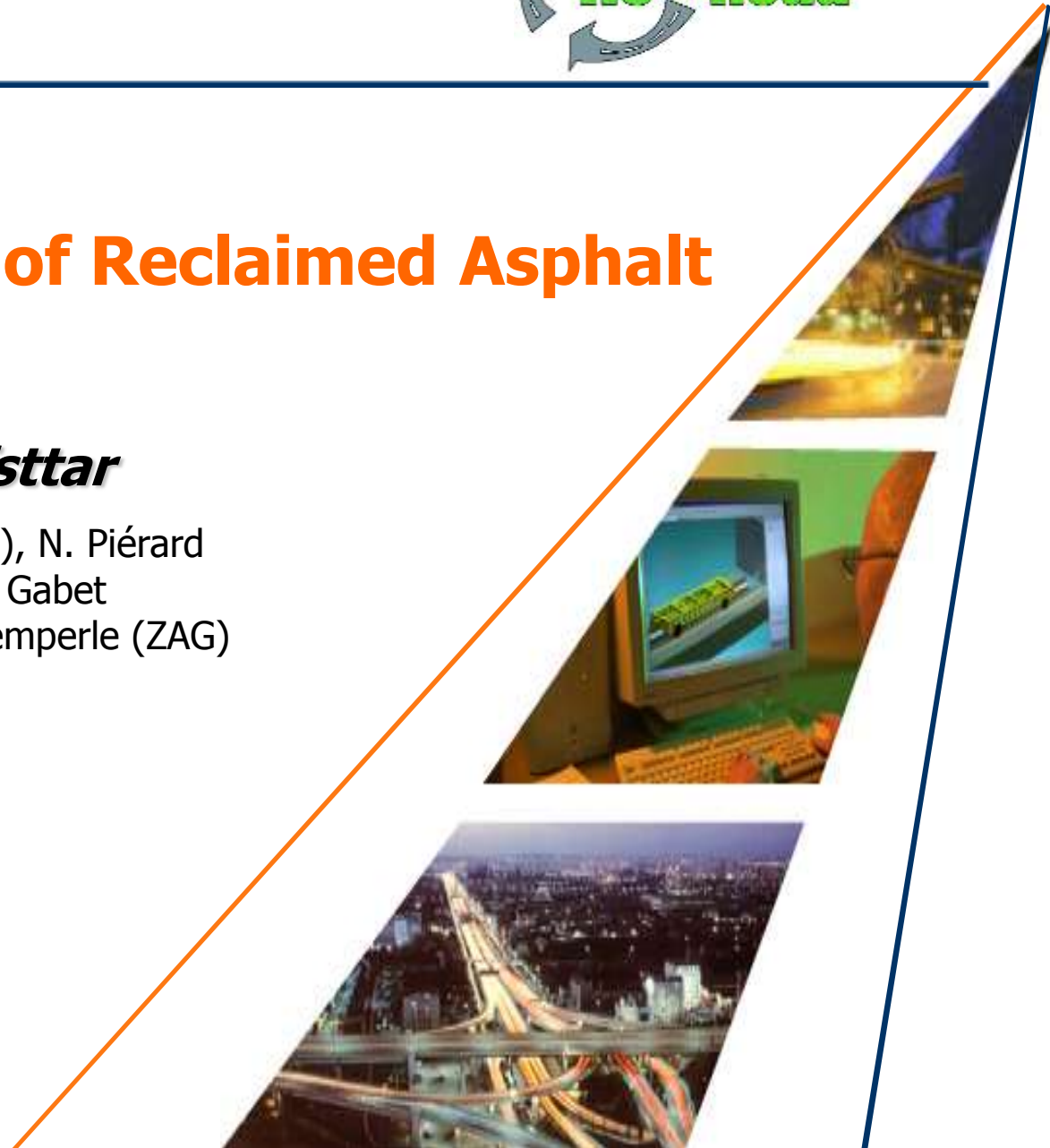


Characterization of Reclaimed Asphalt

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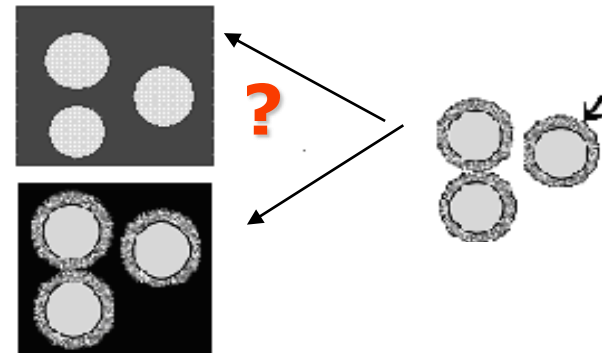
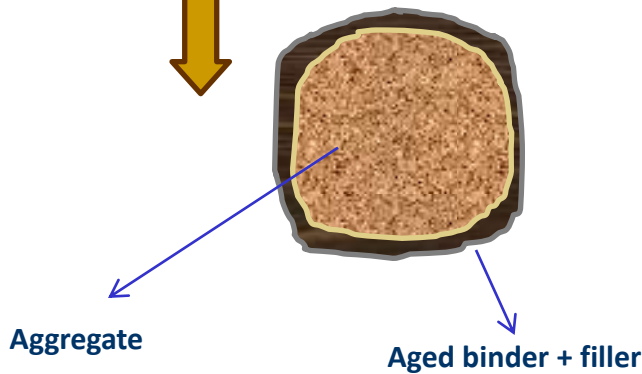


Potential to recycle Reclaimed Asphalts



- intended use of the material
 - content of RA in new mix
- characteristics of RA
 - binder content in the RA
 - nature and ageing of binder of RA
 - pollution level

(* RA = Reclaimed Asphalt)



AGEING DEGREE OF RA BINDER :

Consistency of residual binder

Capacity of the aged binder to act as a binder

IDENTIFICATION OF NATURE OF RA BINDER :

Compatibility with the added binder

Possibility to restore some characteristics

The specific objectives:

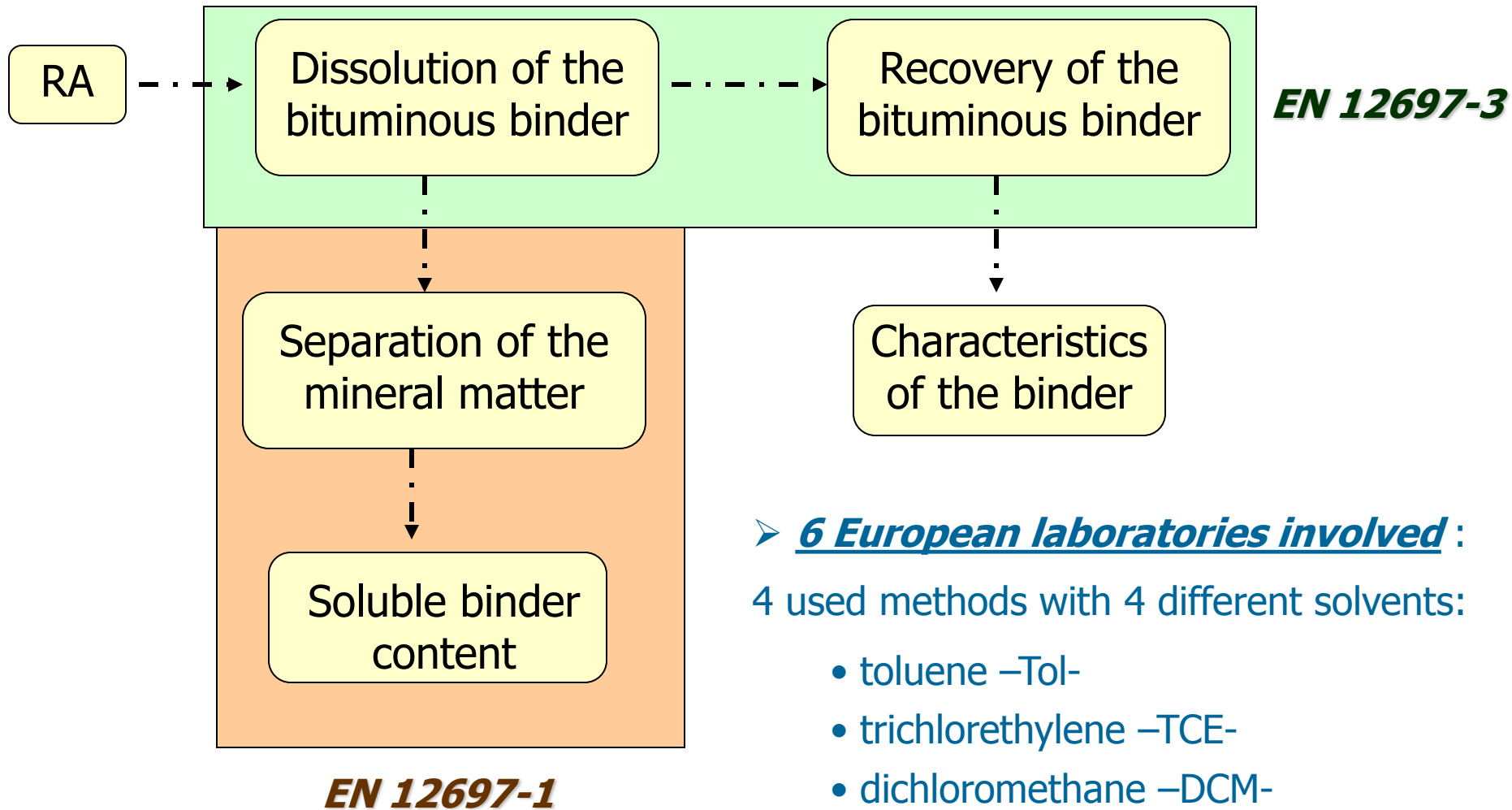
- develop suitable extraction and recovery methods for RA with PmB
- propose testing methodologies for a better characterisation of the bitumen part of a RA
- specify relevant indicators for estimating the recycling potential of a RA

(* PmB = Polymer modified Bitumen)



Methods EN 12697-1: soluble binder content

Methods EN 12697-3 : recovery of the binder



➤ 6 European laboratories involved :

4 used methods with 4 different solvents:

- toluene –Tol-
- trichlorethylene –TCE-
- dichloromethane –DCM-
- perchloroethylene –PCE-

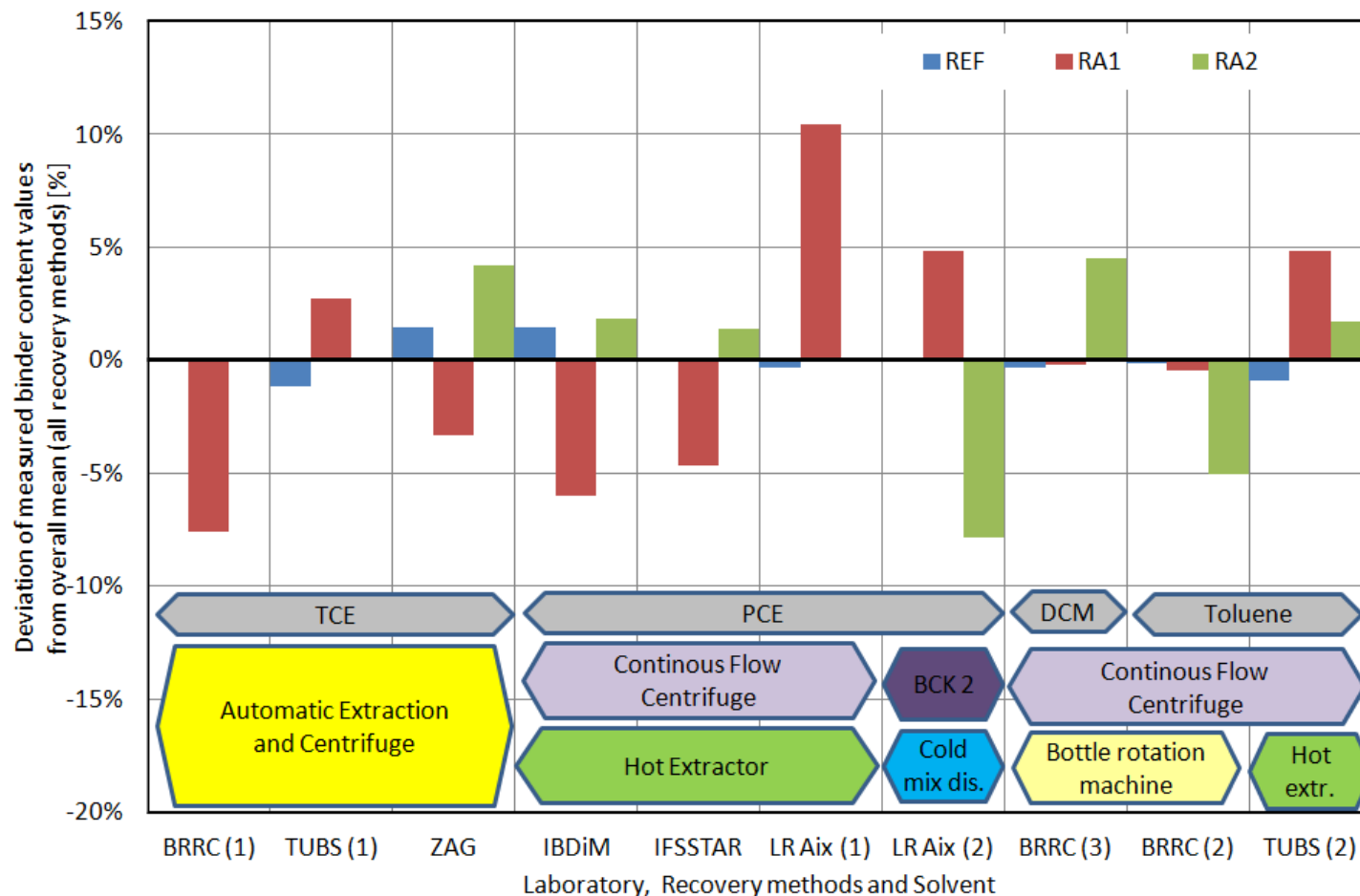
RA taken on site	RA produced in lab	Reference sample
<p>RA of age 10 years and sampled directly from milling :</p> <ul style="list-style-type: none">•“RA1”: the binder used is a physical SBS modified bitumen•“RA2”: the binder used is a chemically linked SBS modified bitumen	<p>RA produced in laboratory according to the RILEM protocol (*) :</p> <ul style="list-style-type: none">•“RA3” : the binder used is a pure binder 35/50 -> binder content = 5,40%•“RA4” : the binder used is a SBS modified bitumen -> binder content = 7,00%	<p>Stone Mastic Asphalt (“REF”) including a PmB and 15% of RA (also with PmB)</p>

() : loose mix aged during 4 hours at 135°C + 9 days at 85°C*

Results of binder content : RA taken on site vs Reference material



➤ Deviation of measured binder content values from overall mean :



Results of binder content : RA taken on site vs Reference material



➤ Statistical analysis of measured binder content values :

	RA1	RA2	REF
Mean	3.78	6.42	6.85
Standard Deviation	0.24	0.33	0.08
Variation coefficient	6.33	5.08	1.18
Minimum	3.40	5.79	6.70
Maximum	4.23	6.89	7.00
Span	0.83	1.10	0.30

➤ High scattering of results for the two RA taken on site (compared to reference mix)



Sampling problem?

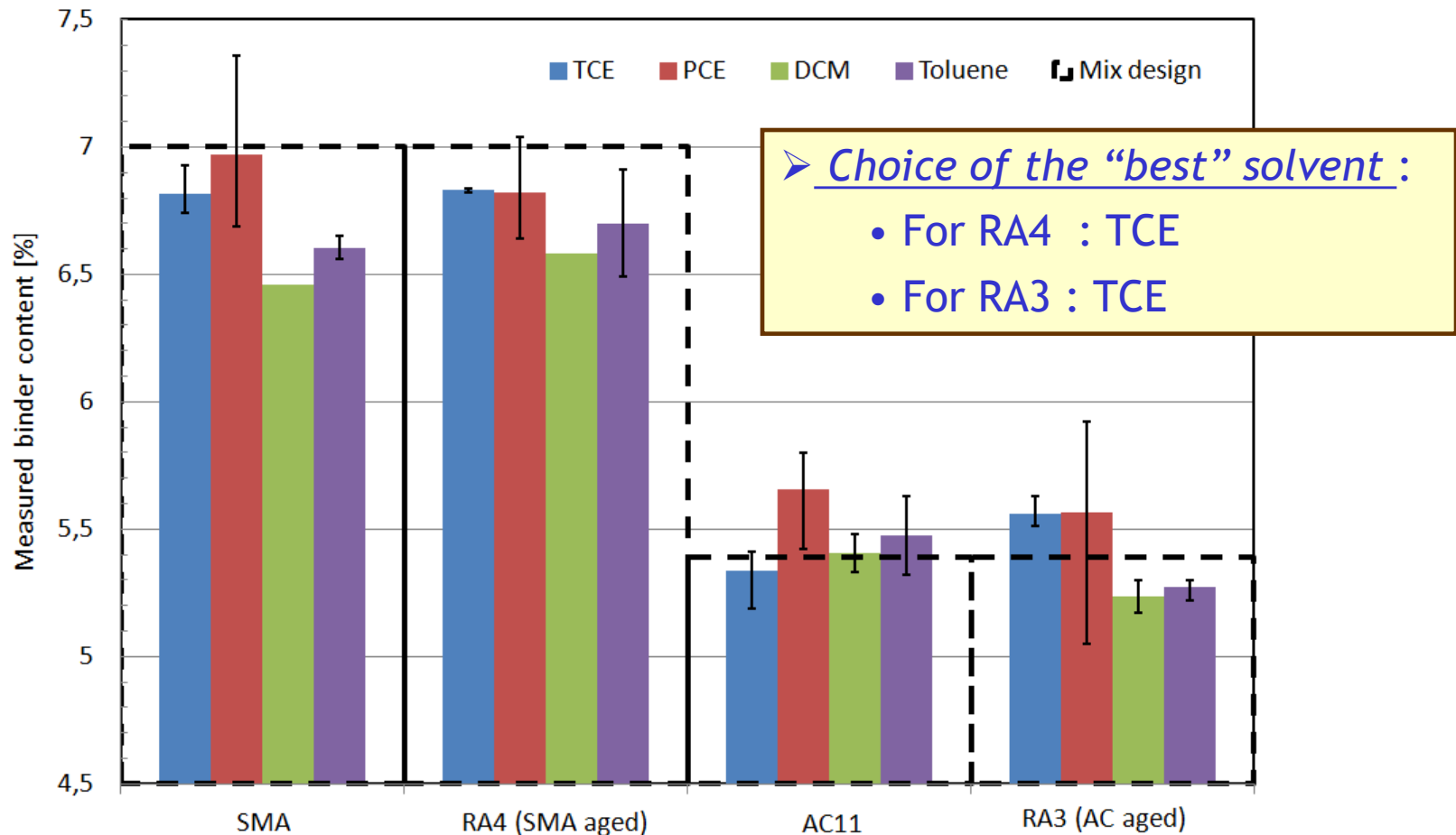
Impact of experimental parameters?



Results of binder content : RA produced in laboratory



➤ Deviation of measured binder content values from « true » value :



Results of binder content : RA produced in laboratory



➤ Statistical analysis of measured binder content values :

	AC11	RA3 (AC11 aged)	SMA	RA4 (SMA aged)
"True value" (mix design)	5.40	5.40	7.00	7.00
Mean	5.49	5.48	6.78	6.77
Standard deviation	0.17	0.24	0.26	0.17
Minimum	5.19	5.05	6.46	6.49
Maximum	5.80	5.92	7.36	7.04
Span	0.61	0.87	0.90	0.55
repeatability (r)	0.28	0.36		
Reproductibility (R)	0.47	0.68	0.73	0.48
Uncertainty U (k=2)	0.20	0.34		



Same standard deviation than for RA taken on site : impact of methods and solvents on the measured soluble binder content

➤ Deviation from « true value »: 0,08 for RA3 and 0,23 for RA4

Analysis of results of binder content : RA taken on site and Reference material



Mixture		REF			RA1			RA2		
Laboratory	Solvent	results	moy	std	results	moy	std	resultss	moy	std
BRRC (1)	TCE	6,87	6,86	0,09	3,48	3,67	0,25		6,65	0,21
		6,83			3,50					
TUBS (1)		6,78			4,11					
		6,76			3,64					
ZAG		6,90		3,50		6,50				
		7,00		3,80		6,80				
IBDIM	PCE	7,00	6,91	0,09	3,40	3,82	0,30	6,40	6,25	0,35
		6,90			3,70			6,60		
IFSTTAR					3,44			6,47		
					3,75					
LR Aix (1)		6,83			4,10					
					4,23					
LR Aix (2)				3,95		5,97				
				3,96		5,79				
BRRC(3)	DCM	6,86	6,83	0,05	3,76	3,77	0,01	6,68	6,67	0,02
		6,79			3,78			6,65		
BRRC(2)	Toluene	6,85	6,81	0,08	3,70	3,86	0,13	6,09	6,42	0,34
		6,82			3,82			6,03		
TUBS (2)		6,87			4,01			6,69		
		6,70			3,90			6,28		
TUBS (2 bis)							6,89			
							6,51			

Impact of method with PCE

Influence of solvent with bottle rotation machine

Automatic extraction and centrifuge apparatus

Hot extractor
Cold mix dissolution of bitumen by agitation

Results of binder content : Conclusions



NORM EN 12697-1: SOLUBLE BINDER CONTENT
« CHOICE OF METHODS AND SOLVENTS »

**No effect on reference mix
including PmB and 15% of RA
(also with PmB)**

**Important impact on RA
with PmB**

Linked to the nature of PmB

**Solvent choice for the
chemically linked SBS
modified bitumen**

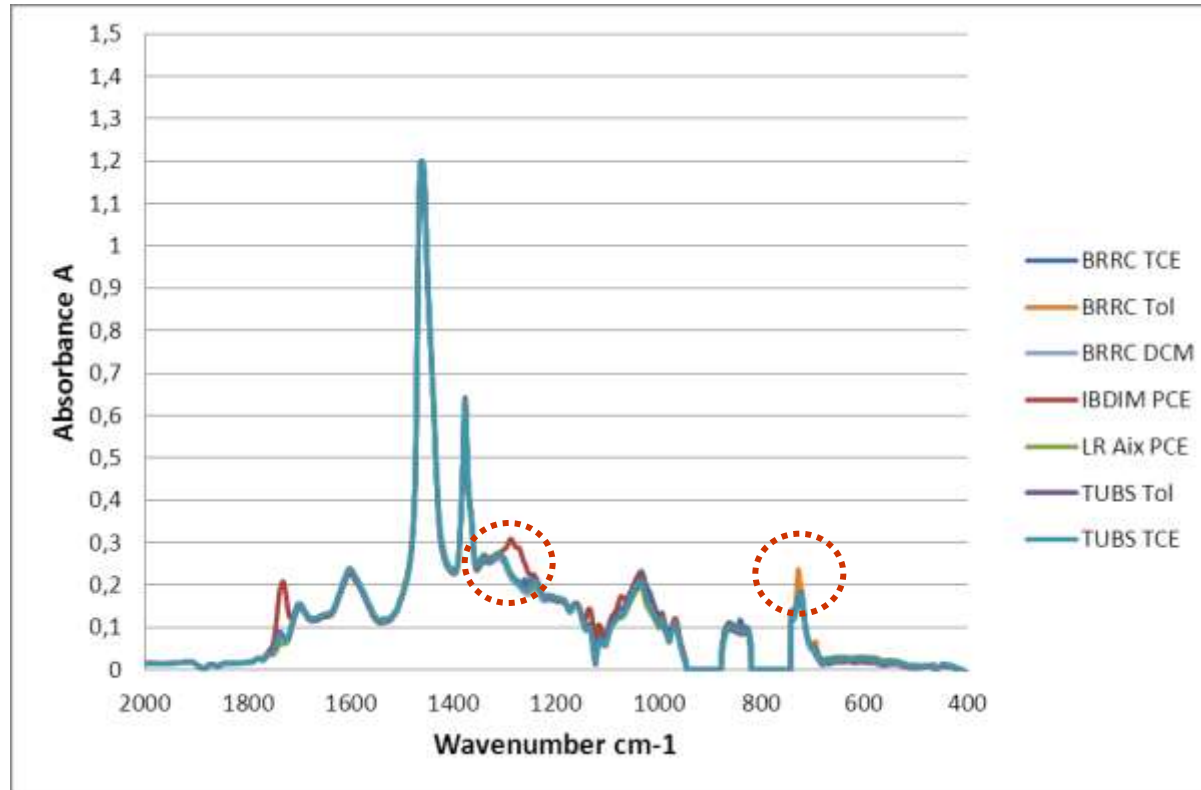
Solubility problems?



Characteristics after binder recovery



➤ Assessment of the recovery quality by infrared spectroscopy :



Quick identification of recovered binders contaminated by solvent

- Structural state of material at the end of its life:
 - *measurement of the consistency of binder* (penetration at 25°C - EN 1426 – and softening point - EN 1427 -);
 - *assessment of quality, nature and ageing level of binder :*
 - Oxydation degree and polymer content by infrared spectroscopy
 - Force ductility at 25 °C (EN 13589) and elastic recovery
 - Complex modulus at 25 and 52°C and 1,6 et 10 Hz
- Reproducibility of tests:
 - one characteristic measured on all recovered binders by a same laboratory
 - homogeneization of binders before analysis at a same temperature (180°C)

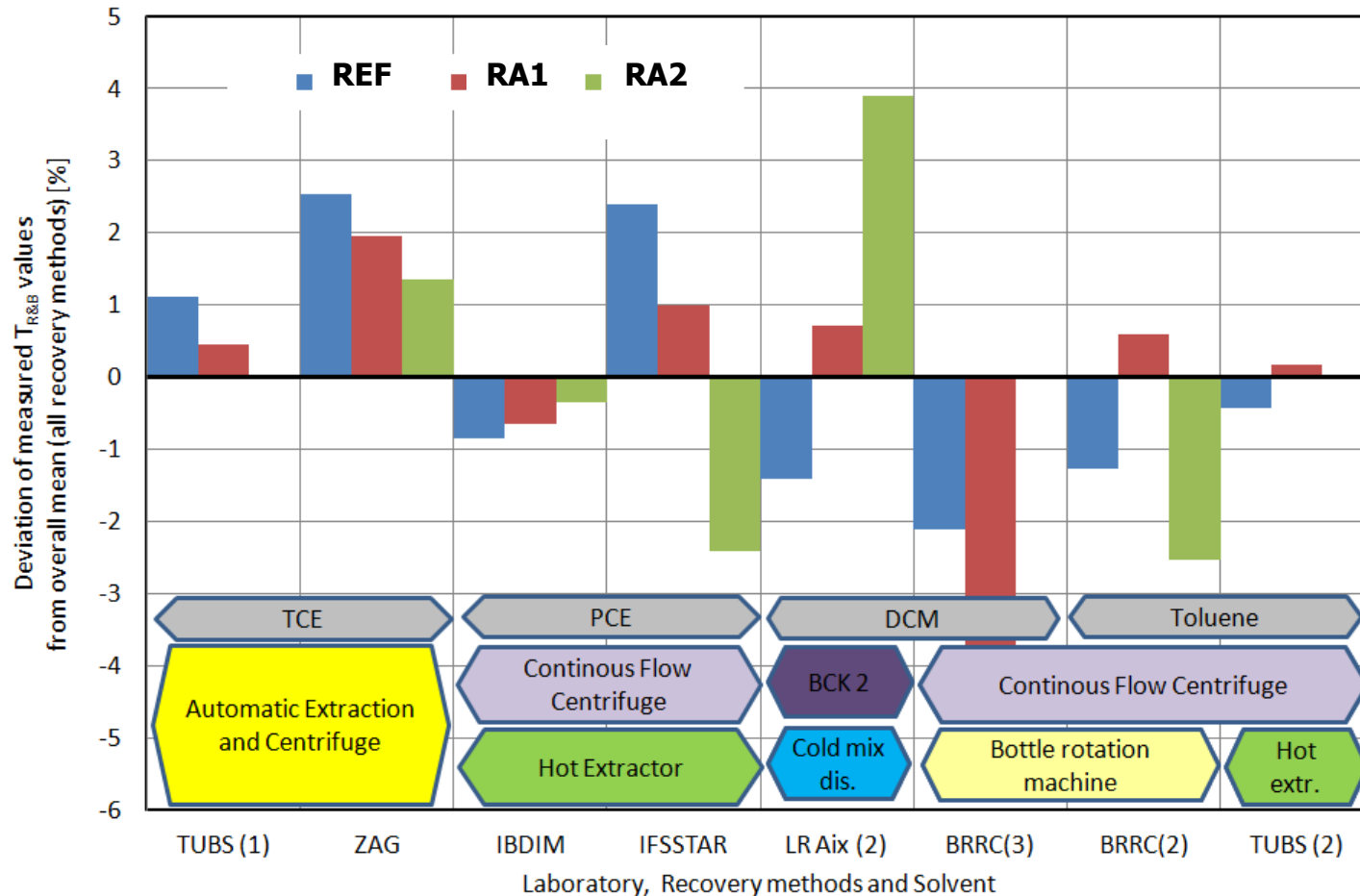


Characteristics after binder recovery :

Consistency : RA taken on site vs Reference



➤ Deviation of measured Ring & Ball temperature values from overall mean :



Characteristics after binder recovery :

Consistency : RA taken on site vs Reference



- Statistical analysis of measured Ring&Ball temperature values :

$T_{R\&B} (^{\circ}C)$	RA1	RA2	REF
Mean	72,6	82,5	71,2
Standard deviation	1,4	2,2	1,3
Variation Coefficient	1,9%	2,7%	1,8%
Minimum	74,1	85,7	73,0
Maximum	69,6	80,4	69,7
Span	4,5	5,3	3,3

- High variability of consistency binders of RA taken on site



Sampling problem?

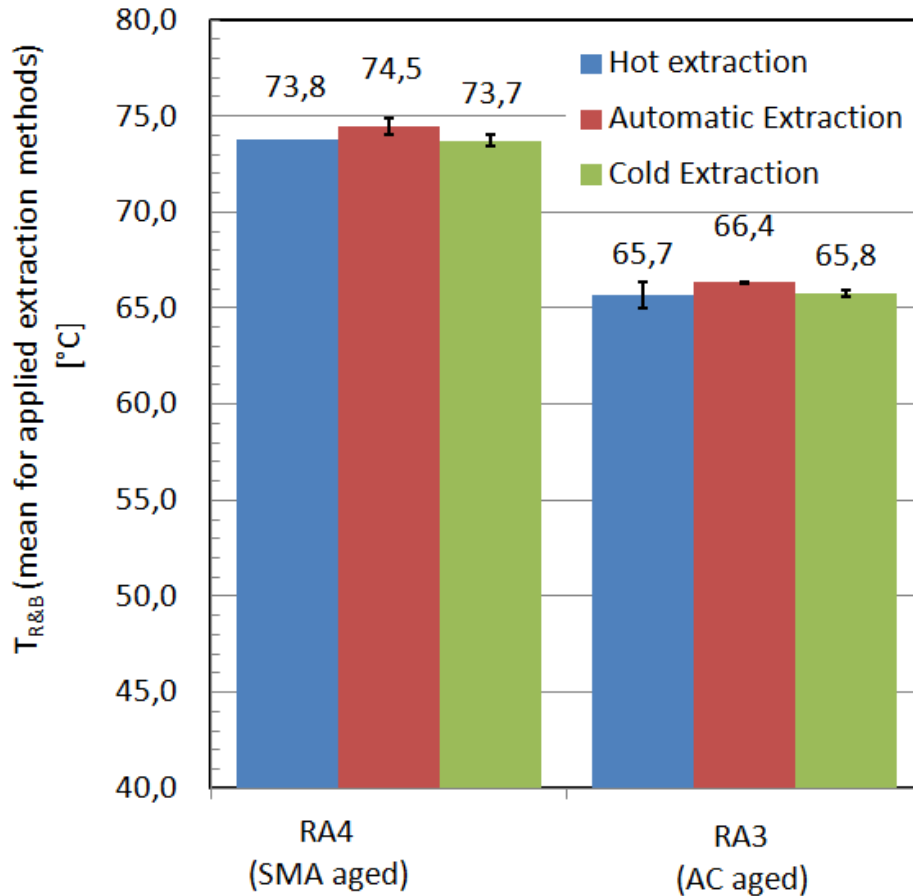
Influence of experimental parameters on binder's recovery?



Characteristics after binder recovery : Consistency : RA produced in laboratory



- Ring & Ball temperature versus applied extraction methods :



➤ *No significant effect of temperature of extraction method*

Characteristics after binder recovery : Consistency : RA produced in laboratory



➤ Statistical analysis of measured Ring & Ball temperature values :

$T_{R\&B}$ (°C)	AC11	RA3 (AC11 aged)	SMA	SMA aged	RA4 (SMA aged) (without recovered binders contaminated)
Mean	56,2	65,9	63,2	72,7	74,0
Standard deviation	0,6	0,5	1,6	2,3	0,6
Variation coefficient	1,1 %	0,8 %	2,5 %	3,2%	0,7%
Minimum	55,6	65,0	61,2	69,2	73,4
Maximum	57,6	66,4	66,0	74,9	74,9
Span	2,0	1,4	4,8	5,7	1,5



- Low impact of methods and solvents on the consistency of binders
- Increase of the softening point around 10°C
- Span of RA3 and RA4 below the reproducibility value of the EN 1427 (R=3.5°C)

Characteristics after binder recovery : Oxidation : RA taken on site vs Reference



➤ Statistical analysis of measured carbonyl content values :

<i>Carbonyl functions</i>	RA1	RA2	REF
Mean	21,5	21,8	14,8
Standard deviation	0,4	0,7	0,6
Variation coefficient	2,0%	3,4%	4,1%
Minimum	21,0	20,8	13,9
Maximum	22,1	22,6	15,3
Span	1,1	1,8	1,7

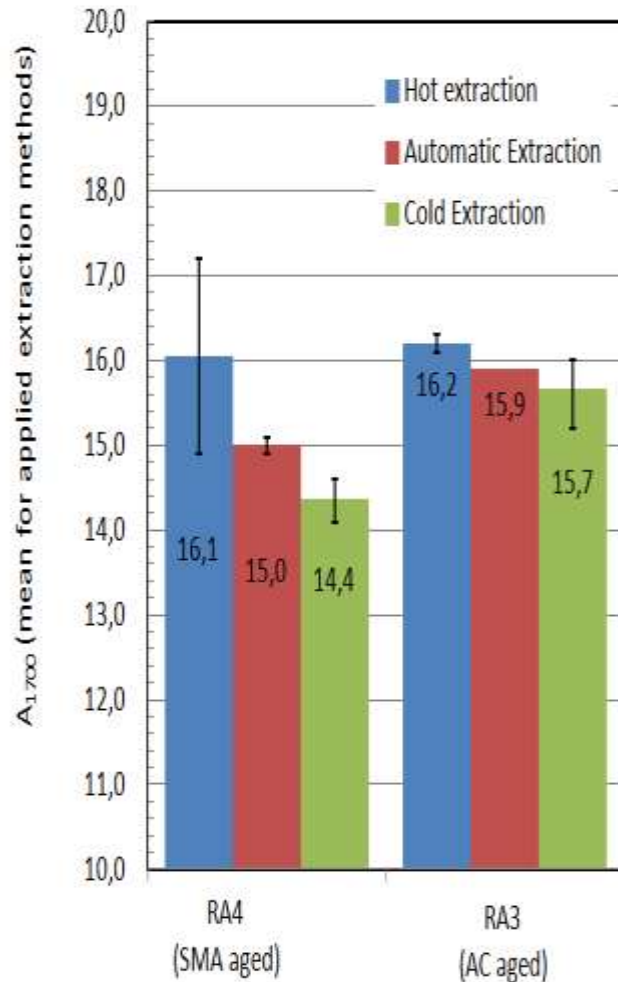


- Low impact of methods and solvents on the oxidation of binders
- High content of carbonyl of RA taken on site compared to the reference material

Characteristics after binder recovery : Oxidation : RA produced in laboratory



➤ Carbonyl content versus applied extraction methods :



➤ *Lower carbonyl content for cold extraction*

➤ *less oxidative ageing*

Characteristics after binder recovery : Oxidation : RA produced in laboratory



➤ Statistical analysis of measured carbonyl content values :

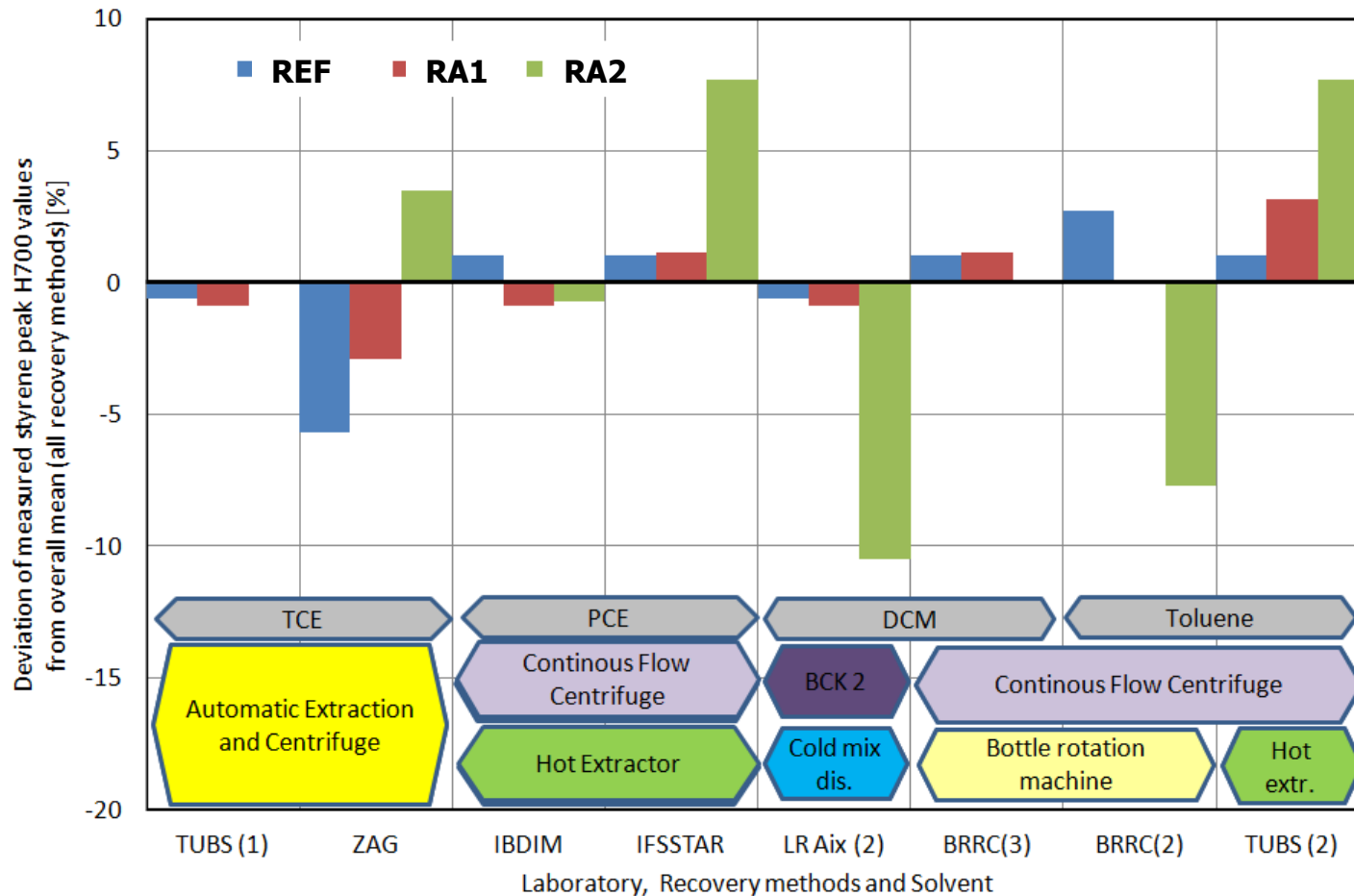
Carbonyl functions	AC11	RA3 (AC11 aged)	SMA	SMA aged	RA4 (SMA aged) (without recovered binders contaminated)
Mean	11,7	15,9	10,9	15,0	14,7
Standard deviation	0,5	0,4	0,6	1,0	0,4
Variation coefficient	4,6 %	2,4 %	5,6%	6,8%	2,7%
Minimum	11,1	15,2	10,1	14,1	14,1
Maximum	12,6	16,3	11,8	17,2	15,1
Span	1,5	1,1	1,7	3,1	1,0

- Low impact of methods and solvents on the oxidation of binders
- Lower content of carbonyl of RA produced in lab compared to the ones taken on site

Characteristics after binder recovery : Polymer content : RA taken on site vs Reference



➤ Deviation of measured styrene peak height from overall mean :



Characteristics after binder recovery : Polymer content : RA taken on site vs Reference



- Statistical analysis of measured styrene peak height :

<i>Styrene presence</i>	RA1	RA2	REF
Mean	0,049	0,072	0,059
Standard deviation	0,001	0,006	0,002
Variation coefficient	2,0%	7,7%	2,5%
Minimum	0,048	0,064	0,056
Maximum	0,051	0,077	0,061
Span	0,003	0,013	0,005

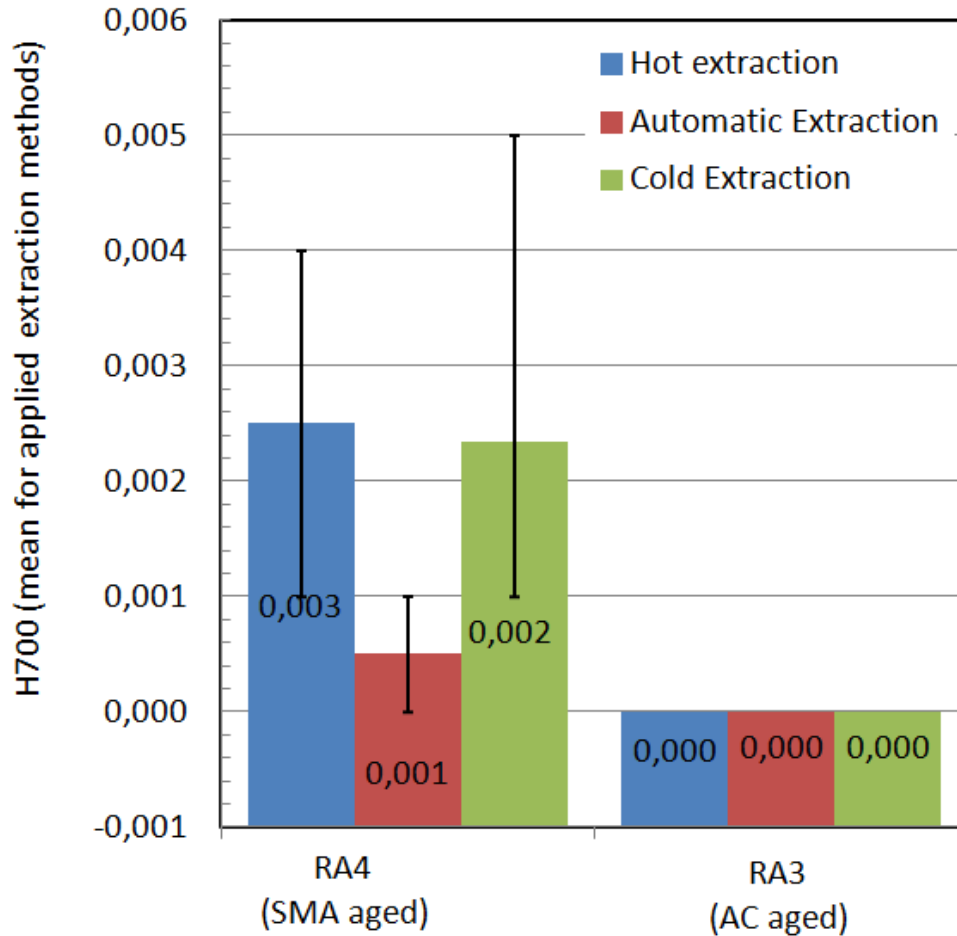


- High impact of methods and solvents on the polymer content for RA with a chemically linked SBS modified bitumen

Characteristics after binder recovery : Polymer content : RA produced in laboratory

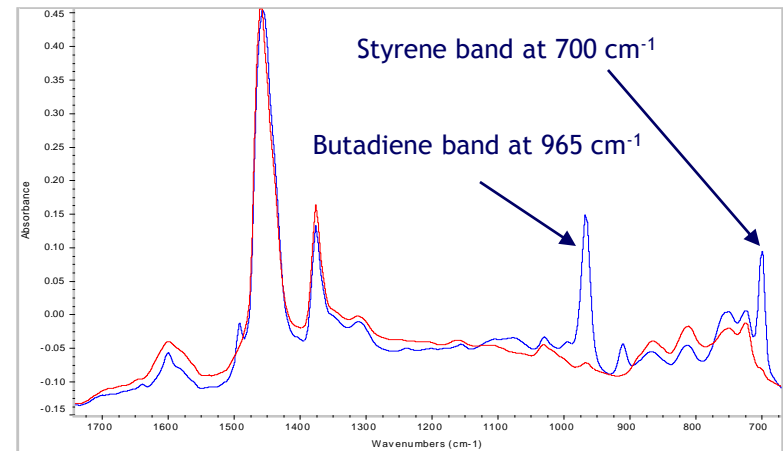


➤ Styrene peak height versus applied extraction methods :



➤ No significant height of styrene peak for RA4 (originally supposed to be a SBS bitumen)

Infrared spectra of SBS modified bitumen



Summary of measured characteristics : Choice of indicators of binder's end of life



Characteristics	RA3	RA4	REF	RA1	RA2
Softening point (°C)	65,9	74,0	71.2	72.6	82.5
Increase of softening point (°C)	<i>9,7</i>	<i>10,8</i>	/	/	/
Content of carbonyls	15,9	14,7	14,8	21,5	21,8
Increase of content of carbonyles	<i>4,2</i>	<i>3,8</i>	/	/	/
Content of sulfoxydes	6,8	6,5	6,4	7,5	6,9
Increase of content of sulfoxydes	<i>1,2</i>	<i>1,4</i>	/	/	/
G* at 25°C and 1.6 Hz	$1,20 \cdot 10^7$	$5,30 \cdot 10^6$	$7,53 \cdot 10^6$	$2,31 \cdot 10^7$	$2,33 \cdot 10^7$
Increase of G* at 25°C and 10 Hz	<i>$0,73 \cdot 10^7$</i>	<i>$3,34 \cdot 10^6$</i>	/	/	/
δ at 25°C and 1.6 Hz	42.6	40,3	44,7	36,7	40,0
Decrease of δ at 25°C and 1.6 Hz	<i>14,3</i>	<i>11,3</i>	/	/	/
G* at 52°C and 1.6 Hz	$1,50 \cdot 10^5$	$1,26 \cdot 10^5$	$1,53 \cdot 10^5$	$3,80 \cdot 10^5$	$3,96 \cdot 10^5$
Increase of G* at 25°C and 10 Hz	<i>$1,17 \cdot 10^5$</i>	<i>$0,93 \cdot 10^5$</i>	/	/	/
δ at 52°C and 1.6 Hz	69,0	55,9	61,9	61,3	56,7
Decrease of δ at 25°C and 1.6 Hz	<i>11,9</i>	<i>8,8</i>	/	/	/



- ⇒ Choice of the most relevant characteristics to assess the end of life of RA binder:
- **carbonyl content** (ageing : physico-chemical state of binder)
 - **complex modulus at 25 and 52 °C** (hardening : rheological state of material)



Input for the choice of adding binder

- ✓ Necessity of collecting data to define the threshold values of material's characteristics that are allowable for recycling
- ✓ Researches to undertake in the future :
 - Improvement of the characterization and technical assessment of RA with polymer modified bitumens
 - Study of the degradation process of polymers in asphalt layers in order to better understand how to restore the polymeric function

Thank you for listening!



More information:

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