iFRAE Delft 2021

Workshop

Changes in binder properties and the role of additives

July 12-15, 2021

Delft, the Netherlands

Editors: Sayeda Nahar (TNO)

Xueyan Liu (TU Delft)

Contributors:

Inge van Vilsteren, RWS

Frédéric Delfosse, Eurovia

Michael Elwardany, FHWA

Dave Mensching, FHWA

Lily Poulikakos, EMPA

Xueyan Liu, TU Delft

Laurent Porot, Kraton Polymer B.V.

Secretaries:

Shisong Ren, TU Delft

Peng Lin, TU Delft

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Profile

Changes in the refining industry have an impact on the bitumen market. These changes are taking place due to economic and regulatory reasons and have drawn considerable attention in terms of variation in the performance properties of the current bitumen. One aspect of this change is the unavailability of certain crude oil sources, which introduces a change in choice of crude oil source and often a mixture of crude oils from multiple sources. Another aspect is the on-going transitions in the refineries. As a result, there are inconsistencies in the crude source and physico-chemical properties of bitumen in the market.

In order to have a better insight into current changes and mapping the potential sources of change in the binder market in relation to property or quality and consistency, the virtual workshop on ‘Changes in binder properties and the role of additives’ was held on July 15th 2021 as a cluster event of iFRAE Delft 2021 under the flagship on KPE-CEAB. This workshop aimed to have an interactive session to discuss the current trends and challenges in the asphalt binder market. Six distinguished speakers were invited who are eminent experts in the field and the workshop was closed with an expert discussion session. There are more than 100 participants originating from 14 different countries who joined this workshop. There were two sessions in the workshop. In the first session, topics related to the changes in the bitumen market and methods to manage the current challenges were discussed. In the follow-up session topics related to additives of the binder and trends in the asphalt recycling practices are discussed.

The Editors would like to thank Organizing Committee of the iFRAE Delft 2021 to provide this virtual conference platform and to give great support for this workshop. The editors would also like to take this opportunity to thank all the invited speakers and guests who participated in the fruitful discussion during the ‘Expert discussion’ of the workshop.

We hope the outcomes of this workshop will help us further understand the influence of change in the binder market in relation to property or quality, the consistency of bitumen binder, and its impact on asphalt performance.

Contents

1 Organizers and Speakers

Organizers:

Sayeda Nahar, TNO, Organizer/Chair

Xueyan Liu, TU Delft, Organizer/Chair/Speaker

Shisong Ren, TU Delft, Organizer

Peng Lin, TU Delft, Organizer

Speakers:

Inge van Vilsteren, RWS, Speaker

Frédéric Delfosse, Eurovia, Speaker

Michael Elwardany, FHWA, Speaker

Dave Mensching, FHWA, Speaker

Lily Poulikakos, EMPA, Speaker

Xueyan Liu, TU Delft, Organizer/Chair/Speaker

Laurent Porot, Kraton Polymer B.V., Speaker

2 Workshop Program

12:30-12:35(GMT) 14:30-14:35(CEST)	Welcome Moderators: Dr. Sayeda Nahar, TNO, The Netherlands Dr. Xueyan Liu, Delft University of Technology
12:35-13:50 (GMT) 14:35-15:50 (CEST)	**Session 1: Changes in the bitumen market and methods to manage the current challenges**
	Dutch challenges due to the changes in the bitumen market: the research project 'Grip on bitumen' *Inge van Vilsteren, RWS*
	Bitumen quality variability and impacts on pavement materials, what is missing in current specifications *Frédéric Delfosse, Eurovia*
	A Tale of Two Deltas: Analysis approach, proposed limits, and validation work to address binder quality-related thermally induced surface damage. *Michael Elwardany, Dave Mensching, FHWA*
13:50-14:00 (GMT) 15:50-16:00 (CEST)	Break
14:00-15:15 (GMT) 16:00-17:15 (CEST)	**Session 2: Binder additives and trends in the asphalt recycling practices**
	Recycling practices and additives in asphalt: Current practices and the future trends *Lily Poulikakos, EMPA*
	Routes to durability and sustainability: Recycling of PmB containing RAP *Dr. Xueyan Liu, TU Delft*
	Designing a toolbox for bitumen to answer the need for tomorrow's pavement *Laurent Porot, Kraton Polymer B.V.*
15:15-15:30 (GMT) 17:15-17:30 (CEST)	**Expert discussion**

3 Workshop Presentations

3.1 Dutch challenges due to the changes in the bitumen market: the research project ‘Grip on bitumen’

Inge van Vilsteren

specialist construction materials, Rijkswaterstaat: Ministry of Infrastructure and the Environment, The Netherlands

Abstract:

More than 90% of the wearing course on the Dutch motorway consists of porous asphalt. Stone Mastic Asphalt and Noise reduction thin layers are the most common wearing course of the national roads, which are mostly expressways. At the regional access roads and ringways, dense asphalt concrete and Stone Mastic Asphalt are used.

The asphalt pavement on the Dutch roads are mainly constructed using penetration grade bitumen. Polymer modified bitumen is used in specific application of wearing courses, but this is not widely used. Nowadays, the Netherlands do not have a bitumen refinery within their borders. Knowing that the refineries changes there processes over the last couple of years, the perception was that the quality of bitumen was not that constant anymore. At the same time asphalt mixtures become increasingly complex and specialized and the roads economic life cycle are becoming increasingly important.

With this growing realization a new research project started; “Grip on Bitumen” (GoB). Within a working group consisting of 20 members from engineering companies, laboratory-facilities, road-constructors, asphalt-producers, bitumen suppliers, branch originations and normative institute, all knowledge was combined. This project team started in 2018 and the last deliverables are due is summer. At the iFRAE one member of Grip on Bitumen will take you through the current challenges in complying with the binder properties in the Dutch context. She will take you through the current findings, the knowledge provided out of parallel projects and the follow up after this.

About the speaker

Inge van Vilsteren is a specialist construction material within the section of Roads and Geo-engineering at Rijkswaterstaat, the Dutch highway authority. Within a team of 35 specialists the section Roads and Geo-engineering is the dedicated advisory-group for around 8000 colleagues. She is a specialist on construction materials in general and in asphalt concrete in particular. With around 90% Porous asphalt as the top-layer paving material on highways, this is a specific knowledge area.

Within the research program of the Section Roads and Geo-engineering, her research topics mostly relate to the development and implementation of innovative asphalt-mixes, with the focus on durability and sustainability. Within Rijkswaterstaat, Inge is one of the knowledge-group members of the internal Program for the Transition towards sustainable roads. She is a member of the National Working Group Asphalt (CROW) for the Dutch implementation of technical requirements for pavement materials. This is a fine combination with the membership of CEN/TC227 working groups and technical committees on the international level involved in developing EN-norms e.g. technical requirements for pavement materials.

Inge is a member of the Dutch mirror-group for CEN/TC336 Bitumen and bituminous binders. Her knowledge of bituminous binders is well used in the binder-project “Grip on bitumen” within a Dutch branch-wide project “Asphalt-Impulse”.

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5th International Symposium on Frontiers of Road and Airport Engineering

Workshop: Changes in binder properties and the role of additives

Dutch challenges due to changes in the bitumen market: the research project ‘Grip on bitumen’

Inge van Vilsteren, Rijkswaterstaat Ministry of Infrastructure and the Environment

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Dutch challenges due to changes in the bitumen market: the research project ‘Grip on bitumen’

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Content

1. Starting point
   – cause for questioning/ grounds for suspicion
2. First collection state of the art
3. Start of Grip on Bitumen
4. Project Leerruimte (Room to learn)
5. Continuation

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Questioning/ suspicion

- EN TC336 and TC227
  – Global bitumen supply/ demand balance is fundamentally changing
  – Refinery closures and investments for IMO2020
  – Investments to convert bottoms to lighter products
  – Combining crudes for better economics will not result in predicted bitumen properties

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Area of management Rijkswaterstaat

90% porous asphalt

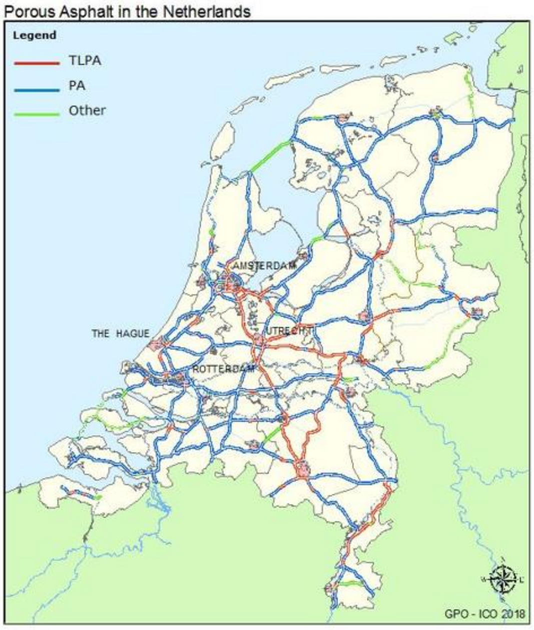
- 70% PA
- 20 % two-layer PA

10% impervious asphalt

- mostly on sliproads
- 9% AC
- 3% SMA / other

Data 2018

Porous Asphalt in the Netherlands



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Questioning/ suspicion

- Bitumen blending
  - The use of different components to meet requirements (bitumen engineering)
  - The use of REOB/VTAE
- EN12591 (bitumen and bituminous binder specifications)
  - Penetration
  - Softening point
  - Resistance to hardening
    - Retained penetration
    - Increase in softening point
  - Fraass breaking point
  - Kinematic viscosity at 135 °C
  - Flash point
  - Solubility

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State of the art report

- policy adviser started to collect knowledge documents and having interviews
- Goal was to get answers;
  - what steps must Rijkswaterstaat take to ensure the quality of asphalt within this bitumen transition
  - Is it a problem? How to address this? What can a government do (and what is not allowed)?
  - Amount of bitumen; are there risks and how to act upon them accordingly
  - Quality; what are the differences and what can be said about the influence on asphalt now and in the future.

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Asphalt-Impulse research project

- 2016 sector-wide exploratory survey
- broad support
- 2017 several meetings to collect and discuss improvement suggestions

Asphalt-Impulse research project

- Aim;
  - “doubling the lifetime of asphalt pavements,
  - halving the scatter in lifetime,
  - halving the CO2- footprint

  at same or lower cost”.

- 6 projects did start;
  - Demonstrable sustainable asphalt mixes,
  - Better asphalt mixes in contracts,
  - **Grip on bitumen,**
  - Functional acceptance,
  - Hightech = Lowcost,
  - Quality assurance and Lifespan prediction model for asphalt mixes.

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Research project Grip on Bitumen

- Problem definition
  - developments bitumen market; there are many publications that show that these developments have unsuspected consequences that, if not known to the user, can cause problems.
- Objectives
  - to make a knowledge document bitumen;
  - to visualize the bitumen properties relevant to asphalt behavior and their distribution;
  - to establish a framework for the evaluation of bituminous binders for use in specific asphalt applications.

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Progress is difficult and slow

- Bitumen supplier, asphalt production plant, contractor ánd the client (road authorities) are all at the table;
  - All have their own interests
- Yes, we do have problems… but to make these concrete…
  - Oral comments; early stage damages & problems within mixing or laying process
  - There is need of concrete cases that could present the real-life examples

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First deliverable knowledge document

- What is bitumen (incl. sweet & sour crude explained and refinery production, REACH, Bitumen blending and REOB
- Changes in production quantities, methods, regulations, production facilities in Europe
- Bitumen specifications & asphalt parameters

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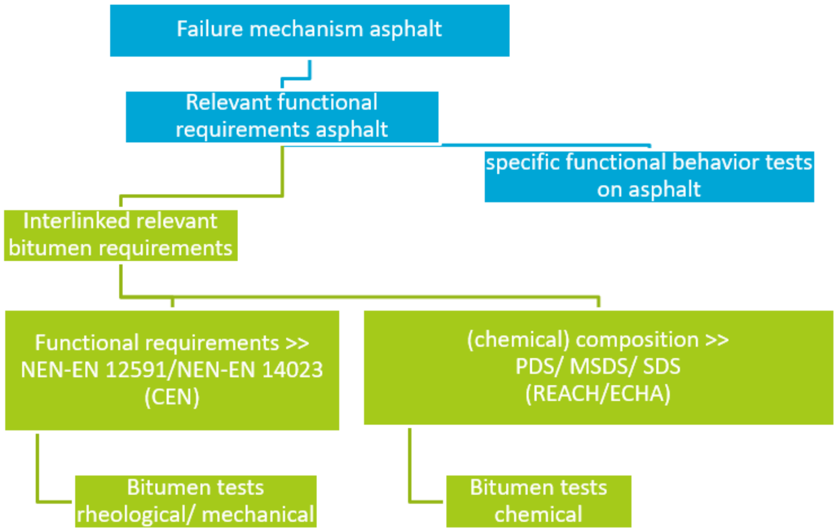
2nd deliverable;
asphalt /bitumen matrix



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asphalt /bitumen matrix

Op asfalt niveau: van bitumen naar asfaltgedrag

	spoorvorming	weerstand tegen vermoeiing	Stijfheid > als relevante 'overkoepelende' eigenschap	scheurtaaiheid > propagatie-energie	scheurdoorgroeisnelheid > direct gerelateerd aan scheurtaaiheid	verdichtbaarheid	verwerkbaarheid	Vorst-dooigedrag	Health, Safety & Environment		aantasting chemicaliën	
T-gevoeligheid (zowel i.h.k.v hard worden als verglazen)	x	x	x	x	x	x	x	x				
Weerstand tegen vervorming (hoge temperaturen)	x		x			x						
viscositeit	x					x	x					
vermoeiing		x	x									
stijfheid	x	x	x			x		x				
thermische krimp								x				
relaxatievermogen	x	x				x		x				
cohesieve sterkte		x		x	x			x				
adhesieve sterkte		x						x				
ductiliteit	x	x		x	x			x				
(scheur-) taaiheid		x		x	x			x				
verandering van gedrag door veroudering (V, KT,LT)	x	x	x	x	x	x	x	x				
Geur									x			
H2S									x			
VOC									x			
PAK									x			
chem. Compatibiliteit											x	
colloidale stabiliteit	x		x									
Chemische samenstelling > basiscomponenten	x											
Chemische samenstelling > Elementanalyse									x			
Chemische samenstelling > Molecuulgrootteverdeling												
Vaste stof gehalte (minerale stof a.g.v. vervuiling)												

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asphalt req/ bitumen tests matrix

						Chemische analyse					
					CEN norm EN	IP469 + IP143 (asfaltenen)	ASTM D2622	EN 13301 / xyleenequivalent	GPC - methode harmoniseren	GC/HPLC	Draeger tubes (H Nox, ...)
	Aantekeningen AI-GOB 22/06/2020	eigenschap	productie relevant	verwerking relevant	gebruik relevant	SARA	Total S	vlekvorming	GPC	PAK (10 - 16)	Headspace indica
			meestal verse bitumen	na RTFOT	na RTFOT + PAV						
T-gevoeligheid (zowel i.h.k.v hard worden als verglazen)	Wat bedoelen we met temp.gevoeligheid >> onderscheid maken tussen temperatuurvensters en relevante eigenschappen. >> Per temp venster zijn specifieke proeven beschikbaar >> Ook proeven beschikbaar die over alle tempvensters dezelfde eigenschap beschrijven	functioneel	x	x	x						
Weerstand tegen vervorming (hoge temperaturen)		functioneel		x							
viscositeit		functioneel	x	x							
vermoeiing		functioneel			x						
stijfheid		functioneel			x						
thermische krimp		functioneel			x						
relaxatievermogen		functioneel			x						
cohesieve sterkte		functioneel			x						
adhesieve sterkte		functioneel			x						
ductiliteit		functioneel			x						
(scheur-) taaiheid		functioneel			x						
verandering van gedrag door veroudering (V, KT,LT)	welke testen na veroudering?	functioneel			x						
Geur		samenstelling	x	x							x
H2S		samenstelling	x	x							x
VOC		samenstelling	x	x							x
PAK		samenstelling	x	x						x	
chem. Compatibiliteit		samenstelling				x					
colloidale stabiliteitsindex		samenstelling	x			x					
Chemische samenstelling > basiscomponenten		samenstelling	x			x					
Chemische samenstelling > Elementanalyse		samenstelling			?						
Chemische samenstelling > Molecuulgrootteverdeling (toegevoegd laagmoleculair bijv REOB)		samenstelling	x					x	x		
EVENTUEEL:											
Vaste stof gehalte (minerale stof a.g.v. vervuiling)		samenstelling									
totaal zwavel (niet voor korte termijn; emissie via H2S gedekt; functioneel is niet bekend; mogelijk u		samenstelling					x				

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Project Leerruimte (Room to learn)

- 2019; huge problems at two big projects
  - Dutch PA16 and TwoLayerPA had damages (high amount of raveling) within days/ weeks after laying
  - & reports of poor workability of Dutch PA16 and TwoLayerPA
- Bitumen research by the contractor

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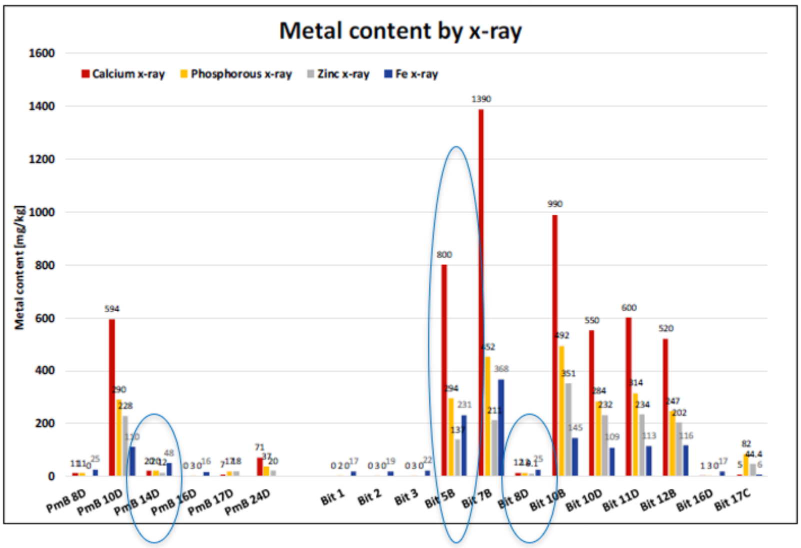
Bitumen research by the contractor



Bron; Contractor concept report provisional results bitumen quality

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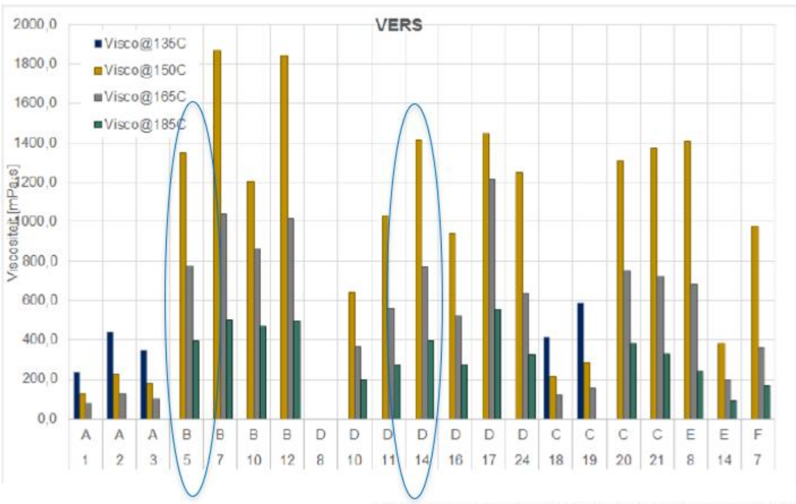
Bitumen research by the contractor

Viscositeit



Bron; Contractor concept report provisional results bitumen quality

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Project Leerruimte (Room to learn)

- Research question;

  how to demonstrate suitability or in-suitability of a specific bitumen, for application in asphalt (verifiable and reliable)
- Lab-research
  - take samples at deliverance at the asphalt plant
  - Sampling bitumen, asphalt mixes production samples, and a sample at the asphalt truck or spreader
  - Selection of chosen relevant bitumen tests
  - Analyse this in combination with information of the production and workability as well as the quality after laying
  - Monitor these roadsections

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Continuation

More to come….

- Grip on Asphalt

- Knowledge-based Pavement Engineering (TU-Delft, TNO, Rijkswaterstaat)
  - Characterization and Evaluation of Asphalt Binder Properties (CEAB)

    (Xueyan Liu, Sayeda Nahar, Peng Li, Liz Mensink, Inge van Vilsteren)
  - Ageing of Asphalt Pavements (AAP+)

    (Aikaterini Varveri, Diederik van Lent, Ruxin Jing, Cecile Giezen, Liz Mensink, Inge van Vilsteren)

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Thank you!
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3.2 Bitumen quality variability and impacts on pavement materials, what is missing in current specifications

Frédéric DELFOSSE

Eurovia Research Centre, Mérignac, France

Abstract

The current situation of revamping and rationalization from refiners creates concerns about the quality and consistency of the delivered bitumen, especially as the current specifications appear insufficient to ensure satisfactory performance of the finished products. In this context, the search for relationships and correlations between bitumen properties and performance of the asphalt mixtures and the pavement has become very relevant. Moreover, with the constant increasing of RAP content in new asphalt mixes coupled with the usage "rejuvenators", some limits of conventional tests methods can be pointed out.

This presentation will highlight some new innovative bitumen and HMA indicators, allowing one to guarantee the durability of the road for the future. A study based from a standard mix design with one type of aggregate (similar volumetric properties) and 16 bitumens from various origins is presented. The characterization of asphalt mixes covered various mechanical tests such as modulus, rutting, fatigue, water sensitivity and thermal cracking.

Also, this study spotlights how crucial it is to consider long-term ageing on the low-temperature end, since their behaviour can highly be impacted for both bitumen and asphalt mixes.

About the speaker

Frédéric Delfosse is a physico-chemical engineer who joined Eurovia in 1998. He is currently the Director of Eurovia's Research Centre near Bordeaux, France. He started his career as research engineer on emulsions and cold mixes for 5 years before becoming a project manager working on different research programs concerning asphalt.

Since 2016, he oversees 33 people working at the Research Centre on various international research projects related to asphalt, such as cold products, innovative additives for the road industry, the development of new mechanical tests for HMA, road marking, aggregates, railway, new innovative sensors to measure road properties in place or even artificial intelligence for autonomous vehicles for instance.

iFRAE DELFT 2021 **5th International Symposium on Frontiers of Road and Airport Engineering**

Workshop: Changes in binder properties and the role of additives

Bitumen quality variability and impacts on pavement materials, what is missing in current specifications ?

F. Delfosse (Eurovia)

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Context : Bitumen Quality and variability

- **European refining outlook: ongoing rationalization**
  - ✓ Changes / evolution in crude oils and refining processes
  - ✓ Additive market expansion – emerging bio additives (polar functions)….
  - ✓ Economics – trading, supply, imports, …
- **European Standard EN 12591: Pen grading limitations to ensure field performance of the finished products**
- **Consequences – Impacts**
  - ✓ Production & supply – Paving & roofing, modified, emulsions…
  - ✓ Binder and mixture properties (ageing…)
  - ✓ Product long-term performance and prediction ?

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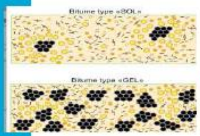
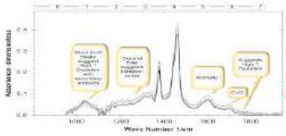
Context : Bitumen Quality and variability

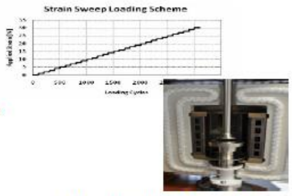
- **Limits of current bitumen specifications what more we should do in future ?**



Bitumen

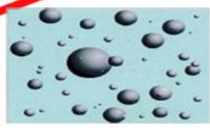


Composition

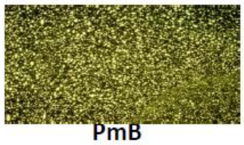


Properties vs. aging

Mixtures



Emulsion



PmB

3

Objectives

- **Define new relevant indicators :**
  - ✓ To mimic fume issue of bitumen on field (limit of RTFO test)
  - ✓ To correlate bitumen quality with Hot Mix Asphalt properties :
    - o Rutting performances/ Low temperature behaviour after ageing/ Modulus….
    - o Impact of additives polarity ? (Rejuvenator or plasticizer)

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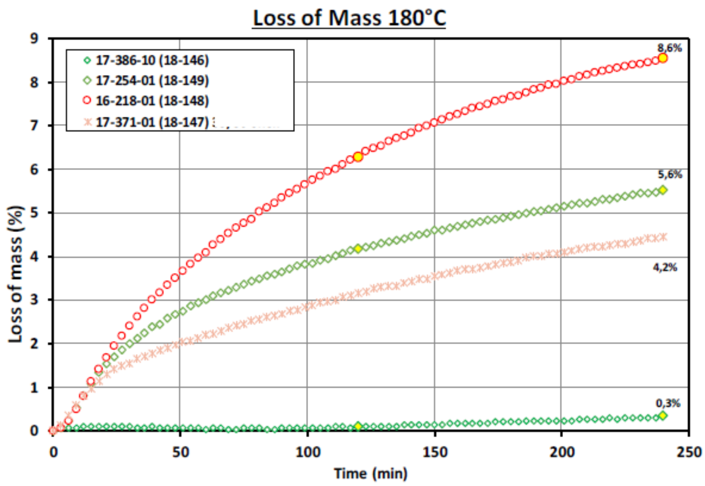
Fume issue

- Bitumen comply with the NF EN 12591 (loss of mass < 0,5 % according to RTFO test (NF EN 12607-1))
- On field , negative feedback is increansingly important (workers constrainst)

- Implementation of a new test with a Thermobalance (Metler HC103) : loss of mass at 160°C and 180°C

Fume issue



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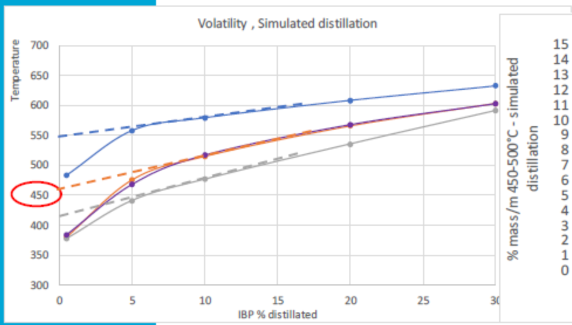
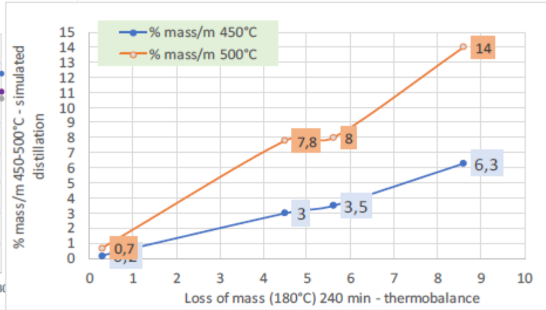
Fume issue

- **Correlation with simulated Distillation by GC ?**





- ✓ Thermobalance test allows a quick answer to classify bitumen susceptibility to generate fumes according to temperature
- ✓ Limits of RTFO test are highlithed . Impact on bitumen durability ?

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HMA properties

	B1	B2	B3	B4	B5	B6	B7	B8
Refining process	Vacuum distilled	Blend	Vacuum distilled	Vacuum distilled	Vacuum distilled	Air rectified	Blend	Blend
Penetration (1/10 mm) : NF EN 1426	40	37	40	22	26	28	55	57
Ring and Ball Temperature (°C) : NF EN 1427	53.4	53	52	59	57.2	61	49	49.2
Superpave performance grading (PG)	70-22	70-16	70-16	76-16	76-16	76-10	64-22	64-16

	B10	B11	B12	B14	B15	B16
Refining process	Blend	Propane deasphalter	Vacuum distilled	Air rectified	Blend	Blend
Penetration (1/10 mm) : NF EN 1426	35	44	50	28	25	24
Ring and Ball Temperature (°C) : NF EN 1427	52.8	51.6	50.2	59.8	57.8	60.3
Superpave performance grading (PG)	70-16	70-16	64-22	76-10	76-16	76-16

- **Bitumen analysis :**
  - ✓ All conventional tests: needle penetration (EN 1426), Ring and Ball temperature (EN 1427), Fraass breaking point (EN12593);
  - ✓ Rheological tests DSR : Isotherms at different temperatures and determination of SHRP criteria
  - ✓ The Multiple Stress Creep Recovery test (MSCRT-EN 16659)
  - ✓ Bending Beam Rheometer (BBR) (EN 14771) on aged samples (RTFOT + 1 PAV)

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HMA properties

AC 14	Content (%)
10/14 Diorite	38.1
6/10 Diorite	9.5
2/6 Diorite	9.5
0/2 Diorite	3.8
Binder	4.9
Manufacturing parameters	
Bitumen and Aggregates Temperatures	160°C
Mixing Time	140s

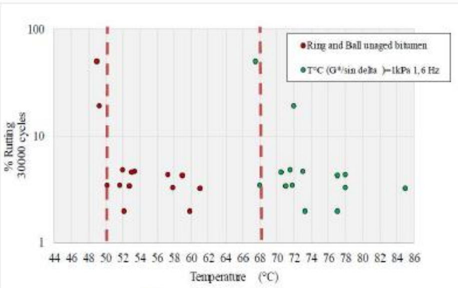
HMA design : VMA = 15,5 ± 1 %; VFA = 78 ± 2 %

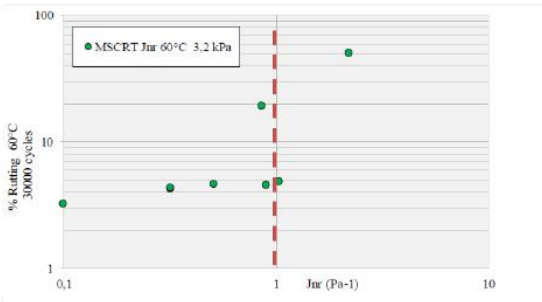
Test	Standard
Wheel tracking test	EN 12697-22
Stiffness Modulus (Test applying indirect tension to cylindrical specimens : IT-CY)	EN 12697-26 Annexe C
Stiffness Modulus (Test applying direct tension to cylindrical specimens : DT-CY)	EN 12697-26 Annexe E
Fatigue Test (Two-point bending test on trapezoidal shaped specimens)	EN 12697-24
Thermal Stress Restrained Specimen (TSRS)	EN 12697-46
Void content by gamma bench	* EN 12697-7
Water sensitivity	EN 12697-12 A / B

*After and before an aging procedure (Rilem : 4h at 135°C+ 9 days at 85°C) 9

HMA properties

- **Rutting Test**



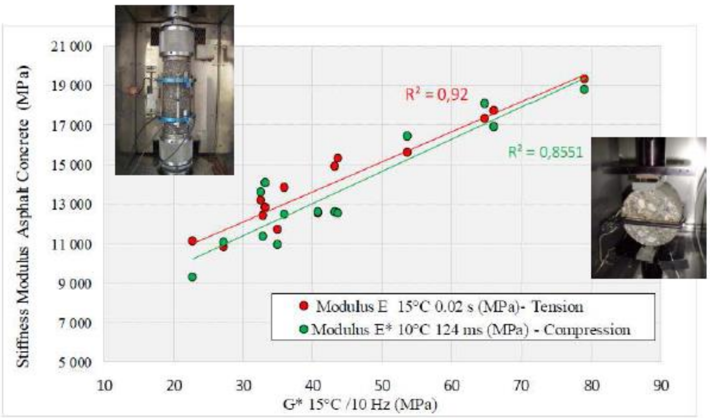


- **Comments :**
  - ✓ For a neat bitumen : R&B Temperature, T(G*/sinδ = 1 kPa) or Jnr are relevant parameter to predict rutting performance
  - ✓ Those thresholds are expected to be aggregate composition dependent

10

HMA properties

- **Stiffness Modulus**



- **Comments :**

➤ Good correlations between stiffness modulus and G* (15°C, 10 Hz)

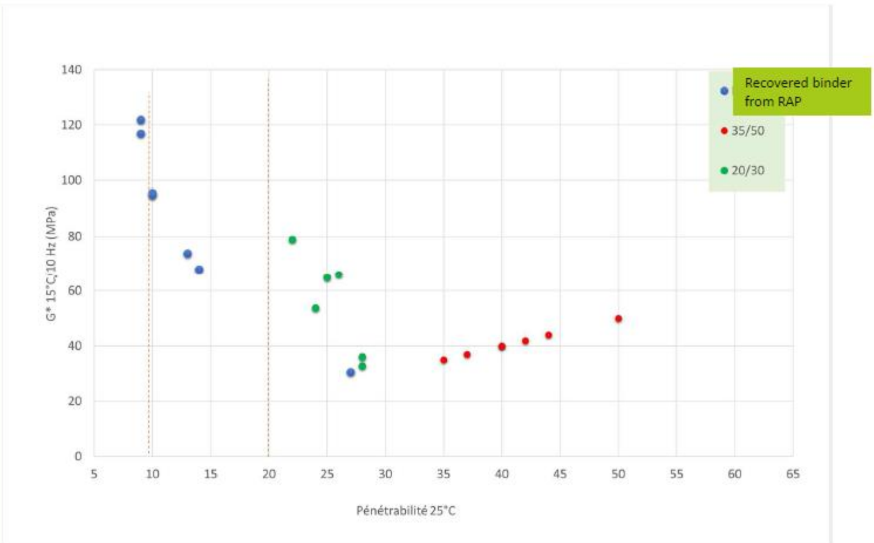
➤ Model has been developed to predict E from VMA, VFA and G* from recovered binder or neat bitumen with an oxidation law

11

HMA properties

GS9

- **Stiffness Modulus**
  - ➤ Limits Pen grade !



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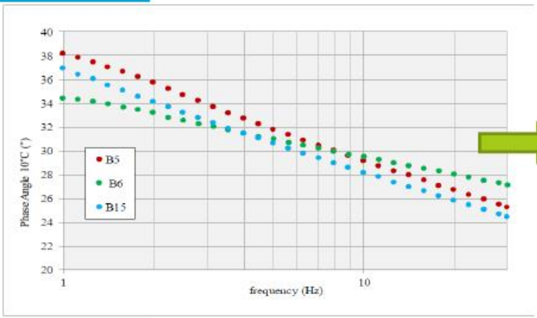
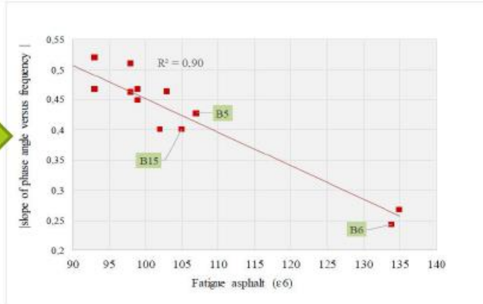
HMA properties

- **Fatigue**





- **Comments :**
  - ✓ The higher the slope (absolute value) of the phase angle isotherm (10°C), the lower the asphalt fatigue resistance (greater sensitivity to loading time).

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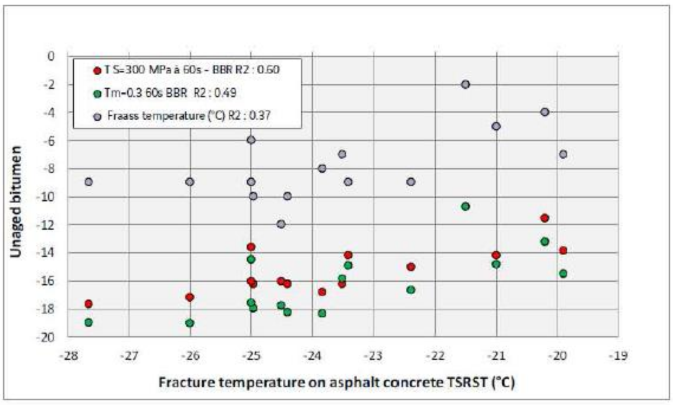
HMA properties

❑ Thermal Stress Restrained Specimen Test



- **Comments :**
  - ✓ For unaged bitumen : Fraass Temperature not correlated with TSRS test. For Ts and Tm not relevant correlations (the temperature difference between bitumen is not important : 4 to 6 °C for Ts or Tm!)

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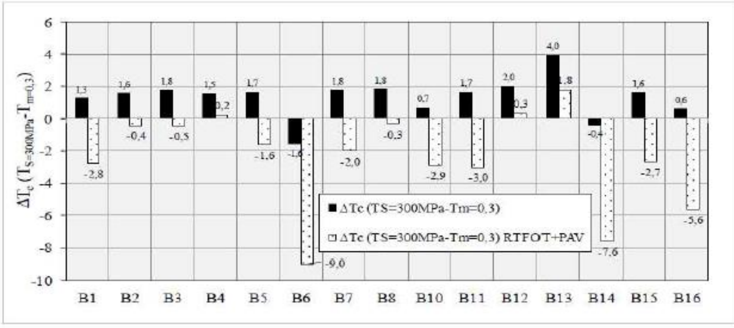
HMA properties

❑ Thermal Stress Restrained Specimen Test



- **Comments :**
  - ΔTc parameter appears as an interesting indicator to illustrate this asphalt oxidation sensitivity . A high |ΔTc| value means a bitumen sensitive to oxidation with a negative impact on the pavement durability. B6 and B14 are strongly m dependant

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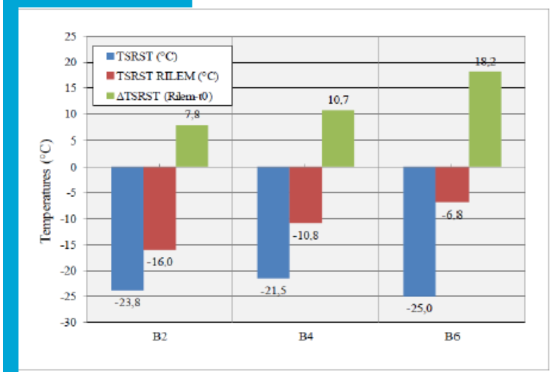
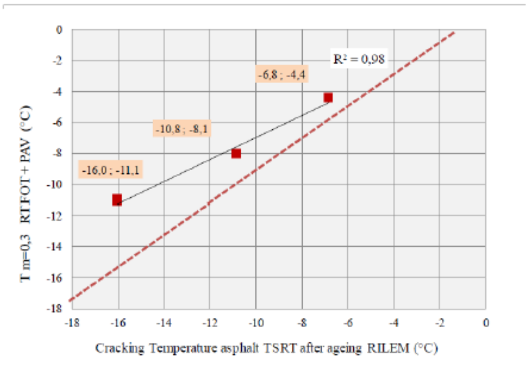
HMA properties

❑ Thermal Stress Restrained Specimen Test





- **Comments :**
  - ✓ These graphs show the importance of testing long-term aged asphalt mixes. HMA with B6 show a very dramatic decrease in its TSRST performance as compared to those made from B2 and B4. These results are well in line with the previous results on binder. On the right we can see the correlation with the Tm value after ageing.

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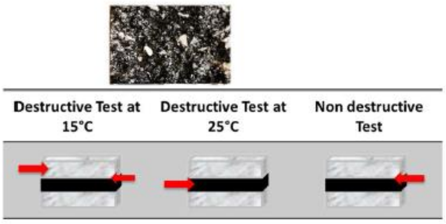
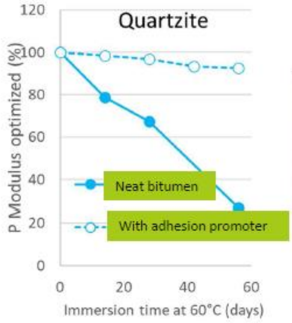
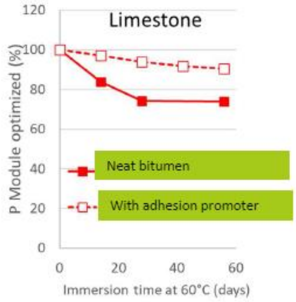
HMA properties

- **Adhesion**
  - J. Vinet Thesis 2016/2019







- Publications/ Conferences:
  - J.Vinet,… « Stripping at the bitumen aggregarate interface : A laboratory method to assess the loss of chemical adhesion », Energy and Fuels 2019, 33, 4, 2641-2650
  - J. Vinet…, « Comparaison statistique de différents essais de tenue à l'eau », RGRA n°966, Sept. 2019
  - J. Vinet…, « Effet de l'eau sur l'interface bitume/granulat et sur un enrobé bitumineux compacté », RGRA n°967, Oct. 2019
  - J. Vinet…, « Advantages of non destructive method in moisture damage evaluation », Eurobitume 2021
  - J. Vinet…, « Tailoring a new laboratory methodology for stripping resistance evaluation of asphalt materials", Petersen conference 2018
  - J. Vinet…, « Laboratory evaluation of moisture damage and impact of additivation", Petersen conference 2019

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HMA properties

❑ Addives impact on HMA properties (Rejuvenator, PG grade…)

✓ All HMA and bitumen tests are performed without water !

✓ Correlations are possible because bitumen is a hydrophobic product.

- Few molecules adsorption on aggregate
- No chemical reaction with aggregate with or without water

➔ HMA is a recyclable product even if the bitumen is aged (UV…) with a dedicated additive (to restore "original" viscoelastic properties)

✓ With a polar additive and according to its content :

- Different reactions are possible :
  - ➔ Adsorption on aggregate surface according to its nature
  - ➔ Chemical reaction between aggregate/ water (hydrolysis) and additive (salt formation…) => Loss of viscoelastic properties
- No more correlation between binder properties and HMA ! Limitations of binder and HMA standardized tests
  - ➔ Publications :
    - ➔ F. Lahjiri,…, "Etude de l'impact physico-chimique des liants dits "régénérants" sur la constructibilité (performance et durabilité) des enrobés recyclés, doctoral thesis, University of Montpellier, 2020
    - ➔ F. Lahjiri,… « Towards a Better Assessment of Recycling Agents Effects on Bitumen During Hot Recycling", ISAP, Padoue 2019
    - ➔ F. Lahjiri,…, "Impact of binder acidity index on the properties of asphalt mixes incorportating additives", Petersen conference 2021

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Conclusion

- This presentation presents some results to show some limits of European tests (Bitumen / HMA) :
  - ✓ Fumes : RTFOT test is not correlated with fume issue on Field. Impact on ageing ?
  - ✓ Correlation between complex Modulus 15°C/10 Hz and stiffness modulus on asphalt
  - ✓ Correlation between slope of the phase angle at 10°C (isotherms) with fatigue tests on asphalt
  - ✓ At higher temperatures, threshold effect with Ring and Ball Temperature, $T(G^*/\sin\delta = 1\text{ kPa})$, JnR with rutting on asphalt
  - ✓ For low temperature behaviour, poor correlations between unaged asphalt mixes (TSRST) and bitumen : BBR critical temperatures or Fraass values. After ageing interesting correlation were obtained between TSRST and the BBR test : ΔT_c and Tm=0.3 parameters. For wearing course at least 2 APV appears as necessary.
  - ✓ To evaluate the water sensitivity, a test from stiffness modulus test after several days in water at 60°C seems relevant (Non destructive test, loading of specimens in the linear deformation domain)

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Conclusion

- ✓ These relations between bitumen and asphalt mix performances must be expected to be also largely dependent on mix composition parameters. Additional tests are in progress to evaluate the impact of VMA and VFA values.

- ✓ Adding of additives can impact these correlations according to their polarity (potential interaction with aggregates / water…) ! Limits of conventional tests are highlighted

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iFRAE
DELFT 2021
5th International Symposium
on Frontiers of Road and Airport Engineering
Workshop: Changes in binder properties and the role of additives
Thank you!
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2
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3.3 A Tale of Two Deltas: Analysis approach, proposed limits, and validation work to address binder quality-related thermally induced surface damage

Michael Elwardany, Dave Mensching, Jean-Pascal Planche, and Gayle King

Federal Highway Administration, and Western Research Institute, USA

Abstract:

Superpave specifications address binder properties that may lead to rutting, transverse cracking, and fatigue damage with varying degrees of success. However, asphalt binder production and formulation has significantly changed and introduced much more variability in terms of quality since the development of Superpave Performance-Grade system because of economic, technical, and environmental reasons. Consequently, aged-induced surface distresses under combined thermal and traffic loading have become the main challenge for highway agencies. Thermally induced surface deterioration appears in the form of traditional transverse cracking, block cracking, and raveling, or accelerating damage at construction joints. This study evaluated the limitations of the proposed linear viscoelastic (LVE) rheological cracking surrogates, such as ΔT_c, R-value, and G-R parameters, and the ability of the Asphalt Binder Cracking Device (ABCD) failure test to overcome these limitations. ABCD is particularly appropriate to rank binder performance because the measured cracking temperature (T_{cr}) encompasses binder LVE properties, failure strength, coefficient of thermal contraction, and cooling rate. The proposed parameter ($\Delta T_f = T_c$ (S=300 MPa) from BBR - T_{cr} from ABCD) relates the failure temperature to the equi-stiffness temperature and gives credit to well-formulated and compatible polymer-modified binders expected to increase binder strength and strain tolerance. This paper proposes a specification framework based on both ΔT_c and ΔT_f, universally applicable, regardless of binder composition. Additionally, preliminary specification limits are proposed based on the analysis of 44 binders, 15 with corresponding field performance data. Obviously as confirmed by a recent stakeholder workshop and industry feedbacks, these preliminary specification limits need further validation and possible adjustments to account for regional experience and local challenges. Current efforts at FHWA TFHRC, in collaboration with various State Highway Agencies (SHA's), are focused to further validate the framework and specification limits.

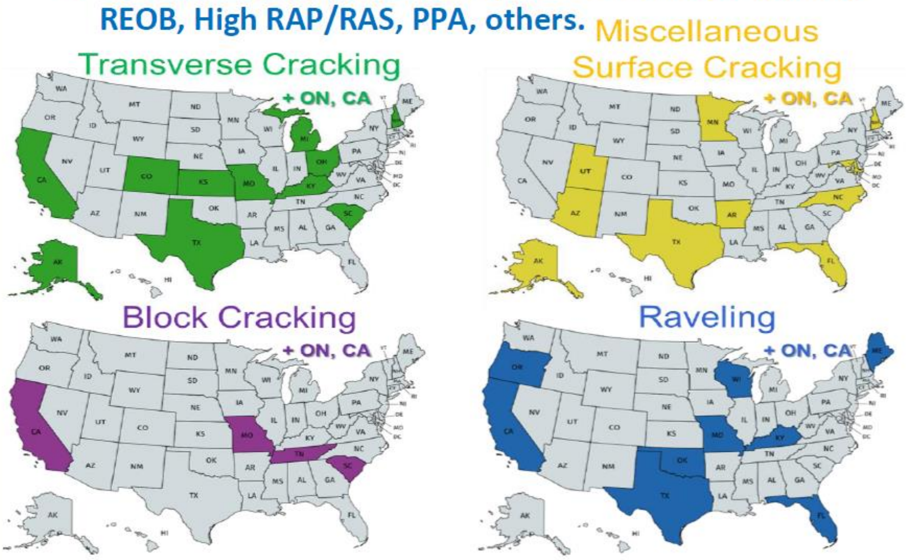
About the speakers

Dr. Michael Elwardany is the manager of the Asphalt Binder and Mixture Laboratories (ABML) at the Federal Highway Administration (FHWA) Turner Fairbank Highway Research Center. He was the program manager for paving asphalts at the Western Research Institute (WRI) for three years. Dr. Elwardany served as project lead to the National Cooperative Highway Research Program (NCHRP) 09-60 Project and the project manager for the Asphalt Industry Research Consortium (AIRC). He is an active member of several Transportation Research Board's Standing Committees, Association of Asphalt Paving Technologists (AAPT), RILEM, and ASCE-Airfield Pavement Committee. Dr. Elwardany holds Master's degrees from University of New Hampshire and a Ph.D. from North Carolina State University. He is a licensed professional engineer in the State of Wyoming, USA.

Dr. David Mensching is the Asphalt Materials Research Program Manager for the Federal Highway Administration (FHWA). He is the director of Turner-Fairbank Highway Research Center's Asphalt Binder and Mixture Laboratory and has research interests in automation and data science, connected pavements, resilience, and performance specifications. He is the chair of the Transportation Research Board's Standing Committee on Binders for Flexible Pavement and an active member of the Association of Asphalt Paving Technologists. Dr. Mensching holds Bachelors and Masters degrees from Villanova University and a Ph.D. from the University of New Hampshire. He is a licensed professional engineer in the Commonwealth of Virginia.

Post-SHRP Era (i.e., Superpave Specs in the 1990's)

- **NCHRP 09-60 Survey in 2017**
  - Potential issues identified by DOT's: Oxidized asphalt, REOB, High RAP/RAS, PPA, others.



Elwardany, M. D., G. King, J. P. Planche, C. Rodezno, D. Christense, R. Fertig III, K. Kuhn, and F. Bhuiyan. 2019. "Internal restraint damage mechanism for age-induced pavement surface distresses: Black cracking and raveling." *Journal of the Asphalt Paving Technologists*, vol. 88, p. 1-47.

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Post-SHRP Market Trends and Consequences

- **Technical, Economic, and Environmental Impacts**
- **Unconventional Binders (sometimes problematic)**
  - Some out of balance and incompatible blends
  - Waxy binders
  - Airblown, oxidized blends
  - Hard Solvent Deasphalting residues w/ soft blends
  - Multigrades and Hardgrades
  - Conversion residues "Visbroken residues" (IMO 2020)
  - Modified binders
    - Polymers: SBS, SBR, Terpolymer, EVA, rPE ...etc.
    - Additives: REOB, PPA, Wax, Biomass
    - Bio-binders and more ...
  - High RAP/RAS
  - Incompatible crudes (Fracking/Heavy)?!

Planche, J.P., Elwardany, M., Adams, J., Boysen, R. and Rovani, J., 2019. Linking Binder Characteristics with Performance: The Recipe to Cope with Changes in Bitumen Binder Quality. In 26th World Road Association (PIARC).

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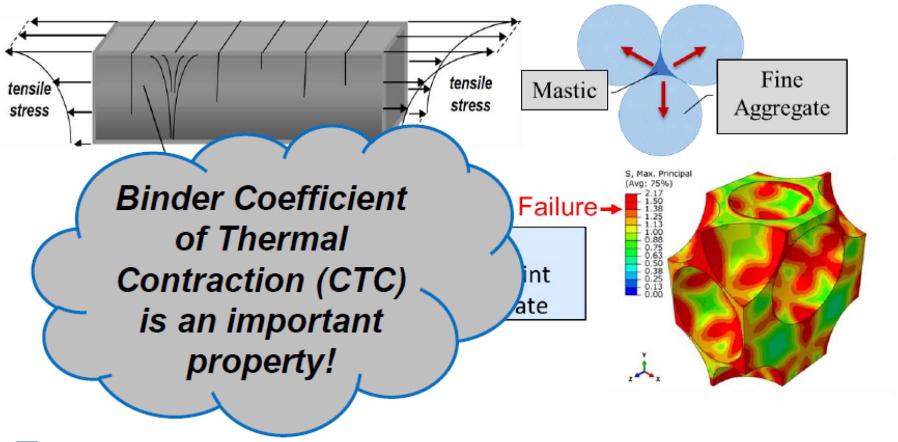
Two Thermally Induced Damage Mechanisms

Mix Restraint (External) and Mastic Restraint (Internal)



- Evidence for internal restraint damage
  - FEA & Mix-BBR(Sliver) Results (Elwardany et al., AAPT 2019)
  - Ohio CTE Device (Behnia et al., 2019)
  - Acoustic Emissions Results (Behnia et al., 2018)

Elwardany, M. D., G. King, J. P. Planche, C. Rodezno, D. Christense, R. Fertig III, K. Kuhn, and F. Bhuiyan. 2019. "Internal restraint damage mechanism for age-induced pavement surface distresses: Black cracking and raveling." *Journal of the Asphalt Paving Technologists*, vol. 88, p. 1-47.

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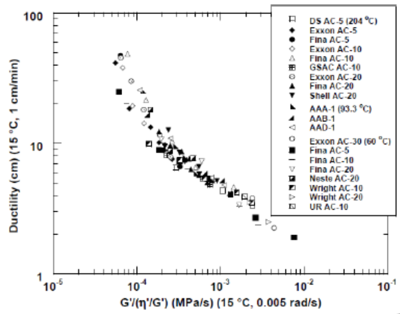
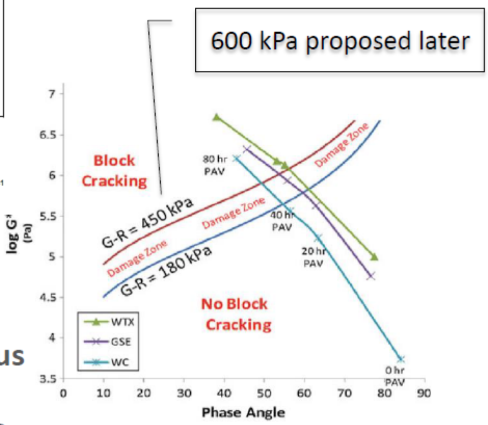
Proposed Rheological Surrogates in the Past Decade

- Black Space Analysis
  - Glover Function (Glover et al., 2005) $G'/(\eta'/G')$
  - G-R Parameter (G. Rowe et al., 2011) $\frac{G^*(\cos\delta)^2}{\sin\delta}$





  - Other proposals
    - Crossover Temp.
    - Crossover Modulus
    - δ at critical G*
    - Temp. at critical δ

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Proposed Rheological Surrogates in the Past Decade

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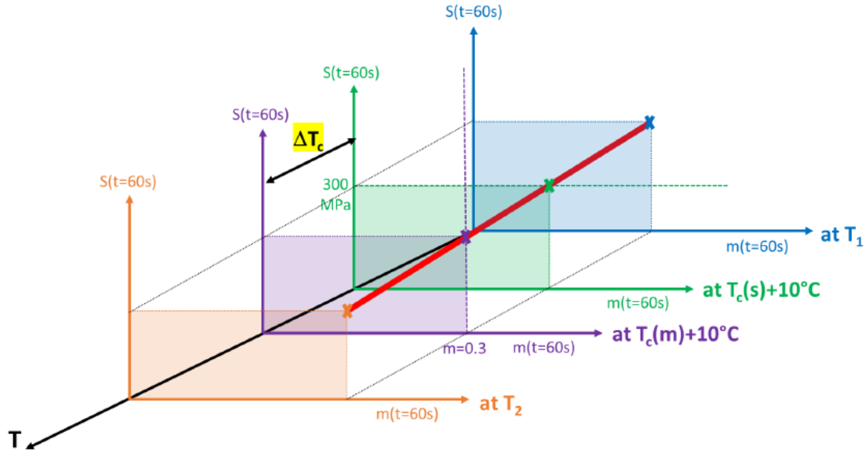
❑ ΔT_c Parameter

- ➢ $\Delta T_c = T_c(S = 300\ MPa) - T_c(m = 0.3)$.
- ➢ ΔT_c is in a third dimension from the Black space.
- ➢ No "real" correlations between ΔT_c and G-R



NCHRP 09-60 workshop under NCHRP 20-44(25), Planche, Elwardany, and King, 2020.

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Proposed Rheological Surrogates in the Past Decade

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❑ History of ΔT_c Parameter

- ➢ (Robertson et al. 2001)
- ➢ Linked to field performance and proposed as a binder spec by (Mike Anderson et al., 2011)
- ➢ Now implemented in at least 10 States/HSA's

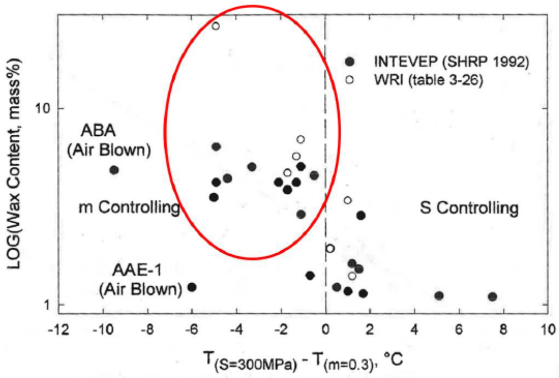

Figure 3-27. Relationship between wax content of asphalts and whether S or m controls the low-temperature SHRP specification.

Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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Proposed Rheological Surrogates in the Past Decade

- **ΔT_c Parameter is fundamentally inter-related to the rheological parameter from CA model (R-value)**
  - **R = 1.94 is equivalent to ΔT_c = 0 (Lesueur et al., 2021)**

$$\Delta T_C = R\frac{E_A}{2.303\overline{R}\log 2} \times \frac{\log\left[\frac{1}{0.3}-1\right]-\log\left[\left(\frac{300}{S_g}\right)^{-\beta}-1\right]}{\left(\log a_{T_{c,(S)}}+\frac{E_A}{2.303\overline{R}T_r}\right)\left(\log a_{T_{c,(m)}}+\frac{E_A}{2.303\overline{R}T_r}\right)} \quad (12)$$

where E_A is the activation energy, $\overline{R}$, the ideal gas constant, $\beta = \log 2/R$, T_r, the reference temperature, S_g the Glassy modulus (in MPa).

  - **ΔT_c is related to $T_{IR} = (T_x - T_g)$ (Elwardany et al., 2019)**
  - **ΔT_c is easier and more practical for implementation**
  - **ΔT_c is related to both low- and intermediate-temperature behaviors.**

Lesueur, D., Elwardany, M.D., Planche, J.P., Christensen, D. and King, G.N. 2021. "Impact of the asphalt binder rheological behavior on the value of the ΔTc parameter", *Construction and Building Materials*, 293, p.123464.

Elwardany, M.D., Planche, J.P. and Adams, J.J. 2019. "Determination of binder glass transition and crossover temperatures using 4-mm plates on a dynamic shear rheometer", *Transportation Research Record*, 2673(10), pp.247-260.

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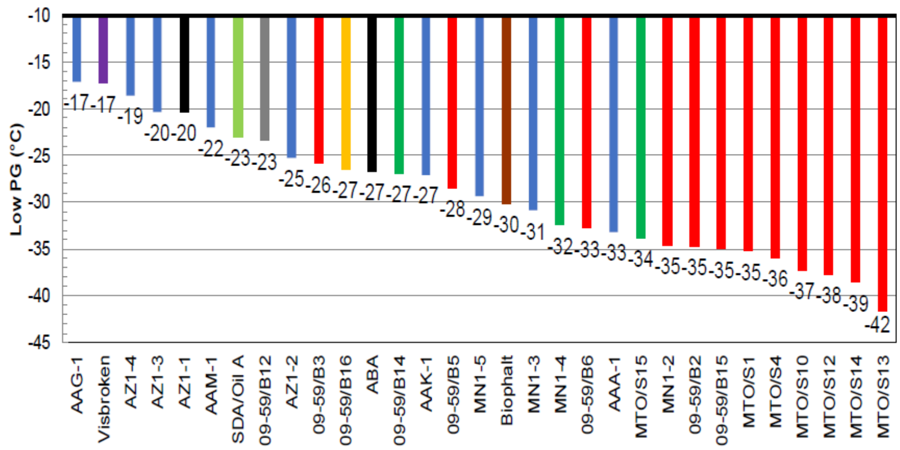
NCHRP 09-60 Binder Database Mapping

- **BBR, Low PG Ranking of 31 Binders after PAV20h Aging**
  - **Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Visbroken.**



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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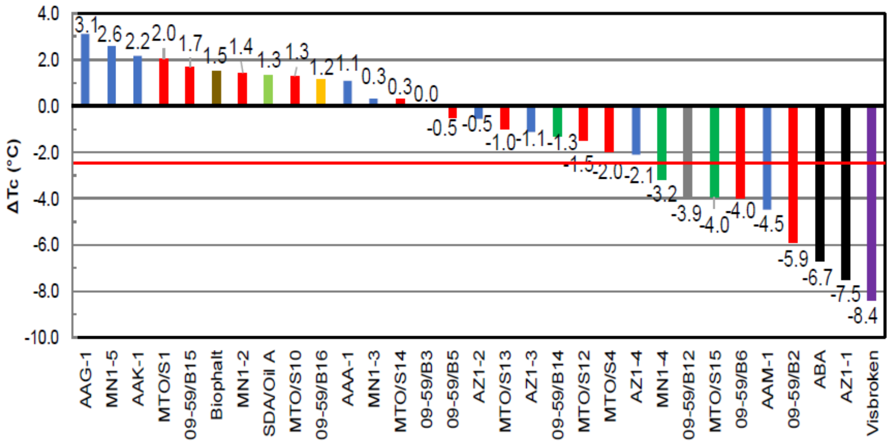
NCHRP 09-60 Binder Database Mapping

- BBR, ΔT_c Ranking of 31 Binders after PAV20h Aging
  - Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Visbroken.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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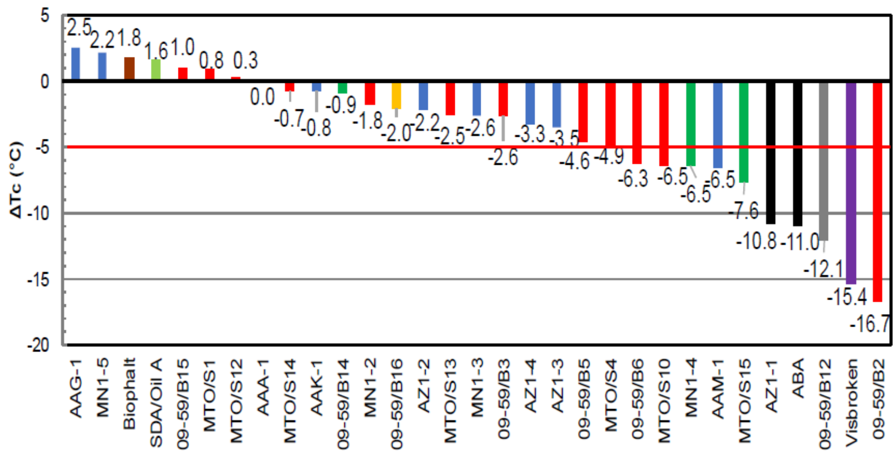
NCHRP 09-60 Binder Database Mapping

- BBR, ΔT_c Ranking of 31 Binders after PAV40h Aging
  - Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Visbroken.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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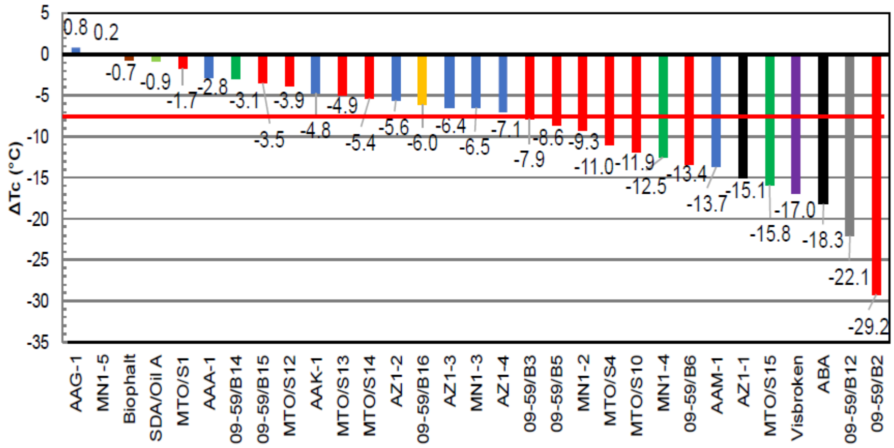
NCHRP 09-60 Binder Database Mapping

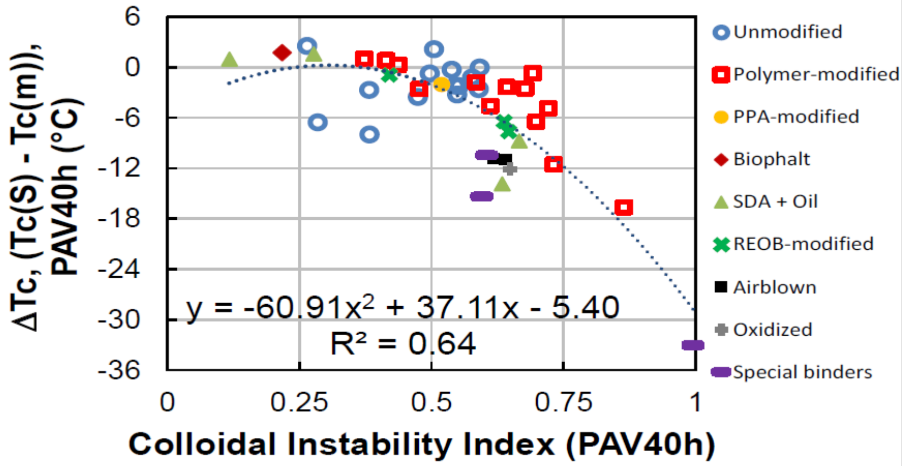
- BBR, ΔT_c Ranking of 31 Binders after PAV40h+PH72h Aging
  - Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Visbroken.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

NCHRP 09-60 Binder Database Mapping

- Correlation between ΔTc and CII after PAV40h
  - Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Special.

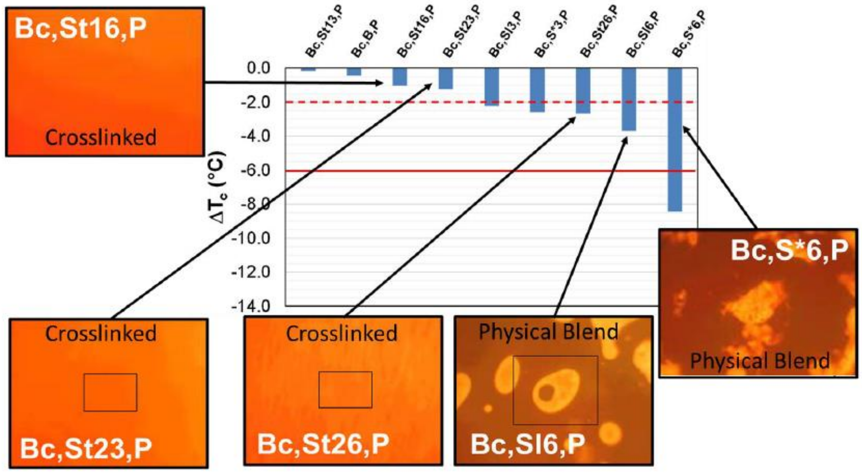


❖ Classical CII= (Sat+Asph)/(Arom+Res)

Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

NCHRP 09-60 Revisiting the TOTAL literature

- **ΔTc Index and phase structure**
  - Data collected from Durrieu et al, Lapalu et al, Mouillet et al, Planche et al, 2004-2008 & Revisited in Elwardany et al., 2020.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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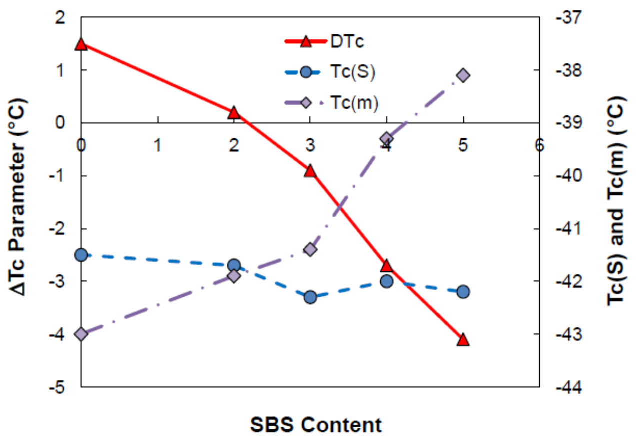
Limitations of Rheological Surrogates for PMA's

- **BBR, ΔT_c Parameter**
  - Systematic SBS-modification Study.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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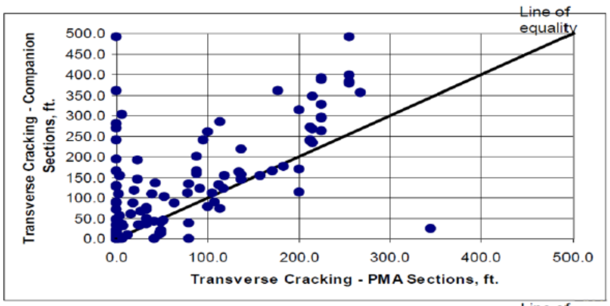
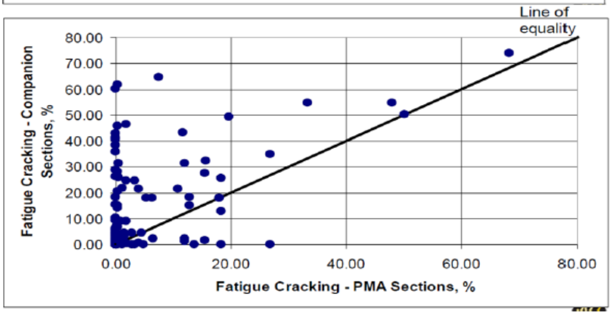
Limitations of Rheological Surrogates for PMA's

- PMA sections vs. companion unmodified sections
  - (Von Quintus, 2005)





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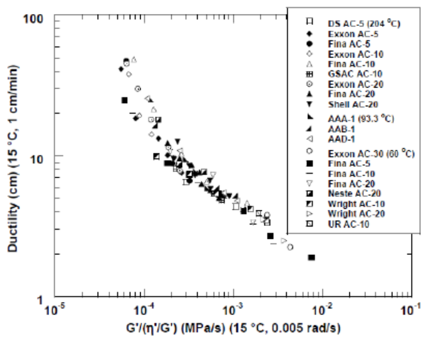
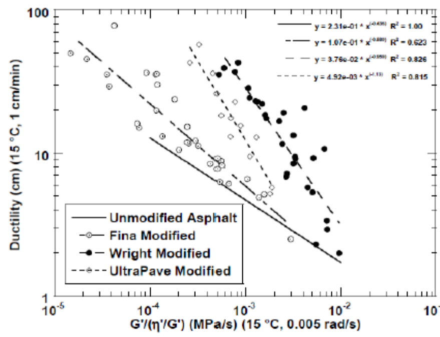
Limitations of Rheological Surrogates for PMA's

- DSR, Glover function and Glover-Rowe Parameter
  - G-R has limited applicability to PMA's (Glover, 2005).





Elwardany, M., J.-P. Planche, and G. King. 2020. "A Tale of Two Deltas" TRB AFK20 sponsored session, Washington, D.C.

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Insights, so far…

- **BBR, ΔT_c Parameter**
  - ✓ Captures relaxation properties of unmodified binders.
  - ✓ Generally, relates to asphalt colloidal structure.
  - ➢ Underestimates the performance of some complex binders such as PMA's.
  - ➢ Fail to capture failure properties outside the LVE domain such as strength/strain tolerance of PMA's.

- **Need to consider failure tests!**
  - ✓ DTT was introduced by SHRP pioneer researchers!
  - ➢ DTT failed due to lack of reproducibility.

- **What do we do now?**

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Western Research INSTITUTE

U.S. Department of Transportation

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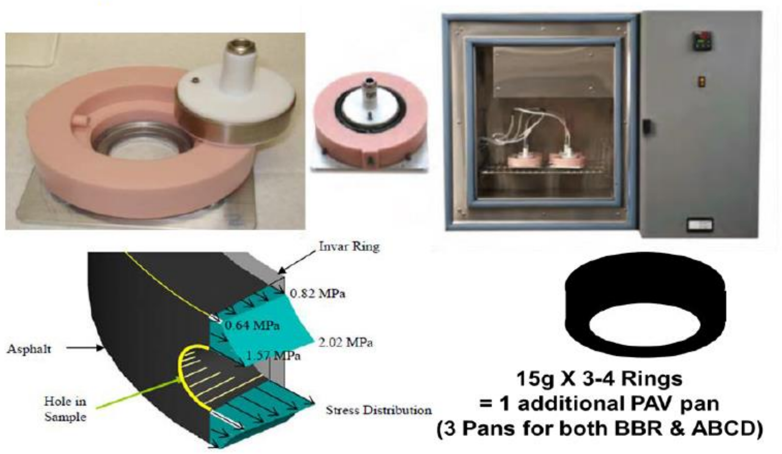
ABCD as a Replacement for DTT (NCHRP 09-60)

- **Asphalt Binder Cracking Device**
  - ➢ Test developed by SS Kim.
  - ➢ Proposed by NCHRP 09-60 as a failure test and a replacement for DTT.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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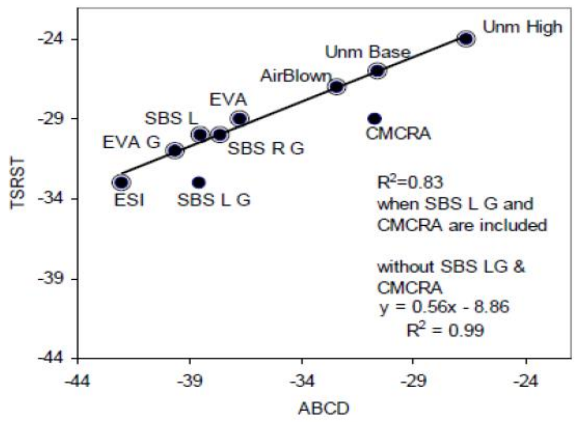
ABCD as a Replacement for DTT (NCHRP 09-60)

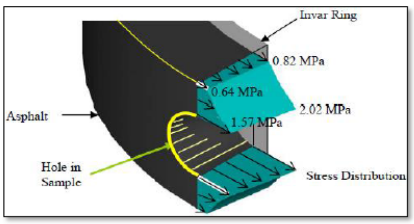
- **Asphalt Binder Cracking Device**
  - Equivalent to the TSRST test for mixtures.
  - Effective and sensitive to well-formulated and compatible PMA's



Kim, S.S. 2007. "*Development of an asphalt binder cracking device*". IDEA Program, Transportation Research Board, 2007.

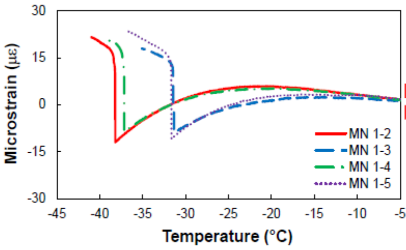
ABCD as a Replacement for DTT (NCHRP 09-60)

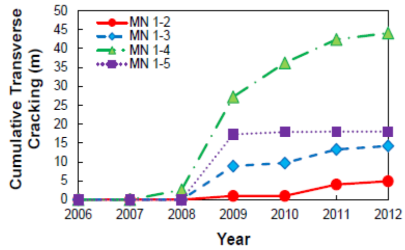
- **Factors affecting ABCD cracking temperature, T_{cr}**
  - CTC controls rate of volumetric changes.
  - LVE rheological properties G* and δ.
  - Strength (Viscoelastic property (t-TSP) with damage).
  - Glass transition temperature (T_g) impacts all parameters.
    - Glass transition takes place over a range of temperatures, confirmed by DSC.
    - Wider T_g region for complex and /or aged binders.



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

A Tale of Two Delta's



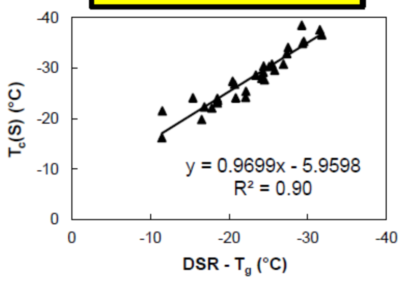


T_g of REOB around -80°c

(Planche et al., TRB 2015)

T_{cr} needs to be normalized to an equi-stiffness temperature! Also, $T_c(S)$ is a practical surrogate for T_g

Asphalt Industry Research Consortium



(Elwardany et al., TRR 2019)

Elwardany, M.D., Planche, J.P. and Adams, J.J. 2019. "Determination of binder glass transition and crossover temperatures using 4-mm plates on a dynamic shear rheometer", *Transportation Research Record*, 2673(10), pp.247-260.

Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". *Construction and Building Materials*, 255, p.119331.

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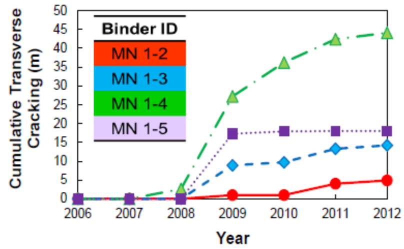
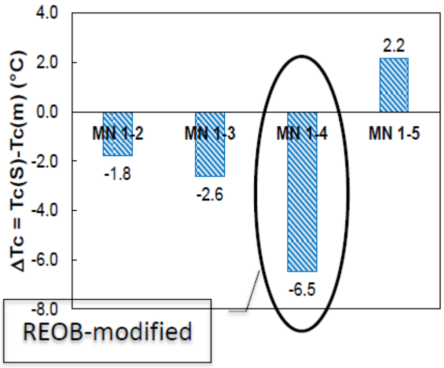
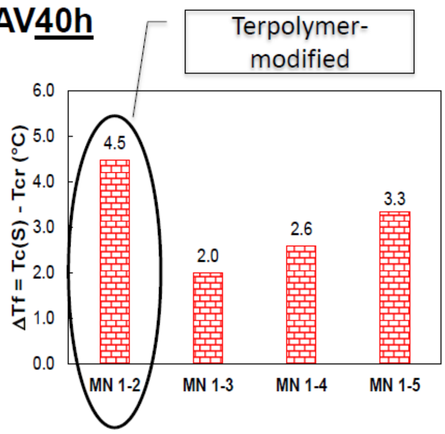
A Tale of Two Delta's

- $\Delta T_f = T_c(S) - T_{cr}$
  - ABCD cracking temperature normalized to equi-stiffness temperature.



- ΔT_c & ΔT_f ranking after PAV<u>40h</u>





Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". *Construction and Building Materials*, 255, p.119331.

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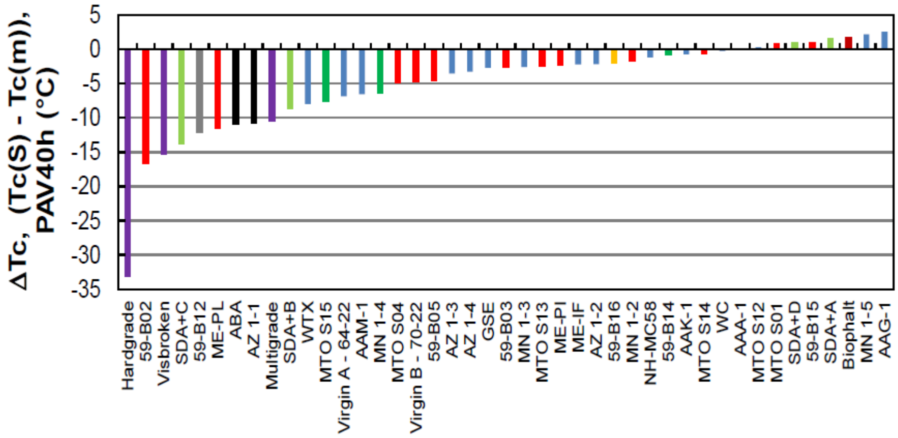
A Tale of Two Delta's

- ❑ **BBR & Corrected 4mm-DSR ΔT_c Ranking after PAV<u>40h</u> Aging**
  - ➢ **Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Special.**



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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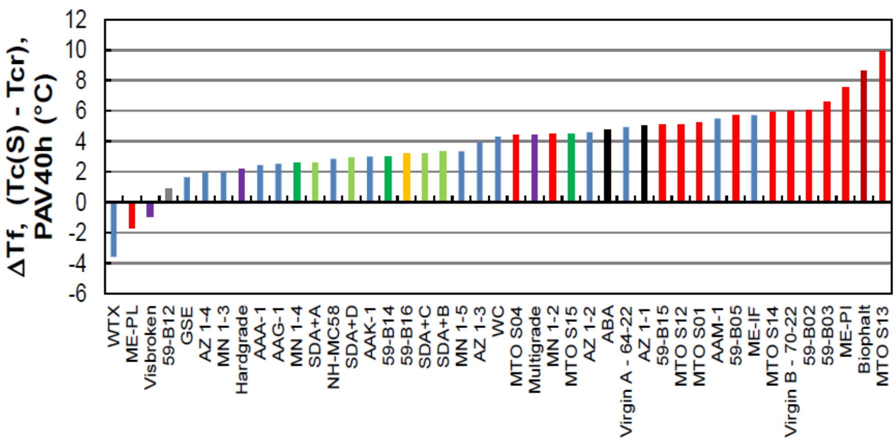
A Tale of Two Delta's

- ❑ **BBR & ABCD ΔT_f Ranking after PAV<u>40h</u> Aging**
  - ➢ **Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Special.**



Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.

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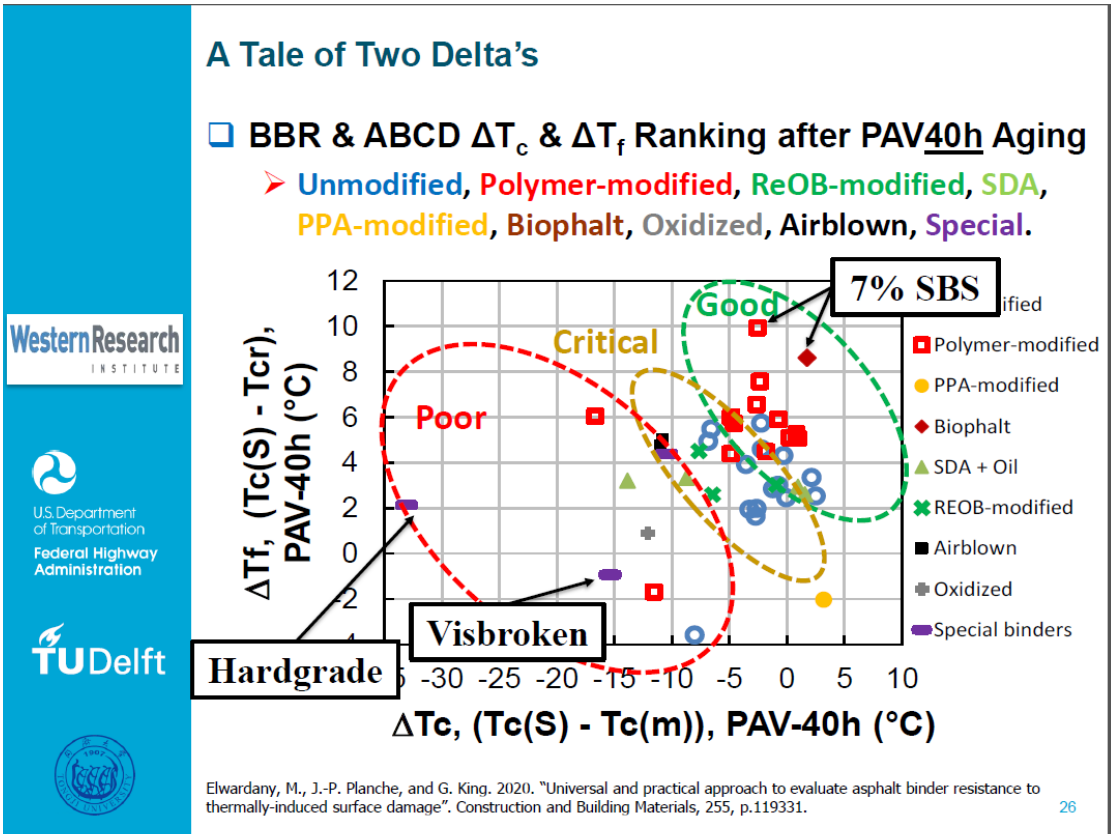
A Tale of Two Delta's
BBR & ABCD ΔTc & ΔTf Ranking after PAV40h Aging
Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Special.
Good
Critical
Poor
7% SBS
Hardgrade
Visbroken
ΔTf, (Tc(S) - Tcr), PAV-40h (°C)
ΔTc, (Tc(S) - Tc(m)), PAV-40h (°C)
Polymer-modified
PPA-modified
Biophalt
SDA + Oil
REOB-modified
Airblown
Oxidized
Special binders
Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.
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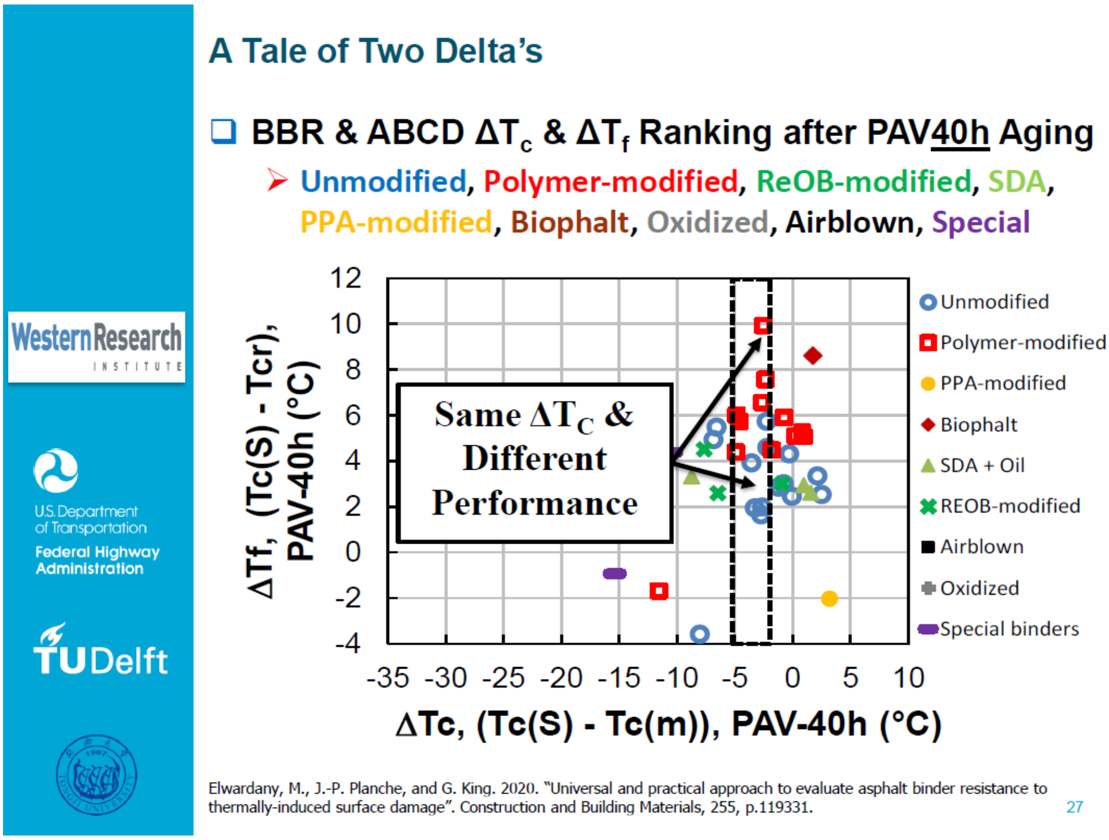
A Tale of Two Delta's
BBR & ABCD ΔTc & ΔTf Ranking after PAV40h Aging
Unmodified, Polymer-modified, ReOB-modified, SDA, PPA-modified, Biophalt, Oxidized, Airblown, Special
Same ΔTC & Different Performance
ΔTf, (Tc(S) - Tcr), PAV-40h (°C)
ΔTc, (Tc(S) - Tc(m)), PAV-40h (°C)
Unmodified
Polymer-modified
PPA-modified
Biophalt
SDA + Oil
REOB-modified
Airblown
Oxidized
Special binders
Elwardany, M., J.-P. Planche, and G. King. 2020. "Universal and practical approach to evaluate asphalt binder resistance to thermally-induced surface damage". Construction and Building Materials, 255, p.119331.
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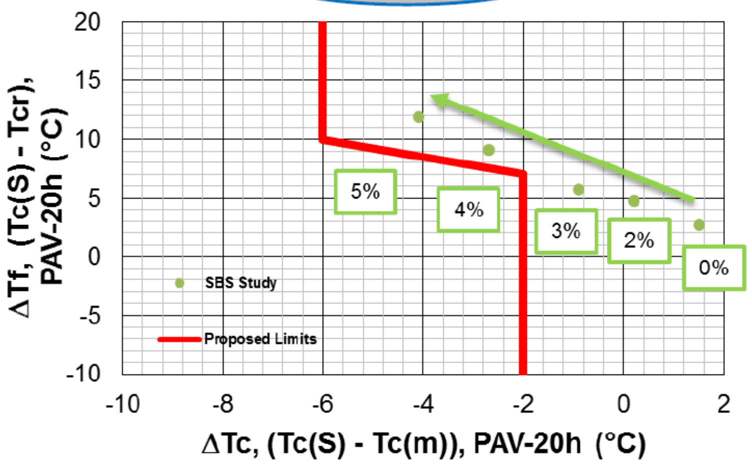
A Tale of Two Delta's

- SBS-modified binder systematic study

Importance of Cracking (Failure) Tests for Universal "Blind" Specs



Elwardany, M., J.-P. Planche, and G. King. 2021. "Proposed Changes to Asphalt Binder Specifications to Address Binder-Quality Related Thermally Induced Surface Damage". Association of Asphalt Paving Technologists Annual Meeting.

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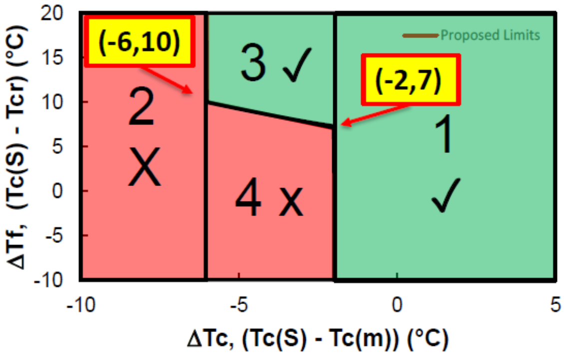
A Tale of Two Delta's

- Framework is later validated after PAV20h aging with various field sections in MN, AZ, TX, ME, and ON.
  - Field Validation will be presented at AAPT – Sept. 2021 in Nashville (Online and in-person meeting).



Proposed specifications framework

- **Addition to current Climate-based PG**
- **Universal - blind**
- **BBR alone when**

ΔT_c > -2°C (Accepted)

ΔT_c <-6°C (Rejected)

- **BBR & ABCD for**

-6°C<ΔT_c <-2°C

ΔT_f min = 7°C at -2°C

ΔT_f min = 10°C at -6°C

Elwardany, M., J.-P. Planche, and G. King. 2021. "Proposed Changes to Asphalt Binder Specifications to Address Binder-Quality Related Thermally Induced Surface Damage". Association of Asphalt Paving Technologists Annual Meeting.

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iFRAE DELFT 2021 **5th International Symposium on Frontiers of Road and Airport Engineering**

Workshop: Changes in binder properties and the role of additives

A Tale of Two Deltas:

Analysis Approach, Proposed Limits, and ***Validation Work*** to Address Binder Quality-Related Thermally Induced Surface Damage

Michael Elwardany, ESC Inc.; David Mensching, FHWA

U.S. Department of Transportation
Federal Highway Administration

TU Delft

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ABBREVIATIONS

- ABCD: asphalt binder cracking device.
- ABR: asphalt binder replacement.
- ALF: accelerated loading facility.
- HMA: hot mix asphalt.
- HP: High Polymer modified asphalt binders.
- PG: performance grading.
- PMA: polymer modified asphalt.
- RAP/RAS: reclaimed asphalt pavement/reclaimed asphalt shingle.
- SBS: styrene-butadiene-styrene polymer.
- WMA: warm mix asphalt.

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FHWA VALIDATION EFFORTS: LESSONS AND GAPS

- **FHWA in collaboration with Rutgers University.**
- **FHWA-ALF Study, RAP/RAS mixtures.**

Source: FHWA.

Source: FHWA.

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FHWA VALIDATION EFFORTS: LESSONS AND GAPS

FHWA-ALF Study, RAP/RAS mixtures.

Recycle Content	HMA/WMA Drum Discharge Temp	149°C (300°F) – 160°C (320°F)		116°C (240°F) – 132°C (270°F)	
	Warm Mix Technology	None		Foam	Chemical
	0%	PG 64-22 L1		N/A	N/A
	20% ABR RAP ≈ 23% RAP by weight	PG 64-22 L6		PG 64-22 L9	PG 64-22 L4
	20% ABR RAS ≈ 6% RAS by weight	PG 64-22 L3	PG 58-28 L7	N/A	N/A
	40% ABR RAP ≈ 44% RAP by weight	PG 64-22 L5	PG 58-28 L8	PG 58-28 L2	PG 58-28 L11

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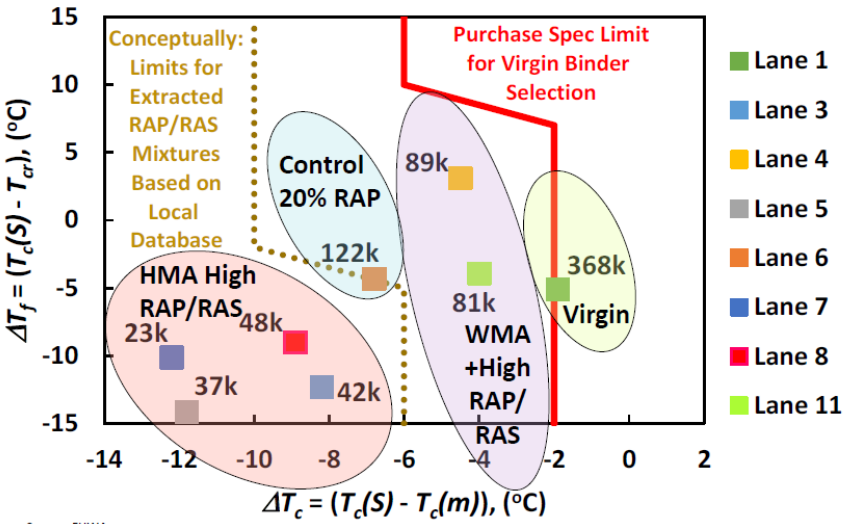
FHWA VALIDATION EFFORTS: LESSONS AND GAPS

SBS Systematic Study, analysis framework.



Source: FHWA.

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FHWA current and future work in collaboration with:

- **Rutgers University**: Field sections at ALF, New Jersey Department of Transportation, and Federal Aviation Administration.
- **University of Waterloo**: Systematic SBS content.
- **University of Nevada Reno**: PMA/HP.
- **Virginia Transportation Research Center**: PMA/HP.
- **International Union of Laboratories and Experts in Construction Materials, Systems and Structures (RILEM) members**: complex binders.
- Others under discussion.

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iFRAE DELFT 2021 **5th International Symposium on Frontiers of Road and Airport Engineering**

Workshop: Changes in binder properties and the role of additives

Thank you!

Michael Elwardany, m.elwardany.ctr@dot.gov
David Mensching, david.mensching@dot.gov

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3.4 Recycling practices and additives in asphalt: Current practices and the future trends

Lily Poulikakos, Ph.D, Senior Scientist

Empa - Swiss Federal Laboratories for Materials, Switzerland

About the speaker

Dr. Lily Poulikakos received her B.S in architectural engineering from the university of Colorado, Boulder USA, M.S. in civil engineering from university of Illinois USA and PhD in civil engineering from ETH Zurich, Switzerland. She is currently a senior scientist at Empa, the Swiss federal laboratories for materials science and technology. Her research focus is on using multi scale characterization methods to study innovative bituminous materials chemically and mechanically. She is a leading member of Rilem as former deputy chair of the technical committee TC-231 NBM on nano bituminous materials and TC-252 CMB chemo mechanical characterization of bituminous materials and currently chair of TC-279 WMR on waste and marginal materials for roads. Dr. Poulikakos is the author of over 100 publications in peer reviewed journals and editor of Elsevier journal Construction and Building Materials CBM.

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Outline

- Introduction and Motivation
- Rejuvenators
- Waste additives
- Facts and Figures
- Rilem TC-279 WMR
- Performance Results
- Environmental assessment
- Life cycle assessment
- Conclusions

2

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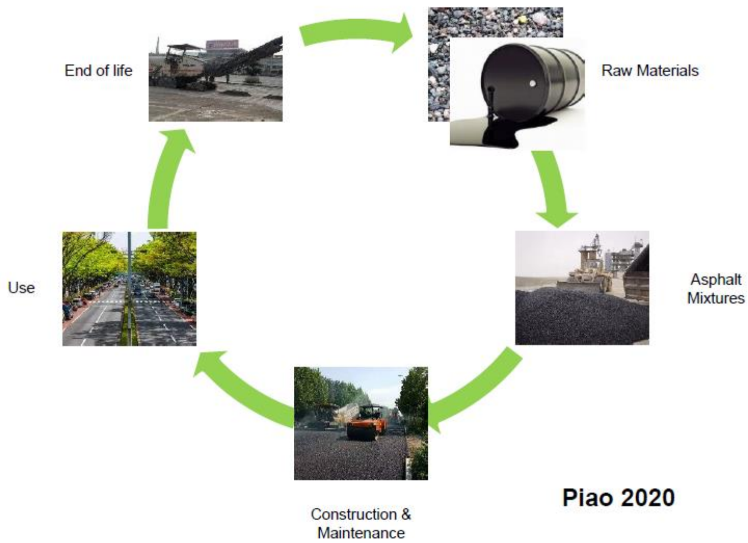
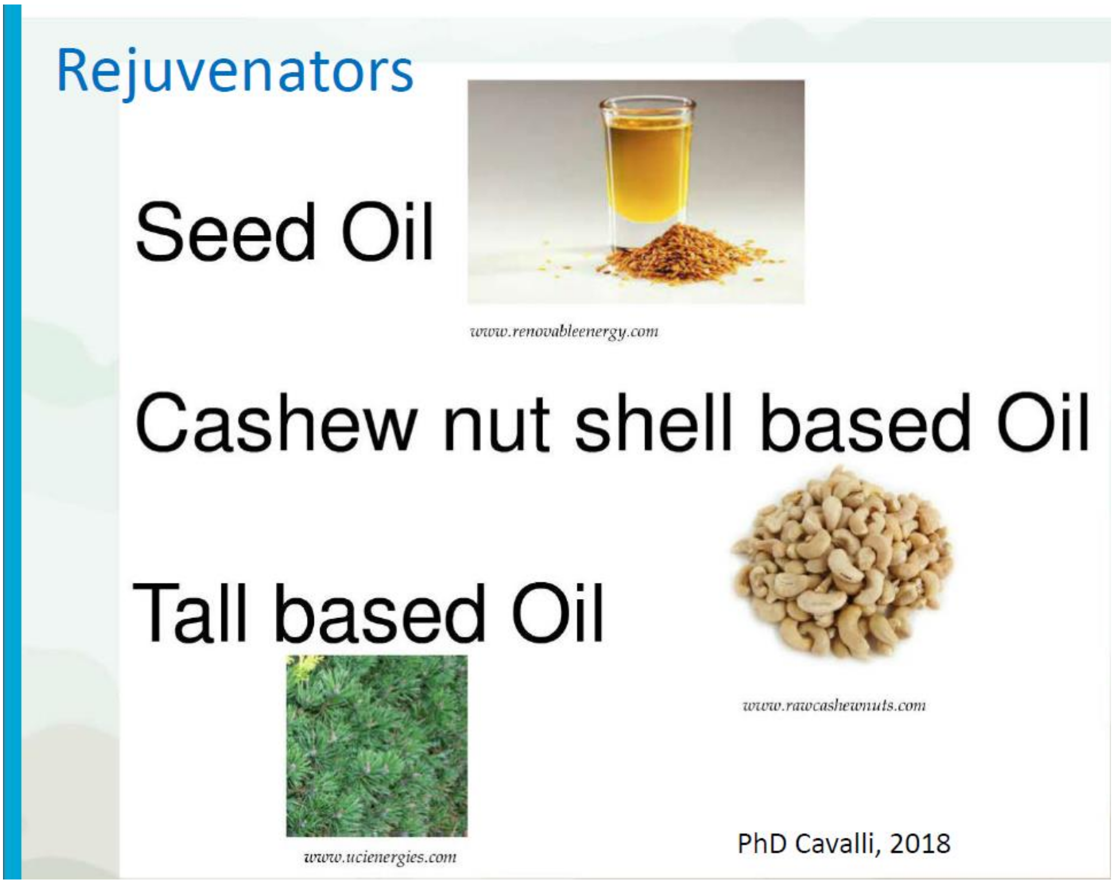
Motivation-Improve Properties

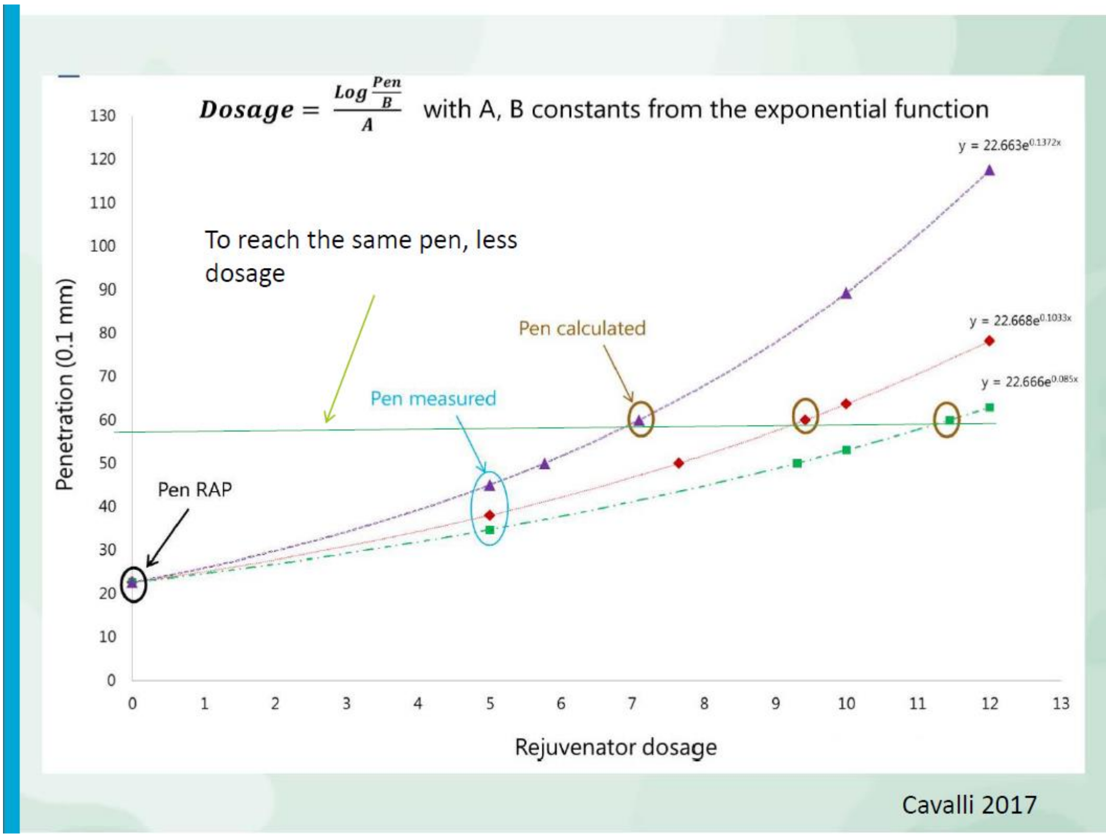
- Circular economy



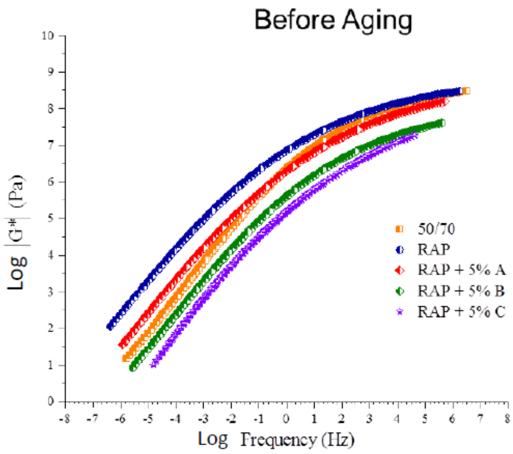
Piao 2020

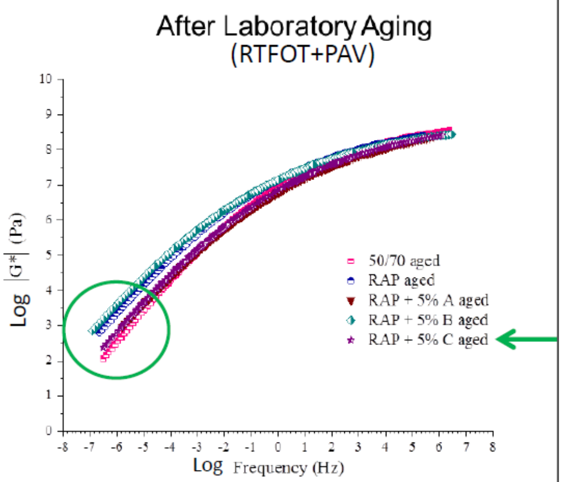
3


Rejuvenators
Seed Oil
www.renovableenergy.com
Cashew nut shell based Oil
Tall based Oil
www.rawcashewnuts.com
www.ucienergies.com
PhD Cavalli, 2018


$Dosage = \frac{Log\frac{Pen}{B}}{A}$ with A, B constants from the exponential function
y = 22.663e^0.1372x
y = 22.668e^0.1033x
y = 22.666e^0.085x
To reach the same pen, less dosage
Pen calculated
Pen measured
Pen RAP
Penetration (0.1 mm)
Rejuvenator dosage
Cavalli 2017

Effect of ageing on the Rheological properties (DSR)

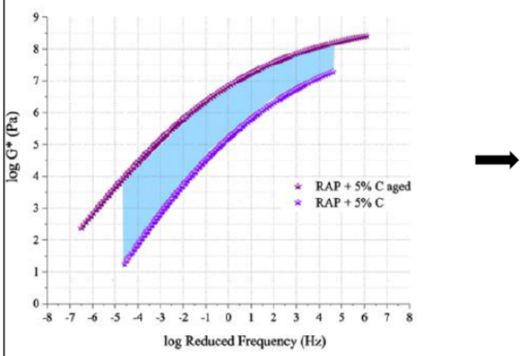


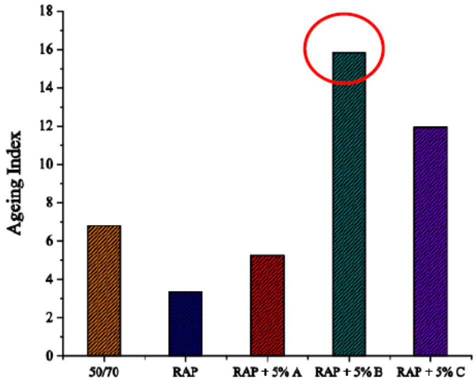


Cavalli MC, Zaumanis M, Mazza E, Partl MN, Poulikakos LD. Effect of ageing on the mechanical and chemical properties of binder from RAP treated with bio-based rejuvenators. Compos Part B Eng 2018;141:174–81. doi:10.1016/j.compositesb.2017.12.060.

Characterising Aging

Empa





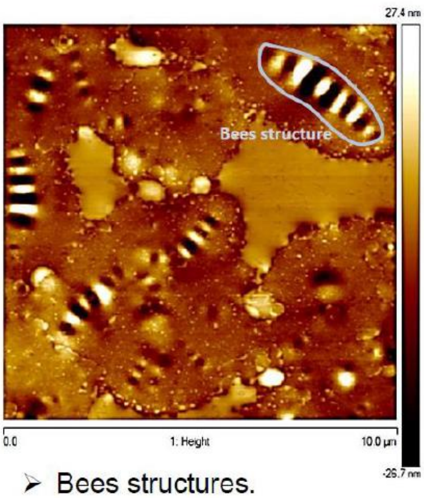
Rejuvenator type affects aging susceptibility

Cavalli MC, Zaumanis M, Mazza E, Partl MN, Poulikakos LD. Effect of ageing on the mechanical and chemical properties of binder from RAP treated with bio-based rejuvenators. Compos Part B Eng 2018;141:174–81. doi:10.1016/j.compositesb.2017.12.060.

Atomic Force Microscopy (AFM)

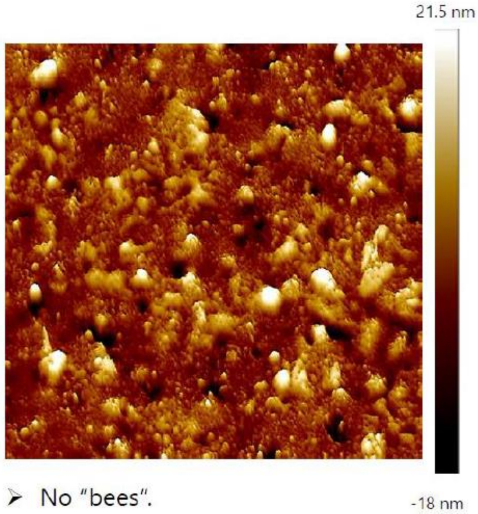
Empa
Materials Science and Technology

Virgin Binder 5070



- Bees structures.

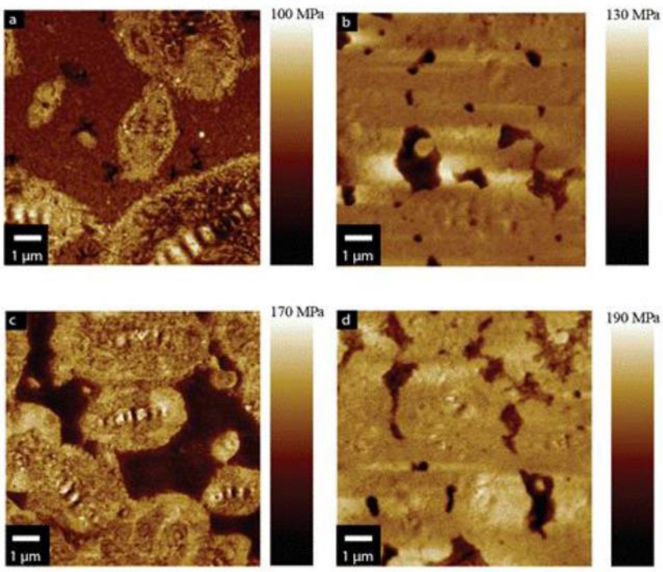
RAP Binder



- No "bees".

Cavalli, M.C., Zaumanis, M., Mazza, E., Partl, M.N., Poulikakos, L.D. Effect of ageing on the mechanical and chemical properties of binder from RAP treated with bio-based rejuvenators (2018) Composites Part B: Engineering, 141, pp. 174-181.

AFM QNM (Quantitative nano mechanical analysis)



a) Virgin binder 50/70 unaged

b) RAP binder unaged

c) Virgin binder 50/70 aged

d) RAP binder aged.

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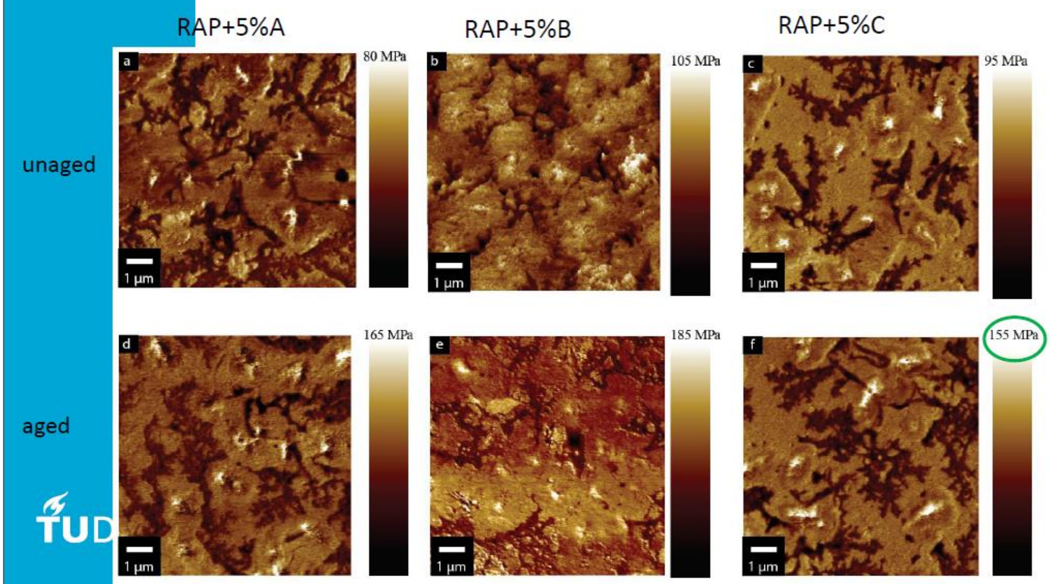
Source: Cavalli, M.C., Zaumanis, M., Mazza, E., Partl, M.N., Poulikakos, L.D. Effect of ageing on the mechanical and chemical properties of binder from RAP treated with bio-based rejuvenators (2018) Composites Part B: Engineering, 141, pp. 174-181.

Surface microstructure using AFM QNM sample size 10µmx10µm



Reached mechanical performance but no restoration of bees

Source: Cavalli, M.C., Zaumanis, M., Mazza, E., Partl, M.N., Poulikakos, L.D. Effect of ageing on the mechanical and chemical properties of binder from RAP treated with bio-based rejuvenators (2018) Composites Part B: Engineering, 141, pp. 174-181.

10

Use of waste materials as additives

11

The Numbers in Europe

Material	Source	Generated Mt/y	Available Mt/y
Concrete	C&D	350	262.5
Ceramics	C&D	200	162
Glass	Var.	20.2	5.4
Steel slag	Steel industry	21.8	15.7
Plastics	MRF	15.1	4.5
Tires	ELV	3.3	1.8

Waste is a substantial problem worldwide

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Technology Readiness Level (TRL)

FNSNF

Urban wastes	Estimated TRL
Crumb rubber (wet process)	7 – 9 (application is partially or completely industrialized)
Crumb rubber (dry process)	5 – 7 (application is validated in the field)
Recycled concrete aggregate (RCA)	< 4 (laboratory scale or lower)
Recycled ceramics	< 4 (laboratory scale or lower)
Recycled glass	< 4 (laboratory scale or lower)
Recycled plastics	5 – 7 (application is validated in the field)

Banke, J., 2017. Technology Readiness Levels Demystified. https://www.nasa.gov/topics/aeronautics/features/trl_demystified.html. (Accessed May 2019).
Piao, Z., Mikhailenko, P., Kakar, M.R., Bueno, M., Hellweg, S., Poulikakos, L.D., 2020. Urban Mining for Asphalt Pavement: A Review. Journal of Cleaner Production 280 (2021)

FNSNF

Suitable waste materials for roads

Table 2
Summary of the effects using waste materials in the asphalt mixture at laboratory scale.

Waste material	Rutting resistance	Moisture resistance	Stiffness modulus	Fatigue resi
CR from ELT (wet process)[a]	↑	≶	≶	↑
CR from ELT (dry process)[b]	↑	↓	↑	≶
RCA[c]	≶	≶	↑	≶
Waste ceramics[d]	≶	≶	↑	↑
Waste PE (wet process)[e]	↑	↑	↑	↑
Waste PE (dry process)[f]	↑	↑	≶	→
Waste PET[g]	↑	↑	≶	↑
Waste PVC[h]	↑	↑	×	×
Waste PP[i]	↑	≶	↑	→
Steel slag[j]	↑	↑	↑	↑

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Source: Piao, Z., Mikhailenko, P., Kakar, M.R., Bueno, M., Hellweg, S., Poulikakos, L.D., (2021). Urban mining for asphalt pavements: A review. Journal of Cleaner Production 280, 124916. https://doi.org/10.1016/j.jclepro.2020.124916.

rilem

Overview of Rilem TC Waste and Marginal Materials WMR

- Chair:Lily Poulikakos, Empa, Switzerland
- Emiliano Pasquini, U Padua, Italy
- The TC will be active for 5 years (2017-2022)
- Combination of literature review, experimental work and test protocol descriptions documented in a STAR report and papers
- Workshop 2020 Rilem symposium Lyon
- Symposium or dedicated TC day at end of TC

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Rilem: International Union of Laboratories and Experts in Construction Materials, Systems and Structures https://www.rilem.net/

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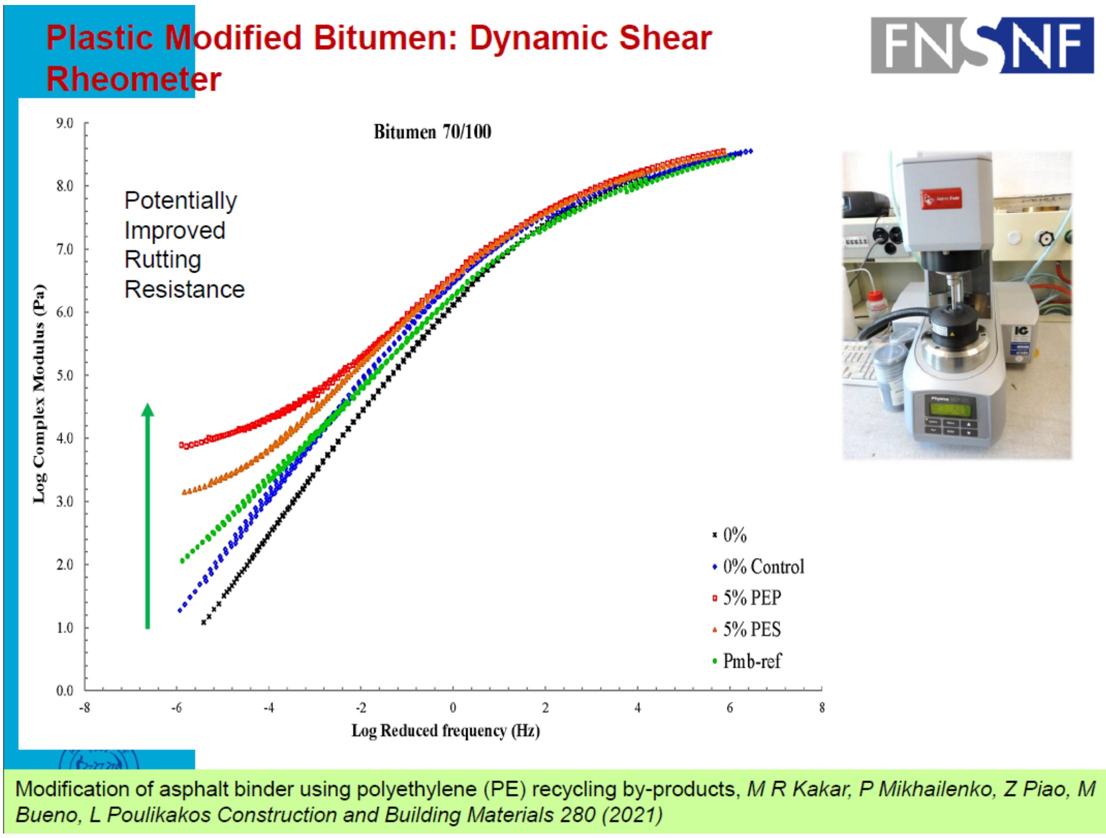
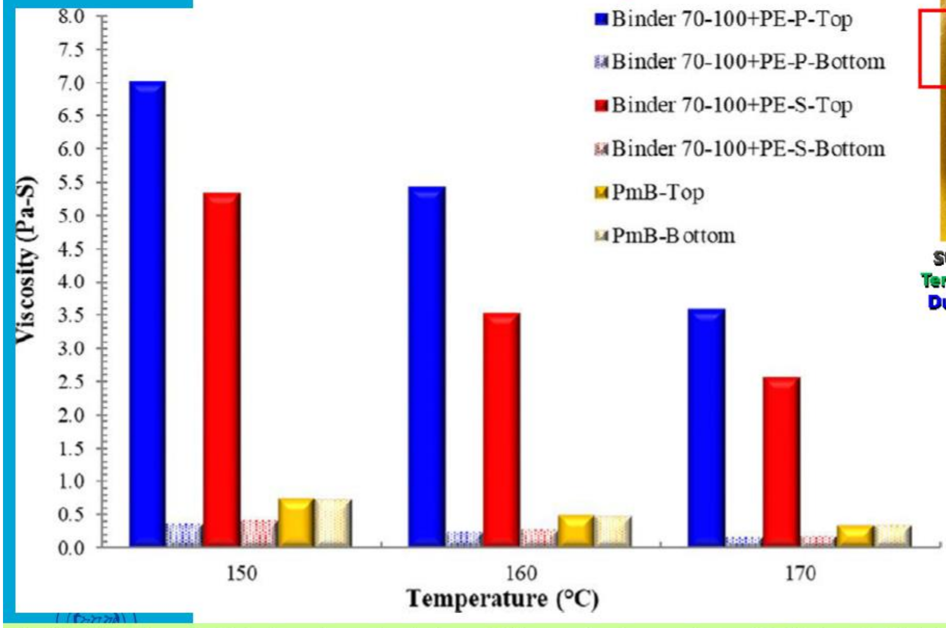
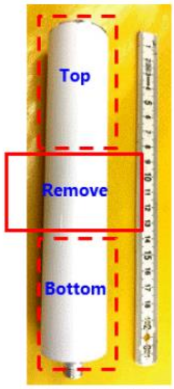
Waste Plastics
Inno-recycling produces 16’000 Tonnes/year of PE „Regranulate“.
20.02.2019
PE-
Pellets
PE-
Shreds
https://innorecycling.ch/
16


Plastic Modified Bitumen: Dynamic Shear Rheometer
FNSNF
Bitumen 70/100
Potentially
Improved
Rutting
Resistance
Log Complex Modulus (Pa)
Log Reduced frequency (Hz)
0%
0% Control
5% PEP
5% PES
Pmb-ref
Modification of asphalt binder using polyethylene (PE) recycling by-products, M R Kakar, P Mikhailenko, Z Piao, M Bueno, L Poulikakos Construction and Building Materials 280 (2021)

Plastics: Stability

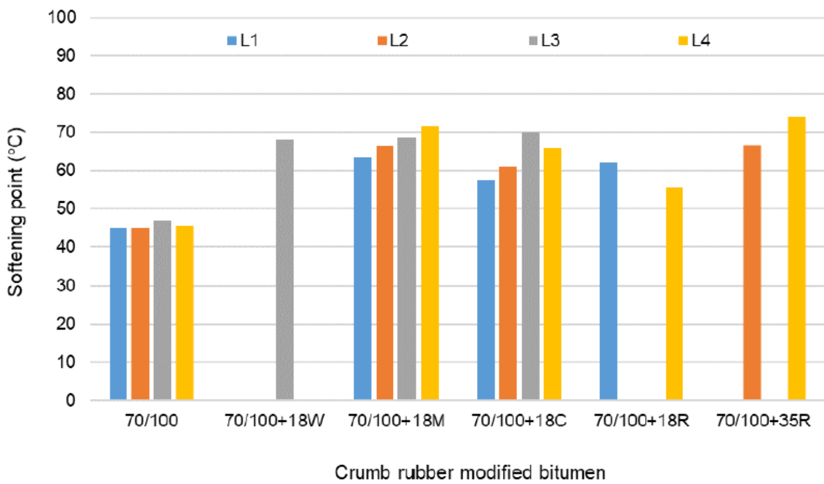




Stability Test Tube
Temperature = 180°C
Duration = 72hours

Modification of asphalt binder using polyethylene (PE) recycling by-products, *M R Kakar, P Mikhailenko, Z Piao, M Bueno, L Poulikakos Construction and Building Materials 280 (2021)*

Round Robin Crumb Rubber Modified Bitumen



Pais et al, submitted

W-Water jet
M-Mechanical grinding
C-Cryogenic
R-RARx

19

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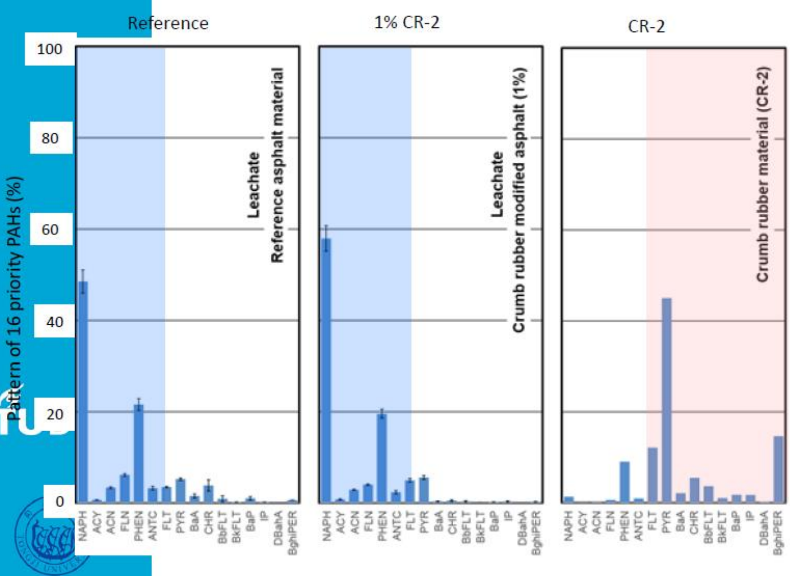
Environment/LCA

20

Comparison of PAH pattern in leachates: Crumb Rubber Modified Mixture

Pattern of 16 priority PAHs in leachates and CR-2 material



-Acidic solution18 h liquid-to-solid ratio 20 to 1

- Soluble 2-3-ring PAHs like naphthalene dominate in leachates
- Less soluble 4-6-ring PAHs dominate in CR-2

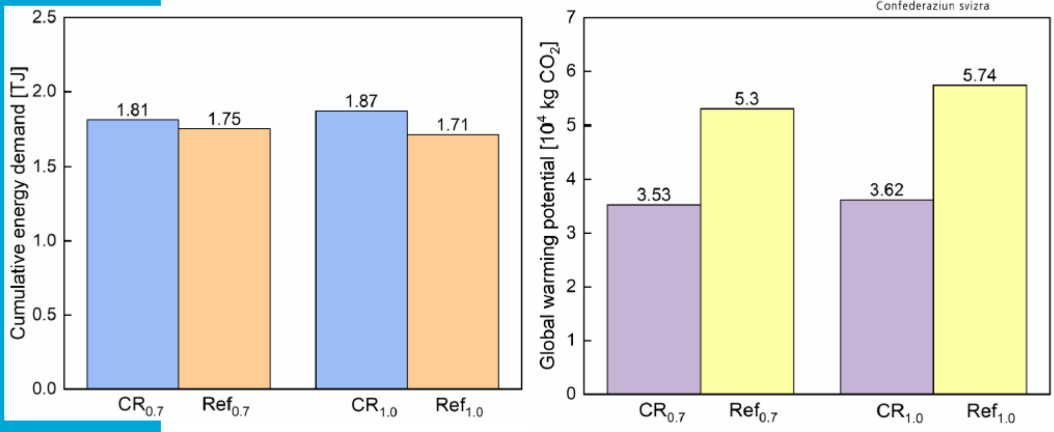
-Leaching of PAHs is asphalt-related

Norbert Heeb

Life Cycle Assessment (LCA): Crumb Rubber

Innosuisse

Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra



(a) (b)

TUDelft

SDA wearing courses. $CR_{0.7}$ =SDA + 0.7% CR, $CR_{1.0}$ =SDA + 1.0% CR; $Ref_{1.0}$ =SDA+ PmB CR

Z Piao, M Bueno, P Mikhailenko, M R Kakar, L Poulikakos, S Hellweg Life cycle assessment of asphalt pavements using crumb rubber: a comparative analysis, Rilem ISBN symposium Lyon 2020

Conclusions

- Additives can have a complex effect on the performance of binder
- Use of waste materials add a degree of complexity
- Appropriate characterization techniques
  - Focus on mixture performance
- Environmental effects
- LCA-cradel to grave, recycling potential

TUDelft

23


iFRAE DELFT 2021
5th International Symposium
on Frontiers of Road and Airport Engineering
Workshop: Changes in binder properties and the role of additives
Innosuisse
BAFU
FNSNF
Thank you!
Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra
rilem
TUDelft

3.5 Routes to durability and sustainability: Recycling of PmB containing RAP

Dr. Xueyan Liu, Associate professor

Delft University of Technology, The Netherlands

Abstract

Recycling of base asphalt pavement with rejuvenators or soft binders has been applied for many decades and a lot of experience has been gained. However, the high-quality recycling technique of polymer modified asphalt (PMA) has still not been sufficiently studied. The aging mechanism of PMA is complex, including the combined effects of the oxidation of bitumen and the degradation of the polymer. The current commercially available rejuvenators are designed mostly for base bitumen and not suitable for the recycling of PMA. For this reason, this research aims at designing an innovative rejuvenator specifically for the recycling of PMA. Firstly, a series of performance-based test methods, including viscoelastic properties, rutting resistance, fatigue resistance, cracking resistance, relaxation ability and aging ability, has been performed to select the appropriate source materials and to determine the optimum ratio between different components. After that, a specific SBS-based rejuvenator was found to be most effective in PMA rejuvenation. To reveal the rejuvenation mechanism, an environment scanning electron microscope (E-SEM) was utilized in investigating the microstructure of the rejuvenated binder. The results illustrated that E-SEM method can distinguish the influence of rejuvenator dosage, rejuvenator types, and addition of fresh bitumen on the morphology of aged PMB, which can help us to have a better understanding of the rejuvenation mechanism.

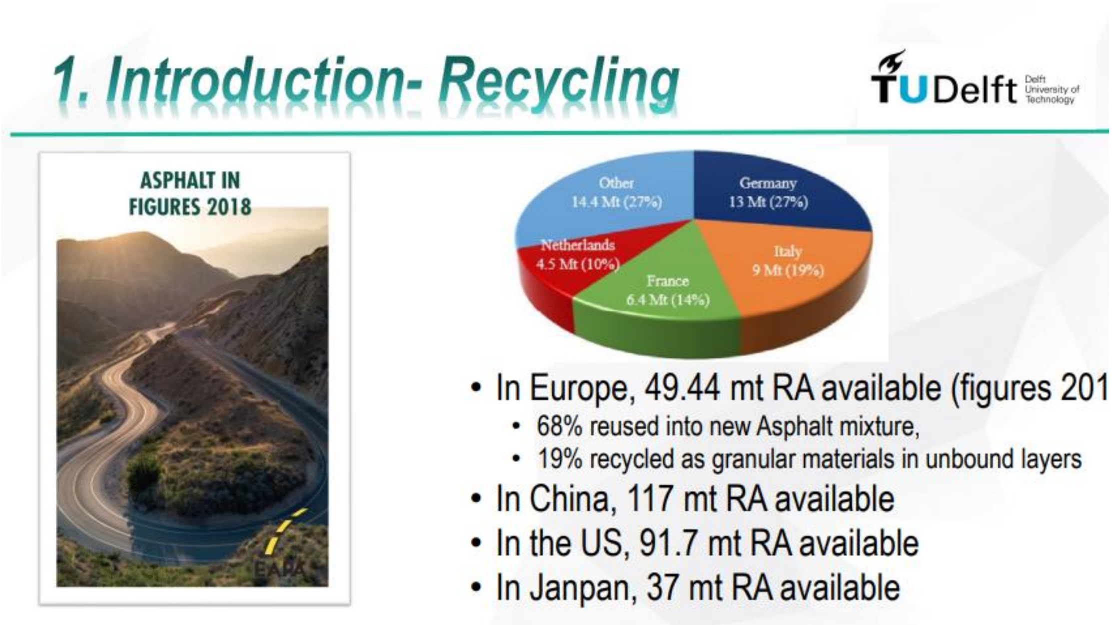
About the speaker

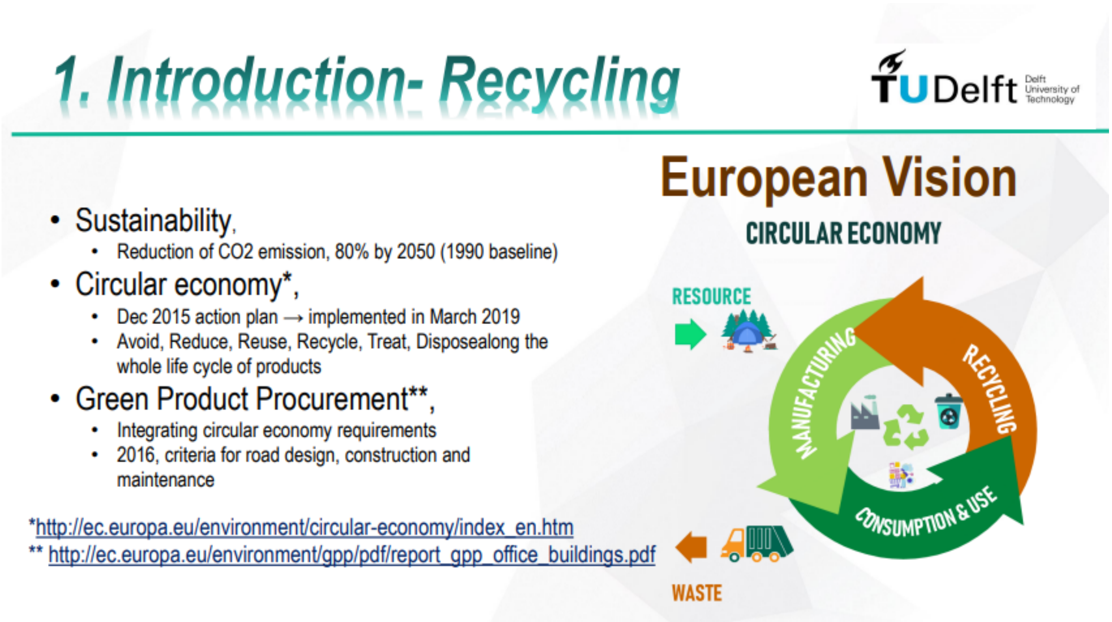
Dr. Xueyan Liu is currently an associate Professor in the Section of Pavement Engineering of the Faculty of Civil Engineering & Geosciences of TU Delft. He works in the areas of constitutive modelling, numerical modelling and material experimental characterization. Within the research program of the Section Pavement Engineering, his research topics mostly relate to the development and implementation of constitutive models for the simulation of the static and dynamic response of various pavement engineering materials like soils, asphalt concrete, liner and reinforcing systems etc. and sustainable development technologies, i.e., multiscale modelling of asphaltic materials, warm/cold asphalt concrete technology, durability of asphalt surfacings on orthotropic steel deck bridge, accelerated pavement test, pavement continuous monitoring and sustainable development technologies. Dr. Liu was granted his doctoral thesis in 2003. During the same period, Dr. Liu participated also in the team that developed the ACRe model for Asphalt Concrete Response currently implemented in 3D Computer Aided Pavement Analysis system (CAPA-3D).

Dr. Liu has published more than 100 technical and journal papers on the mechanics and the finite element modelling of granular, concrete and asphaltic materials. Dr. Liu is a member of RILEM Technical Committee 272-PIM Phase and Interphase behaviour of Bituminous Materials and a member of Delft Centre for Materials (DCMat). He is also a member of ISAP, AAPT, APSE and IACMAG. Dr. Liu is an Editorial Board Member of Geomaterials (GM). Dr. Liu was appointed as Board member of the International Association of Chinese Infrastructure Professionals (IACIP) and member of the Academic Committee of the Key Laboratory of Road Structure and Materials Transportation Industry of the China Ministry of Transport. He is also actively involved in organizing inter/national workshops and conferences and was invited as Scientific/Technical committee member of several international conferences.


iFRAE
DELFT 2021
5th International Symposium
on Frontiers of Road and Airport Engineering
Workshop: Changes in binder properties and the role of additives
Routes to durability and sustainability: Recycling of PmB containing RAP
Xueyan Liu, Peng Lin, Sandra Erkens
Delft University of Technology, the Netherlands
TUDelft


01
Introduction


1. Introduction- Recycling
TUDelft Delft University of Technology
ASPHALT IN FIGURES 2018
EAPA
Other 14.4 Mt (27%)
Germany 13 Mt (27%)
Netherlands 4.5 Mt (10%)
Italy 9 Mt (19%)
France 6.4 Mt (14%)
• In Europe, 49.44 mt RA available (figures 201
• 68% reused into new Asphalt mixture,
• 19% recycled as granular materials in unbound layers
• In China, 117 mt RA available
• In the US, 91.7 mt RA available
• In Janpan, 37 mt RA available


1. Introduction- Recycling
TUDelft Delft University of Technology
• Sustainability,
• Reduction of CO2 emission, 80% by 2050 (1990 baseline)
• Circular economy*,
• Dec 2015 action plan → implemented in March 2019
• Avoid, Reduce, Reuse, Recycle, Treat, Disposealong the whole life cycle of products
• Green Product Procurement**,
• Integrating circular economy requirements
• 2016, criteria for road design, construction and maintenance
*http://ec.europa.eu/environment/circular-economy/index_en.htm
** http://ec.europa.eu/environment/gpp/pdf/report_gpp_office_buildings.pdf
European Vision
CIRCULAR ECONOMY
RESOURCE
MANUFACTURING
RECYCLING
CONSUMPTION & USE
WASTE

1. Introduction- Recycling

Asphalt Recycling in the Netherlands

- 71% of the RAP is used in HMA and WMA recycling
- 11% of the RAP is used in Cold recycling
- 18% RAP is used in other applications.

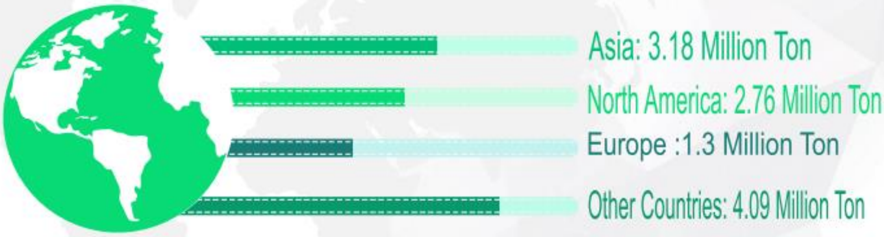
New goals In the Netherlands

- 50% CO_2 and raw material reduction in 2030
- Be circular (no waste, no use of raw materials) in 2050

1. Introduction- Marketing

TU Delft Delft University of Technology

PMB Market Distribution



PMB in Global Market

More than 80% of PMB was used in pavement construction.

Global Polymer Modified Bitumen Market Professional Survey Report 2018

1. Introduction- Challenge

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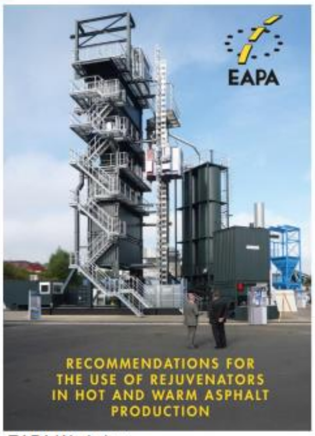

EAPA Workshop
Use of rejuvenators in asphalt mixtures
Padova (Italy), 10th - 11th September 2019

1. INTRODUCTION

Now

The reuse of RAP with standard bituminous binders up to 50 % addition rates is successfully achieved.

Challenges

Over recent years more PMB has been used in asphalt production. However, 'standard technology' may not be adequate for recycling such kind of RAP.

additives restore the rheological behaviour of the aged binder from reclaimed asphalt when blent with the (pre-heated) reclaimed asphalt. Rejuvenators also can avoid the need of using a soft paving grade bitumen to meet the requirements.

1. Introduction- Goal

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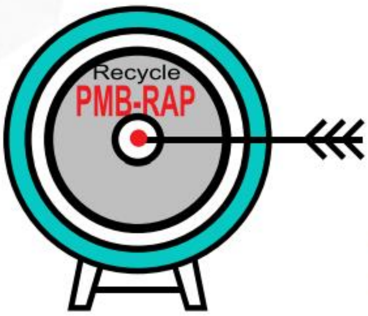


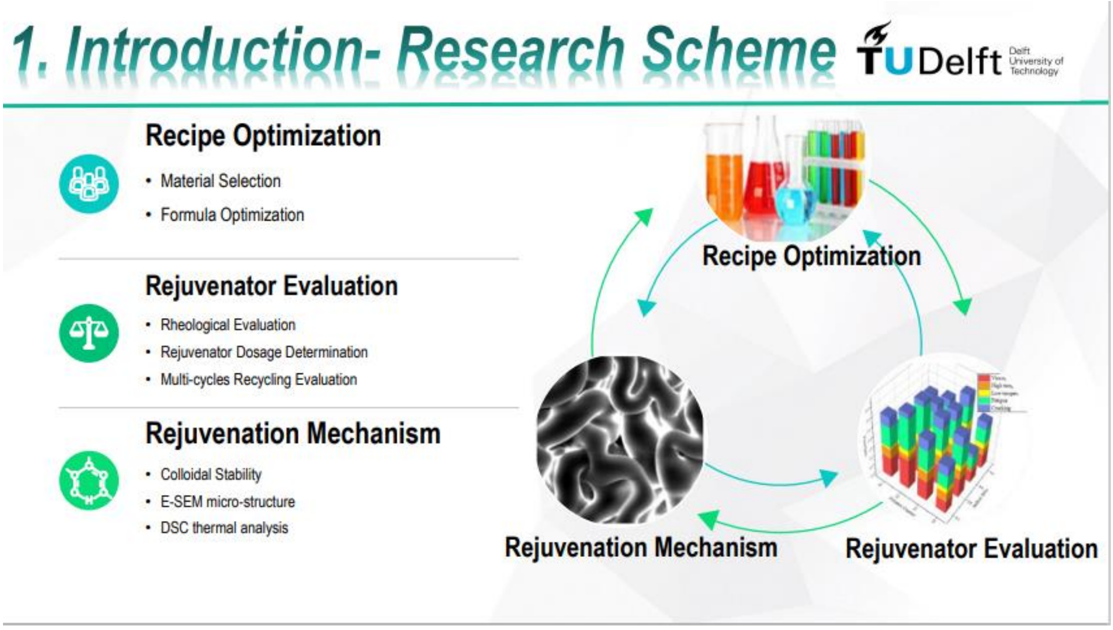
High –quality Recycling of PMB-RAP

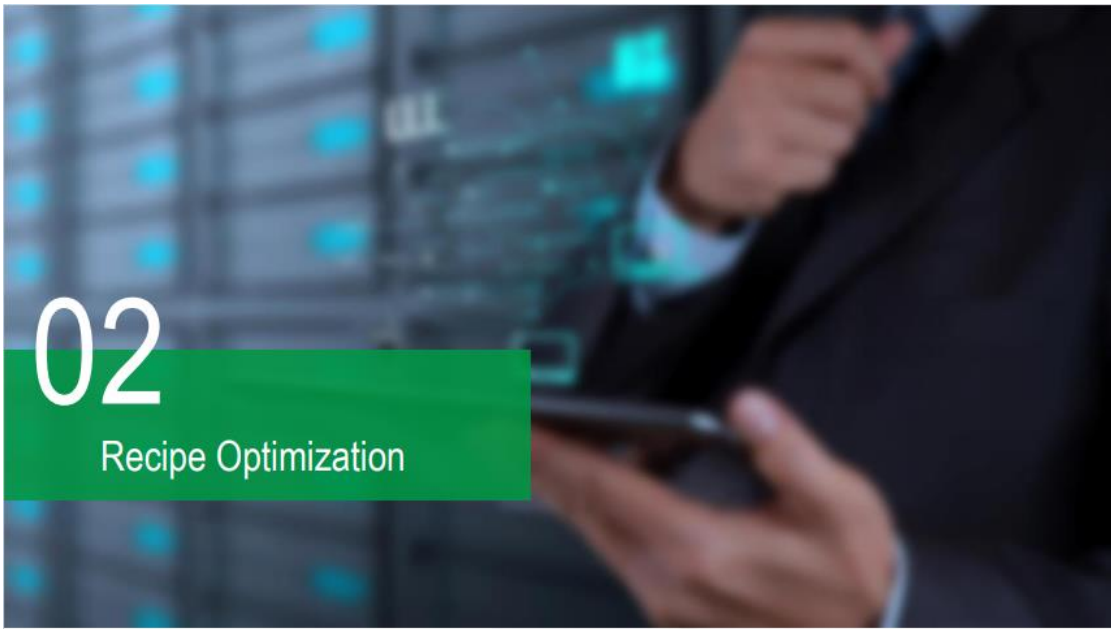
Improve Quality

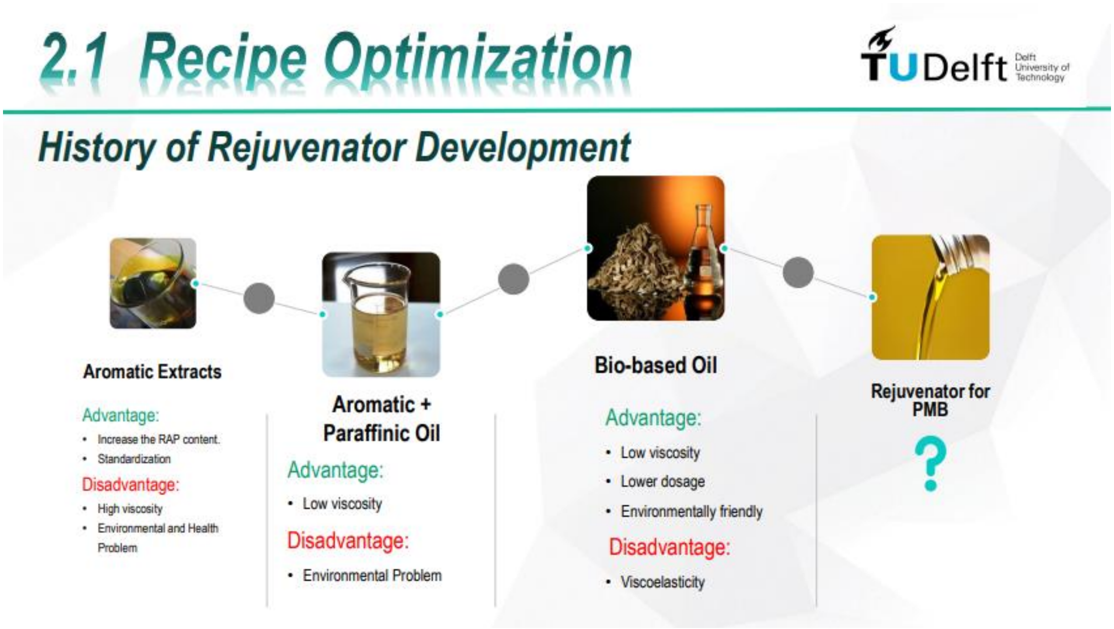
- **Activate** the **aged PMB in** the RAP
- **Fully use** the remaining polymer
- **Longer service life** of pavement

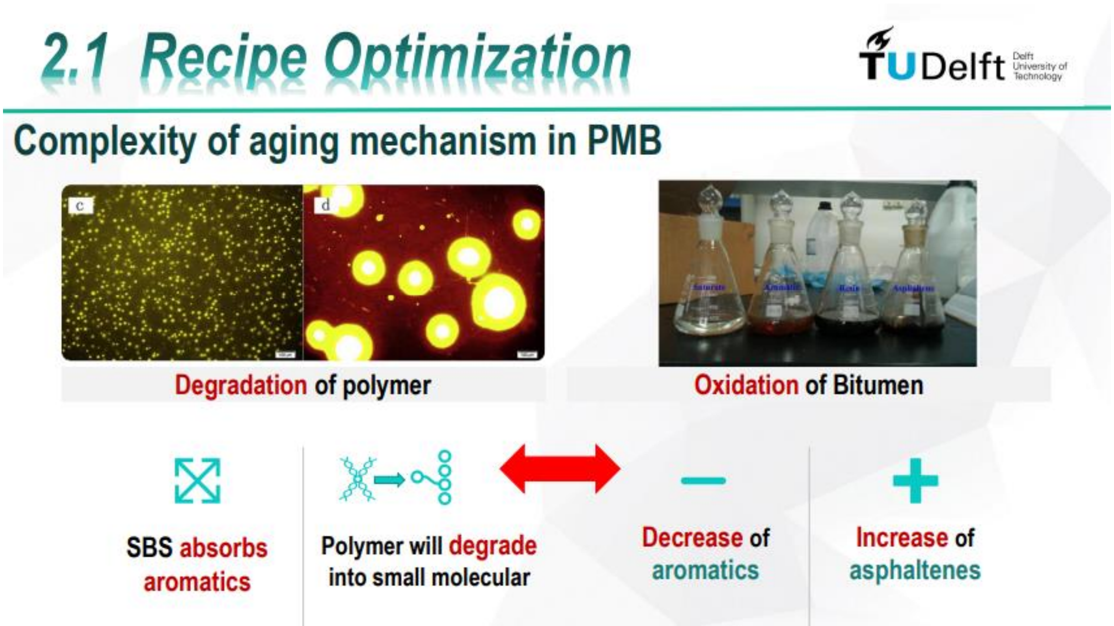
Increase Profit

- **Increase** the **PMB-RAP content**
- **Societal benefits**
- **Decrease the release of CO_2**


1. Introduction- Research Scheme
TUDelft Delft University of Technology
Recipe Optimization
• Material Selection
• Formula Optimization
Rejuvenator Evaluation
• Rheological Evaluation
• Rejuvenator Dosage Determination
• Multi-cycles Recycling Evaluation
Rejuvenation Mechanism
• Colloidal Stability
• E-SEM micro-structure
• DSC thermal analysis
Recipe Optimization
Rejuvenation Mechanism
Rejuvenator Evaluation


02
Recipe Optimization


2.1 Recipe Optimization
TUDelft Delft University of Technology
History of Rejuvenator Development
Aromatic Extracts
Advantage:
• Increase the RAP content.
• Standardization
Disadvantage:
• High viscosity
• Environmental and Health Problem
Aromatic + Paraffinic Oil
Advantage:
• Low viscosity
Disadvantage:
• Environmental Problem
Bio-based Oil
Advantage:
• Low viscosity
• Lower dosage
• Environmentally friendly
Disadvantage:
• Viscoelasticity
Rejuvenator for PMB
?


2.1 Recipe Optimization
TUDelft Delft University of Technology
Complexity of aging mechanism in PMB
c
d
Degradation of polymer
Oxidation of Bitumen
SBS absorbs aromatics
Polymer will degrade into small molecular
Decrease of aromatics
Increase of asphaltenes

2.1 Recipe Optimization

TUDelft Delft University of Technology

Challenges in rejuvenation of PMB

1. Hardening due to the oxidation of base bitumen
2. Property loss due to the degradation of polymer
3. Colloidal stability loss in the recycled PMB

2.1 Recipe Optimization

TUDelft Delft University of Technology

Procedure of recipe optimization

Materials Selection

Determination of component ratio in rejuvenator

Mechanical property characterization


Material Selection
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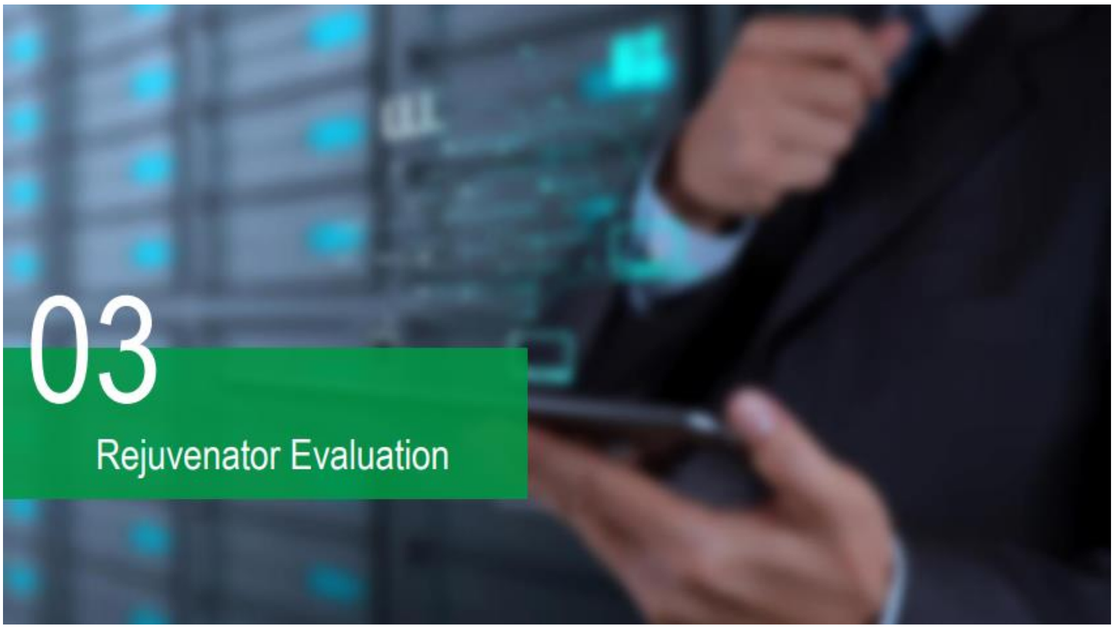
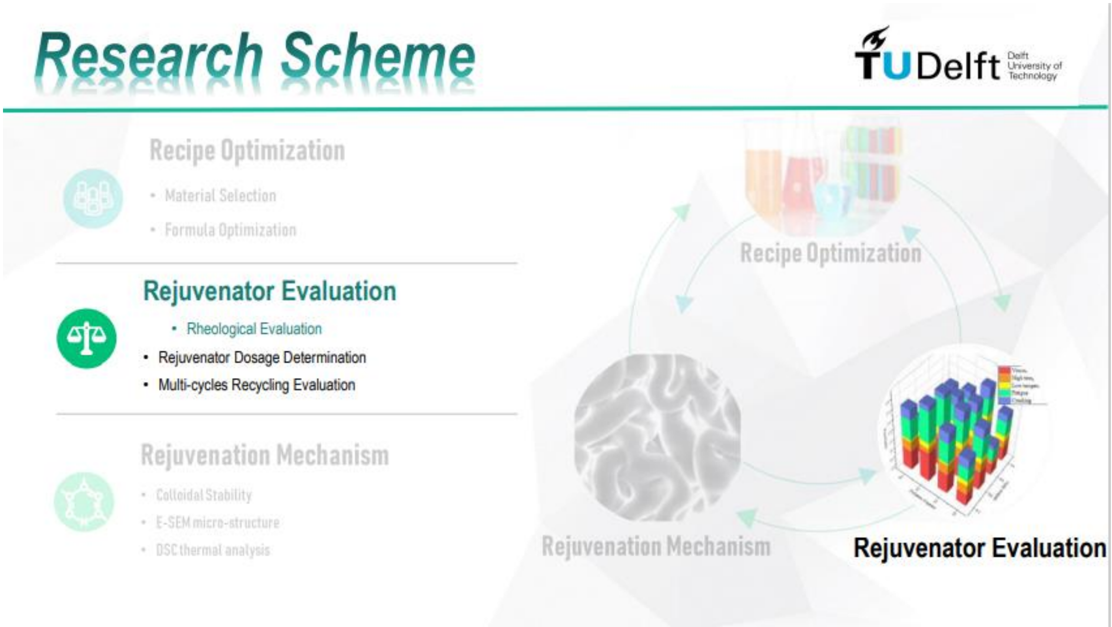
EBA-based Rejuvenator
A
SBS-based Rejuvenator
B
Terminal blend (TB)-based Rejuvenator
C
TU Delft
TB/SBS-based Rejuvenator
D
Bio-based Rejuvenator
Rich in Saturates
E
Aromatic-based Rejuvenator
Rich in Aromatics
F


03
Rejuvenator Evaluation


Research Scheme
TUDelft Delft University of Technology
Recipe Optimization
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• Colloidal Stability
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• DSC thermal analysis
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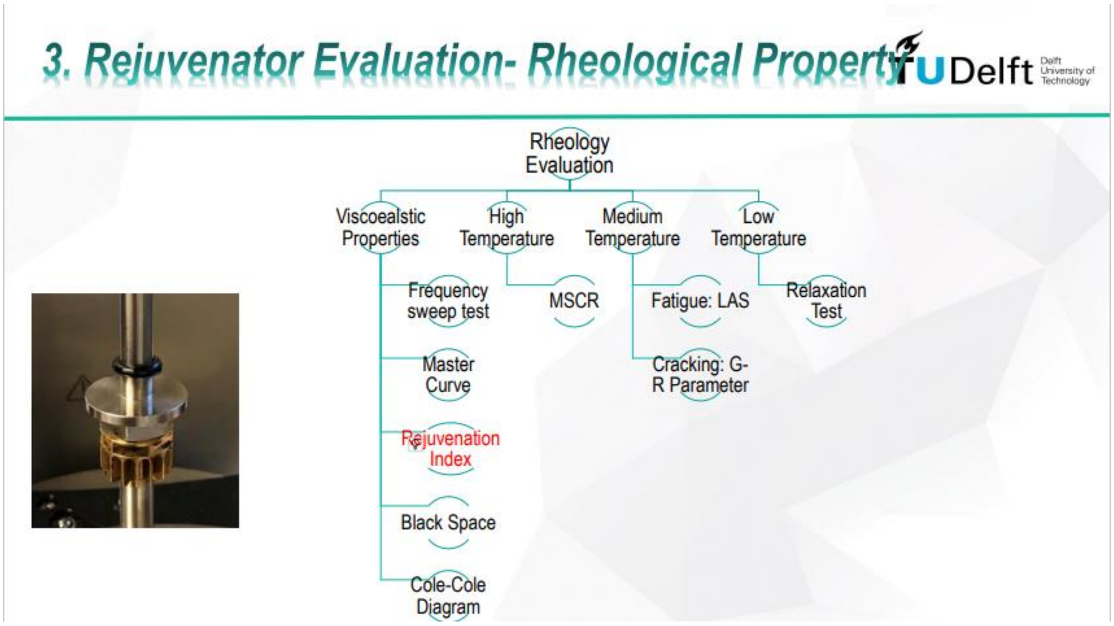
Rejuvenator Evaluation


3. Rejuvenator Evaluation- Rheological Property
TUDelft Delft University of Technology
Rheology Evaluation
Viscoealstic Properties
High Temperature
Medium Temperature
Low Temperature
Frequency sweep test
MSCR
Fatigue: LAS
Relaxation Test
Master Curve
Cracking: G-R Parameter
Rejuvenation Index
Black Space
Cole-Cole Diagram

3. Rejuvenator Evaluation- Rheological Property

Rejuvenation Indexes

$$A_{MH} = \int_{0}^{4} \log G^*(\xi) d\xi$$

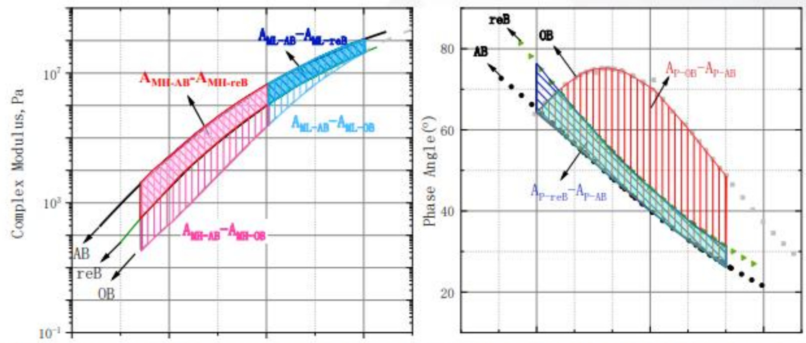
$$A_{ML} = \int_{-5}^{0} \log G^*(\xi) d\xi$$

$$A_P = \int_{-5}^{4} \delta(\xi) d\xi$$

$$I_{MH} = \frac{A_{MH-AB} - A_{MH-reB}}{A_{MH-AB} - A_{MH-OB}} \times 100\%$$

$$I_{ML} = \frac{A_{ML-AB} - A_{ML-reB}}{A_{ML-AB} - A_{ML-OB}} \times 100\%$$

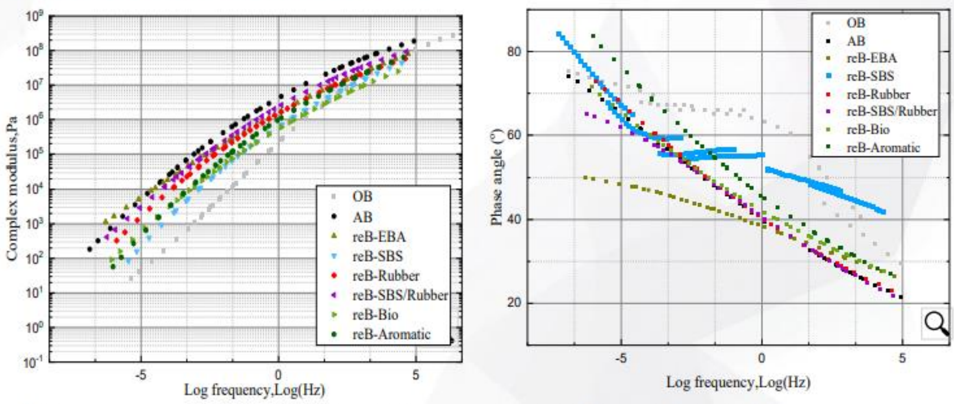
$$I_P = \frac{A_{P-reB} - A_{P-AB}}{A_{P-OB} - A_{P-AB}} \times 100\%$$



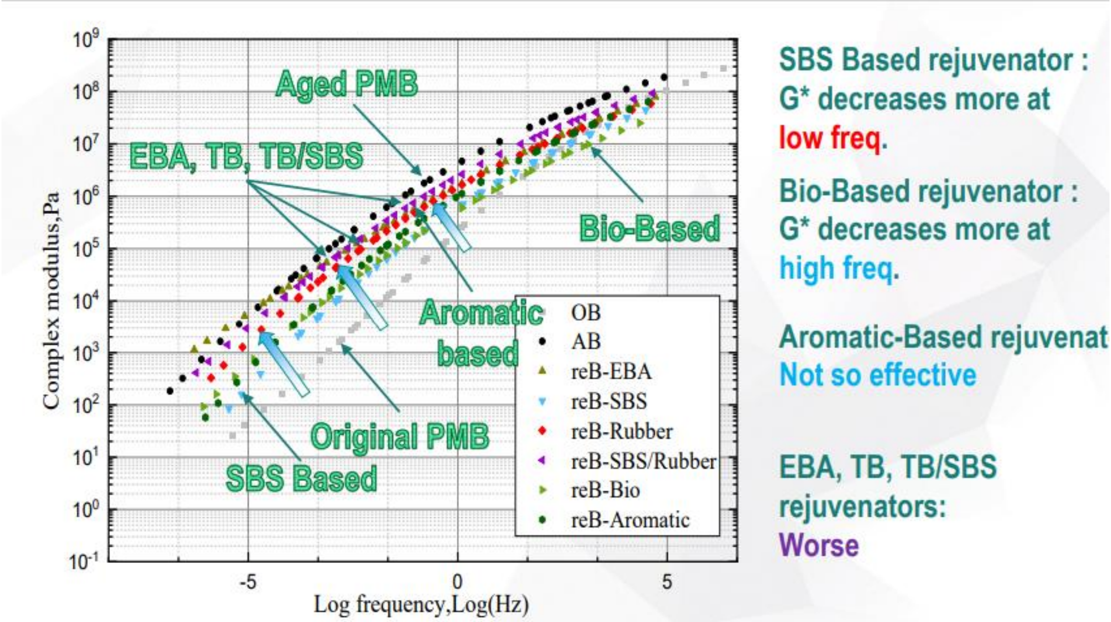
Indexes were defined as the ratio of the integral area difference of G* & δ between aged and rejuvenated bitumen in the selected frequency ra

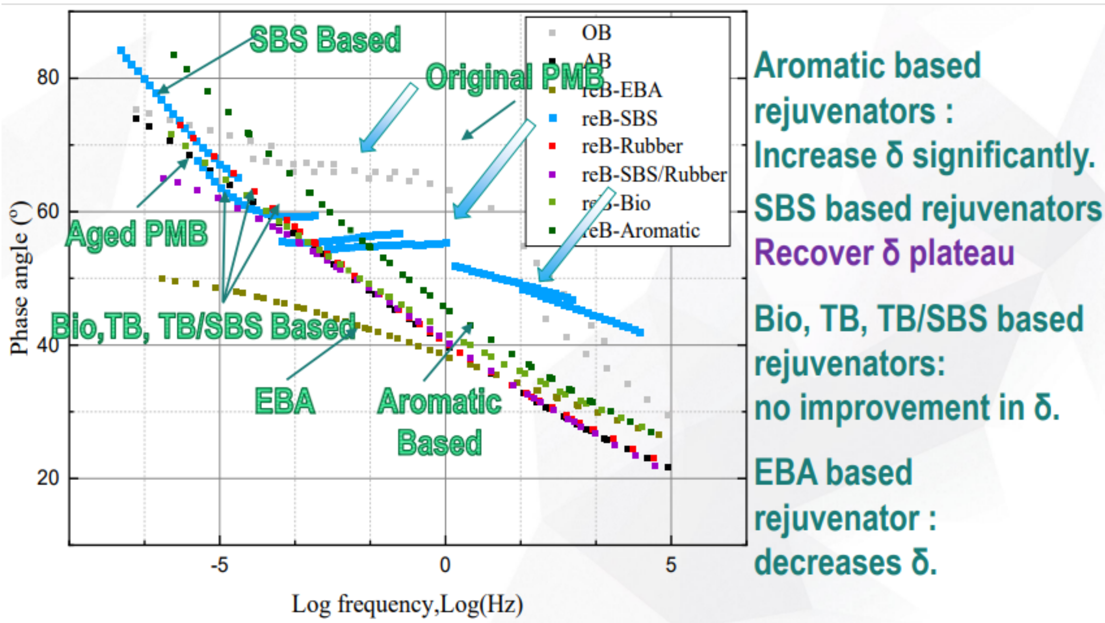
3. Rejuvenator Evaluation- Rheological Property

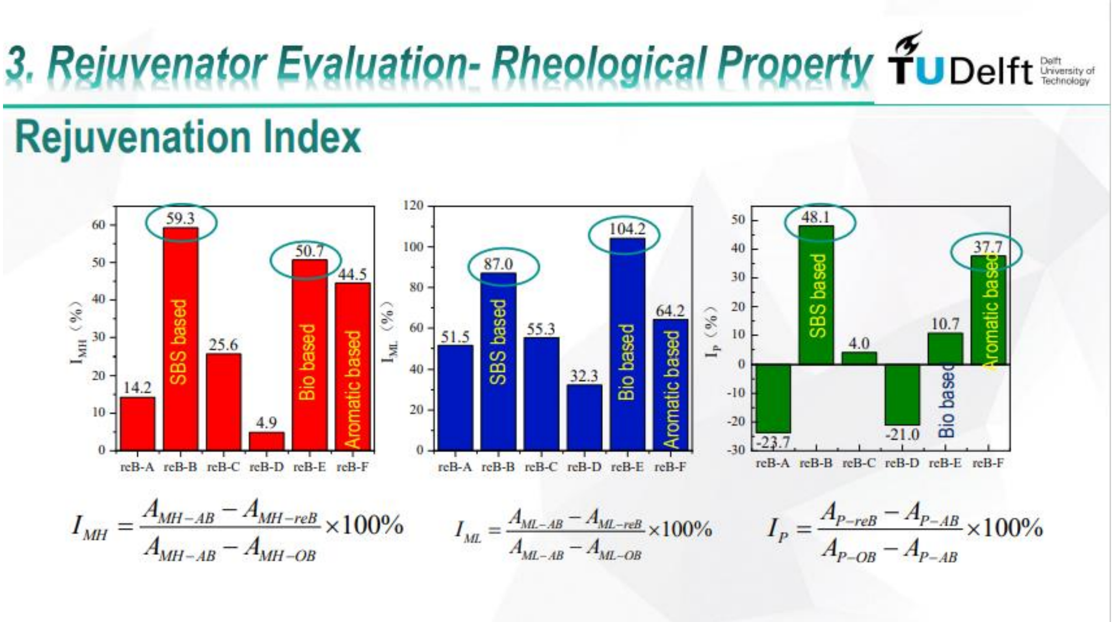
Viscoelastic-Master Curves



DSR frequency sweep tests between 0.01 and 10 Hz were performed at 0, 20, 30, 40, 60 and 80ºC
the reference temperature is 30 ºC.


Complex modulus,Pa
Log frequency,Log(Hz)
Aged PMB
EBA, TB, TB/SBS
Bio-Based
Aromatic based
Original PMB
SBS Based
OB
AB
reB-EBA
reB-SBS
reB-Rubber
reB-SBS/Rubber
reB-Bio
reB-Aromatic
SBS Based rejuvenator : G* decreases more at low freq.
Bio-Based rejuvenator : G* decreases more at high freq.
Aromatic-Based rejuvenat
Not so effective
EBA, TB, TB/SBS rejuvenators: Worse


Phase angle (°)
Log frequency,Log(Hz)
SBS Based
Original PMB
Aged PMB
Bio,TB, TB/SBS Based
EBA
Aromatic Based
OB
AB
reB-EBA
reB-SBS
reB-Rubber
reB-SBS/Rubber
reB-Bio
reB-Aromatic
Aromatic based rejuvenators : Increase δ significantly.
SBS based rejuvenators Recover δ plateau
Bio, TB, TB/SBS based rejuvenators: no improvement in δ.
EBA based rejuvenator : decreases δ.


3. Rejuvenator Evaluation- Rheological Property
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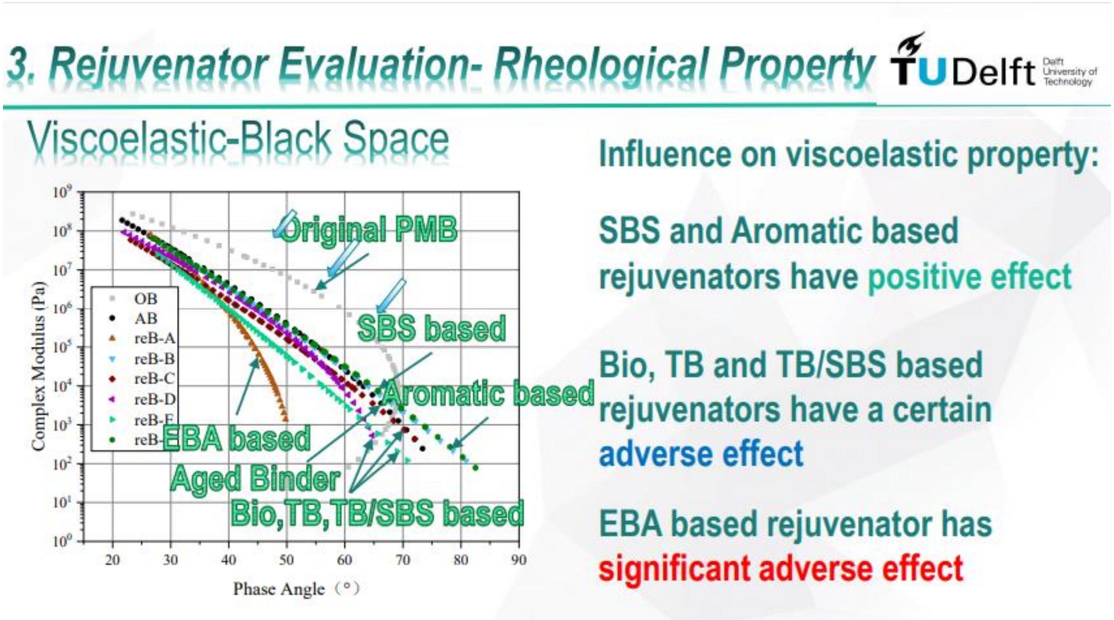
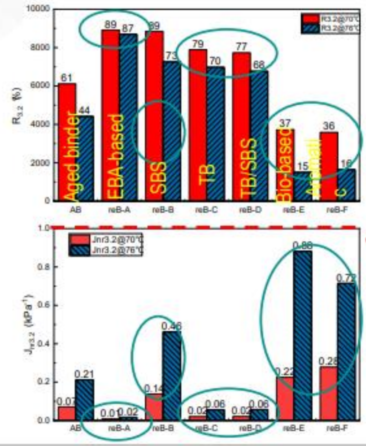
Delft University of Technology
Rejuvenation Index
59.3
SBS based
25.6
14.2
4.9
50.7
Bio based
44.5
Aromatic based
I_{MH} (%)
reB-A reB-B reB-C reB-D reB-E reB-F
51.5
87.0
SBS based
55.3
32.3
104.2
Bio based
64.2
Aromatic based
I_{ML} (%)
48.1
SBS based
4.0
-23.7
-21.0
10.7
Bio based
37.7
Aromatic based
I_P (%)
$I_{MH}=\frac{A_{MH-AB}-A_{MH-reB}}{A_{MH-AB}-A_{MH-OB}}\times100\%$
$I_{ML}=\frac{A_{ML-AB}-A_{ML-reB}}{A_{ML-AB}-A_{ML-OB}}\times100\%$
$I_P=\frac{A_{P-reB}-A_{P-AB}}{A_{P-OB}-A_{P-AB}}\times100\%$


3. Rejuvenator Evaluation- Rheological Property
TU Delft
Delft University of Technology
Viscoelastic-Black Space
Original PMB
SBS based
Aromatic based
EBA based
Aged Binder
Bio,TB,TB/SBS based
OB
AB
reB-A
reB-B
reB-C
reB-D
reB-E
Complex Modulus (Pa)
Phase Angle (°)
Influence on viscoelastic property:
SBS and Aromatic based rejuvenators have positive effect
Bio, TB and TB/SBS based rejuvenators have a certain adverse effect
EBA based rejuvenator has significant adverse effect

3. Rejuvenator Evaluation- Rheological Property

igh Temperature Properties

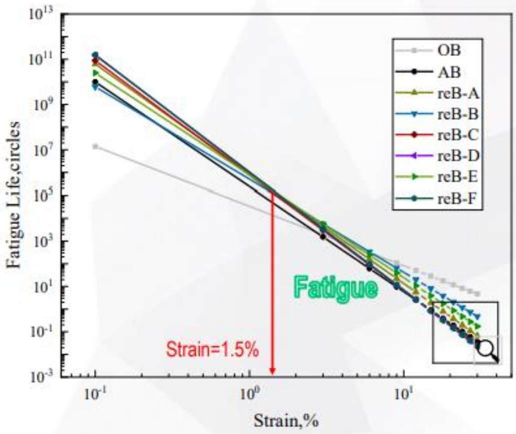


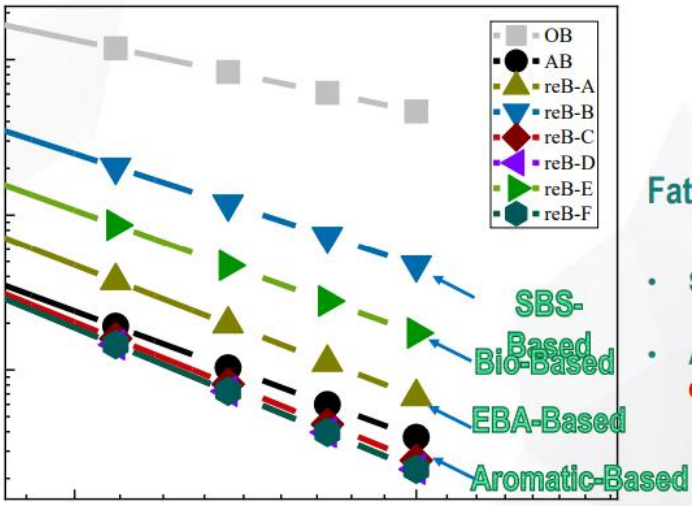
- EBA, TB, TB/SBS based rejuvenators have advantages in MSCR results.
- SBS based rejuvenator shows better strain recovery capacity compared with the reference rejuvenators.
- $J_{nr3.2}$ values of all binders at 76℃ are much lower than the maximum allowable value (2 kPa^{-1}) for the 'H' traffic level.

3. Rejuvenator Evaluation- Rheological Property

edium Temperature-Fatigue

LAS Test @ 10Hz & 20 ℃





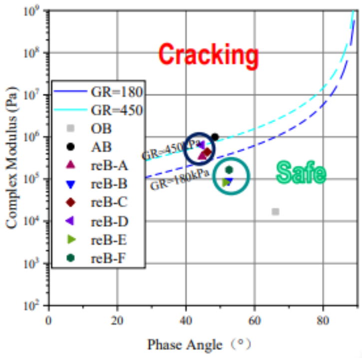
Fatigue resistance :

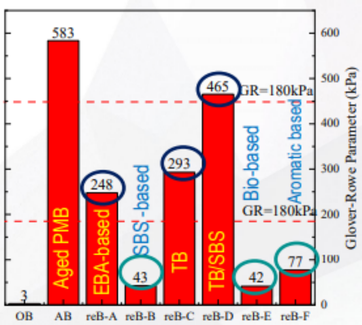
- SBS based > Bio-based > EBA base
- Aromatic, TB, TB/SBS have **adverse effect**

3. Rejuvenator Evaluation- Rheological Property

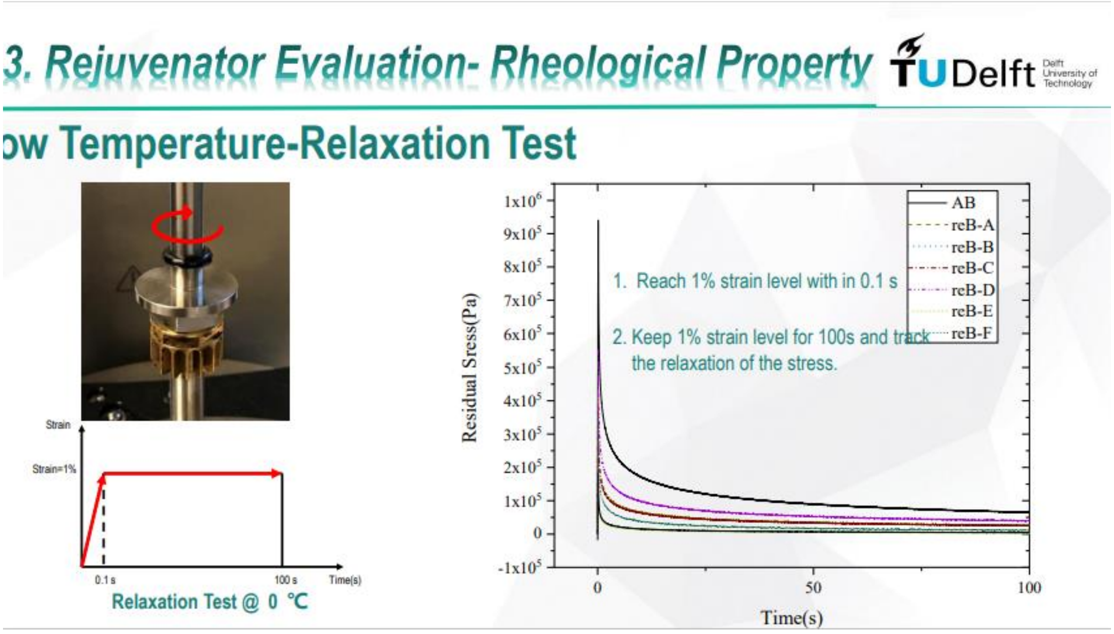
TU Delft Delft University of Technology

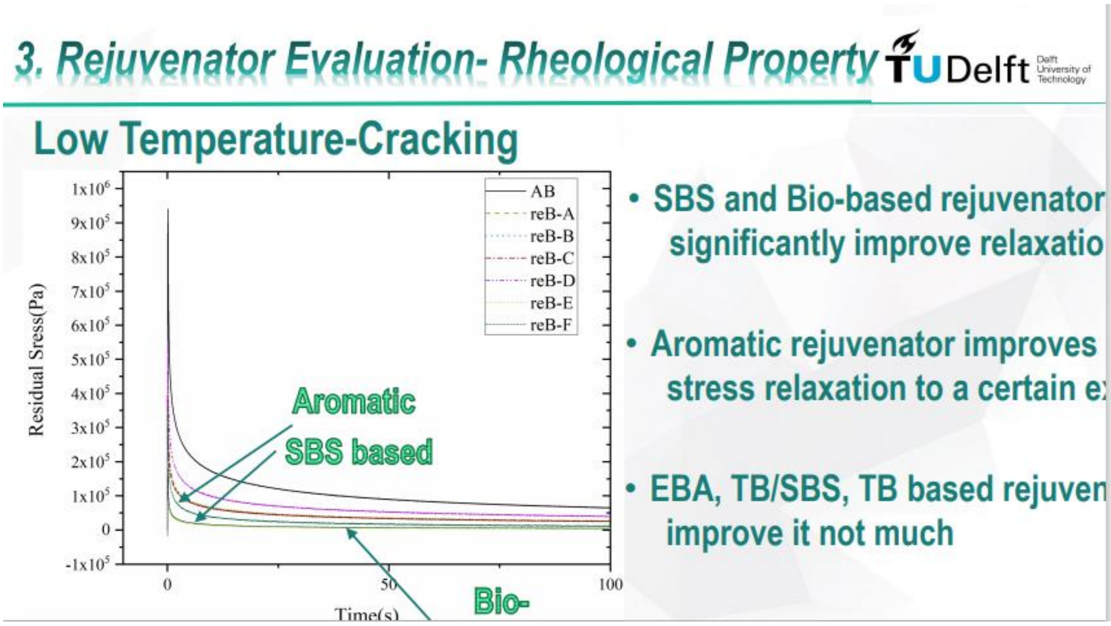
Medium Temperature-Cracking evaluation with G-R parameter

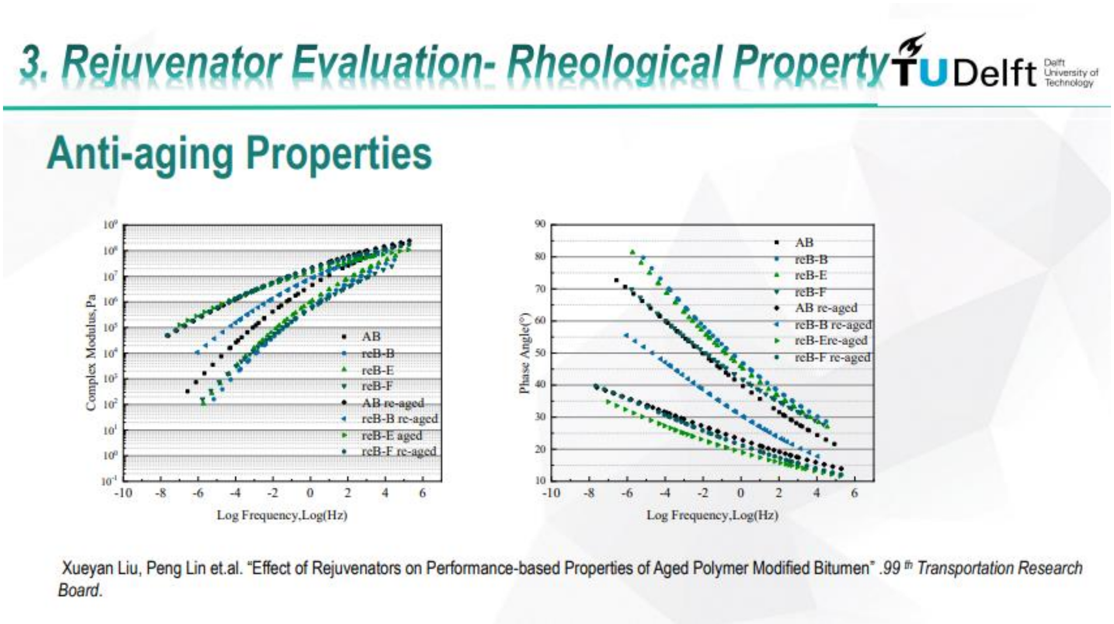


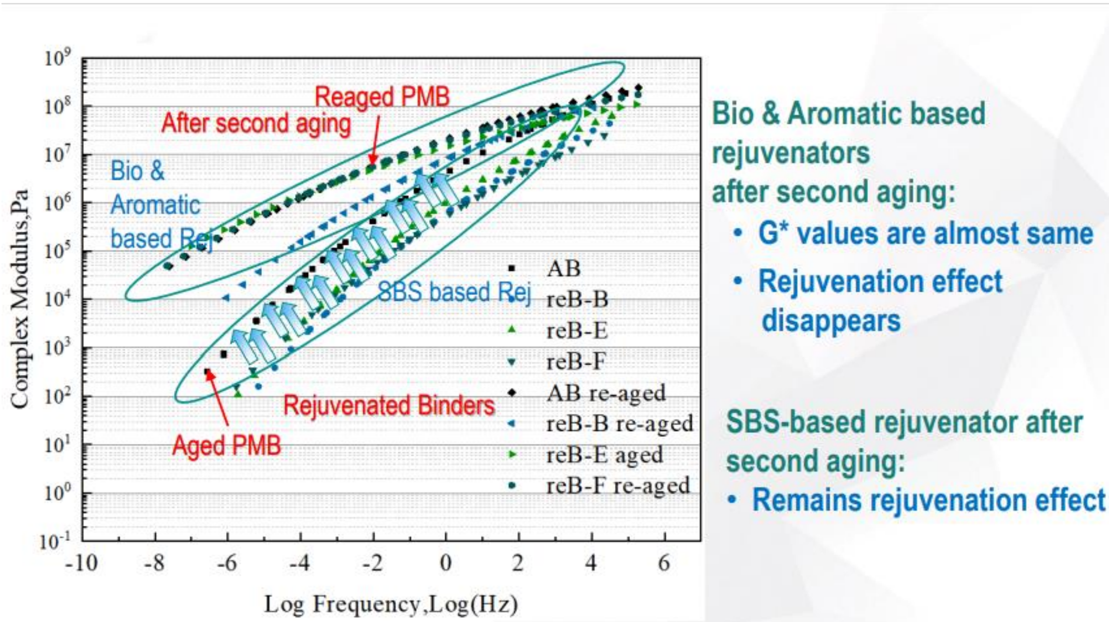


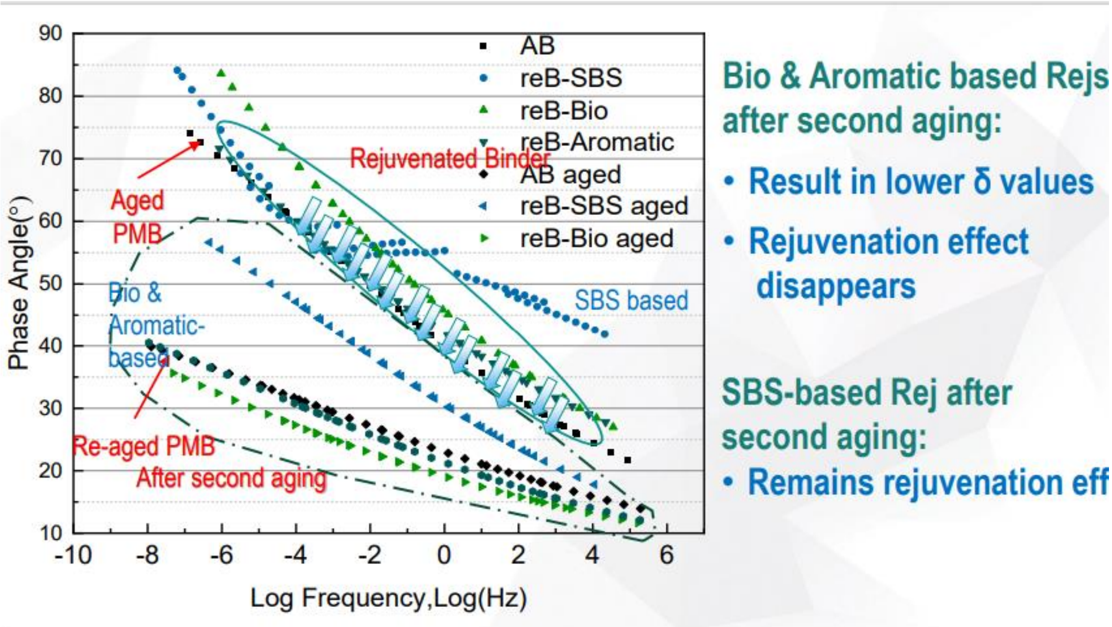
SBS, Bio, Aromatic based rejuvenators significantly improve cracking resistance

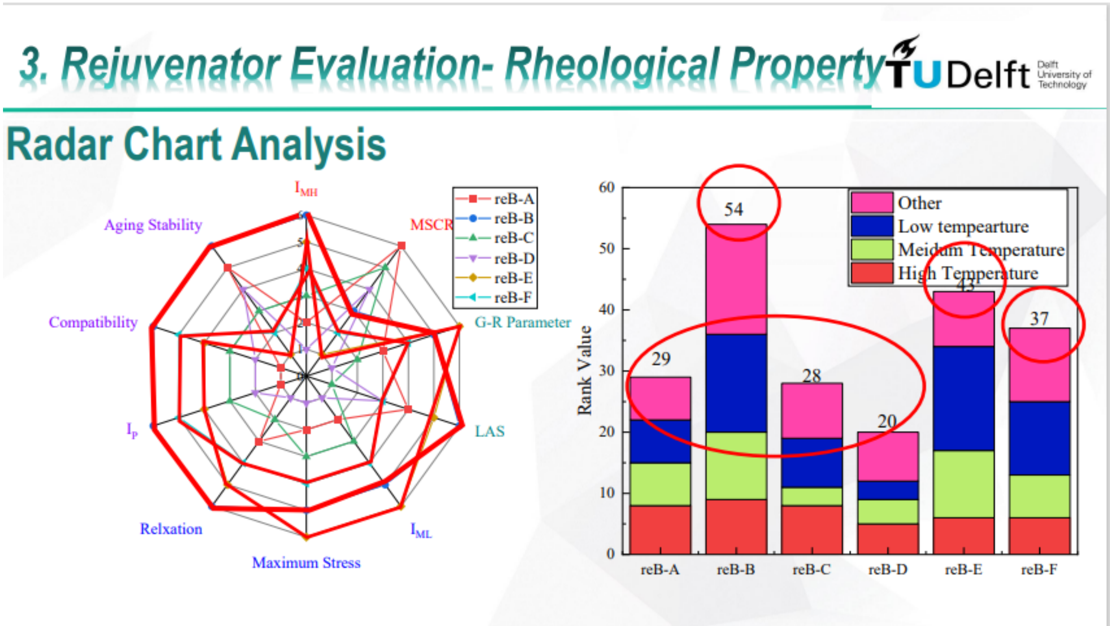

3. Rejuvenator Evaluation- Rheological Property
TUDelft Delft University of Technology
ow Temperature-Relaxation Test
Strain
Strain=1%
0.1 s
100 s
Time(s)
Relaxation Test @ 0 ℃
Residual Sress(Pa)
Time(s)
AB
reB-A
reB-B
reB-C
reB-D
reB-E
reB-F
1. Reach 1% strain level with in 0.1 s
2. Keep 1% strain level for 100s and track the relaxation of the stress.

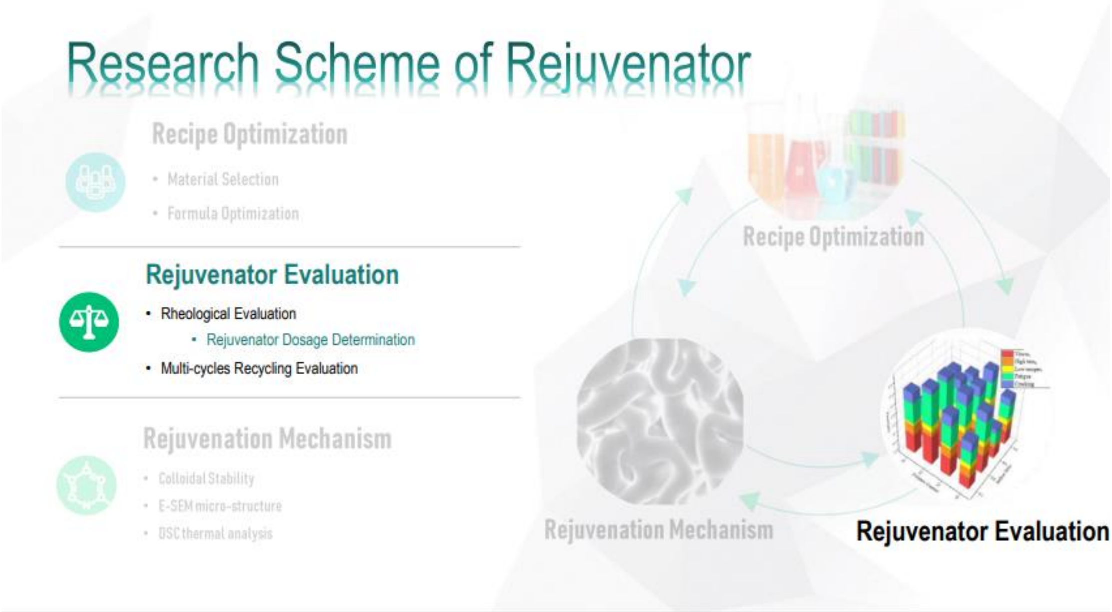

3. Rejuvenator Evaluation- Rheological Property
TUDelft Delft University of Technology
Low Temperature-Cracking
Residual Sress(Pa)
Time(s)
AB
reB-A
reB-B
reB-C
reB-D
reB-E
reB-F
Aromatic
SBS based
Bio-
• SBS and Bio-based rejuvenator significantly improve relaxatio
• Aromatic rejuvenator improves stress relaxation to a certain e
• EBA, TB/SBS, TB based rejuven improve it not much

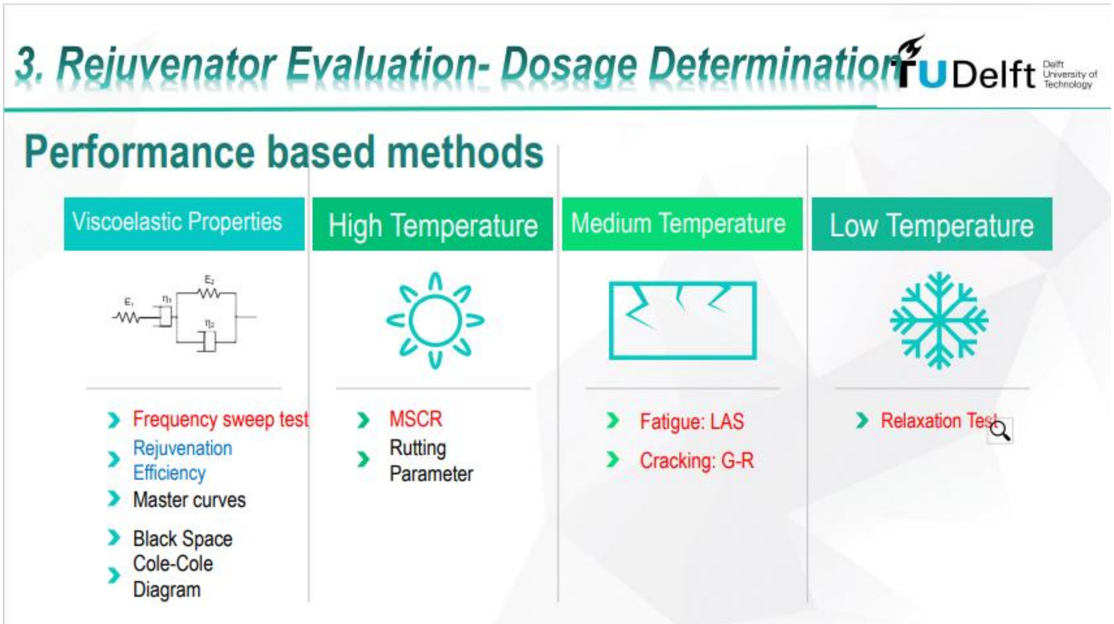

3. Rejuvenator Evaluation- Rheological Property
TU Delft Delft University of Technology
Anti-aging Properties
AB
reB-B
reB-E
reB-F
AB re-aged
reB-B re-aged
reB-E aged
reB-F re-aged
Complex Modulus,Pa
Log Frequency,Log(Hz)
reB-Ere-aged
Phase Angle(°)
Xueyan Liu, Peng Lin et.al. "Effect of Rejuvenators on Performance-based Properties of Aged Polymer Modified Bitumen" .99 th Transportation Research Board.


Reaged PMB
After second aging
Bio & Aromatic based Rej
SBS based Rej
Rejuvenated Binders
Aged PMB
AB
reB-B
reB-E
reB-F
AB re-aged
reB-B re-aged
reB-E aged
reB-F re-aged
Complex Modulus,Pa
Log Frequency,Log(Hz)
Bio & Aromatic based rejuvenators after second aging:
• G* values are almost same
• Rejuvenation effect disappears
SBS-based rejuvenator after second aging:
• Remains rejuvenation effect

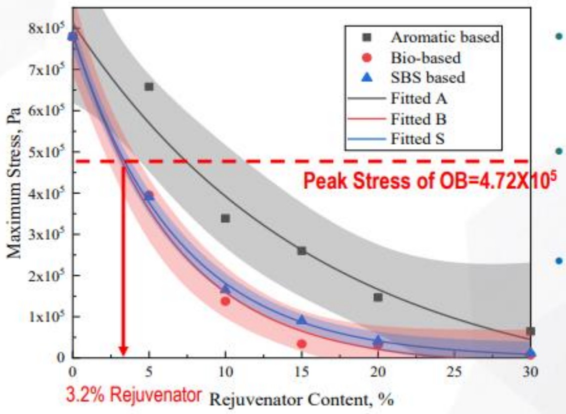

Bio & Aromatic based Rejs after second aging:
• Result in lower δ values
• Rejuvenation effect disappears
SBS-based Rej after second aging:
• Remains rejuvenation eff
Phase Angle(°)
Log Frequency,Log(Hz)
AB
reB-SBS
reB-Bio
reB-Aromatic
AB aged
reB-SBS aged
reB-Bio aged
Rejuvenated Binder
Aged PMB
Bio & Aromatic-based
SBS based
Re-aged PMB
After second aging


3. Rejuvenator Evaluation- Rheological Property
TU Delft Delft University of Technology
Radar Chart Analysis
I_{MH}
MSCR
G-R Parameter
LAS
I_{ML}
Maximum Stress
Relxation
I_p
Compatibility
Aging Stability
reB-A
reB-B
reB-C
reB-D
reB-E
reB-F
Rank Value
Other
Low tempearture
Meidum Temperature
High Temperature
29
54
28
20
43
37


Research Scheme of Rejuvenator
Recipe Optimization
Material Selection
Formula Optimization
Rejuvenator Evaluation
Rheological Evaluation
Rejuvenator Dosage Determination
Multi-cycles Recycling Evaluation
Rejuvenation Mechanism
Colloidal Stability
E-SEM micro-structure
DSC thermal analysis
Recipe Optimization
Rejuvenation Mechanism
Rejuvenator Evaluation

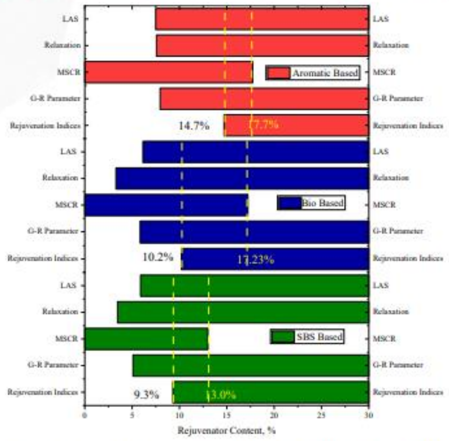

3. Rejuvenator Evaluation- Dosage Determination
TU Delft
Performance based methods
Viscoelastic Properties
High Temperature
Medium Temperature
Low Temperature
Frequency sweep test
Rejuvenation Efficiency
Master curves
Black Space
Cole-Cole Diagram
MSCR
Rutting Parameter
Fatigue: LAS
Cracking: G-R
Relaxation Test

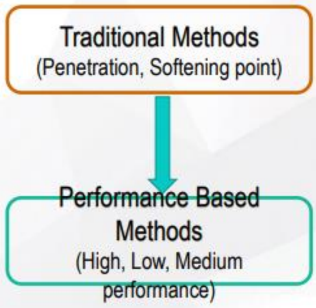
Low Temperature- Relaxation



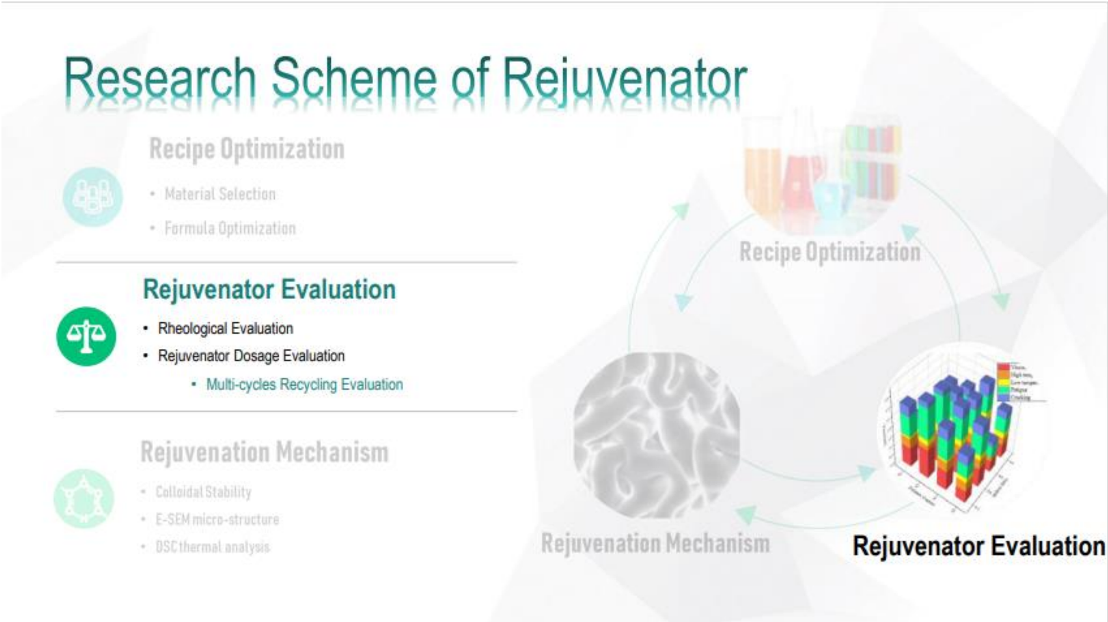
- The maximum stress change shows an exponential relationship with rejuvenator dosage,
- SBS and Bio-based rejuvenators are more efficient than Aromatic-based rejuvenator in decreasing maximum stress
- Calculate the minimum rejuvenator dosage based on the peak stress equals to the one occurred in the original fresh binder

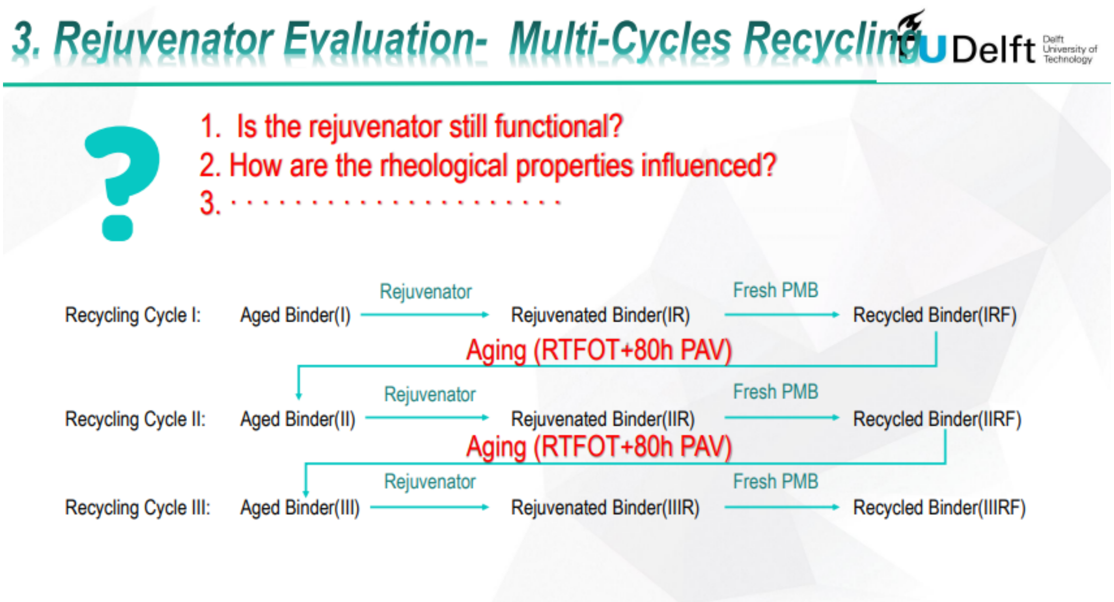
Rejuvenator Dosage Determination

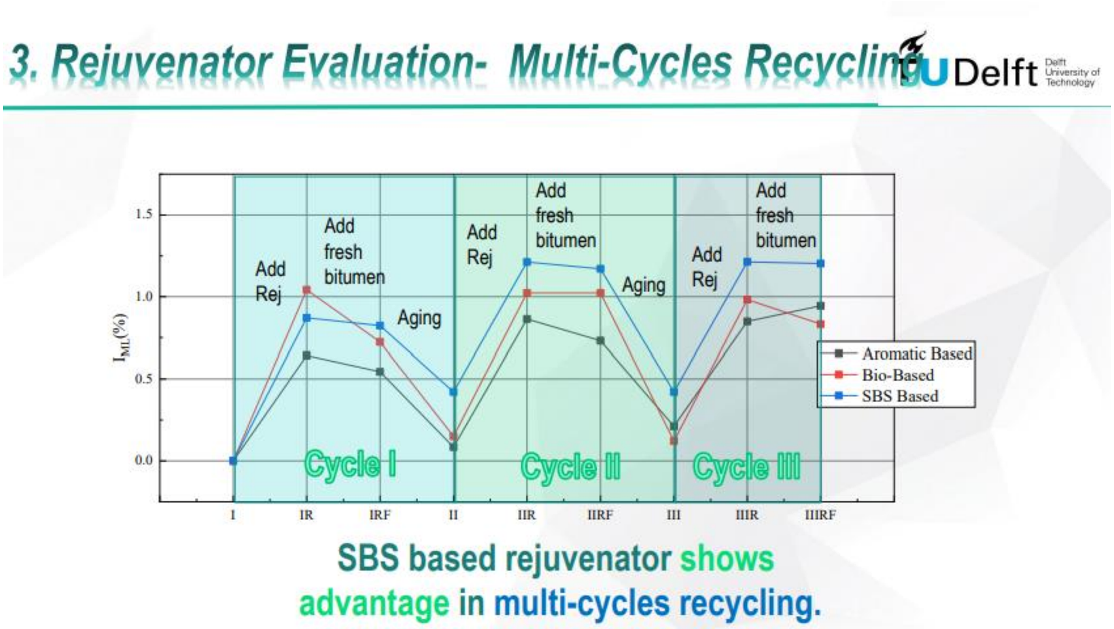




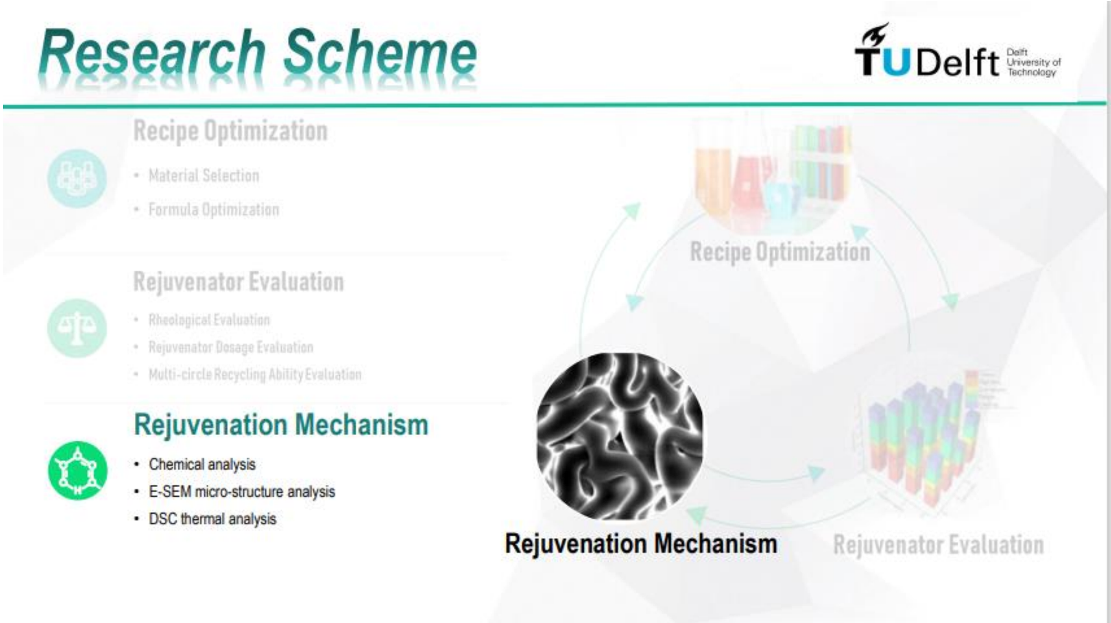
Peng Lin, Xueyan Liu, Panos Apostolidis, Sandra Erkens, Shisong Ren, Shi Xu, Tom Scarpas, and Weidong Huang. "On the Rejuvenator Dosage Optimization for Aged SBS Modified Bitumen." Construction and Building Materials 271 (February 15, 2021): 121913.

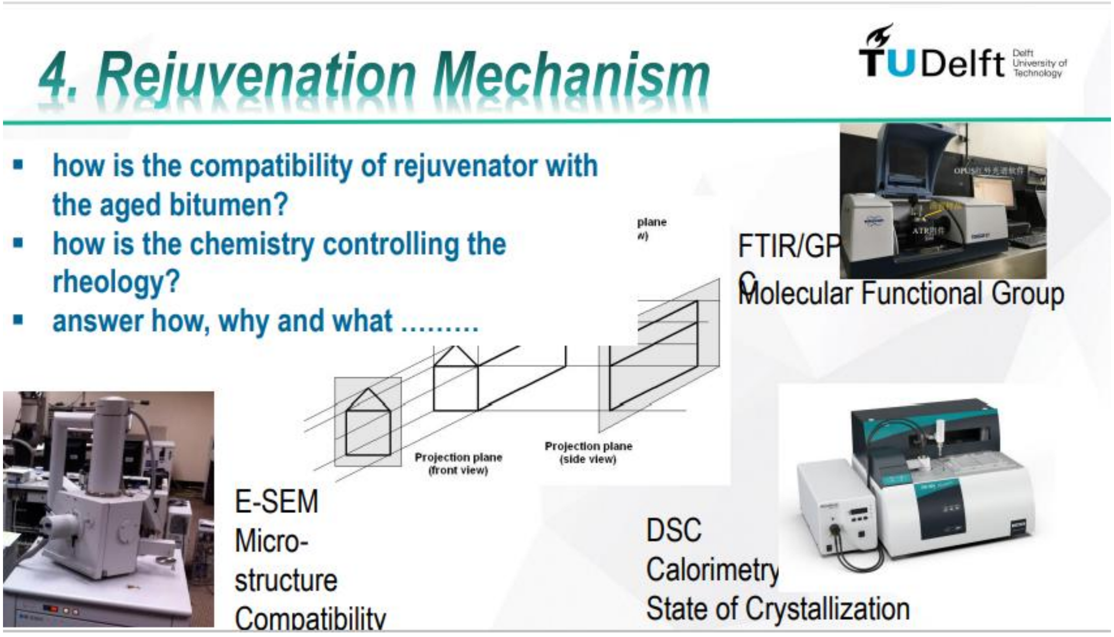

Research Scheme of Rejuvenator
Recipe Optimization
Material Selection
Formula Optimization
Rejuvenator Evaluation
Rheological Evaluation
Rejuvenator Dosage Evaluation
Multi-cycles Recycling Evaluation
Rejuvenation Mechanism
Colloidal Stability
E-SEM micro-structure
DSC thermal analysis
Recipe Optimization
Rejuvenation Mechanism
Rejuvenator Evaluation

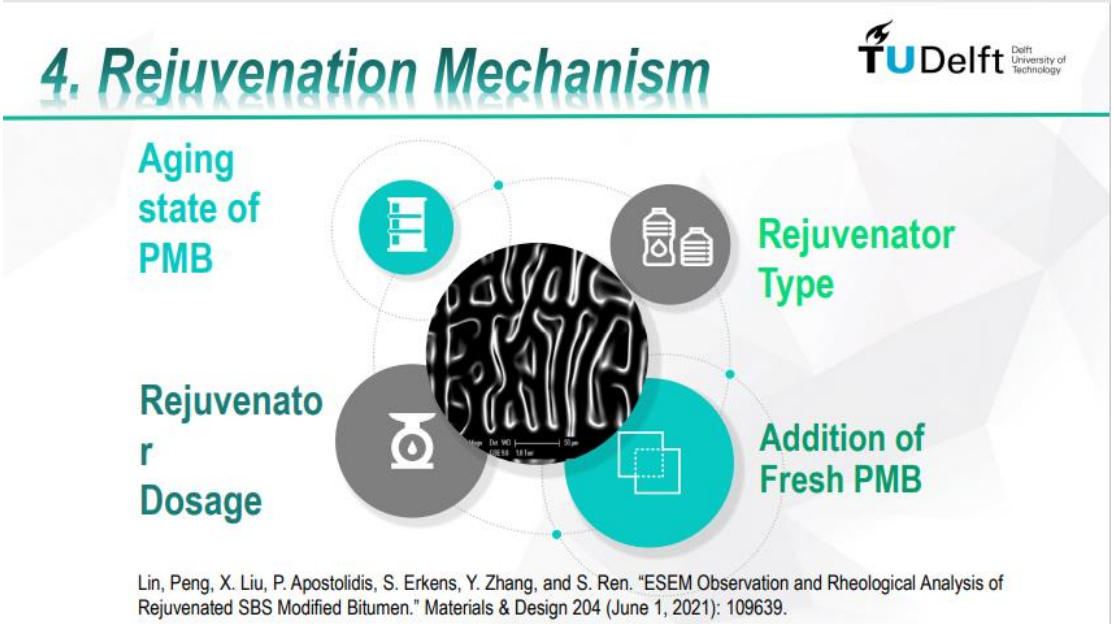

3. Rejuvenator Evaluation- Multi-Cycles Recycling
TU Delft
1. Is the rejuvenator still functional?
2. How are the rheological properties influenced?
3. . . . .
Recycling Cycle I: Aged Binder(I) Rejuvenator Rejuvenated Binder(IR) Fresh PMB Recycled Binder(IRF)
Aging (RTFOT+80h PAV)
Recycling Cycle II: Aged Binder(II) Rejuvenator Rejuvenated Binder(IIR) Fresh PMB Recycled Binder(IIRF)
Aging (RTFOT+80h PAV)
Recycling Cycle III: Aged Binder(III) Rejuvenator Rejuvenated Binder(IIIR) Fresh PMB Recycled Binder(IIIRF)

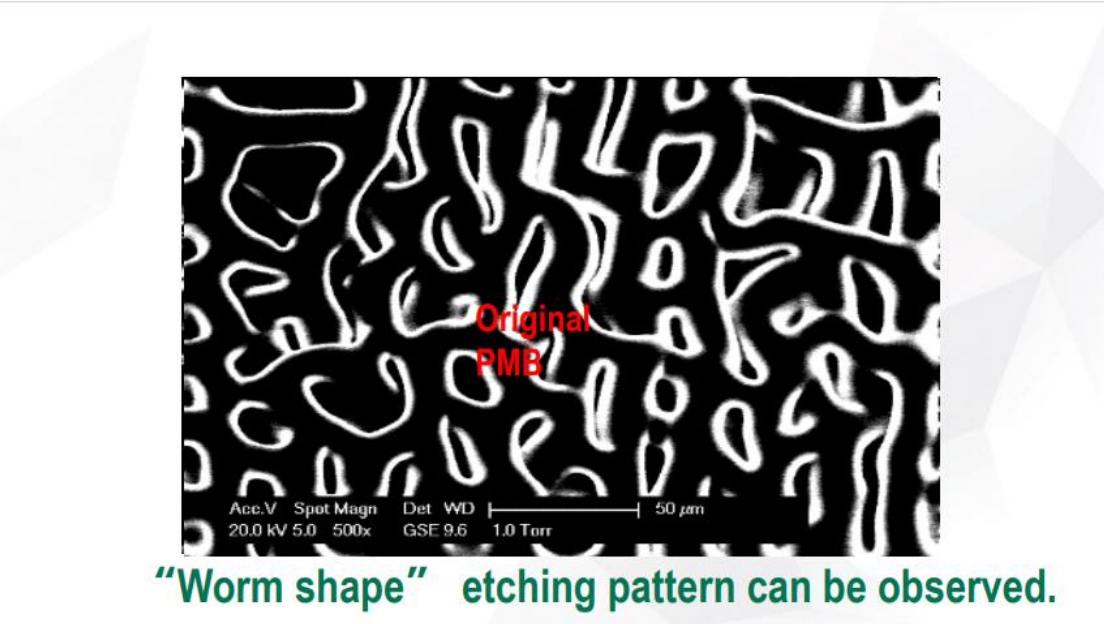

3. Rejuvenator Evaluation- Multi-Cycles Recycling
TU Delft Delft University of Technology
Add Rej
Add fresh bitumen
Aging
Cycle I
Cycle II
Cycle III
I_ML(%)
1.5
1.0
0.5
0.0
I
IR
IRF
II
IIR
IIRF
III
IIIR
IIIRF
Aromatic Based
Bio-Based
SBS Based
SBS based rejuvenator shows advantage in multi-cycles recycling.

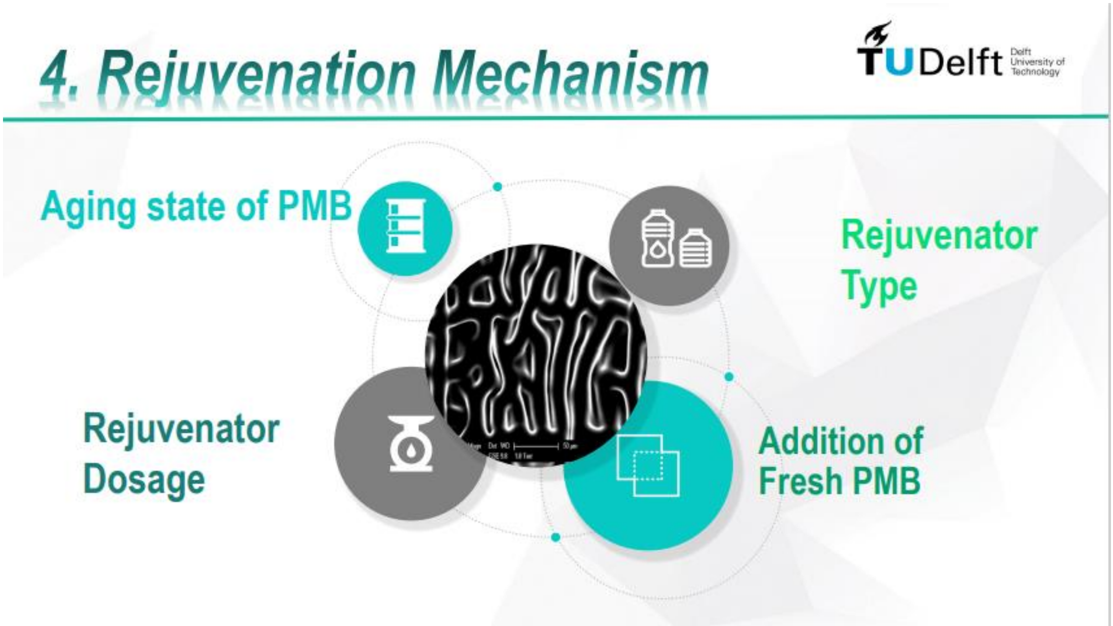

04
Rejuvenation Mechanism

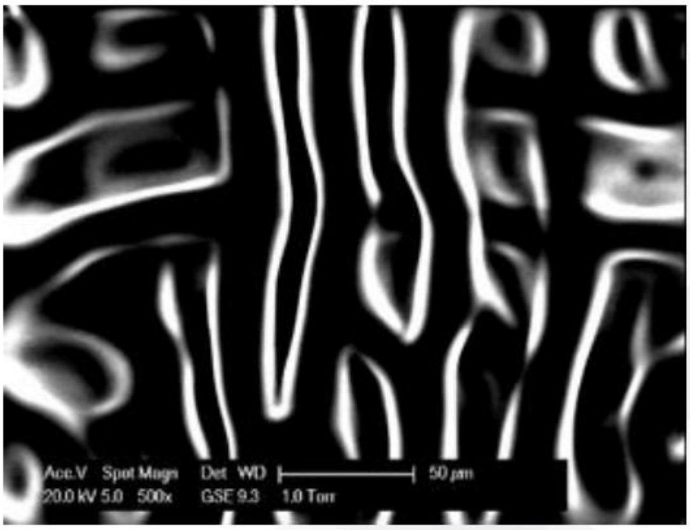

Research Scheme
TUDelft Delft University of Technology
Recipe Optimization
• Material Selection
• Formula Optimization
Rejuvenator Evaluation
• Rheological Evaluation
• Rejuvenator Dosage Evaluation
• Multi-circle Recycling Ability Evaluation
Rejuvenation Mechanism
• Chemical analysis
• E-SEM micro-structure analysis
• DSC thermal analysis
Recipe Optimization
Rejuvenation Mechanism
Rejuvenator Evaluation


4. Rejuvenation Mechanism
TUDelft Delft University of Technology
▪ how is the compatibility of rejuvenator with the aged bitumen?
▪ how is the chemistry controlling the rheology?
▪ answer how, why and what ....
FTIR/GPC
Molecular Functional Group
Projection plane (front view)
Projection plane (side view)
E-SEM
Micro-structure
Compatibility
DSC
Calorimetry
State of Crystallization

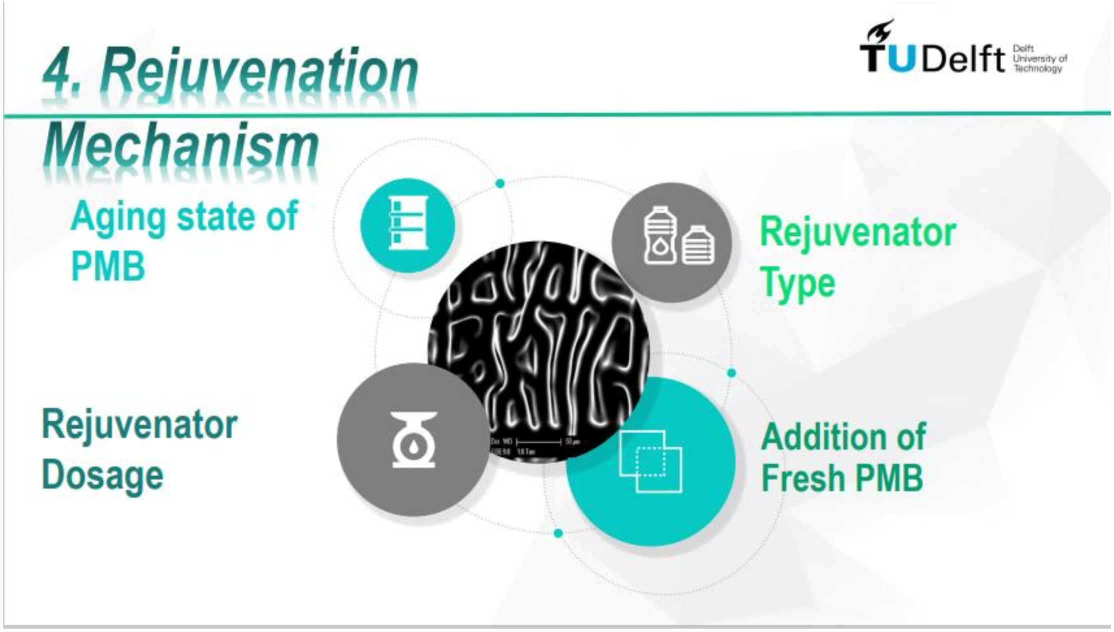

4. Rejuvenation Mechanism
TUDelft Delft University of Technology
Aging state of PMB
Rejuvenator Type
Rejuvenato r Dosage
Addition of Fresh PMB
Lin, Peng, X. Liu, P. Apostolidis, S. Erkens, Y. Zhang, and S. Ren. "ESEM Observation and Rheological Analysis of Rejuvenated SBS Modified Bitumen." Materials & Design 204 (June 1, 2021): 109639.

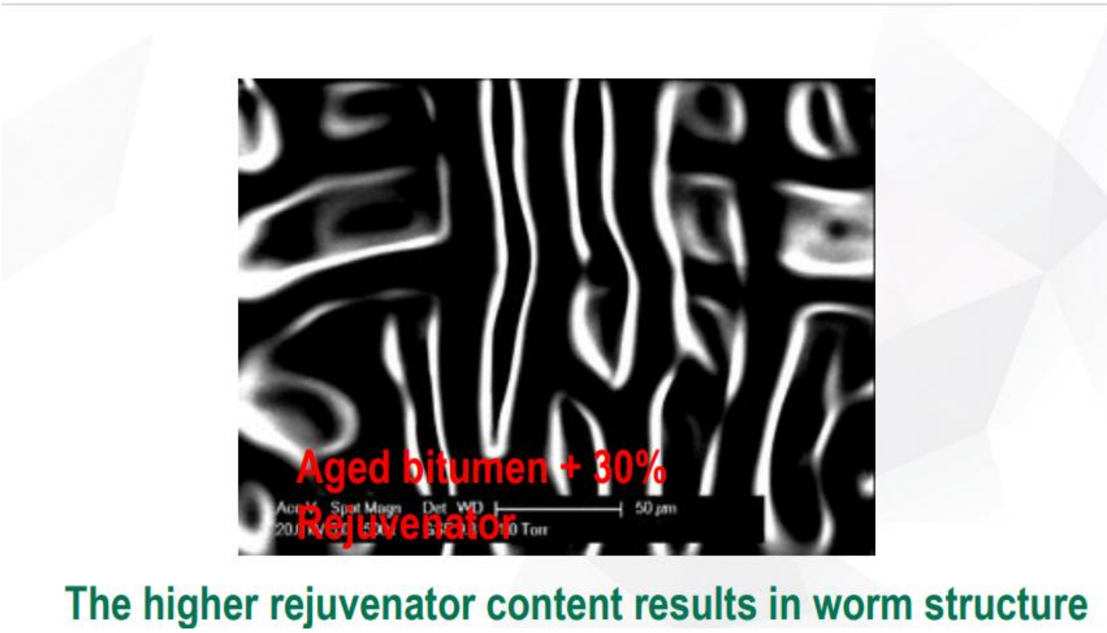

Original PMB
Acc.V Spot Magn Det WD 50 µm
20.0 kV 5.0 500x GSE 9.6 1.0 Torr
"Worm shape" etching pattern can be observed.

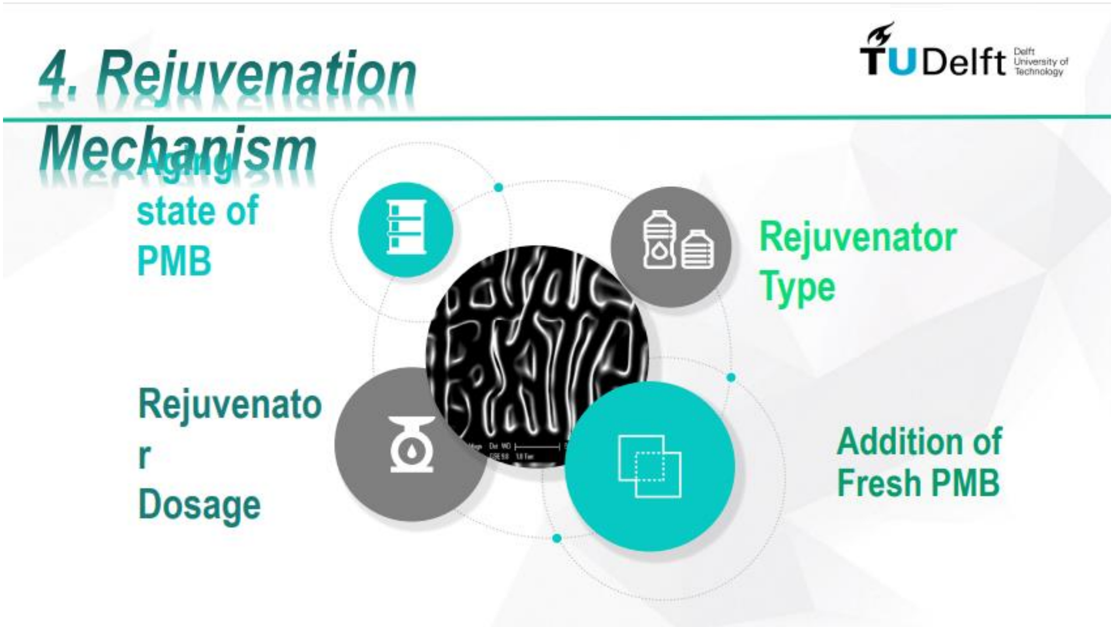




Rejuvenator types influence the morphology of aged PMB


4. Rejuvenation Mechanism
TUDelft Delft University of Technology
Aging state of PMB
Rejuvenator Type
Rejuvenator Dosage
Addition of Fresh PMB


Aged bitumen + 30% Rejuvenator
50 μm
The higher rejuvenator content results in worm structure


4. Rejuvenation Mechanism
TU Delft Delft University of Technology
Aging state of PMB
Rejuvenator Type
Rejuvenato r Dosage
Addition of Fresh PMB


Aged bitumen + Bio-based Rej+Fresh bitumen
Det WD 50 µm
GSE 9.5 1.0 Torr

4. Rejuvenation Mechanism

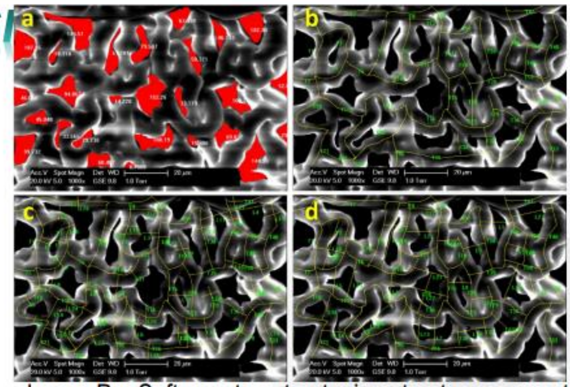
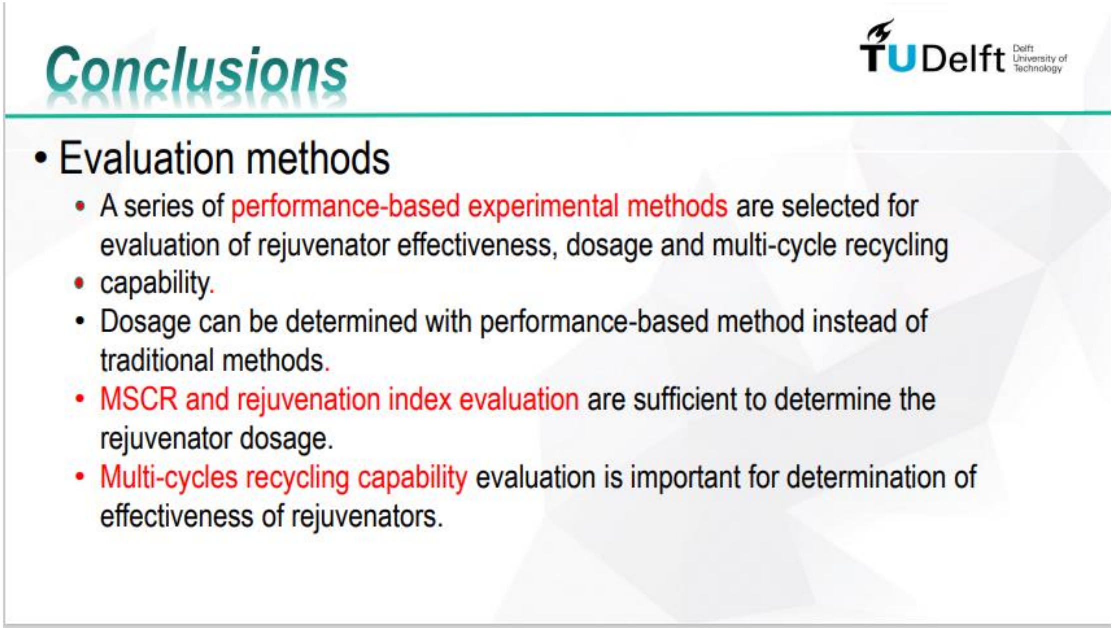


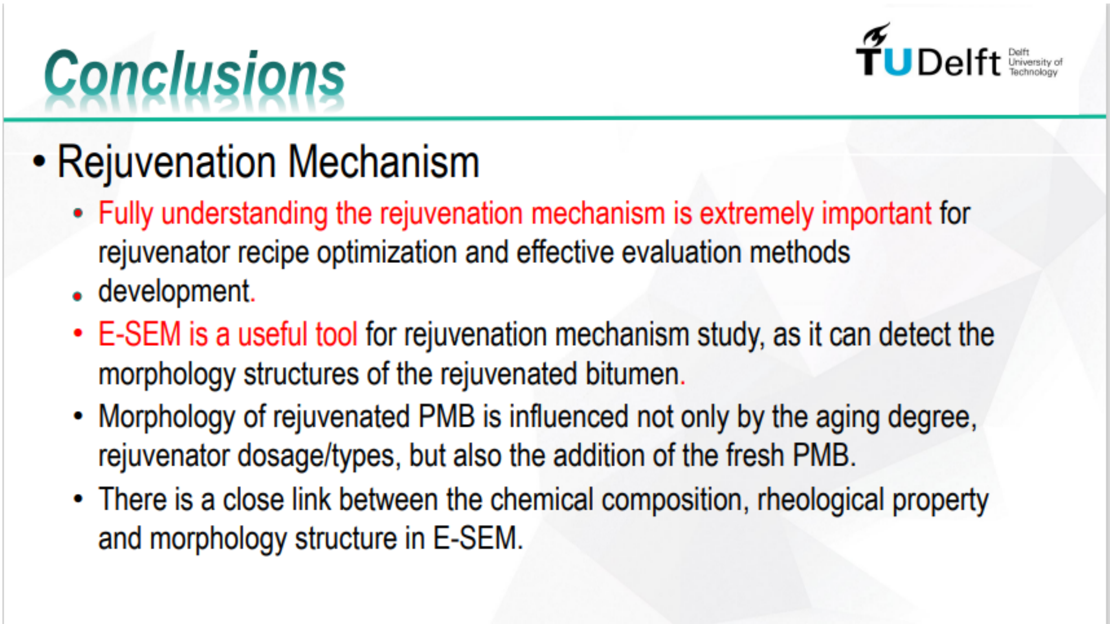
Image Pro Software to extract microstructure parameters:
(a: area coverage; b: length; c: diameter; d: distance…)

Lin, Peng, X. Liu, P. Apostolidis, S. Erkens, Y. Zhang, and S. Ren. "ESEM Observation and Rheological Analysis of Rejuvenated SBS Modified Bitumen." Materials & Design 204 (June 1, 2021): 109639.

Conclusions

- Recipe Optimization
  - Recipe optimization should consider material selection, component ratio determination and functional property balanced at different temperature conditions.
  - SBS-based rejuvenator is one of the most effective rejuvenators for the aged PMB binders.
  - SBS based rejuvenator shows advantages in multi-cycles recycling capability.
  - Bio-based rejuvenator shows advantage in reducing G* at low temperature, but drawbacks in aging resistance and viscoelastic property.
  - Aromatic based rejuvenator shows advantage in viscoelastic property, but drawback
  - in other properties.
  - EBA, TB, TB/SBS rejuvenators show advantage in high-temp property, but drawback in other properties.


Conclusions
TU Delft Delft University of Technology
• Evaluation methods
• A series of performance-based experimental methods are selected for evaluation of rejuvenator effectiveness, dosage and multi-cycle recycling
• capability.
• Dosage can be determined with performance-based method instead of traditional methods.
• MSCR and rejuvenation index evaluation are sufficient to determine the rejuvenator dosage.
• Multi-cycles recycling capability evaluation is important for determination of effectiveness of rejuvenators.


Conclusions
TU Delft Delft University of Technology
• Rejuvenation Mechanism
• Fully understanding the rejuvenation mechanism is extremely important for rejuvenator recipe optimization and effective evaluation methods
• development.
• E-SEM is a useful tool for rejuvenation mechanism study, as it can detect the morphology structures of the rejuvenated bitumen.
• Morphology of rejuvenated PMB is influenced not only by the aging degree, rejuvenator dosage/types, but also the addition of the fresh PMB.
• There is a close link between the chemical composition, rheological property and morphology structure in E-SEM.


iFRAE
DELFT 2021
5th International Symposium
on Frontiers of Road and Airport Engineering
Workshop: Changes in binder properties and the role of additives
Thank you !
CO2
TUDelft

3.6 Designing a toolbox for bitumen to answer the need for tomorrow's pavement

Laurent Porot

Kraton Polymer B.V., Amsterdam, the Netherlands

Abstract

The asphalt industry is facing some key challenges. There is a need to move towards more sustainable and environmental friendly solutions to construct the pavements for tomorrow. This has to answer the market needs for greater performance with improved warranties, increased safety and less impact on environment, all in required budget constraints. At the same time, there is an even greater diversity in binders, petroleum based binders from different sources or processes for which bitumen quality may be affected. Up to now, specifications and characterisation for asphalt binders have been designed for known petroleum-based bitumen. With complex binders, more fundamental understanding and properties have to be considered to really capture the long-term benefits in road and airport engineering. And finally, beyond the technical requirements, sustainable aspects need to be part of the design including circularity, environmental impacts, health and safety amongst others. This is an important paradigm where new technologies are needed and adjustment of designing materials.

Thus, the need for new solutions are becoming increasingly common practice. Designing the exact solution may depend on various parameters such as the nature of the modifier, the dosage level, or the expected effects on the binder, on the asphalt mix and finally on the pavement. It can be viewed as a toolbox where different options can be selected and combined together to adjust the properties of the binders that fits the need for pavement applications.

Through some examples with the specific use of polymers and bio-based additives, an example of general framework will be discussed to be served as a toolbox to design materials to bring the frontiers of road and airport engineering a step further to the future.

About the speaker

Laurent is Market Development Manager at Kraton, based in the Netherlands, in charge of technical development for polymer and pine chemical additives in paving and roofing application. He has a master degree of civil engineering from Ecole Nationale des Ponts et Chaussees, France. With 30 years of experience, he has capitalised a worldwide expertise on pavement engineering with pavement design, materials, job works and research & development.

He is member of numerous international scientific committees and representative in industry association. He has been working with key research institutes in the field of asphalt and pavement materials within projects and inter-laboratory experiment. With a robust technical background he extended his learning on environmental impacts and Life Cycle Assessment. He is passionate about interacting with people to design more sustainable solutions.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DELFT 2021
5th International Symposium
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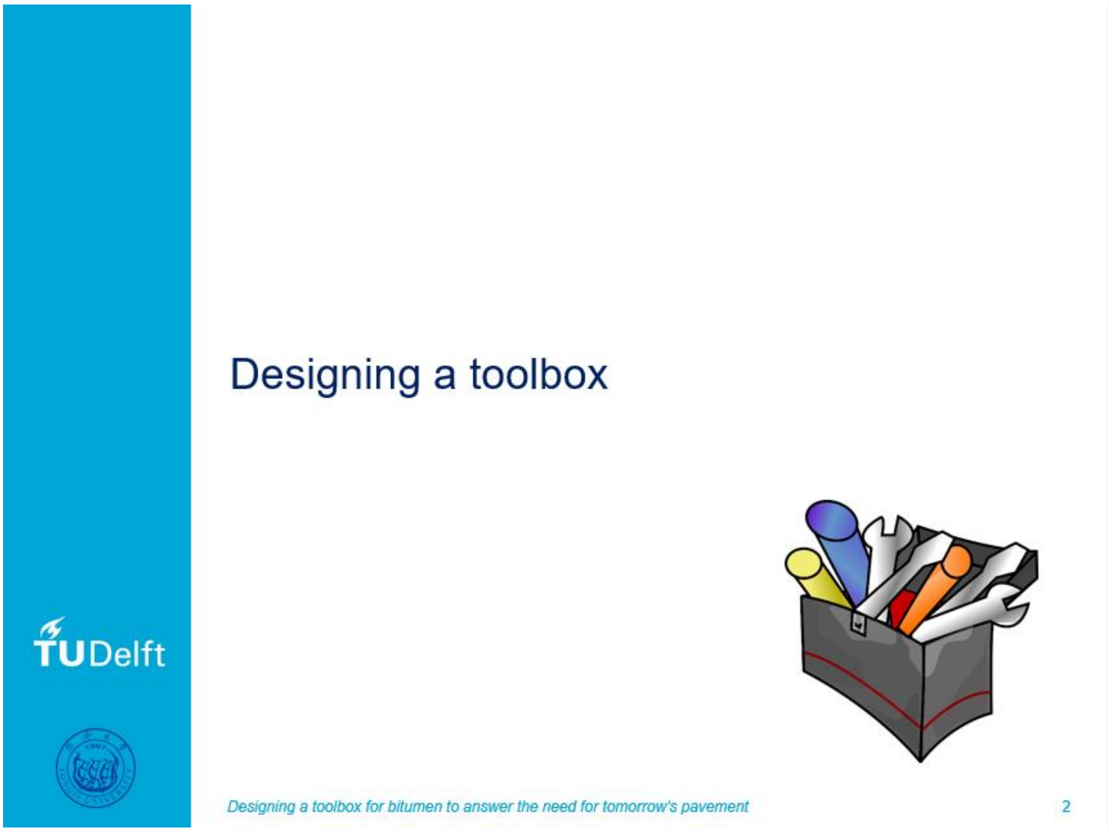
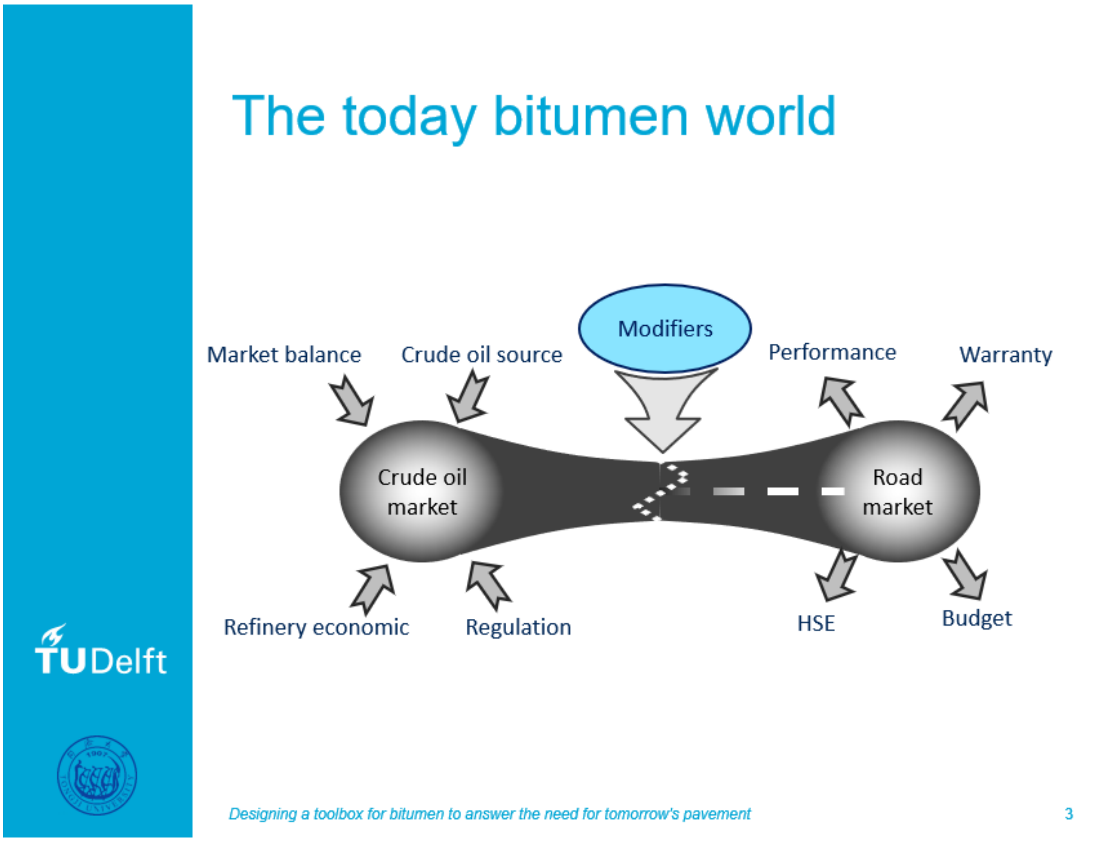
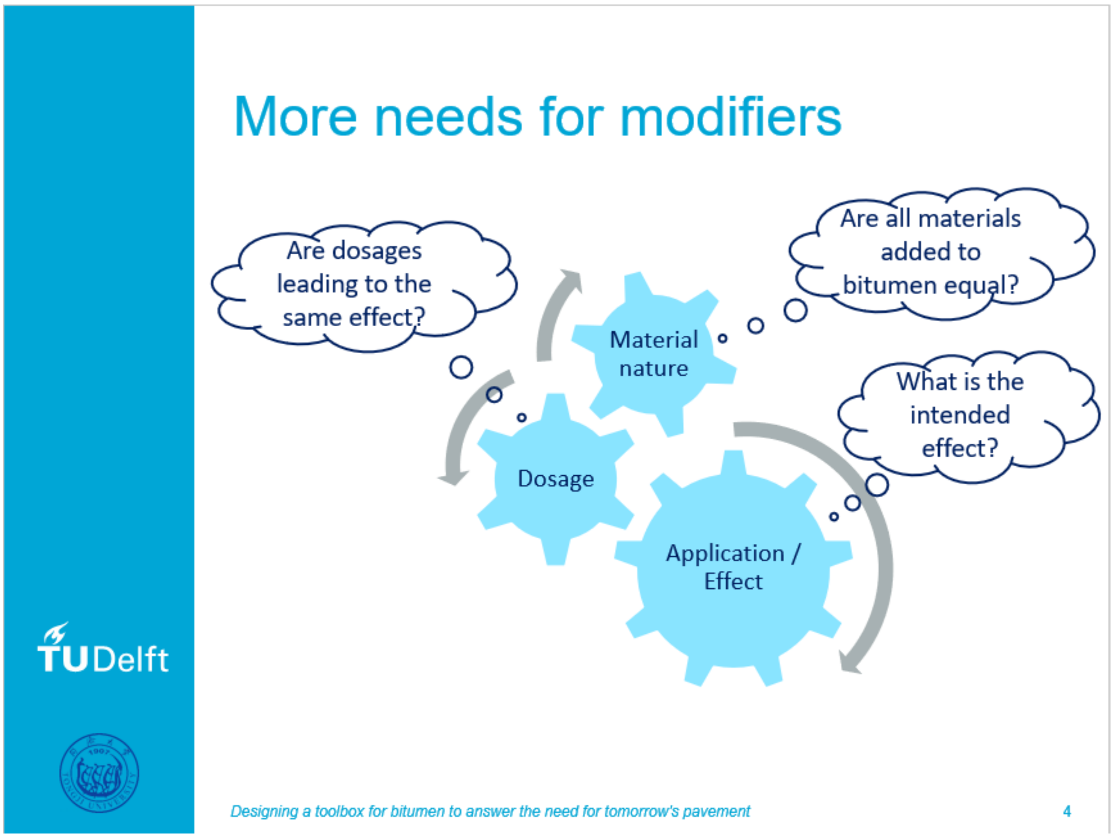
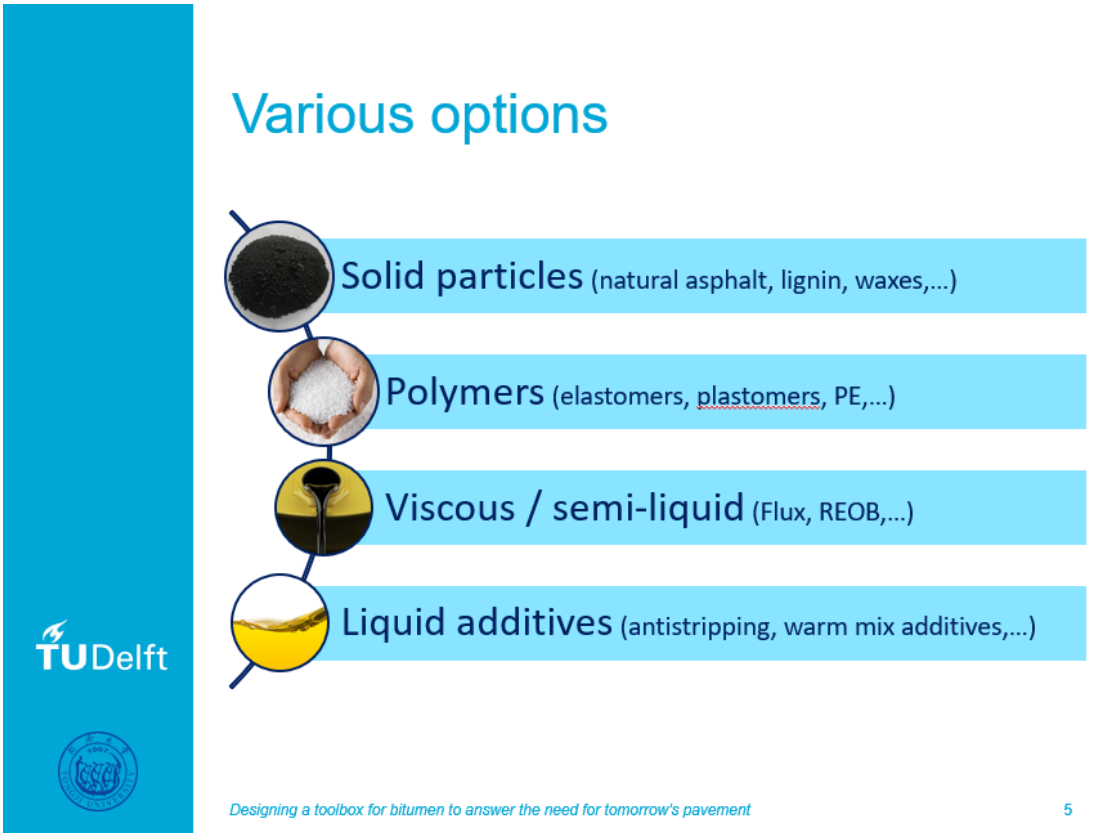
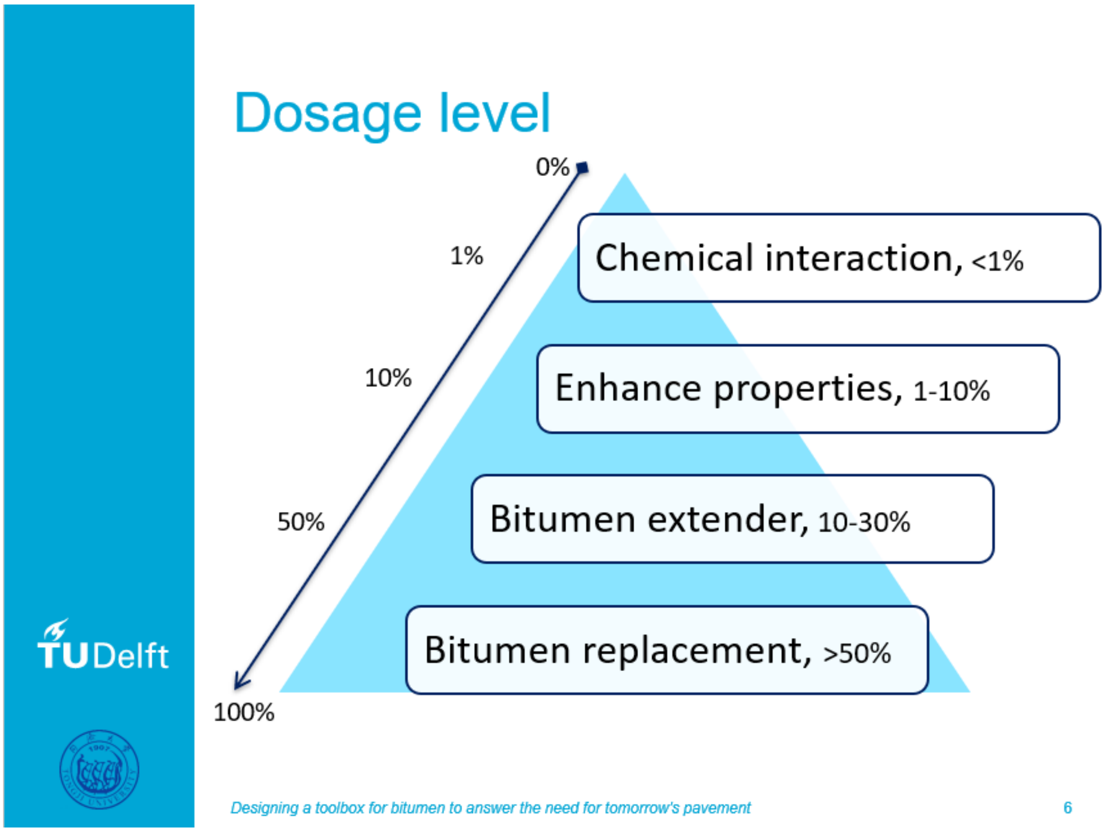
Workshop: Changes in binder properties and the role of additives
Designing a toolbox for bitumen to answer
the need for tomorrow's pavement
Laurent Porot, Kraton Polymers B.V. the Netherlands
KRATON
TUDelft
CO2

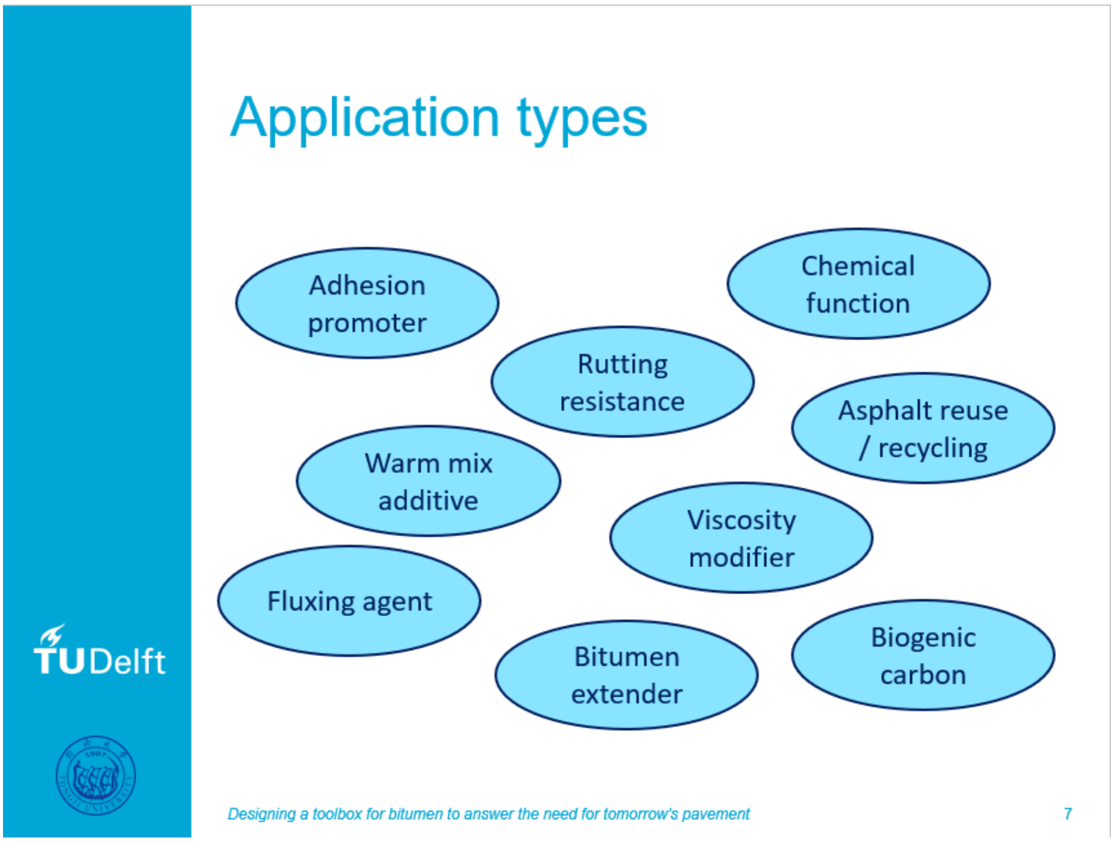

Designing a toolbox
TUDelft
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
2

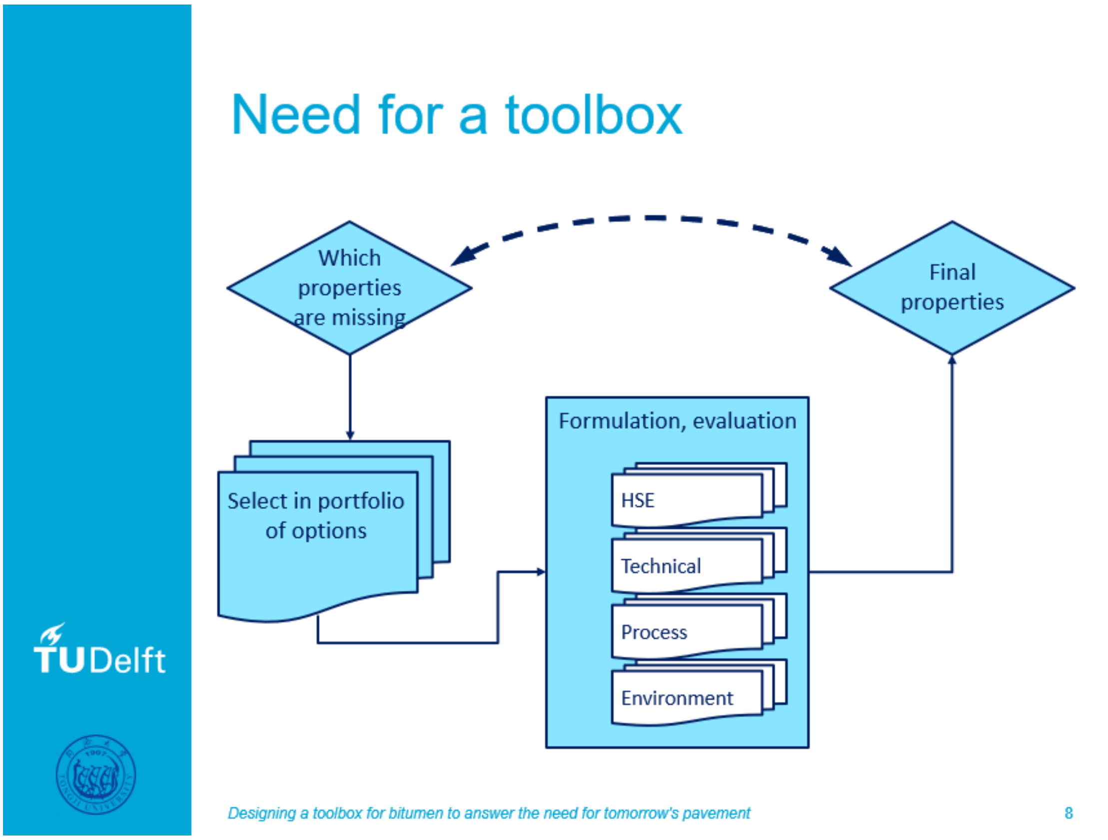

The today bitumen world
Modifiers
Market balance
Crude oil source
Performance
Warranty
Crude oil market
Road market
Refinery economic
Regulation
HSE
Budget
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
3


More needs for modifiers
Are dosages leading to the same effect?
Are all materials added to bitumen equal?
Material nature
What is the intended effect?
Dosage
Application / Effect
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
4


Various options
Solid particles (natural asphalt, lignin, waxes,...)
Polymers (elastomers, plastomers, PE,...)
Viscous / semi-liquid (Flux, REOB,...)
Liquid additives (antistripping, warm mix additives,...)
TUDelft
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
5


Dosage level
0%
1%
10%
50%
100%
Chemical interaction, <1%
Enhance properties, 1-10%
Bitumen extender, 10-30%
Bitumen replacement, >50%
TUDelft
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
6


Application types
Adhesion promoter
Chemical function
Rutting resistance
Asphalt reuse / recycling
Warm mix additive
Viscosity modifier
Fluxing agent
Bitumen extender
Biogenic carbon
TU Delft
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
7


Need for a toolbox
Which properties are missing
Final properties
Select in portfolio of options
Formulation, evaluation
HSE
Technical
Process
Environment
TU Delft
Designing a toolbox for bitumen to answer the need for tomorrow's pavement
8

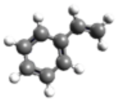
Designing a toolbox

Example with SBS polymer

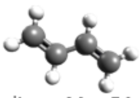
TU Delft

What is SBS

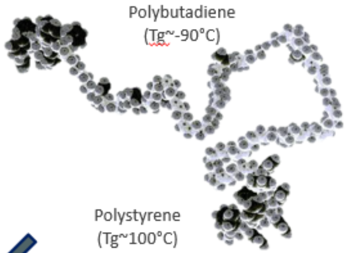
- **S**tyrene-**B**utadiene-**S**tyrene polymers
  - Thermoplastic elastomer block copolymers
  - From monomers, Styrene and Butadiene



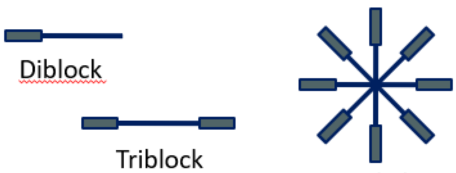
Styrene, Mw=104



Butadiene, Mw=54



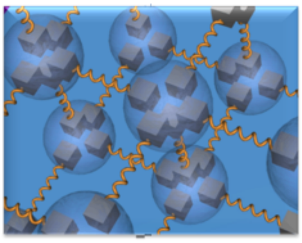
  - Different structures



TU Delft

SBS polymer in bitumen

- SBS dissolves in bitumen to form an elastic network
  - Leading technology for bitumen modification
- Key features in bitumen
  - Greater resistance to rutting
  - Improve durability
  - Extended life time by 20-30% as compared to non modified*
  - Recyclable

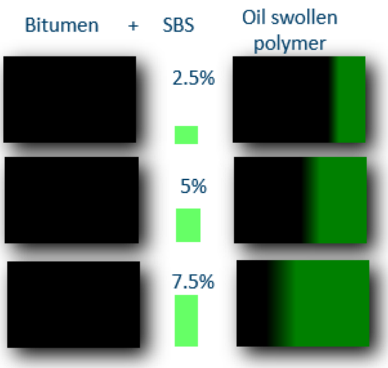


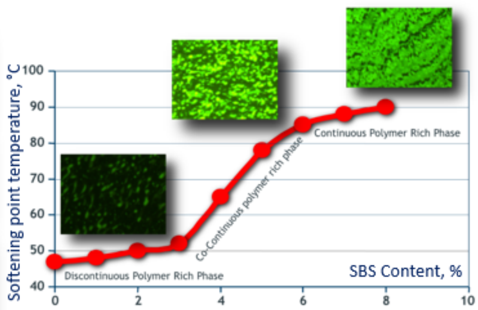
Elasticity is key

* Asphalt Institute, ER-215 Engineer's Report: Quantification of the effects of PMA for Reducing Pavement Distress, 2005

How SBS behaves in bitumen

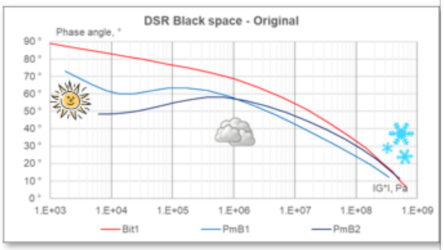
- SBS swells in oil phase, increasing volume by 7-10 times
  - Balance between bitumen / polymer rich phase
  - Direct influence in elastic behaviour





Overall effect on properties

- Effect on rheology
  - Higher elasticity with rubber plateau at high Temperature



- Effect on specification
  - Increase high T
  - Maintain low T
  - Reduce temperature susceptibility

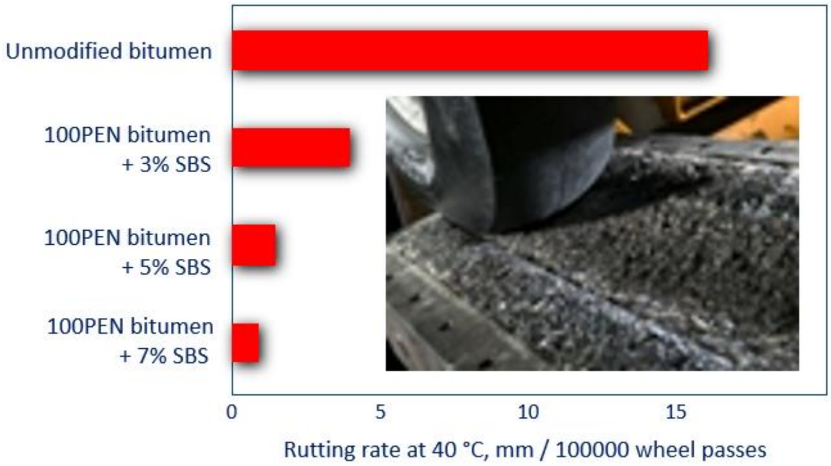
Bit1 is 35/50 pen bitumen, PmB1 is standard PmB, PmB2 is highly modified PmB

From "Characterisation of complex polymer modified bitumen with rheological parameter", L. Porot et al, EATA 2021, doi.org/10.1080/14680629.2021.1910070

Designing a toolbox for bitumen to answer the need for tomorrow's pavement 13

Effect on asphalt mix

- Wheel tracking test – rutting resistance



Designing a toolbox for bitumen to answer the need for tomorrow's pavement 14

Designing a toolbox

Example with SBS polymer

Examples with pine chemistry additives

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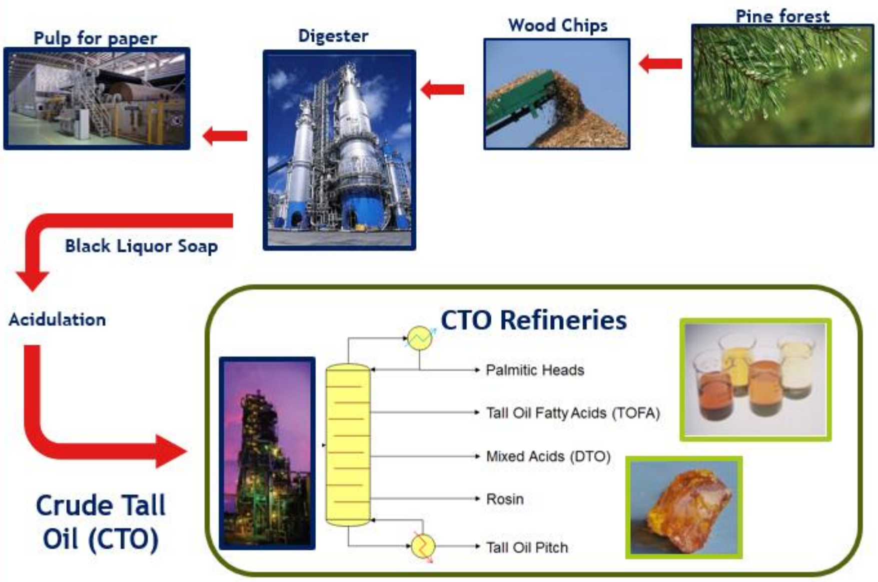
What is Pine chemistry?



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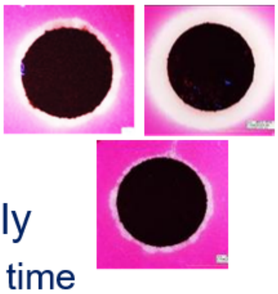
Pine chemistry in bitumen

- HSE benefits
  - Products from bio-renewable feedstock
  - No known harmful additional emission (PAH, VOC)
- Compatibility with bitumen
  - "Young" bitumen like
  - Molecule structure similar to bitumen ones
- Consistent quality and supply
  - Pine trees have long growing time
  - Processed products to meet specification

Example from exudation droplet test



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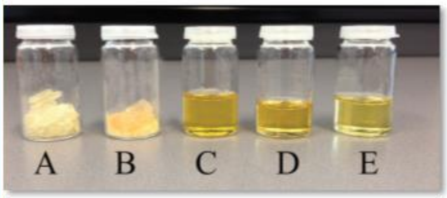
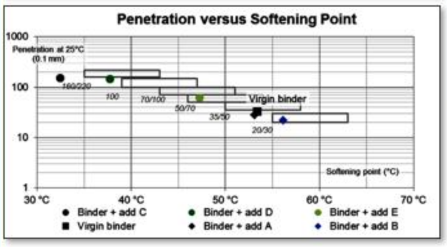
Effect on physical properties

- Enable to control independently the properties of bitumen





From "Effect of bio-baed additives on bitumen properties" A. Grilli et al, RMPD Vol20-8 2019, doi.org/10.1080/14680629.2018.1474790

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Dope for bitumen emulsion

- Chemical dope for bitumen in emulsion
  - Mimic the functionality of naphthenic bitumen
  - Low effective dosage <1%
- Effect on bitumen emulsion
  - Maintain good storage stability
  - Enable better control of breaking index
  - No impact on physical properties

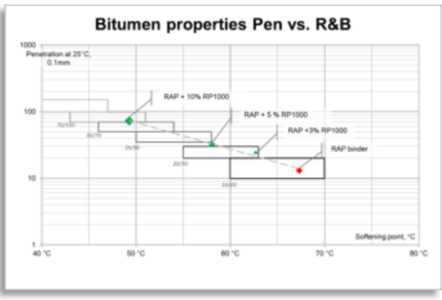
Bitumen emulsion properties EN 13808

	Unit	no additive	1% additive
Efflux time	s	47	46
Breaking index	s	160	135
Settlement tendency	%	43.9	26.4


Breaking index


Storage stability

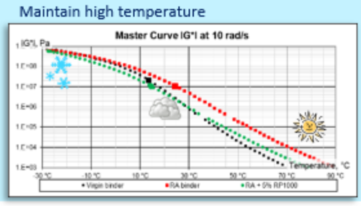
Asphalt Reuse Additive for RA



5% dosage improves aged binder by 2 grades

Properties in wide conditions

- Improve low temperature
- Restore intermediate temperature
- Maintain high temperature



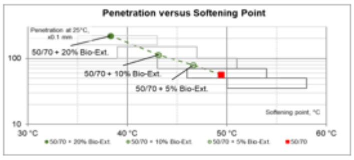
Restore long-term flexibility

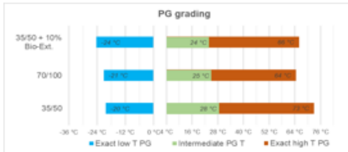
- Low temperature after long-term aging (BBR after PAV)

From "Asphalt and binder evaluation of asphalt mix with 70% reclaimed asphalt" L. Porot EATA 2017, doi.org/10.1080/14680629.2017.1304259

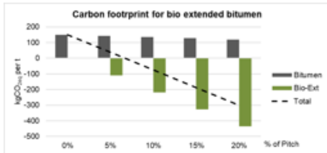
Bio-bitumen extender

- Effect on physical properties
  - A dosage of 5% reduces by 1 grade softer
  - Various applications as visco-grade, extender





- Environmental effect
  - Comes with carbon credit



Carbon offset between 5% and 10%

From "Pitch in bitumen application", L. Porot et al, August 2020 doi:10.13140/RG.2.2.21500.36487

Designing a toolbox

Example with SBS polymer

Examples with pine chemistry additives

Key takeaways

The need for toolbox

- Constant changes
  - In material properties: bitumen, mix design
  - In specifications: regulation
  - In demand: future mobility, circularity
- A path for advanced technologies
  - Existing solutions
  - New developments
- Evaluation framework
  - Going beyond standard specifications

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Examples of possible technologies

- Polymer modified Bitumen to enhance high traffic road performances
- Bitumen rheology modifiers
- Chemical dopes to address chemical functions
- Asphalt recycling additive to take recycling to the next level
- Bio-bitumen extender to improve the carbon footprint
- …

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Evaluation framework

- **HSE:** Safety data sheet, fume emissions, …
- **Technical:** compatibility, durability, specifications, …
- **Processability:** use conditions, handling, …
- **Environment:** EPD/LCA, recyclability, …

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iFRAE
DELFT 2021
5th International Symposium
on Frontiers of Road and Airport Engineering
Workshop: Changes in binder properties and the role of additives
Designing a toolbox for bitumen to answer
the need for tomorrow's pavement
Thank you!
Laurent Porot, Kraton Polymers B.V. the Netherlands
TUDelft

4. Workshop Discussion

Topics discussed during the workshop varied from the changes in binder properties to analysis approach, recycling technology and polymer additives design. Some points that came up were:

- During production of asphalt with reclaimed asphalt more benzene is measured in the emissions than with production of asphalt without reclaimed asphalt. What is the reason of this? Does the benzene come from aged bitumen in reclaimed asphalt?
- ➢ *Based on the report from the organization regarding the benzene research. https://www.bouwendnederland.nl/actueel/nieuws/19495/onderzoek-benzeen-wijst-de-weg-naar-circulaire-asfaltproductie-binnen-uitstootnorm.*

- There was also a problem with smell during asphalt production and application.
- ➢ *There is a report from the contractor that they had the problem with the mixing, and more energy is needed when compaction. And indeed, there were several mentions about the not nice smell. Those are the contractors' information and again I don't know which place and which bitumen specimen it was. We need to look into this, but most times we just see that the report will be done later or they will come together. And then we see the problem, then you go look into this. And then you go back to check what was happening. They are still working on this.*

- You mentioned that some of correlations have not been validated. So, are you still working for validation.
- ➢ *We have additional programs. but we do that step by step. And new indicators will be proposed to validate the correlations. We have lots of research points. We want to work with lots of people.*

- Why do we still use old fashioned tests (more than 100 years old!) like penetration and T ring & ball to characterize pen grade bitumen and PmB and for the bitumen requirements, while we have nowadays very good sophisticated tests which have a better predictability for field performance. DSR and other tests give a better understanding of the bitumen quality.
- ➢ *From DSR, DSC, we have lots of new tests. We cannot predict the final products. We select the bitumen with different properties. Lots of conventional tests not allow the durability evaluation, and we need new test to select the base bitumen although some of them are not in application.*

- Do you establish some link between contraction of variation with some molecule's unbalances (SARA) into colloidal matrix and/or specific hierarchical organization of molecules?
- ➢ *We come up with the specification free more that can be used with performance evaluation could be run in other's lab not just FHWA. So, we want to have something simple, practical and does not require chemistry PhD to run these types of tests. So that is our idea, and that's why we use ABCR and BBR to study them. There were some correlations with*

chemical properties. However, we found some correlations with SARA fractions. Delta Tc can capture the unbalanced binders and delta T_f was given advantage to the polymer modification and high stress binders. We need both parameters and one of them would not be efficient. And the whole idea is that we had to have some failure properties to control the binder property. In the US, binder is the only construction material and without studying the failure properties, we study the G and phase angle in linear viscoelastic region, but never go beyond the linear viscoelastic region to find some failure properties.*

- How is the TRL established? Civil technical performance at point of laying, life-time effects? or even re-recycling?
  - *So, the technology readiness level is defined actually by NASA. We only look at the mixture performance and not binder, and he identified if there were several failed performances, then he put that into for example level 7 to 9. So, that is based on what was published and the literature published available.*

- Looking at the mixture performance is indeed a good approach. But…. we should take in account that with using different materials, the failure mechanism could change as well. Did EMPA do some research on this aspect?
  - *We have done research on PE, CR and RCA mixtures. Some are published some in the pipeline. Regarding failure mechanism you are right but we tried to reach the reference performance. Our reference mixture is a semi dense asphalt with pmb.*

- In the RAP you don't see bees. Can they be influenced by extraction of the aged bitumen with solvent?
  - *The solvent is not remaining in the aged binder because it goes to heat and evaporated. But I believe that there is difference between what we extracted still have filler, and we do not reproduce the virgin binder. We only use toluene and I do not know if other colleagues use different solvents.*

- The properties of AC with waste materials, is this only initial properties or also over time/aging? Does it address AC or also PA?
  - *I showed some binder results and some mixture results yesterday. And there we did not aged binder. The asphalt mixture performance is reviewed in the paper, and they are all from the literature published on laboratorial produced mixtures.*

- Do you know where these three rejuvenators would be located in terms of their polarity or in the SARA fractions? Also, do you consider that the bee structure has an Impact on mechanical Performance of the material? (Does it matter whether it is there or not)
  - *That's a big million-dollar question. We have done some works to look at the surface properties, published in TRR. There was a correlation. However, we do not have the same material. We still have the oxidation indicators with sulfoxide and carbonyl index from FTIR test. So, we look at different material and the bee structure can have the influence on mechanical performance of materials.*

- I wonder if you and your team have plans on conducting LCA and LCCA. Will you and your team approach "lifetime" in your LCA and LCCA? And how?
  - *We have a current PhD looking at LCA cradle to grave. SARA has been developed for bitumen, petroleum-based product, and is not suitable for other product in nature. it all depends on the solvents used.*

- So, you did not use the EU "translation" of the NASA TRL definitions?
  https://doi.org/10.1016/j.conbuildmat.2021.122492
  https://doi.org/10.1016/j.jclepro.2020.124916
  https://doi.org/10.1016/j.conbuildmat.2020.120166
  We used the original Nasa definition see link above. The Paper lists the criteria for using the results in the literature. essentially, we looked for comparison with ref material.
  Please see link below for specimen preparation including extraction of RAP and rejuvenation
  https://doi.org/10.1080/14680629.2019.1691042

- What was the procedure to let the rejuvenator be effective on the RA? And how did you reclaim the binder?
  - *I think the important thing you have to understand is this rejuvenator cooperate with bitumen which you would like to use because bitumen quality is change. In this case, we use the aged binder at lab and no extraction. The extraction will bring some other things to the aged binder and influence the result.*

- What are the advantages of considering stress relaxation instead conventional MSCR in case of rejuvenation?
  - *We try to look at the higher temperature response with MSCR. So, the stress relaxation is used to evaluate the low temperature property.*

- How is the ageing behavior of pine wood additives and how does it affect the ageing behavior when blended with Bitumen?
  - *That's something we have to consider that case at least from pine chemistry the good thing is that the molecule is known for 15-20 years. So, the aging behavior of pine wood additives depends on the processing, which differs with different applications.*

- In your opinion, what is the best approach to explain the chemical reactions between additives and asphalt binder that result in rheological changes? From my point of view this is very challenging.
  - *All chemical analysis from bitumen and no necessary for non-bitumen products. So, if you look to SARA analysis, GPC, whatever, we are very close to the results outcomes. I would like to say that physical properties are related to the rheological properties, for example G* or BBR from us.*

- A lot of additives are suggested for being an improvement for the asphalt. What is your point of view on this? (should some additives be blocked?)

- *We have lots of additives we can choose in the market. All of benefits and drawbacks should be on basis of which properties you want to work for. And sometimes, many effects just focusing on viscosity or aging. So, we have no unique factor answer and that's why I do the presentation about the design of toolbox today.*