating has several draw backs.
    - For the purpose of RECYPMA it is decided, that superheating of virgin aggregate will not be applied. Heating the virgin aggregate for 5-10 °C higher than the mixing temperature to compensate for temperature loss during transfer from the oven to mixer is not considered as superheating.
    - In RECYPMA project a high content of polymers from the RPMA is desired and lead to utilise a high percentage of RA in general in the mix. This increased amount of RA leads to a decreasing amount of virgin aggregate to be heated to high temperatures. With a relative small amount of virgin aggregate these temperatures can become unrealistically high to accommodate the heat demand. This is neither practical in the laboratory with the demand for oven controlled at different temperatures nor desirable in the asphalt plant as it tends to burn the nylon filter bags in the filter house.
    - The pre-dried or dry reclaimed asphalt shall be heated for a short period of time – not more than two hours – at a temperature below or equal to the mixing temperature. This time period will depend on the amount of reclaimed asphalt in the final mixture. The possibility or desire to use superheated virgin aggregates to provide the necessary heat will also have an impact on achieving the wanted mixing temperature
- The virgin aggregates are heated to the mixing temperature or superheated to a specific temperature if this scenario is chosen for an extended period – maximum 8 hours – prior to the actual mixing procedure.
- The virgin bituminous binder in sufficient amounts and in container sizes suitable for the mass of asphalt to be produced is heated to the mixing temperature for the shortest time applicable for the purpose. If the virgin binder is supplied in large buckets the binder must be melted, homogenised and poured into suitable container sizes prior to mixing the asphalt.
- If cellulose fibres are part of the mix design the fibres need to be taken apart in rather small lumps and dried at (110 ± 5) °C for approx. t wo hours prior to mixing The fibres must be completely dry, as moist fibres will not be coated during mixing. Due to the less efficient laboratory mixing compared to full scale mixing at the asphalt plant you

can't rely on "dry-mixing" to disintegrate larger lumps of fibres. (This can be detected by microscopic techniques).

- The mixing temperature is chosen according to either
    - the rheology of the virgin binder,
    - the rheology of the reclaimed binder
    - the rheology of the expected resulting binder or
    - the mix temperature stated by the binder supplier (in case of modified binder)

  The choice depends on which situation that provides the most limiting condition with respect to binder rheology. This is for instance influenced by the ratio between reclaimed and new binder and their properties or stage of ageing.

- The following mixing step and times necessary to achieve a homogeneous mix is an initial suggestion which must be adjusted for local experience:
    - Pour all the dry virgin aggregates (including fibres!) in the laboratory mixer and "dry-mix" for 30 s. Additional fillers can either be added at this stage or after addition of the new binder according to local experiences.
    - If lumps of fibres are recognisable after this period prolong the time by not more than 30 seconds. This can be a trade-off of additional crushing of the aggregates and an unintended production of fines and loss of heat if superheated aggregates are used.
    - Add reclaimed asphalt and mix for extra 30 s.
    - Add new binder and mix for additional 90 s. Special fillers can as earlier mentioned be added in this stage. Since this is the local procedure used at UNIZA this approach will be followed in the RECYPMA project.
    - Prolong this period to achieve full coverage of the aggregates but do not prolong it more than absolutely necessary.

4 Experience with utilisation of RA containing PMB in full scale production

4.1 Introduction

Recycling of reclaimed asphalt (RA) is a technology that has been around for several decades. The incentive and innovation took off on an increased scale following the energy crisis in 1972 and especially the next in 1979. In the last decades the concept of sustainability has come in focus including emphasis of saving and recycling of natural resources (virgin aggregates) and the reuse of waste materials from other industries and areas of activity (building demolition waste etc.).

The ever increasing traffic has in the same period imposes tougher demands on the bituminous binders in asphalt materials. These demands could not in all cases be provided by the straight run paving grade binders from processing selected crude oils. For many high trafficked roads in Europe the use of more expensive modified bituminous binders has been cost-effective in order to meet the challenge of rutting (permanent deformations). A large group of modified binders consists of polymer modified binders where selected, engineered polymers have been added to the refined bitumen. The purpose has been to provide a three dimensional network in the binder phase resulting in the required engineering properties of the binder and the asphalt material in which it has been used. One of the main families of polymers in road building is SBS which is an abbreviation for a co-block polymer of Styrene – Butadiene – Styrene monomers.

The use of polymers for this purpose has for the last decades increased and contributed to prolonged durability of the asphalt pavement. This is one of the main reasons why the challenge of recycling of reclaimed polymer modified asphalt has suffered a kind of time lag. Polymer modified bituminous binders are used in new pavements in a much higher percentage of the market compared with the composition of available reclaimed asphalt coming in for re-processing. This situation is now rapidly changing in many European countries.

Many of the problems that the asphalt contracting industry has faced during the process of introducing recycling of normal standard asphalt with paving grade binders have been solved overcome or minimized. It has also imposed minor problems to introduce reclaimed material with polymer modified binders in very small quantities. The effect of the polymer modified binder could just be added as "inert material" – perhaps with no detrimental properties but also not contributing with the potential of the polymers. When the polymer modified reclaimed asphalt was retrieved in small quantities, the incentive has not been eminent for yet another parameter to sort and separate the materials. This has been the situation when the amount of polymer modified asphalt in reclaimed materials has been minute and dispersed.

When larger homogeneous asphalt pavements (e.g. high ways and motorways) have been scheduled to be recycled, the incentive to reuse the aggregates and the potential benefit of the polymers of the old materials has become an obvious route. The quantities of homogeneous material could support the initial analysis and development of a focused mix design for an optimisation of resources and cost-effective production. Material handling in a cost-effective manner complying with all the restraints imposed from society with respect to sustainability, environment and working conditions of the employees has become a key issue for the industry.

This issue has been foreseen for many years, but due to the time lag created by the enhanced durability mentioned above, the recycling of reclaimed polymer modified asphalt (RPMA) is now presenting a variation and additional optimisation parameter on the already acquired knowledge of recycling technology. This means that we have to improve our knowledge through focused activities on how to recycle RPMA in order to let the polymers reveal their full potential.

The present state of the art report on utilisation of RA containing PMB in full scale production is focused on the challenge when recycling of polymers present in the RA has been given priority. For this reason the state of the art only embraces documentation especially high lighting this issue ("the added value/impact of polymers") and not recycling technology in general.

It has been difficult to find documentation for general or specific use of polymer containing reclaimed asphalt which has been recycled on the sole purpose to utilise and document the benefit of polymer. Several reasons can be part of the explanation for this fact and can be following:

- Reclaimed asphalt containing PMB is perhaps available in small volumes and is not kept separate from other types of RA of similar aggregate type. The combined RA is "only" recycled for the purpose of the aggregate and not the "diluted" polymer content.
- Reclaimed asphalt containing PMB will most probably originate from an old surface layer. Apart from recycling porous asphalt into a new stone mastics asphalt the use of recycling material in thin surfacing for high trafficked roads (noise reducing pavements) is seldom possible due to material constraints or allowed by the customer (road authority).
- Full scale recycling jobs are more often case stories in construction magazines rather than the object from scientific research projects (the gap between academia and practitioners).

4.2 Re-Road - Recent state of the art on related issues

The state of the art on recycling including special focus on reclaimed asphalt with polymers has been documented in the European 7th framework programme Re-Road where information was gathered as a result of two subtasks with respect to specific issues through detailed questionnaires and interviews. The next two subparagraphs show the scope information gathering in the Re-Road project as introduction to the extract of information related to reclaimed asphalt containing polymers in the following subchapter 4.3.

4.2.1 "Handling of reclaimed asphalt"

Subtask 4.2 concerned "Handling of reclaimed asphalt" with questions in categories like:

- Validation of the RA "at the gate of the asphalt plant or pre-processing facility
    - Contaminations like tar, asbestos, soil, etc.
    - Potential positive component like polymer modified binder or wear resistant aggregate
- Interim storage
- Interim transport
- Crushing operations
- Sieving/crushing operations
- Environmental survey
- Material consumption and
- Mix plant conditions

The partners of this task covered Denmark, Sweden, United Kingdom and Slovenia with additional information from Finland, Norway, Ireland and contacts in Austria and Italy.

4.2.2 "Introduction of reclaimed asphalt in the mixing process"

Subtask 4.3 concerned "Introduction of reclaimed asphalt in the mixing process" with questions in categories like:

- Re-Road focuses on recycling in stationary asphalt plant but is "in-situ" recycling also performed like (reshape, regrip, remix, remix+, etc.)
- "Mix in plant"-recycling
    - Type of asphalt plant configuration
    - Maximum and optimal percentage of reclaimed asphalt
    - Capacities of plants under these conditions
    - Mixing conditions
    - The use of additives
- "Mix in plant"-recycling – experience with or how to handle moisture in reclaimed asphalt
- "Mix in plant"-recycling – experience with recycling PMBs
    - RPMA handled differently from ordinary RA?
    - Occurrence of special problems linked to polymers being present like sticking problems and how to avoid it
- "Mix in plant"-recycling – configuration of asphalt plant and technology for recycling covering both batch plants and continuous plants
- Asphalt plant modification to improve recycling – experience and trends
- Asphalt plant data with respect to parameters like energy, CO_2 etc.

The partners of this task covered Belgium, Denmark, Germany, Portugal, Sweden, United Kingdom and Slovenia with additional information from Finland, France, Ireland, Norway, Spain, The Netherlands and contacts in Austria and Italy.

4.3 Experience with recycling reclaimed polymer modified asphalt.

All the information from the collected questionnaire responses and interviews are documented in a project report [32] available at the FEHRL website. The documented experience with recycling old asphalt materials which contain polymer modified bitumen documented in [32] is very limited for several reasons. The results of this are mentioned in the following paragraphs in condensed form.

4.3.1 Recognition and availability of reclaimed polymer modified asphalt

One of the reasons why it is hard to extract experience exclusively on recycling old polymer containing asphalt materials is due to characterisation of the materials prior to the mix design. It is important to know before milling that some valuable polymer containing materials can be "harvested" by selective milling in order to reutilize the polymer in the reclaimed asphalt. If reclaimed asphalt containing polymers is not kept separate or combined with similar type of materials (both with respect to polymer and aggregate quality) in your RA management you are losing potential assets. It is important to keep to polymer content of the reclaimed asphalt as high as possible by avoiding "dilution" of the polymer. The new asphalt containing RA shall have at least a polymer content of 3 % in the resulting binder before you have any hope to reach an effect of the old polymer irrespectively whether the polymer has degraded over time or not. This means if you wish to recycle RA containing high level of polymers with a standard paving grade bitumen you must have at least a RA content of 40 % to see any effects. In this situation it means that cold feed recycling (where 15-20 % RA can be reached) will provide you no benefit from the polymers.

When the content of RA shall be at least 40 % in the new mix this will almost certainly require at plant configuration that allows heating of the RA material like in a parallel drum or similar means to heat the material close to the mixing temperature. But such high percentages will often give difficulties to abide to other requirements of the mix composition. Ra containing polymers can be used at lower percentages if the added virgin binder itself is polymer modified. The challenge is then to document the effect of the old polymer from the RA as opposed to the new added polymer.

The knowledge of PMB in the old pavements can originate from documents in the road administrations' archives from, when it was paved, or from recent analysis from cores taken prior to milling. It is not only important to know that polymers are present but also the magnitude of the content.

Additionally the age or state of hardening is important in the first assessment. Some countries have placed limitations to the level of which hardening of the old binder is allowed to proceed and still is recognised as a binder, that can be rejuvenated. Above a certain stage (often by a limit of the softening point ring and ball) the old binder – when assessed as recovered binder – must be considered as "inert material". This will indicate that the binder is deemed beyond a level, where the binding properties can be reconstituted as part of an effective binder. On an even higher level of ageing the "inert material" can even be considered to be detrimental to the durability of the new asphalt due to its brittle nature.

Another reason for very little experience is documented may be due to the market situation. In some countries PMB was introduced in surface layers in the middle or late 1980's for heavy duty application. It improved the durability over the alternative standard binder, which means that some of the first polymer containing surface layers have created a time lag and are just now on the brink of being scheduled for recycling. The introduction and use of polymer in surface layers in Denmark in the 80's and 90's were quite successful but the asphalt sector estimated that the average reclaimed asphalt that are finding its way to the asphalt plants for recycling perhaps only contain polymers in 2 – 5 % of the cases., This percentages is expected to increase rapidly over the next few years since history is catching up on the mentioned time lag. For this reason it has not until now been economically feasible to separate PMB containing reclaimed asphalt from the traditional type of RA. The only cases were, when the source was a huge milling job of a homogeneous stretch of old polymer containing surface layer, where the incentive was both aggregate quality and premium binder.

Even though the asphalt companies would try to separate smaller amount of reclaimed polymer modified asphalt, they could run into problems with their environmental certificate, which in many countries is setting a maximum capacity for handling/processing at a specific site.

4.3.2 Limitation due to grading curve

The next point is that the grading curve may contain an amount of fines grained materials (sand and fillers) that limits the recycling percentage dramatically. For a dense graded material the grading curve may show an even higher level of fines after the milling or scarifying process. If the modification level of polymer is low, the content of fine grained material can in the new mix design end up in a percentage where no effect can be expected, unless the new virgin binder also is chosen as a polymer modified binder. In this case you may have an additional effect of the old polymer. If you chose standard paving grade bitumen as the new virgin binder, you have "diluted" the polymer below a limit, where no effect can be expected irrespectively whether or not the old polymer can have an effect. In "diluted stage" the polymer concentration cannot reach the level, where a three dimensional polymer network is formed throughout the binder phase. In this case the small amount of polymer is present but with no effect. The consequence is, that recycling reclaimed polymer modified asphalt with low level of modification by cold mix addition – with respect to utilise the polymer in the old asphalt - can only be done, if you decide to add a new virgin PMB. The reason behind this is that superheating the virgin aggregates is limited to maximum 15 - 20 % of RPMA in order not to burn the nylon filters in the baghouse filters with this asphalt plant configuration.

High level of polymer modification of the old material can be problematic to obtain, when utilizing the polymer together with virgin non polymer-containing bitumen. You are forced to have an asphalt plant configuration with a parallel drum mixer for heating the reclaimed asphalt containing polymer to almost mix temperature to achieve higher percentages of RA. But through this route it is possible to obtain a level, where the effect of the old polymer is detectable. If irrespectively of your plant configuration you are forced to use a PMB as new virgin binder it is perhaps difficult later to detect whether or not you can observe the added effect of the old polymer over the new added one.

4.3.3 Transport of hot reclaimed asphalt inside the asphalt plant

Heating reclaimed polymer modified asphalt like old stone mastic asphalt in parallel drums to 120 – 135 °C can create a special problem. The Belg ians have encountered a particular problem when using RA with PMB (e.g. originating from SMA surface courses). PMBs in Belgium commonly contain SBS polymers which stick together at temperatures of about 120 – 135 °C. To circumvent this sticking problem, the RA has to be mixed with normal or low mortar content RA before adding it to the parallel drum. This has the drawback of "dilution" of the polymer to a level, where no polymer effect can be detected (unless the new added binder is a PMB.

Perhaps the sticking problem is more evident when polymers are present. It is a recognised problem how to deal with binder or mortar rich materials in recycling; especially using a parallel drum or another means to heat the RA separately before adding it into the mixer (e.g. other techniques than cold feed addition directly into the mixer). Danish asphalt contractors reported in the interviews that they normally in their pre-processing of the RA avoided to establish a 0/6 mm fraction of surface layers, because this material would easily "cake" together during interim storage. If such a fraction was produced, they have avoided the problem by adding virgin aggregate of the same fraction to "diminish" the "caking" problem, but it was more a remedy than a good solution to the problem.

Another angle on this stickiness problem is the experience from asphalt plant configurations concerning transport of hot mix. Transport shall always be either horizontal on conveyor belts or vertically in cup elevators. Sliding material in an angle between 0 and 90 ° shall be avoided if you want to minimize or exclude deposits of material (e.g. material sticking to the equipment). One manufacturer of asphalt plants has introduced a ramp for sliding hot mix material but the plane is also covered with non-stick coating and electrical heated.

The inside of a parallel drum involves some lifting of the materials in shovels but also some sliding action, when reclaimed materials are heated to intermediate temperatures prior to the real mixing process. The fouling inside of the drum and shovels especially when the parallel drum for smaller productions has not reached the optimal working temperature is one of the reasons why some of the Danish contractors are abandoning the use of parallel drum for other means of heating the reclaimed asphalt. The market situation with small job sizes and frequent mix changes during the production day is supporting this trend, as well as the manual labour needed to clear a parallel drum of this fouling material. One contractor mentioned it took approx. two weeks out of operation to clear a parallel drum.

4.4 Conclusion on practical experience with utilisation of RA containing PMB in full scale production

The market situation for reclaimed asphalt containing polymers will in many European countries make it a rapid increasing commodity. As polymer modified asphalt often is associated with surface layers that contain aggregates of premium quality there are incentive for increased focus of the potential in these materials.

Good recordkeeping of old pavement at the road administration or by the contractor can limit the need for analysis prior to milling job to investigate whether or not a particular reclaimed asphalt contain polymers.

If problems with selective milling and interim storage of smaller amount of materials can be diminished or solved there is a good potential for future use of the polymers in reclaimed asphalt and the properties, they can contribute to the new asphalt.

5 Conclusions of the state of the art

The ERANET Road project RECYPMA (Recycling Polymer Modified Asphalt) is focused on the challenge of recycling of polymers present in the RA; among others by providing tools (mixing rules for the reclaimed and virgin binder) to facilitate a well-documented use in mix design of these materials. As a preliminary step a state of the art report to home in on specific points that are necessary to deal with the special issues that concerns the utilisation of polymer modified binders.

For this reason the state of the art only embraces documentation especially high lighting this issue ("the added value/impact of polymers") and not recycling technology in general.

The items of the state of the art are the following three challenges that are identified as important points in the objective of the project:

1. An updated state of the art on practical laboratory extraction methods that will allow characterisation of the bulk properties of an aged polymer modified bitumen. (The objective is to ensure that EN 12697-2 and -3 still seem to be the potential best extraction method for practical use) [Task 2.2]
2. An updated state of the art will be performed on conditions for laboratory mixing of RA containing PMB as opposed to full scale handling of the same materials in asphalt plants [Task 2.3]
3. From a framework on the main asphalt plant configurations for recycling hot mix asphalt a state of the art will be performed with respect to experience with utilisation of RA containing PMB [Task 2.1].

The report's conclusion on each item can be summarized to:

1. It is possible with the dominant polymer in asphalt materials SBS – Styrene-Butadiene-Styrene) to use the solvent dichloromethane within the European standards EN 12697-1 and -3 for extraction and recovery of a representative binder from the reclaimed polymer containing asphalt.
2. A best practise laboratory mixing procedure (mixing order, steps and duration) is suggested, which after a necessary adaption of local conditions can be used to determine the optimum properties of a new mix containing reclaimed polymer modified asphalt.
3. Full scale recycling, which focuses on the optimal use of the potential of the old polymer modified asphalt, is still in its birth – apart from individual large scale rehabilitation job on very homogeneous stretches. Recent survey of the situation reveals that material handling from production of the reclaimed polymer modified asphalt and pre-processing needs further improvement to fully utilise the potential. Suggested areas are:
   - better track records of produced pavement, that will reduce the need for analysis and increase the feasibility for selective milling (separate surface layers from layers beneath)
   - separate crushing and storage of different types of high quality reclaimed asphalt (both polymers and aggregate for surface layers)

The three items are addressed in individual chapters of the report.

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Mix design of bituminous materials including reclaimed asphalt pavement

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Microscopy analysis of Asphalt Mixtures Using Reclaimed Asphalt containing Polymer Modified Binder.

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Mix design and performance of asphalt mixes with RA

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6.2 Annexes

This annex is a supplement to paragraph 6.1. Some of the sources of information can be personal communication – either orally or by e-mail. If many details in such a communication for public use are presented, the main body of that e-mail will be documented here for future reference.

6.2.1 Personal communication

From: **Cliff Nicholls, TRL, UK**

Quote:

TRL has participated in recycling polymer-modified asphalt, with a different polymer in the virgin binder, and has monitored it. This is described in TRL645-05 "Feasibility of recycling thin surfacing back into thin surfacing systems". Authors: I Carswell and J C Nicholls (TRL Limited), R C Elliott (Scott Wilson Pavement Engineering Limited), J Harris (Lafarge Aggregates Limited) and D Strickland (Shell Bitumen). The report was prepared for the Highway Agency.

Chapter 4 describe the Residual binder and its properties. Neither extraction method nor the used solvent is explicitly stated in the beginning of the chapter. It can however – due to comparison with recovery data from 1993/94 and onwards – be deducted from the discussion in paragraph 4.2.3 that the used extraction method is BS 598-102 which was described to be replaced by EN 12697-1.

The investigations were performed on different materials. Reclaimed porous asphalt was originally produced with BP Olexibit 100 and the trials used three binders as new virgin binder (BP Olexibit 100, Shell Cariphalte Aroflex and Shell Mexphalte 40/60 pen grade). The test programme for mixed binders focuses to a large extent on the effect of binder drainage.

The main report about it construction and its early life was in Chapter 7 of TRL645.

6.2.2 Personal communication

From: **Erik Jan Scholten, Kraton Polymers, The Netherlands**

Quote:

I have discussed this matter with my colleague Willem Vonk, technology manager in our research organization. He took part in a lot of R&D that was conducted in the time when we were part of Shell. See below his comments:

> *Dear Mr. Nielsen*
>
> *I can share some experience we have gained in the days we were still part of the Shell group.*
>
> *In those days we were making use of the support of our asphalt technical facilities and we executed many binder recoveries and as we were heavily investing in gaining knowledge on every aspect of bitumen/SBS performance, we generated quite some comparative data viz-a-viz the unmodified base bitumens. In none of the experiments with SBS I got any feedback from these guys that there were anomalies in the binder extraction/ recovery of the PMB compared to the unmodified bitumen. They also used an in-house test these days, in which they kept a non-compacted mix for a certain period of time at high temperatures, after which they recovered the binder and even then, no anomalies were detected. In those days they were doing the extractions with*

di-chloromethane, mainly because of low temperatures needed to release the solvent from the dissolved binders.

There have been issues when we tested competitive systems, but only in case of the use of polyethylene, scrap polyolefins and crumb rubber, as they were not or not sufficiently dissolved in the solvent. I can also imagine that such things could in theory happen when blends are reacted with sulphur, as this reaction can potentially lead to some gel formation, which will not be fully dissolved and thus filtered out.

And although I agree with you that if one would execute a study on recovery of PMBs it should be on SBS modified asphalt, as it is much more commonly used, I still would think that there should be no issue: SBS has no functional groups and does as such not form strong bonds with the aggregate surface. The fact that it has such high compatibility with bitumen is also indicative for the rather similar solution parameters with which one would expect also the same dissolving characteristics for both components in a certain solvent. However, with some of the other polymers being used in the industry (certainly for crumb rubber) one can be absolutely sure that recovery will not yield the same binder as being present in the asphalt mixture.

I also made a quick scan in the readily accessible literature on binder recovery and there were hardly any that addressed the problem you described.

I hope that the above makes sense and is helpful for your investigations.

Willem Vonk

6.2.3 Personal communication

From: **Nathalie Pierard, BRRC, Belgium**

Quote:

BRRC has done a research project on the extraction and recovery of PmB from bituminous mixes (EN 12697-1 and 3). The objectives were:

- to evaluate in the case of bituminous mixtures with PmB (SBS, EVA) the effect of :
  - Extraction on the determination of the binder content (EN 12697-1, part B2 : extraction with manual continuous flow centrifuge)
  - Extraction and recovery on the properties of PmB (EN 12697-3: recovery with rotary evaporator)

The materials were freshly produced, but even though it did not involve reclaimed asphalt the findings can be relevant to RECYPMA.

In the project BRRC studied:

- the effect of the dissolution of PmB alone following by their recovery (following EN 12697-3) (the goal was to approach the impact of the dissolution of the PmB on their properties (different solvents and dissolution method tested)) ==> the results were published at Belgian Road Congress* in 2009 (only in French) and resume in the introduction of a paper for EATA 2010.
- the effect of the extraction and the recovery of PmB (different solvents and dissolution method tested)
  - on bituminous mixes produced in laboratory ==> EATA paper**
  - on bituminous mixes taken at the finisher during the laying

Conclusions of this project are summarized in a publication (Bulletin CRR*** (only in French)) and a PowerPoint in English (presented to the meeting of the Technical committee at BRRC, 02 March 2011).

Concerning the method used in the labs of BRRC to characterize reclaimed asphalt containing PmB (on loose asphalt mixes)

- for the dissolution, we used the "rotating bottle machine" (EN 12697-1 B.1.4) : 2 H of dissolution in toluene
- for the extraction, we used the EN 12697-1, part B2 : extraction with manual continuous flow centrifuge
- for the recovery, we used the condition described for toluene in the EN 12697-3.

Another source of information is the Re-Road project : a paper was submitted by Virginie Mouillet and all WP 1 partners on the first results (for E&E congress at Istanbul).

* [9]

** [10]

*** [11,12]

6.2.4 Personal communication

From: **Konrad Mollenhauer, TU Braunsweig, Germany**

Quote:

I discussed your request with my colleagues. Especially Prof. Renken gave interesting information.

> *For the recovery the German standard method using Trichloroethylene in automatic hot extraction devices according to EN 12697-1 has proven suitable for aged asphalt containing polymer modified binders. A reference project will be published soon, in which the extraction procedure was applied on several reclaimed asphalts originating from highly-aged porous asphalt courses.*
>
> *(reference in German:*
>
> *Grönniger, J., Renken, P. und Wistuba, M. 2009. Verwendung von Fräsasphalt aus Offenporigen Asphaltdeckschichten auf möglichst hohem Wertschöpfungsniveau. Schlussbericht, Forschungsvorhaben FE 07.0212/2006/CGB i.A. des Bundesministeriums für Verkehr, Bau- und Stadtentwicklung, Technische Universität Braunschweig, Institut für Straßenwesen, Braunschweig. Forschung Straßenbau und Straßenverkehrstechnik Heft 1035)*
>
> *Of course the use of Tri has important disadvantages for health and safety. In Germany its use is only allowed in “closed automats”, which are usually installed in fume hoods.*
>
> *The alternative solvent to use is Toluene in manual cold and hot extraction. Though, the experience is that usually not all polymers can be extracted totally / the recovery according EN 12697-3 doesn’t work properly. One reason for this could be that the temperature/pressure for recovery according to EN does not meet the requirements for this type of solvent. This experience was not analysed in detail, as far as I know, but is a general experience.*

I hope this little information will be of some help.

6.2.5 Personal communication

From: **Torbjørn Jørgensen, Statens Vegvesen, Norway**

Quote:

In Norway 3-5 laboratories are capable of extracting and recovering PMB from asphalt samples.

Our recommended procedure is

1) extraction in an apparatus such as the “Infratest Asphalt Analyser”, using dichloromethane as solvent (EN 12697-1)

2) Recovery from extract using an rotary evaporator (EN 12697-3). The distillation conditions are fine-tuned by dissolving a 100 g sample of bitumen (or PMB) in dichloromethane, and after 1-2 hours do the recovery of binder from the extract. Penetration (+ softening point, elastic recovery etc.) of the original PMB compared to the recovered PMB should be within the reproducibility limit of the test method.

Although we haven’t done any systematic investigation of the accuracy of the recovery test, we believe we get reasonable reliable results on recovered SBS-modified PMB’s.

The last 5 years wax-addition to PMB’s is used in several applications (e.g. mastic asphalt). Wax may be difficult to extract, and we are not sure if recovered PMB with wax is representative of the binder in the asphalt pavement. If the use of Warm Mix Asphalt (WMA) adding wax to the PMB increases, I foresee difficulties in binder control and judging binder aging in the asphalt pavement.

We may have to say that the recovered PMB represent the extractable binder only.

If the project looks for an innovative technique, Supercritical extraction with CO2 may be checked up. We had it demonstrated in 1995, and succeeded to extract a few mL of bitumen. There was a limit on the sample size (and hence price pr. analysis), so the method was never developed. Perhaps technology and commercial apparatuses are more achievable today? I don’t know if this method will work on PMB’s.