EUROPEAN PROJECT RE-ROAD: ROUND ROBIN TEST ON EXTRACTION AND RECOVERY METHODS FOR RECLAIMED ASPHALTS WITH POLYMER MODIFIED BITUMENS

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ABSTRACT

The recycling of Reclaimed Asphalt (RA) in new hot-mix asphalt (HMA) is a common practice throughout Europe. However, Reclaimed Asphalts (RA) are complex materials and the use of significant proportions of RA involves a more accurate control of their characteristics. This is an essential step for asphalt mix design and a key factor to obtain good performances. At this time there is clearly a lack of knowledge and adequate test methods to analyse RA with Polymer modified Bitumens (PmBs). The present European test methods for extraction and recovery of binder in RA is suitable for RA with pure binders but give only indicative guidelines for RA containing PmB.
To develop techniques for increasing the recycling rates of RA especially in surface asphalt mixes, the binder characterisation test methods are analysed and optimised during European research project Re-road. This contribution presents the results of a round robin test on:
-soluble binder content using different normalized extraction methods and solvents according to EN 12697-1
-characteristics of recovered PmB using different normalized recovery methods and solvents according to EN 12697-3
Six laboratories participated in the round robin test, in which three different bituminous materials have been analysed: one Stone Mastic Asphalt including 15% of RA and two other RA with physical and cross-linked elastomer modified bitumens. The round robin test has contributed to the evaluation of present means and their (dis)advantages for studying RA containing Polymer modified Bitumen (PmB), the ultimate aims being to improve the characterization of RA containing PmB.

Keywords: reclaimed asphalt pavement (RAP), recycling, polymer modified bitumens, binder content, extraction

1. INTRODUCTION

Most of European road network is paved with asphalt material. The dismantling and end of life strategies for these pavements are very divergent among the EU member states and the associated countries. In general the share of recycling the reclaimed asphalt in new asphalt courses is rather lower than it could be technically. The Re-road project[1] aims to address these problems with a holistic approach to the technical and environmental aspects of all steps in the recycling procedures of asphalt material (Kalman, 2009). The overall objectives of the project is to be able to raise the level of re-use of hot asphalt asphalt to 99% with a minimum of downgrading of the material and a minimal introduction of virgin material into the mixes made with reclaimed asphalt. For surface courses the objective is to recycle reclaimed asphalt at highest possible quality, preferably with a content of reclaimed asphalt of 40 % or higher.

However, Reclaimed Asphalts (RA) are complex materials and the recycling of significant proportions of RA involves a more accurate control of their characteristics. These last ones are essential for asphalt mix design and a key factor for correct performance of new asphalt mixtures containing large content of RA.

Consequently, it is very important to focus on issues that are specifically related to the characterization and technical evaluation of RA and particularly RA containing modified binders for which there is clearly a lack of knowledge and adequate test methods to sample and to analyse them. One of the topical problem is related with the determination of the binder content for which the present European test methods for extraction and recovery of binder in RA are normative for RA with pure binders and give only indicative guidelines for RA containing PmB. As binder extraction and recovery is essential in order to determine the basic characteristics of RA, it is necessary to precise suitable methods for RA with PmB. This is the aim of this paper that presents first research results of a round robin test on:

- soluble binder content using different normalized extraction methods and solvents according to EN 12697-1,
- characteristics of recovered PmB (namely penetration, softening point, oxidation degree) using different normalized recovery methods and solvents according to EN 12697-3 (rotary evaporator).

Six laboratories[2] participated in the round robin test, in which three different bituminous materials have been analysed: one Stone Mastic Asphalt including 15% of RA and two other RA with physical and cross-linked elastomer modified bitumens. The exploitation of this round robin test is described below. It has contributed to the evaluation of present methods and their (dis)advantages for studying RA containing Polymer modified Bitumen (PmB) and is going to permit to select and/or develop suitable extraction and recovery methods in order to obtain the correct binder content and the correct properties of PmB in RA.

2. DESCRIPTION OF THE EXPERIMENTAL PART

2.1 Materials

In order to assess the influence of extraction/recovery methods on the binder content and bitumen characteristics of RA, three different reclaimed asphalt mixtures have been sampled and tested with the methods used in the different partner laboratories:

- One Stone Mastic Asphalt (called “SMA8”) with modified bitumen as virgin binder and including 15% of RA (also with PmB) was sampled from a mixing plant. This first asphalt mixture is used as reference sample because it is a new mixture including a PmB and a low content of RA. Hence, it can be assumed that this asphalt mixture will have a good homogeneity and properties not very affected by aging. So, if a difference is observed, it can be assumed to be due to the extraction and recovery procedures of binder.
- Two Reclaimed Asphalts with Polymer modified Bitumens. The polymer SBS (Styrene-Butadiene-Styrene) being the most common polymer modifier used at this time, only RA with elastomer modified bitumens have been selected:

[1] Funded by the European Community’s Seventh Framework Program FP7/2007-2013 (n° SCP-GA-2008-218747)
[2] ISBS from Germany, IBDiM from Poland, IFSTTAR from France, LRPC-Aix from France, BRRC from Belgium and ZAG from Slovenia

- One French Porous Reclaimed Asphalt (called "PRA") that was sampled in 2009 directly after milling from porous asphalt layer of age 10 years; the binder used for this RA is a physical SBS modified bitumen.
- One Reclaimed Asphalt (called "RA(V1)") was sampled in 2004 directly after milling from porous asphalt layer of age 10 years; the binder used for this RA is a chemically linked SBS modified bitumen.

2.2 Methods of determination of soluble binder content

In this study, each laboratory has measured the binder content according to the European standard EN 12697-1. The dried asphalt mixture is weighted and set in a cold or hot solvent. Then, mineral particles are separated from the bituminous binder. The binder content is calculated by measuring the weight of the obtained aggregates.

The European standard EN 12697-1 allows different methods for the extraction of the binder. They are to be combined with different methods for separation of the mineral matter. Different solvents have to be used according to differing work safety regulations. Consequently, each partner has measured the binder content according to its own methods (Table 1) chosen from EN 12697-1. So, the same samples, representative of the considered RA, are tested by different laboratories, with different methods and different solvents.

Standard method :	BRRC	IBDiM	IFSTTAR	LR-Aix	TUBS	ZAG
1. EN-Methods for binder extraction						
Hot extractor		X	X	X (1)	X (2)	
Soxhlet						
Bottle rotation machine	X (2,3)					
Centrifuge extractor						
Cold mix dissolution of bitumen by agitation				X (2)		
Alternative method :						
Automatic extraction and centrifuge apparatus	X (1)				X (1)	X
2. EN-Methods for the separation of mineral matter						
Continuous flow centrifuge	X (2,3)	X	X	X (1)	X (2)	
Pressure filter						
Bucket centrifuge type 1						
Bucket centrifuge type 2				X (2)		
Alternative method :						
Automatic extraction and centrifuge apparatus	X (1)				X (1)	X
3. Solvent						
Toluene (Tol)	X (2)				X (2)	
Trichlorethylene (TCE)	X (1)				X (1)	X
Dichloromethane (DCM)	X (3)					
Perchlorethylene (PCE)		X	X	X (1,2)		

Table 1 : EN- methods for determination of the soluble binder content described in EN 12697-1 and their use by each partner[3]

As can be seen, four methods of those described in the standard are used for the binder extraction by the laboratories involved and the continuous flow centrifuge is the most used method for the separation of the mineral matter. Half of the laboratories also use an automatic extraction and centrifuge apparatus. Altough some parameters such as pressure or temperature are controlled, the standard EN 12697-1 allows the use of an automatic device if it can be demonstrated that it provides the same results as one of the methods described in table 1.

Concerning the choice of solvent, four solvents are used in the different partners' laboratories.

Each laboratory has measured the binder content of the asphalt samples with 2 repetitions.

2.3 Binder characterization

2.3.1 Extraction and recovery methods

[3] Number between brackets associates the EN- methods used for extraction and separation with the solvent used in an complete procedure.

First of all, an important step before characterizing binders is the recovery process, that is done by each partners' laboratory according its method chosen from EN 12697-1 (see table 1) and EN 12697-3. In fact, EN 12697-3 describes a procedure to recover bitumen from an asphalt mixture. It consists first to separate and to extract the binder from the bituminous mixture by dissolution in an adequate solvent and by the use of a method to separate it from the mineral matter (according to EN 12697-1 standard). In a second step, bitumen is recovered by distillation using a rotary evaporator.

To recover the binder, the methods used for the separation and the extraction of the soluble binder are the same as the ones mentionned in the table 1 for most of partners involved in this round robin test except for :

- BRRC : Only the bottle rotation machine was used to dissolve the binder in toluene or dichoromethane and the separation of the mineral was done using the continous flow centrifuge.
- LR-Aix : the binder was extracted from the mixture by dissolution at 30°C in dichloromethane and was separated from the mineral part using a bucket centrifuge type 2.

2.3.2 Binder characterization

As the composition of RA binder is an important issue related to the possible re-use of RA and in particular its capacity to mix with the fresh added bitumen by the exchange of viscosities, different characteristics have been assessed by laboratory testing after recovery of binder from RA (according to EN 12697-3). These characteristics are strongly related to the assessment of the end of life of RA, namely the physico-chemical state of the binder and its ageing level. Hence, a representative panel of relevant laboratory procedures has been proposed, that consists in measurement of usual properties of a binder (penetration, softening point) and laboratory assessment of the quality, nature and ageing level of a bituminous binder (oxidation degree, polymer content, complex modulus, ductility force, elastic recovery, repartition of molecular sizes). It has to be noted that, in order to avoid bias effects due to reproducibility scattering caused by the test method, one single partner's laboratory has performed one type of test for all recovered binders.

Among the different characteristics, this paper will only discuss the impact of the recovery method and solvent used on three measured characteristics described below:

- Consistency parameters : measurements of penetration at 25°C (1/10 mm) according to EN 1426 and ring and ball softening point (°C) according to EN 1427.
- Oxidation degree : it is determined by Fourier Transform Spectrometry, which permits to follow the binder oxidation with ageing, based on oxidation peaks observed in IR spectrometry, that is, the peaks at 1700 and 1030 cm^{-1} (Mouillet *et al.*, 2010). The first peak is characteristic of the presence of carboxylic functions in the binder, while the second characterizes sulfoxide functions. Both are indicators of binder ageing, as they reflect the degree of oxidation. These chemical indicators are determined with an infared spectrometer (Perkin Elmer Spectrum One) (Piérard & Vanelstraete, 2009). The binders are dissolved to a concentration of 75 g of binder/l of CCl_4. Each spectrum is normalized as follows: correction of the baseline between 1885 and 459 cm^{-1} and absorbance coefficient of a standard bitumen peak situated between 1400 and 1500 cm^{-1} brought to 1.2.

  To evaluate the oxidation degree, the following surface area peaks are studied:
  - A1700 (area comprised between 1530 and 1770 cm^{-1}) indicating the presence of carbonyl functions (ketones, esters, carboxylic acids);
  - A1030 (area comprised between 1000 and 1105 cm^{-1}) indicating the presence of sulfoxides compounds.

3. RESULTS AND DISCUSSION

3.1 Soluble binder content

To consider the influence of different experimental parameters on the results, the values of binder content obtained by each partner's laboratory have been classified according to the method and solvent used (see table 2). The relative differences of measured binder content evaluated in the various methods from the total mean are shown in Figure 1.

Considering the results, the solvent and the method used does not seem to have an impact for the SMA8 mix sample, for which the single values vary less than 1,5 % relative from the total mean value. This is not the case for the RA materials. The impact of the solvent/method is more pronounced for the RA(V1) with chemically linked SBS modified bitumen (0.4% by mass) than for the PRA with a physical SBS modified bitumen (0.2% by mass).

The deviation of the test results indicates relative differences of the single test results up to ± 10 % of the mean value for PRA samples and ± 7,5 % for sample RA(V1) (Figure 1). For most methods, the test results indicate deviations of the mean binder contents in both directions. For example method IBDiM results in higher binder contents compared to the total mean for samples SMA8 and RA(V1) (with chemically linked SBS modified bitumen) but in lower binder contents for sample PRA (with a physical SBS modified bitumen). The discrepancy in relative deviation for the single extraction and recovery methods indicates that the effect of test method maybe less than the inhomogeneity of the tested RA samples.

The general precision of the test method according EN 12697-1 for asphalt mixes with unmodified binder is:

- Experiment 1, according EN 12697-1, clause 8.1: reproducibility R = 0.5 % by mass, s_R = 0.18 % by mass.
- Experiment 2, according EN 12697-1, clause 8.2: repeatability for a asphalt mix with D ≤ 11 mm: ; reproducibility R = 0.42 % by mass, s_R = 0.15 % by mass.
- Experiment 3, according EN 12697-1, clause 8.3: R = 0.34 % by mass, s_R = 0.12 % by mass.

The standard deviation calculated from all the results for the three samples in this study are:

- SMA8: s_D = 0.08 % by mass,
- PRA with a physical SBS modified bitumen: s_D = 0.24 % by mass,
- RA(V1) with chemically linked SBS modified bitumen: s_D = 0.33 % by mass.

The total standard deviation evaluated for sample SMA8 is below the reproducibility standard deviations according to EN 12697-1. For the two RA samples, the standard deviations exceed the three reproducibility limits according to EN 12697-1.

The larger dispersion in the results for the case of RA is also already observed between two results of a same laboratory for a given method. The repetability standard deviation with the general precision of the test method according EN 12697-1 for asphalt mixes with unmodified binder are:

- Experiment 1, according EN 12697-1, clause 8.1: r = 0.3 %, s_r = 0.11 % by mass.
- Experiment 2, according EN 12697-1, clause 8.2: for a asphalt mix with D ≤ 11 mm: ; reproducibility r = 0.28 %, s_r = 0,10 % by mass.
- Experiment 3, according EN 12697-1, clause 8.3: r = 0.23 %, s_r = 0.08 % by mass.

For a given laboratory, a given solvent and a given method, deviations of the mean value (determined from two tests) were in all cases (except one) smaller than the values given in EN 12697-1 for the new SMA8 whereas for both RA mixes the deviations are more often larger than the ones given in this standard.

Mixture				SMA8			PRA			RA(V1)		
Laboratory	**Separation method**	**Solvent**	**Calculation**	**Results**	**Mean of the results**	**Average per solvent**	**Results**	**Mean of the results**	**Average per solvent**	**Results**	**Mean of the results**	**Average per solvent**
BRRC (1)	**Automatic extraction and centrifuge**	**TCE**	**D**	6.87	6.85±0.02	6.86±0.09	3.48	3.49±0.01	3.67±0.25	--	--	6.65±0.15
			D	6.83			3.50			--		
TUBS (1)			**D**	6.78	6.77±0.01		4.11	3.88±0.24		--	--	
			Rt	6.76			3.64			--		
ZAG			**D**	6.90	6.95±0.05		3.50	3.65±0.15		6.50	6.65±0.15	
			D	7.00			3.80			6.80		
IBDIM	**CFC**	**PCE**	**D**	7.00	6.95±0.05	6.91±0.09	3.40	3.55±0.15	3.82±0.30	6.40	6.50±0.10	6.25±0.35
			D	6.90			3.70			6.60		
IFSTTAR			**Rm**	--	--		3.44	3.60±0.16		6.47	6.47	
			Rm	--			3.75			--		
LR-Aix (1)			**D**	6.83	6.83		4.10	4.17±0.07		--	--	
			D	--			4.23			--		
LR-Aix (2)	**BCK type 2**		**Rm**	--	--		3.95	3.96±0.01		5.97	5.88±0.09	
			Rm	--			3.96			5.79		
BRRC (3)	**CFC**	**DCM**	**D**	6.86	6.83±0.04	6.83±0.04	3.76	3.77±0.01	3.77±0.01	6.68	6.67±0.02	6.67±0.02
			D	6.79			3.78			6.65		
BRRC (2)	**CFC**	**Tol**	**D**	6.85	6.84±0.02	6.81±0.08	3.70	3.76±0.06	3.86±0.13	6.09	6.06±0.03	6.42±0.34
			D	6.82			3.82			6.03		
TUBS (2)			**D**	6.87	6.79±0.09		4.01	3.96±0.06		6.69	6.49±0.21	
			Rt	6.70			3.90			6.28		
TUBS (2bis)			**D**	--	--		--	---		6.89	6.70±0.19	
			Rt	--			--			6.51		
Overall Mean				**6.85±0.80**			**3.78±0.24**			**6.42±0.33**		

Extraction method:

- Auto. extr. & centrifuge (orange)
- Hot extractor (green)
- Cold mix dissolution (cyan)
- Bottle rotation machine (yellow)

Separation method:

- CFC — Continuous flow centrifuge
- BCK type 2 — Bucket centrifuge type 2

Calculation of the binder content:

- D — Difference method
- Rm — Recovery method from a portion (mass calculation)
- Rt — Total recovery method

Table 2 : Classification of the different results according to the methods and the solvent[4]

[4] Number in brackets refers to table 1.

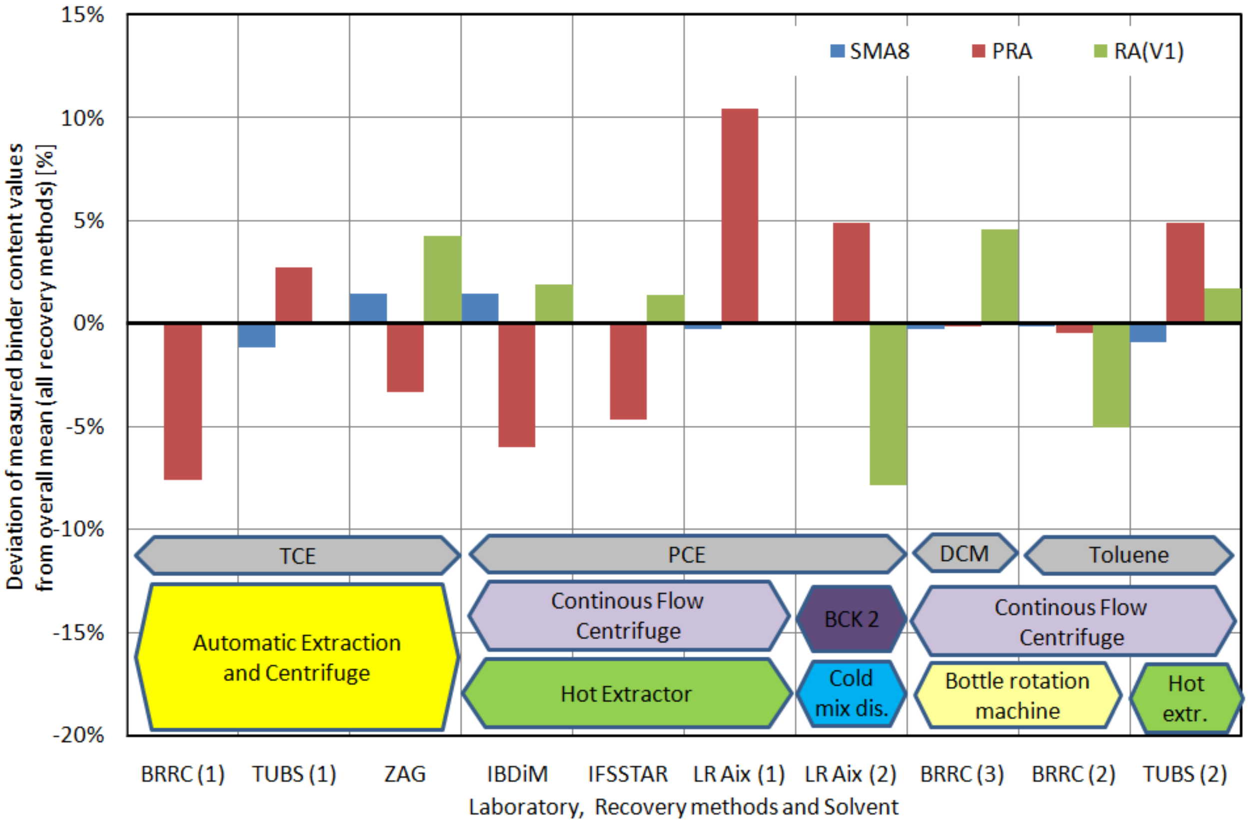

Figure 1 : Deviation of measured binder content from the mean binder content

3.1.1 Impact of the nature of binders

Apparently, the testing method and the solvent have no impact on a fresh asphalt mixture with modified bitumen and including a low content of RA. Problems appear for RA with Polymer modified Bitumen. More scattered results are obtained for RA with a physical SBS modified bitumen ("PRA"), but the largest span is obtained for RA with chemically linked SBS modified bitumen ("RA(V1)"). Due to their huge aging state, the binder from RA with PmB is more difficult to solubilise by solvents than from fresh asphalt (higher content of asphaltenes and of polar functions and hardening of the binder). Consequently their recovery is more difficult than for fresh asphalt with PmB, and the choice of the testing method and of the solvent seems linked with the type of binder (physically bond or cross-linked PmB).

3.1.2 Impact of the extraction method in the case of Reclaimed Asphalt

For a given solvent, it appears that the used testing method for extraction does not lead to the same results. For instance, when perchlorethylene is used (e.g. LR Aix (2) and IFSTTAR or IBDiM), very different results are found on both reclaimed asphalts, depending on the extraction method (cold or hot dissolution). Also for a given method and a given solvent (for example the automatic centrifuge with trichloroethylene), the standard deviation on the average of all the results is quite large. That can be explained by the number of washes which can be different between different labs and which is really important to fully remove the bitumen from the mixture.

3.1.3 Impact of the used solvent for a given test method in the case of Reclaimed Asphalt

For a given method, it appears that the used solvent can lead to different results depending on the type of PmB.
Table 2 shows clearly that different binder contents are found for the Reclaimed Asphalt with chemically linked SBS modified bitumen ("RA(V1)") depending on the solvent used (for a same test method) (e.g. BRRC with Toluene and Dichloromethane). This is however not the case for RA with a physical SBS modified bitumen ("PRA"). This could be

due to solubility problems of chemically linked SBS modified bitumen linked to the nature and temperature of the solvent.

3.2 Consistency of recovered binders

In one laboratory, penetration and Ring and Ball temperature $T_{R\&B}$ were measured on binder samples recovered by the various methods and using various solvents. The results of these tests are summerised in table 3.

Lab / Sample	**Solvent**	**Extraction method**	**SMA8**		**PRA**		**RA(V1)**	
			Pen [1/10mm]	**$T_{R\&B}$ [°C]**	**Pen [1/10mm]**	**$T_{R\&B}$ [°C]**	**Pen [1/10mm]**	**$T_{R\&B}$ [°C]**
TUBS (1)	TCE	Automatic extraction and centrifuge (hot dissolution)	21	72.0		73.0	10	80.0
ZAG			14	73.0	10	74.1	9	83.8
IBDIM	PCE	Hot extractor	18	70.6		72.2	10	82.2
IFSSTAR			21	72.9		73.4	12	80.5
LR-Aix (3)	DCM	Cold dissolution	20	70.2	12	73.2	12	85.7
BRRC (3)		Bottle rotation machine (cold dissolution)	21	69.7		69.6	9	
BRRC (2)	Tol		22	70.3		73.1	13	80.4
TUBS (2)		Hot extractor	20	70.9		72.8	12	
Mean:			**19.6**	**71.2**	**11**	**72.7**	**10.9**	**82.5**
St. Dev.			**2.6**	**1.3**	**1.4**	**1.4**	**1.6**	**2.3**

Table 3 : Binder properties measured on recovered binders

The total mean values of the binder properties are:

- SMA8: penetration = 19.6 1/10 mm and $T_{R\&B}$ = 71.2 °C
- RA(PA): penetration = 11.0 1/10 mm and $T_{R\&B}$ = 72.7 °C
- RA(V1): penetration = 10.9 1/10 mm and $T_{R\&B}$ = 82.5 °C

The relative deviation of the measured values from the total mean are shown in figure 2 (for penetration) and figure 3 (for $T_{R\&B}$).

For the penetration test results, the relative deviations range between -30 % and +20 % of the total mean value with similar scattering for all three samples. The use of TCE in automatic extraction and centrifuge seems to result in lower penetration values compared to other extraction and recovery methods, whereas the extraction with Toluene and DCM results in comparatively high penetration values. For softening point Ring and Ball $T_{R\&B}$ the test results scatter up to ± 3 %. The general slight tendencies to high viscosity indicators (high $T_{R\&B}$) for the automatic extraction with TCE as well as lower viscosity for Toluene hot extraction as already indicated by the penetration test results are approved. However all these statements are to be taken with care as the results obtained for the different solvents are in this case often obtained by different laboratories.

Despite the fact that the results on the binder samples recovered by various methods in 6 laboratories show scattering higher than the repeatability and reproducibility limit of the binder test method, no common trends can be indicated according to single extraction and recovery methods. Especially the results show that the distillation temperature applied for binder recovery which is lowest for DCM and highest for PCE doesn’t effect the conventional binder properties.

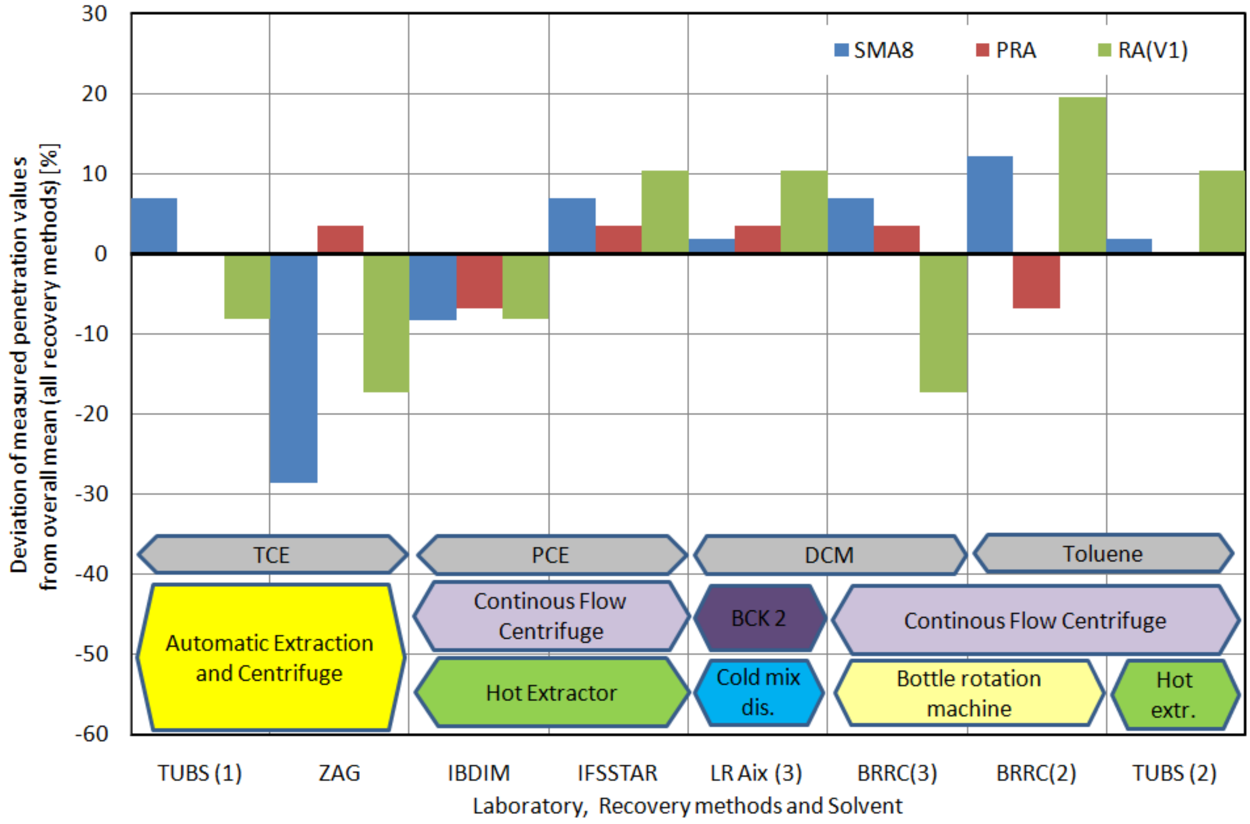

Figure 2 : Deviation of measured penetration values from the overall mean

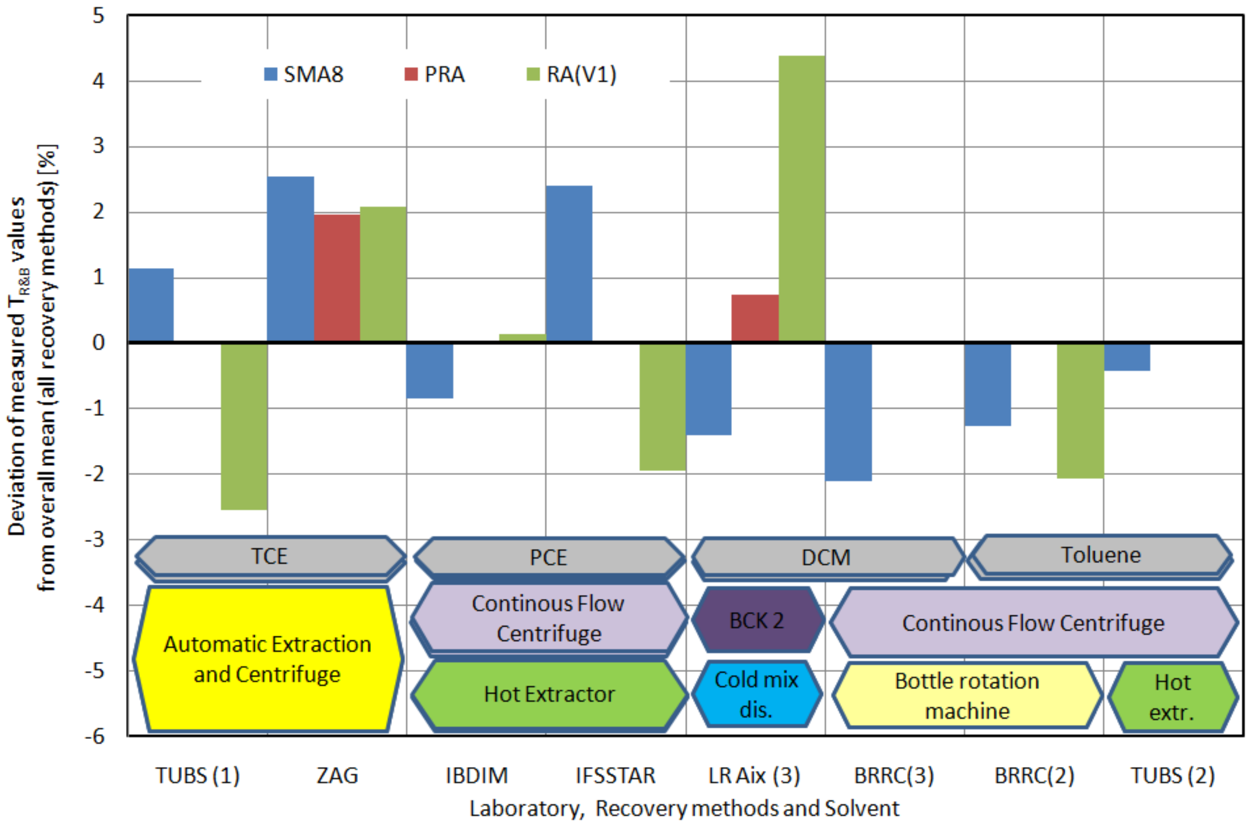

Figure 3 : Deviation of measured $T_{R\&B}$ values from the overall mean

3.3 Oxidation degree of recovered binders

The aging level of the binder in the reclaimed asphalt is often the predominant parameter determining the possibility to reuse the material in new hot asphalt mixes or to recycle it with less added-value as unbound material in the road base. One way to approach this aging level is to evaluate the oxydation state of the recovered binder by Fourier Transform Spectrometry. The global results are presented at the table 4.

Peak area of chemical functions	**Carbonyl (A_{1700})**			**Sulfoxide (A_{1030})**		
	SMA8	PRA	RA(V1)	SMA8	PRA	RA(V1)
Mean (m)	14.8	21.5	21.8	6.4	7.5	6.9
Standard deviation (sd)	0.6	0.4	0.7	0.3	0.2	0.5
Variation coefficent (sd/m)	4.1%	2.0%	3.4%	4.5%	3.2%	7.8%
Maximum	15.3	22.1	22.6	6.9	7.7	7.6
Minimum	13.9	21.0	20.8	6.0	7.0	6.2
Span	1.7	1.1	1.8	0.9	0.7	1.4

Table 4 : Main results of oxidation degree of recovered binder for each material

Table 4 shows that the two RA (PRA and RA(V1)) have more important carbonyl and sulfoxides functions than the new SMA with 15% of RA. This is consitent with the ageing occurring during the service life of RA (Choquet, 1991).

The comparison of the variation of the three different peaks area values with the penetration values shows that the penetration decreases in the same proportion (45% lower for both RA than for SMA) as the carbonyl area increases (45-47% higher for both RA). The softening point does not seem correlate to the oxidation state of the bitumen.

4. CONCLUSIONS

This experimental campaign allowed to evaluate the adequacy of the methods and solvents proposed in the EN 12697-1 for the determination of the soluble binder content of different types of asphalt mixtures containing PmB (new mix and reclaimed asphalts). In this standard a large range of methods and solvents are described and can be chosen to carry out the test.

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 combinaisons 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.

The next step that will be undertaken is to select suitable experimental parameters (solvent, methods and temperature of dissolution) of extraction and recovery methods in order to obtain the “true” binder content and the “true” properties of the PmB from RA. This study will be performed on RA of controlled quality (production in laboratory of an artificial RA with exactly known binder content and binder properties) .

It would be also interesting to continue this study by considering only one method with different solvents and only one solvent with different methods, to assess the impact of these parameters on the test. As the binder content of reclaimed asphalt is important to determine the amount of reclaimed asphalt re-usable in a new pavement, there is a big interest in this topic. This can lead to improve EN 12697-1 in the specific case of binder content determination of reclaimed asphalt containing PmB.

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