InnovA58 materials research

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Scope of InnovA58

CONTEXT

- ‘InnovA58’ - innovation test section is a location for demonstrating sustainable road materials.
- Four surface layers have been constructed, using recycled asphalt (60% PR) and four rejuvenators in sustainable asphalt mixtures (DZOAB).

OBJECTIVE

- To evaluate the effectiveness of different rejuvenator systems for DZOAB with RAP

SCOPE

- The scope of the research focuses on two parts: a material research and an emissions research.

Overview of the mixture systems

- **Mixture 1:** DZOAB16 60% PR with bitumen 70/100 + rejuvenator 1 (V1)
- **Mixture 2:** DZOAB16 60% PR with bitumen 70/100 + rejuvenator 2 (V2)
- **Mixture 3:** DZOAB16 60% PR with bitumen 70/100 + rejuvenator 3 (V3)
- **Mixture 4**: DZOAB16 60% PR with bitumen 70/100 + rejuvenator 4 (V4)

B- 70/100

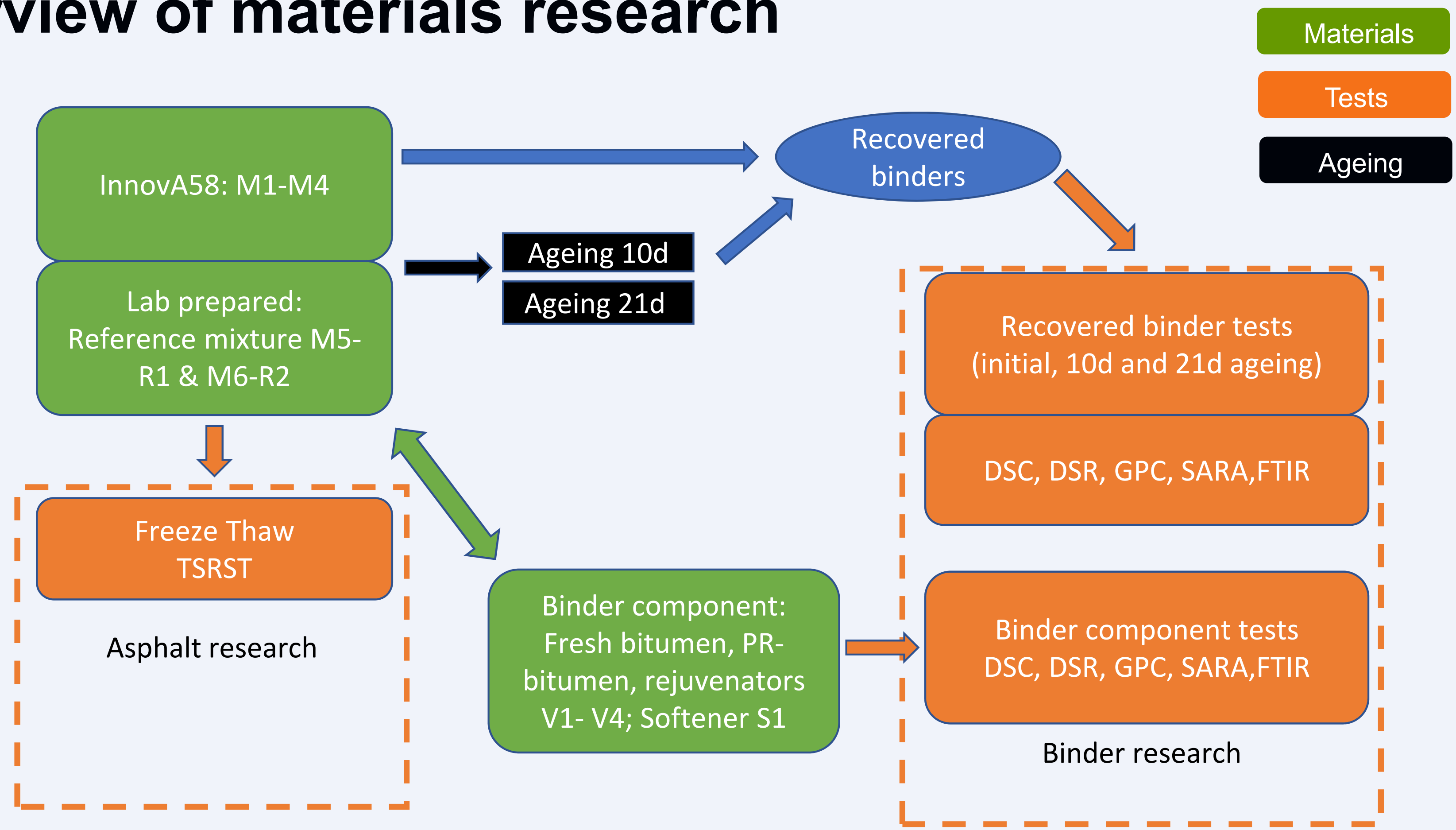
- **Mixture 5:** DZOAB16 60% PR with bitumen 160/220 (R1)
- **Mixture 6:** DZOAB16 60% PR with bitumen 70/100 + 'softener' (R2)

E- 160/220

C- 70/100

- Mixtures 1 to 4 are from InnovA58
- Mixtures 5 and 6 are the references, prepared in the lab

Overview of materials research



Binder research: Goals

1. To fingerprint the rejuvenators and the softener
2. To assess effectiveness of the rejuvenators
   a) initial properties
   b) influence of ageing on (initial) properties

Binder composition:

- M1 to M4: rejuvenator + old bitumen PR + fresh bitumen70/100
- M5-R1: old bitumen PR + fresh bitumen 160/220
- M6-R2: softener + old bitumen PR + fresh bitumen 70/100

Binder research: Overview of test methods

Binder

- DSR
- DSC
- GPC
- SARA
- FTIR

Binder
Bitumen
- B- 70/100
- C- 70/100
- E- 160/220

Rejuvenators
- V1
- V2
- V3
- V4

Softner
- S1

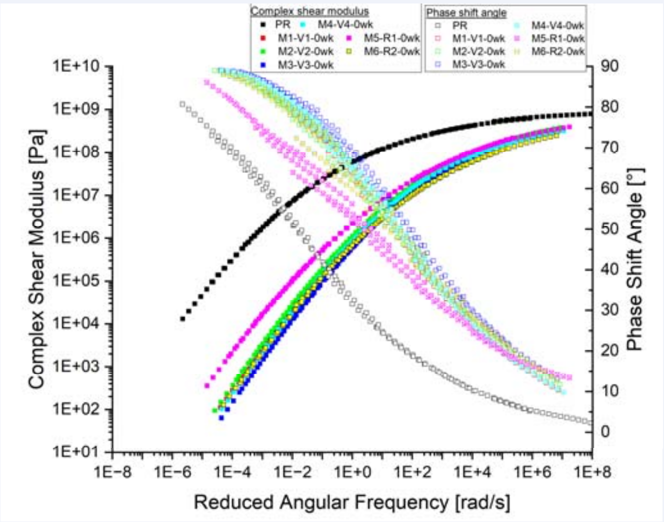
Binder (Extracted)
Recovered bitumen
- PR
- 4 mixtures and 2 references
  - t=0
  - t= *10 days ageing
  - t= *21 days ageing

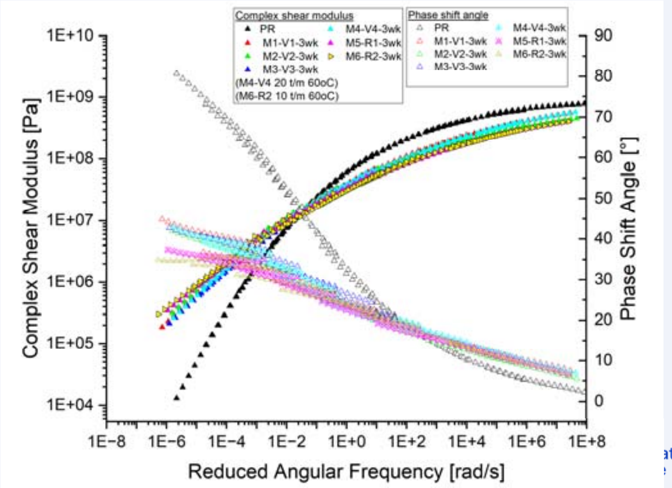
*laboratory ageing, following the TNO protocol
(85° C, 150 bar-AAP+, TKI accelerated ageing)

DSR master curves

- **Initial properties:**
  - o M1 to M4 binders show a comparable rheological response.
  - o M5-R1 binder (with soft bitumen) shows higher stiffness compared to the other binder systems
- **10 days of ageing:**
  - o M1 to M4 and M5-R1 binders show similar G* and δ but M6-R2 shows more stiffening.
- **3 weeks of ageing:**

o G* profiles indicate higher stiffness values than the PR binder and 'δ's show higher elasticity at low frequency.

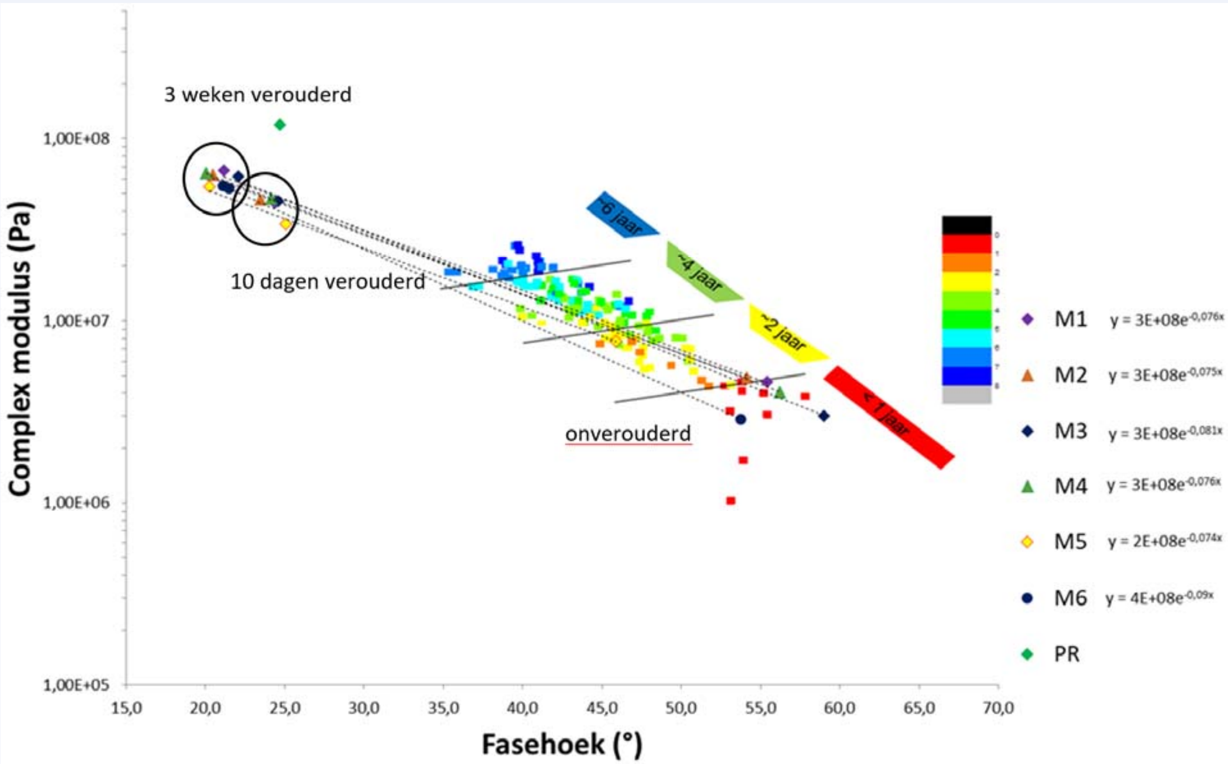




DSR- G* and δ at 10 rad/s, 20 °C

Binder	Initial		Aged			
			10 days		3 weeks	
	|G*| [Pa]	δ [°]	|G*| [Pa]	δ [°]	|G*| [Pa]	δ [°]
M1-V1	4,60E+06	55,37	4,43E+07	24,37	6,64E+07	21,17
M2-V2	4,87E+06	54,09	4,63E+07	23,48	6,32E+07	20,45
M3-V3	3,03E+06	58,99	4,56E+07	24,60	6,19E+07	22,07
M4-V4	4,08E+06	56,21	4,68E+07	24,19	6,44E+07	20,04
M5-R1	7,77E+06	45,89	3,38E+07	25,09	5,42E+07	20,28
M6-R2	2,90E+06	53,69	5,51E+07	21,10	5,33E+07	21,46
PR	1,19E+08	24,68				

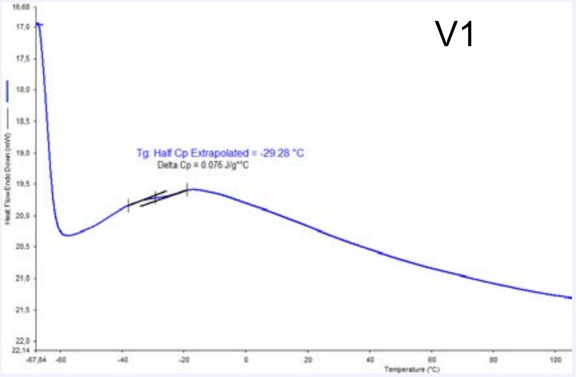
DSR- BSBD

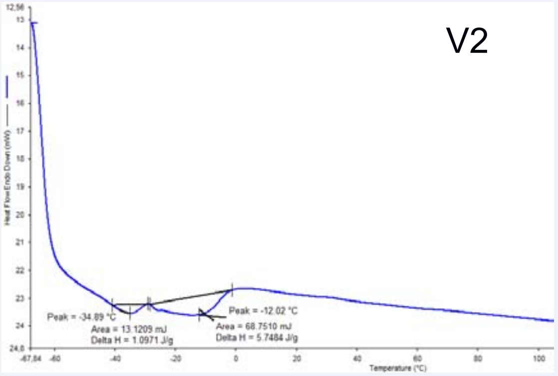


- PR binder shows 15-17 years of field ageing and follows the trend in the BSDB database
- M1 to M4 binders, at initial condition, have properties comparable to 1 to 2 years of field ageing and M5-R1, 3-4 years ageing
- After 10 days and 3 weeks of ageing; binders simulates ~15-17 years of ageing.
- After ageing, all binders show a shift in the trend line suggesting a lower phase angle for the same value of the complex shear modulus.

DSC

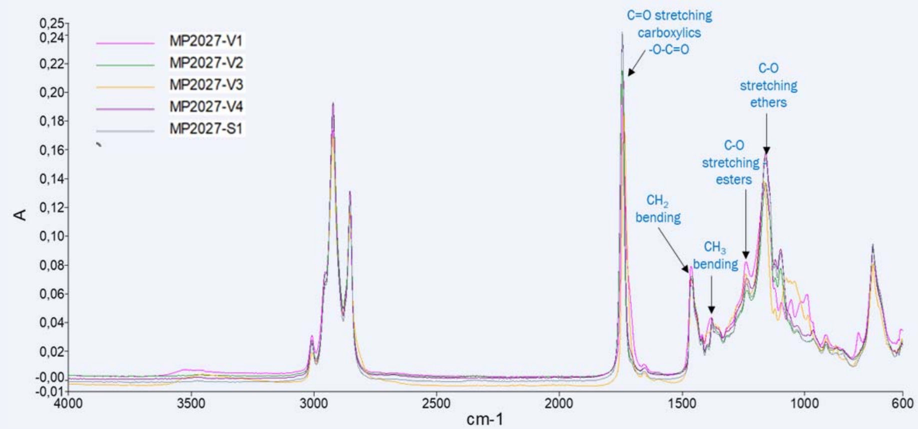
- DSC data provides thermal fingerprint of each rejuvenator.
- The Tg of the rejuvenated binders were lower than the PR binder showing restoration.
- With ageing, the Tg of the binders increased and 10 day and 3 week aged binder showed similar Tg values. (supported by rheology).



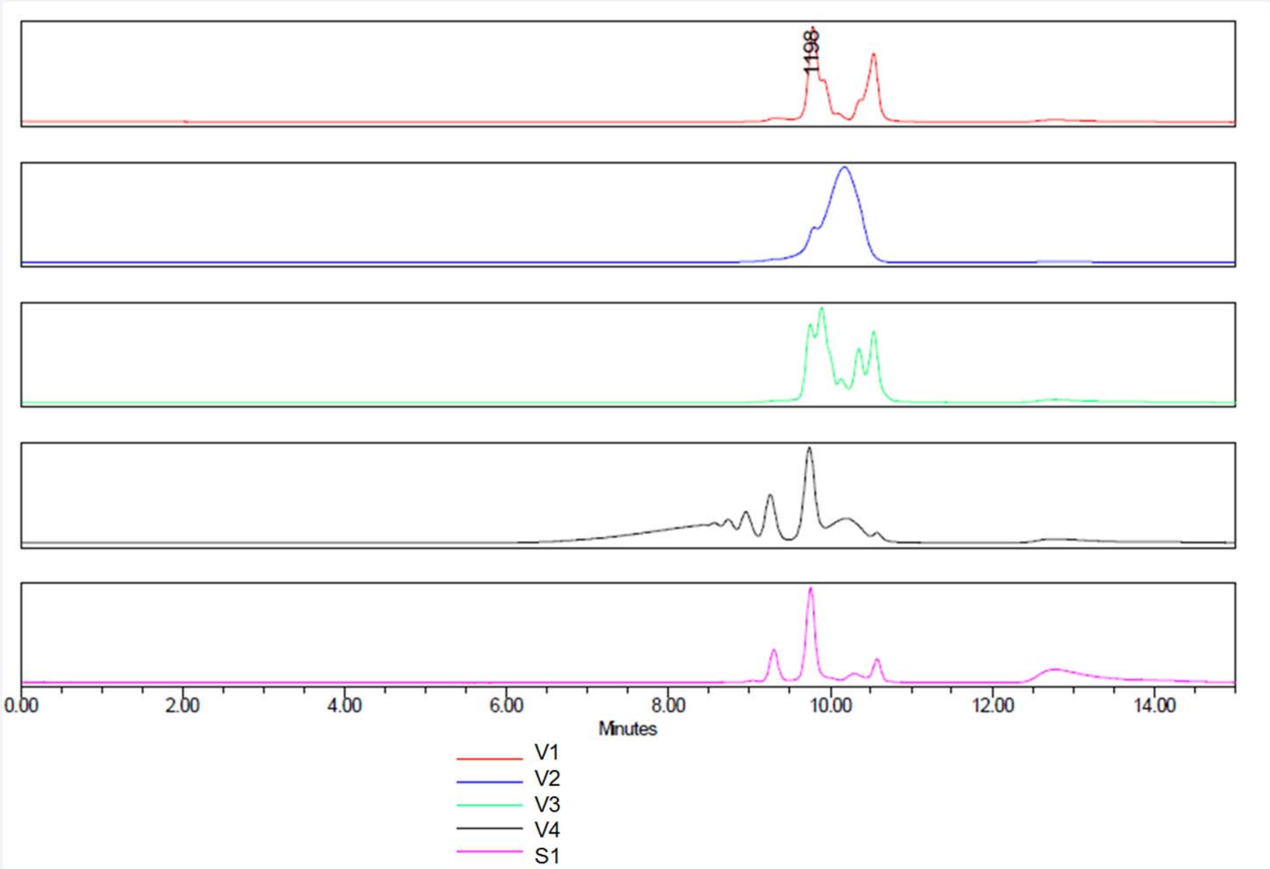


FTIR

- FTIR identifies characteristic functional groups (i.e. carboxylic groups, aliphatic chains and ester and ether groups) present in rejuvenators and the softener.
- M1-M4 binders at initial condition, shows trace peak of characteristic functional group (the peak at 1740 cm-1).
- After 10 days of ageing M1 and M4 showed the peak.
- After 3 weeks of ageing, the carboxylic group was not present.
- C=O, S=O:
  - The carbonyl and sulfoxide peaks were higher after 10 days and 3 weeks of ageing.
  - After 3 weeks of ageing, carbonyl peaks were comparable to PR binder but the sulfoxide peaks were higher.

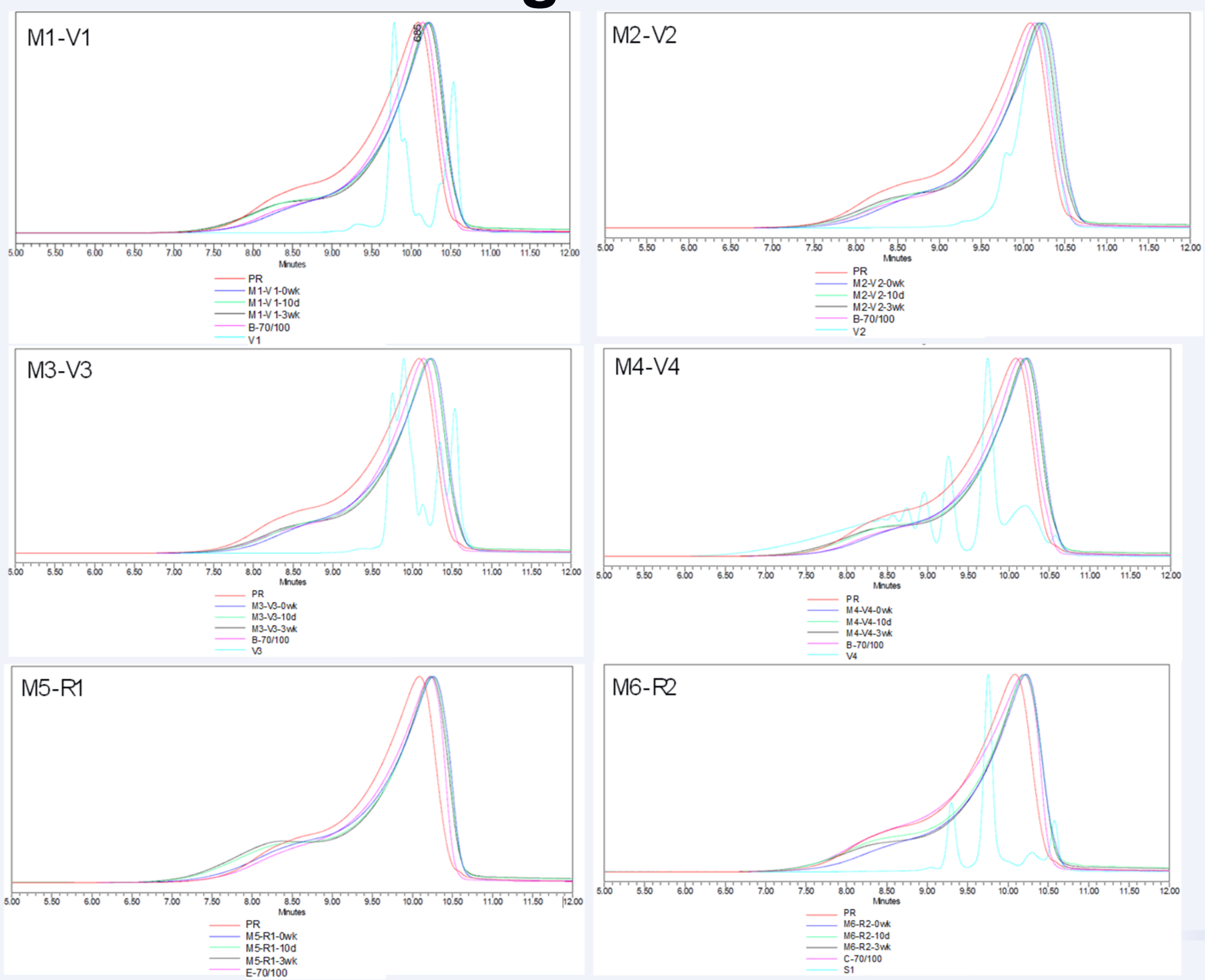


GPC: Chromatograms of rejuvenators/ softener



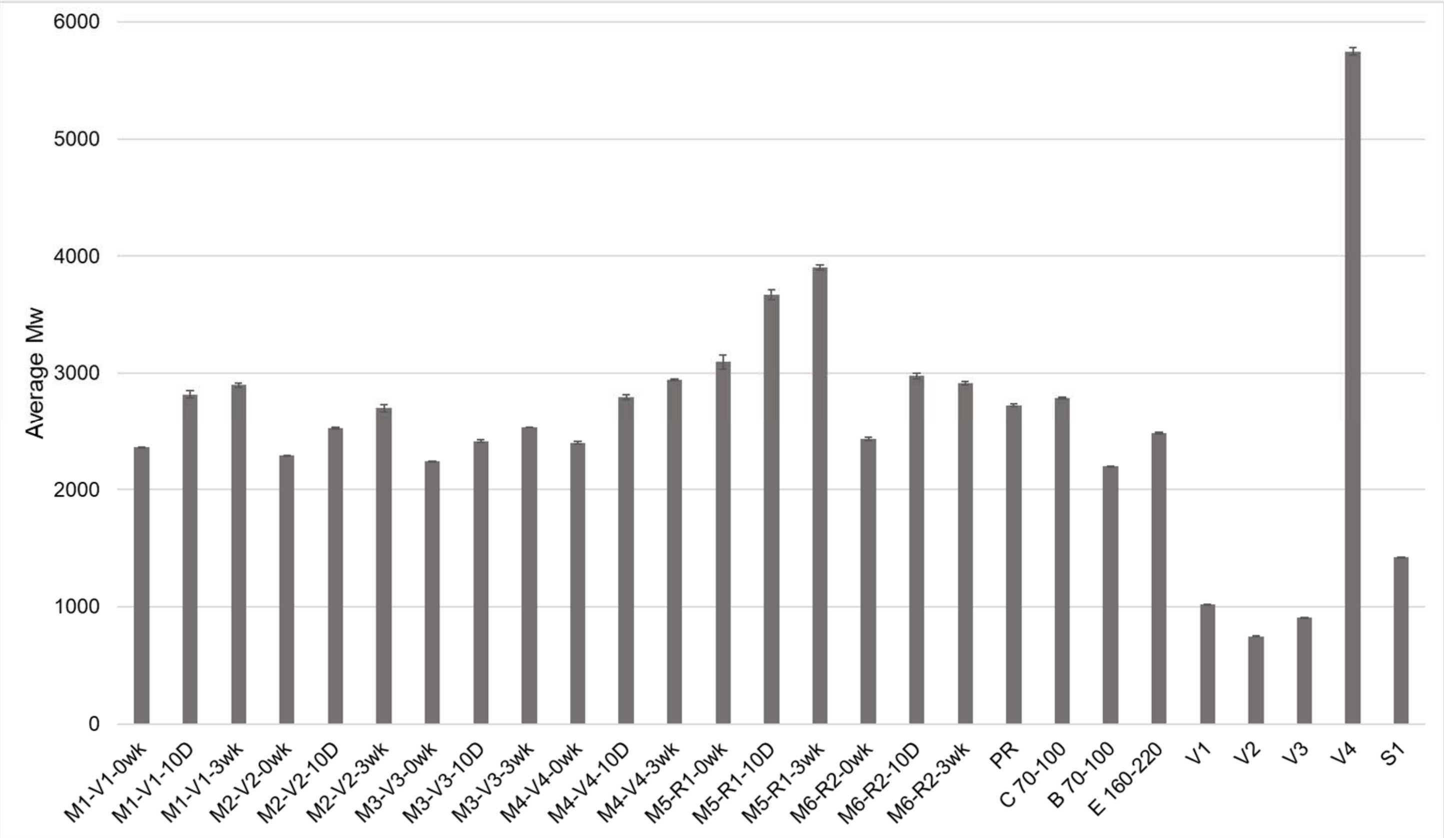
The GPC chromatograms serves as molecular mass distribution fingerprint for each rejuvenator / softener

GPC: Chromatograms of the recovered binders



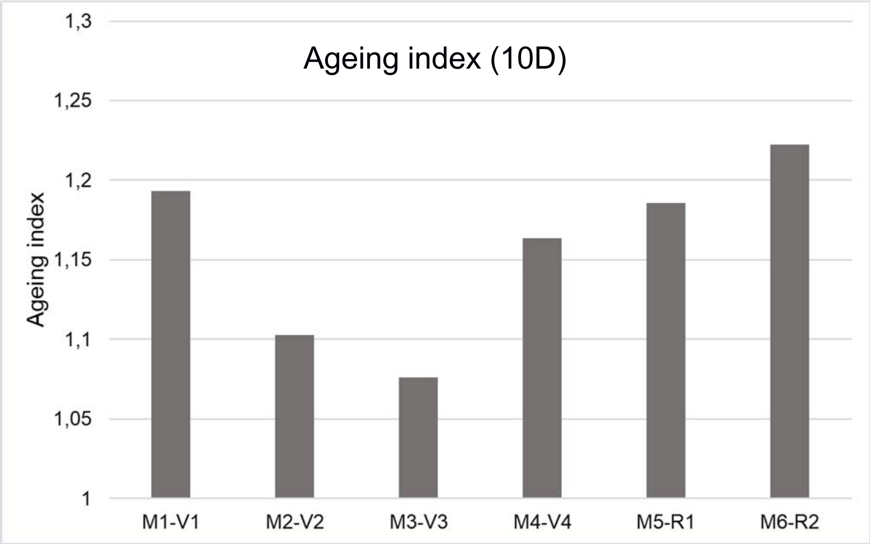
- After ageing, the average molecular weight- M_w of the binders increases.
- After 3 weeks of ageing, M5-R1 binder show higher intensity in high molecular mass regions than the PR binder

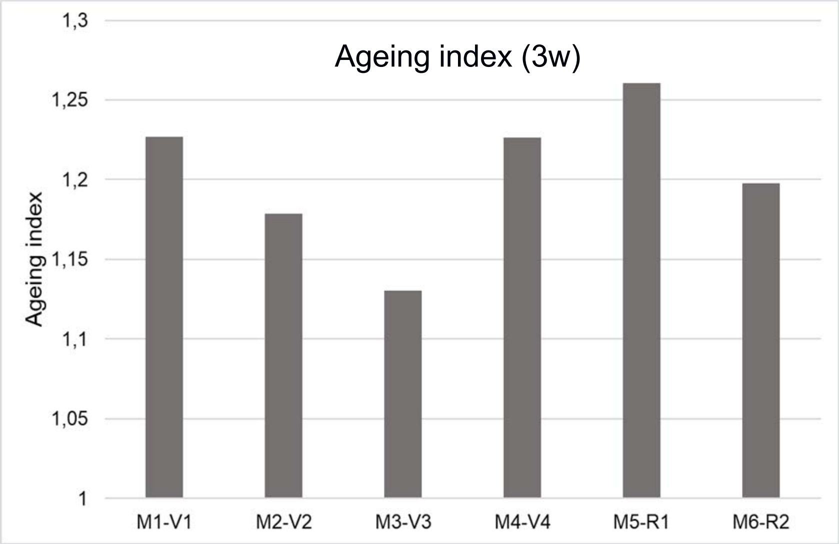
Comparison of GPC data: Mw



M_w and polydispersity increase with ageing

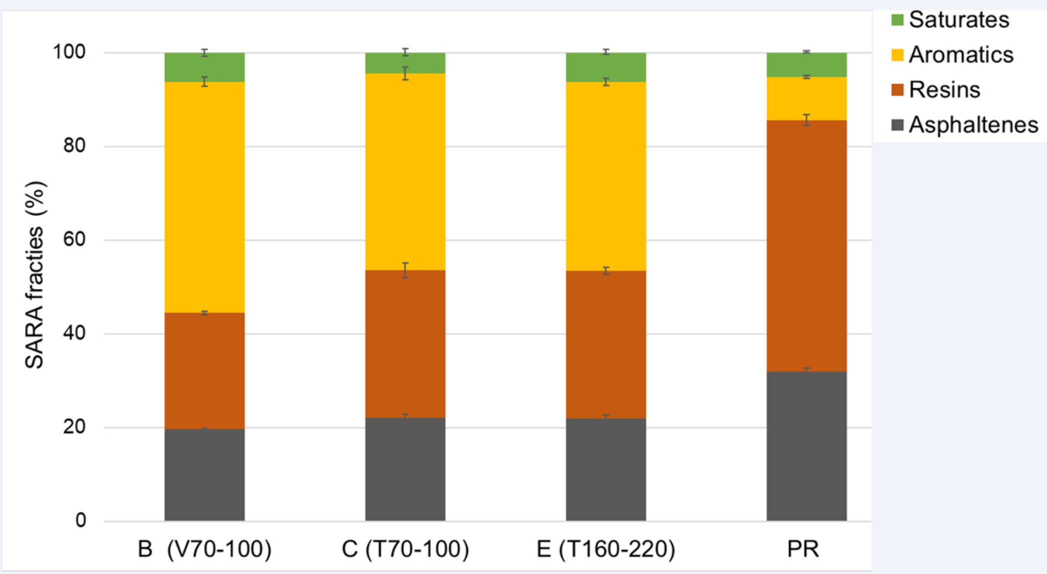
GPC: Ageing index based on Mw



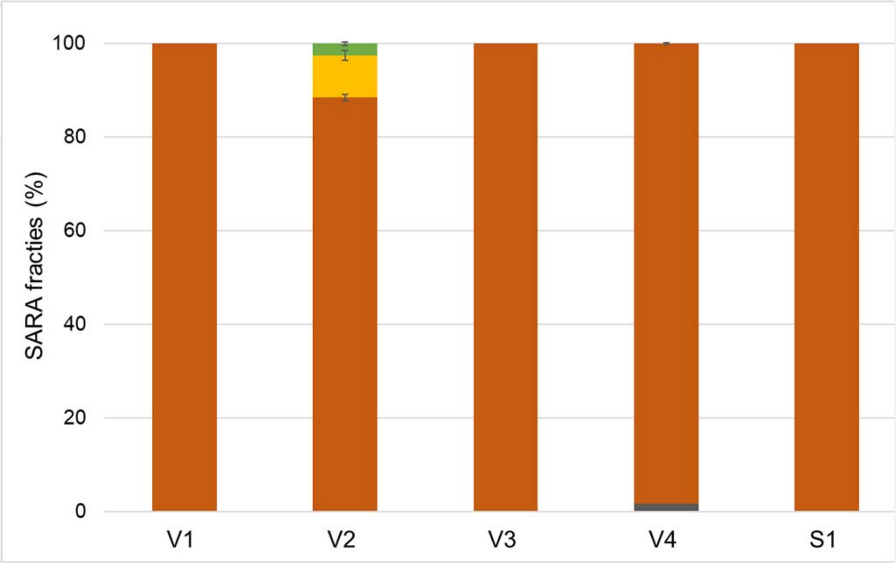


- Ageing index= $M_{w\text{-aged}}/M_{w\text{-fresh}}$
- 4 Rejuvenated mixtures exhibit different Mw and ageing index after ageing
- For 3 weeks, M1 and M4 show similar ageing degree, M5-R1 shows the highest.

SARA fractions: Fingerprinting the binder components

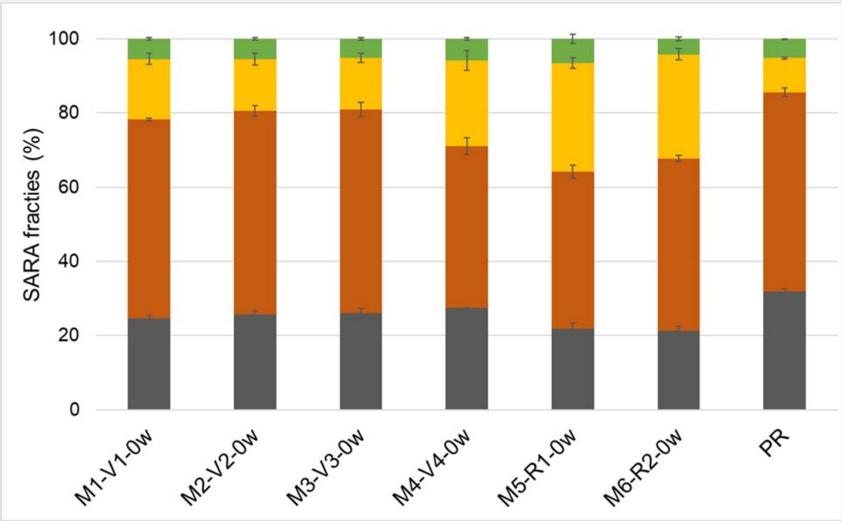


Bitumen

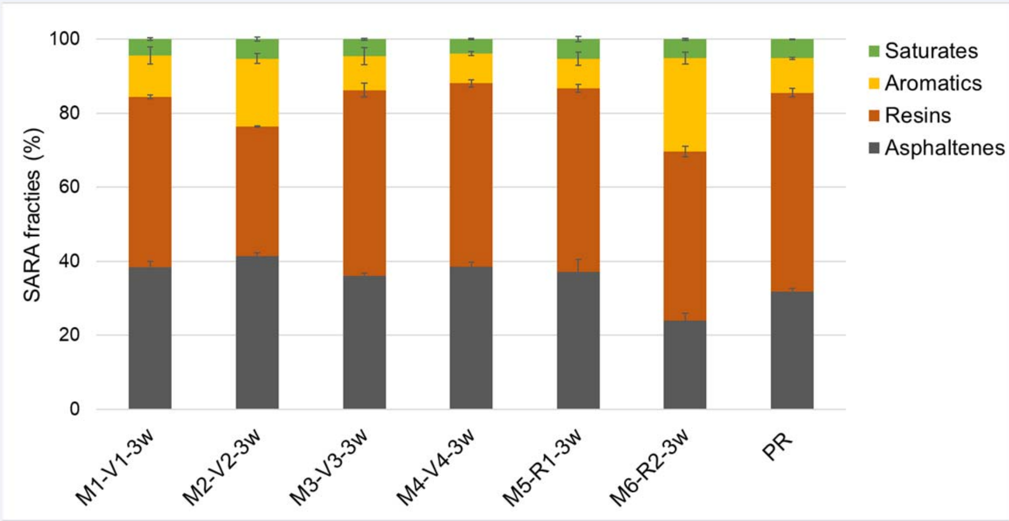


Rejuvenators/softener

SARA fractions



Fresh

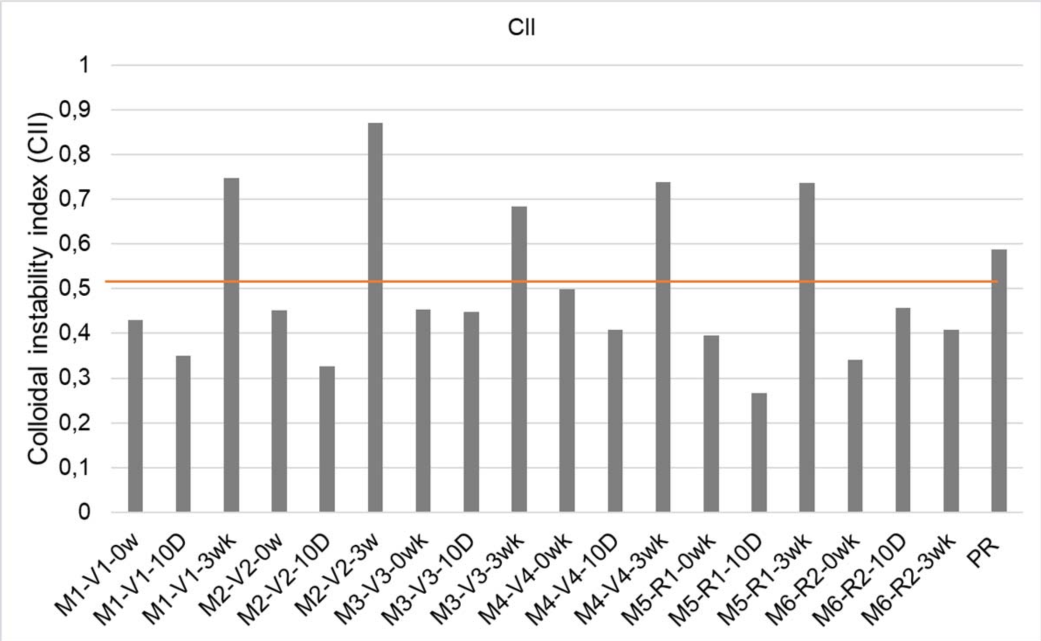


21 days aged

- M1 - M4 binders could restore the SARA fractions of the PR bitumen.
- Aromatics fraction was not completely restored.
- Ageing causes changes in the SARA fractions, mainly in resin, asphaltenes and aromatics.

Colloidal instability index

- $CII = \frac{(asphaltene + saturates)}{(resin + aromatics)}$



Austroads2006 recommends a CII value of 0.22 < CII <0.55 for conventional bitumen

- **Colloidal instability index (CII):**
  - Ageing after 3 weeks: M1-M4 binders are less stable compared to the PR binder

Asphalt research: Overview of the test methods

- Asphalt
  - Freeze-thaw test
  - (m)-TSRST
  - Visual inspection

Freeze-thaw test: protocols I and V from the InfraQuest frost damage research (*Giezen, et al., 2013).

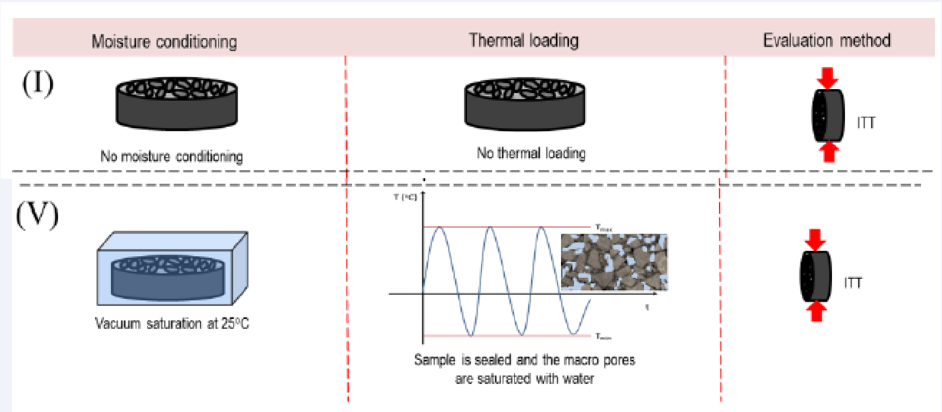
Thermal Stress Restrained Specimen Test (m-TSRST): (NEN-EN 12697-46:2020, procedure 8.2)

Visual inspection and microscopy

- *Visual inspection:* after freeze-thaw conditioning and ITT test.
- *Microscopy:* on thin sections using polarization and fluorescence microscopy (PFM).

Freeze-Thaw

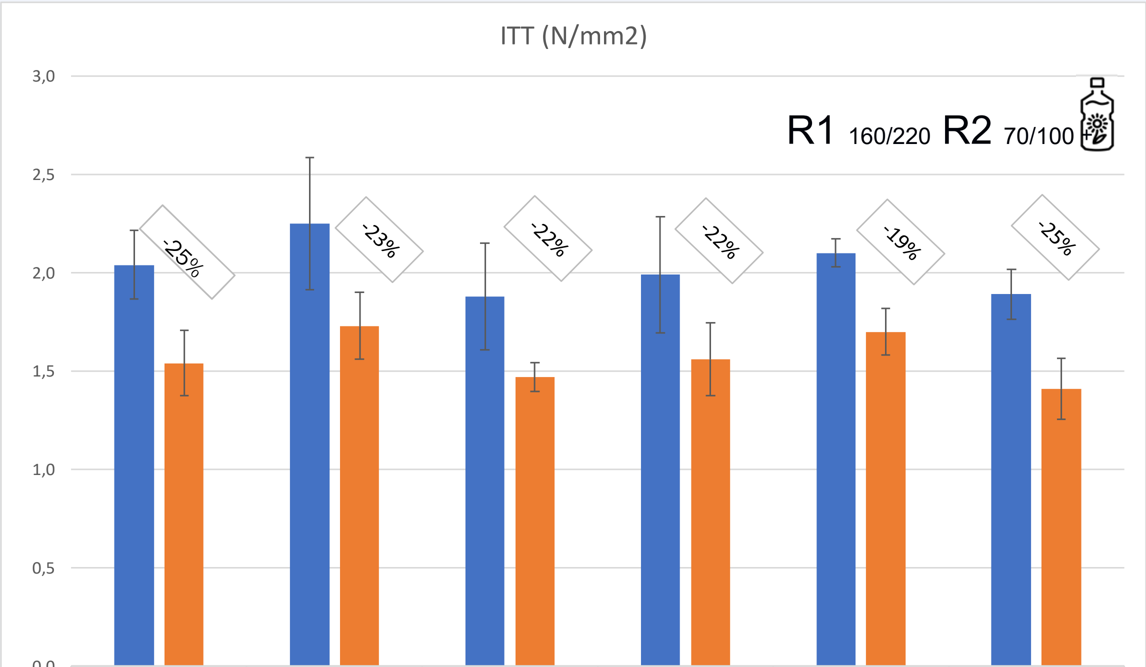
- *Protocol I:*
  - Asphalt cores are stored at 5°C.
- *Protocol V:*
  - combination of moisture and temperature load (i.e. temperature variations).
  - 1st the cores were pre-conditioned in freshwater to saturate the micropores.
  - After pre-conditioning, the cores were placed in a climate chamber for the frost-thaw load where macropores were saturated with water.



Protocol(I)	Specimens remained in storage at 5°C during the temperature loading of the specimens in protocol V.		
Protocol (V)	Number of thermal cycles	24	Cycles
	Duration per cycle	48	hours
	Total duration	48	days

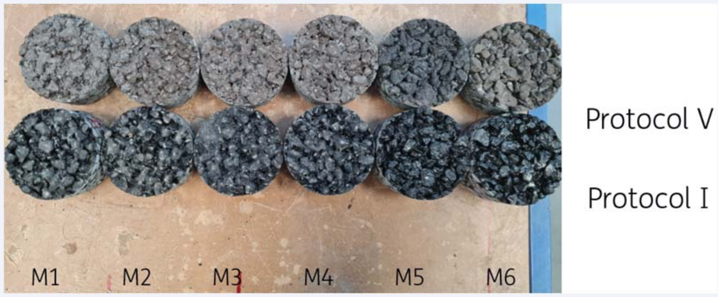
Temperature (interval)	Duration (Total 48 h)
+18 to -15 °C	4 h
-15 °C	12 h
-15 to +18°C	2 h
+18°C	6 h
+18 to -5 °C	2 h
-5 °C	14 h
-5 to +18 °C	2 h
+18 °C	6 h

Freeze-Thaw: average ITS value



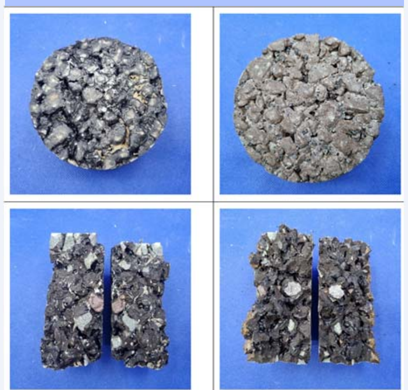
	Mengsel 1	Mengsel 2	Mengsel 3	Mengsel 4	Mengsel 5	Mengsel 6
I	2,0	2,3	1,9	2,0	2,1	1,9
V	1,5	1,7	1,5	1,6	1,7	1,4

- ITS values for protocol I and V are comparable
- A decrease in average ITS value was observed for all mixtures after freeze-thaw loading
- The spread in the results is higher for the field mixtures (M1 to M4) than the lab mixtures M5-R1 and M6-R2.
- Protocol V causes discoloration of the specimens. M1-M4 and M6-R2 show a light brown discoloration, while M5-R1 shows a gray discoloration



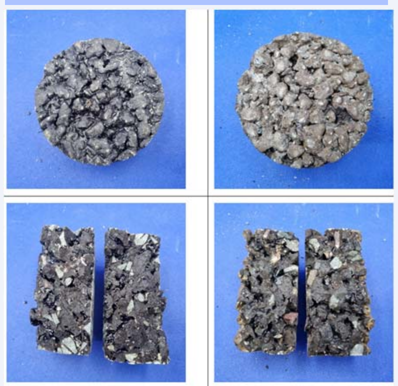
Visual assessment after ITT

M1



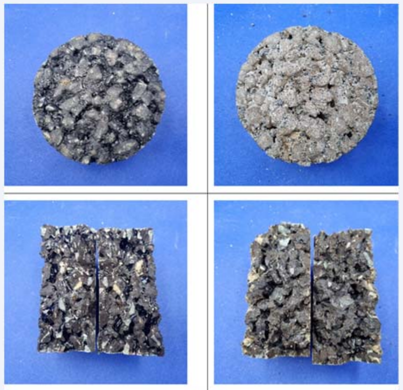
Mengsel 1, protocol I, 67A | Mengsel 1, protocol V, 74A

M2



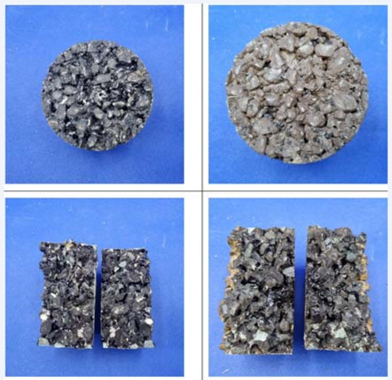
Mengsel 2, protocol I, 29C | Mengsel 2, protocol V, 37C

M3



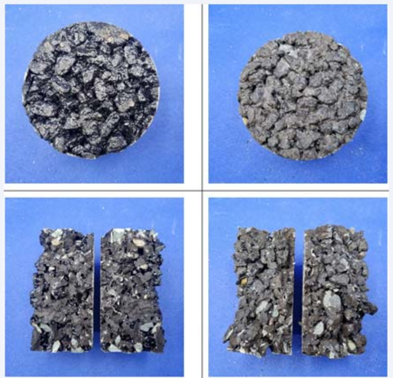
Mengsel 3, protocol I, 55A | Mengsel 3, protocol V, 56C

M4



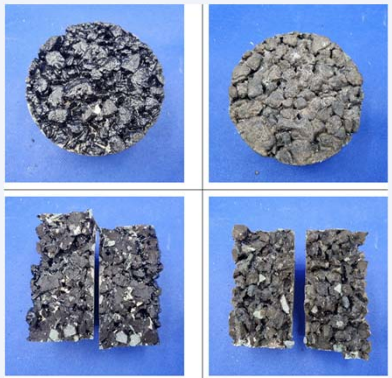
Mengsel 4, protocol I, 8A | Mengsel 4, protocol V, 18C

M5



Mengsel 5, protocol I, 65844 | Mengsel 5, protocol V, 65865

M6



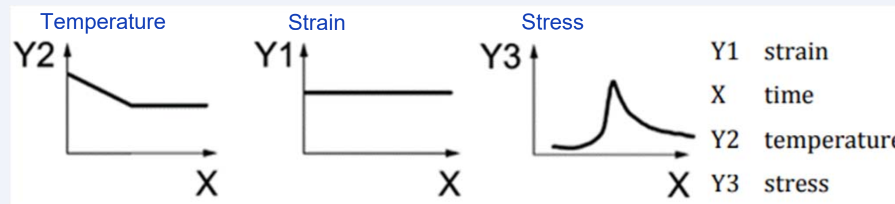
Mengsel 6, protocol I, 65812 | Mengsel 6, protocol V, 65833

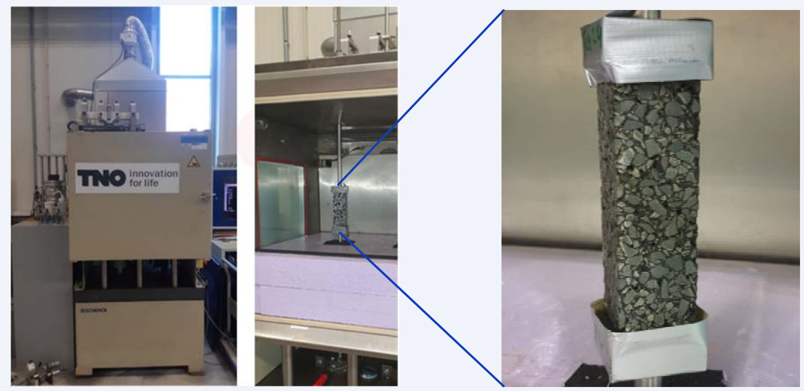
- Specimens fractured vertically along the aggregate
- M1 to M6 show the same fracture patterns (combination- c) in both protocols
- Specimens show cohesive failure, where few aggregates were broken
- The discoloration occurred on the surface not affecting the bulk

m-TSRST

- Prismatic specimen is clamped, fixed to its ends and subjected to a linear temperature decrease.
- Thermal conditioning
  - Start temperature +20 °C
  - End temperature -15 °C

    12 h
  - Isothermal conditioning: -15 °C for 8 h (relaxation)
  - Three tests performed per mixture

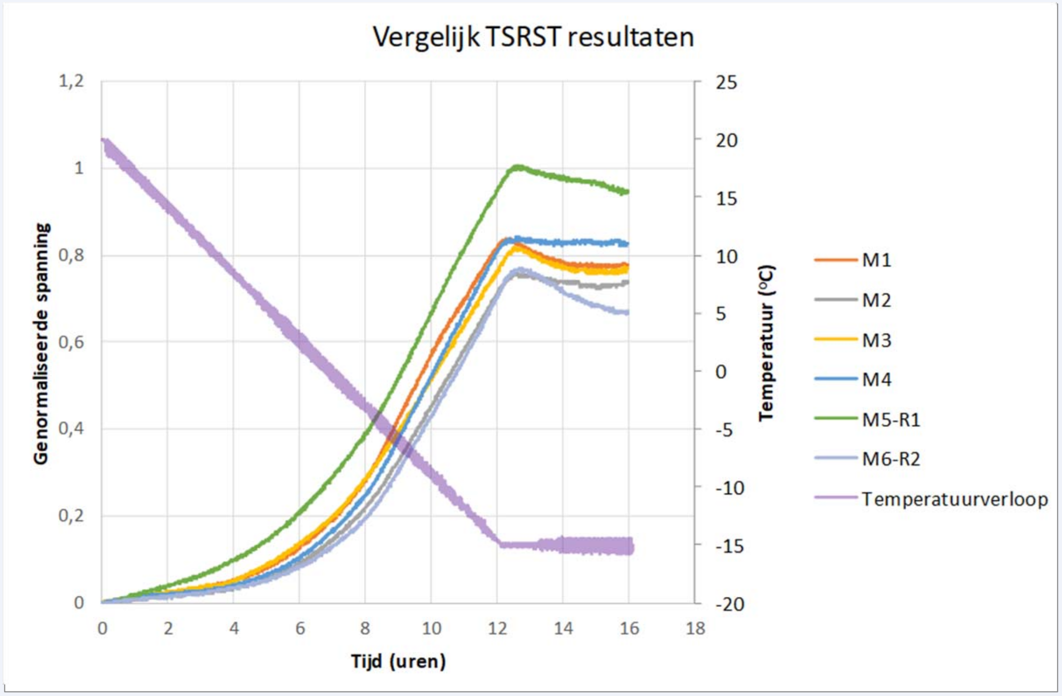




Instron-1 setup

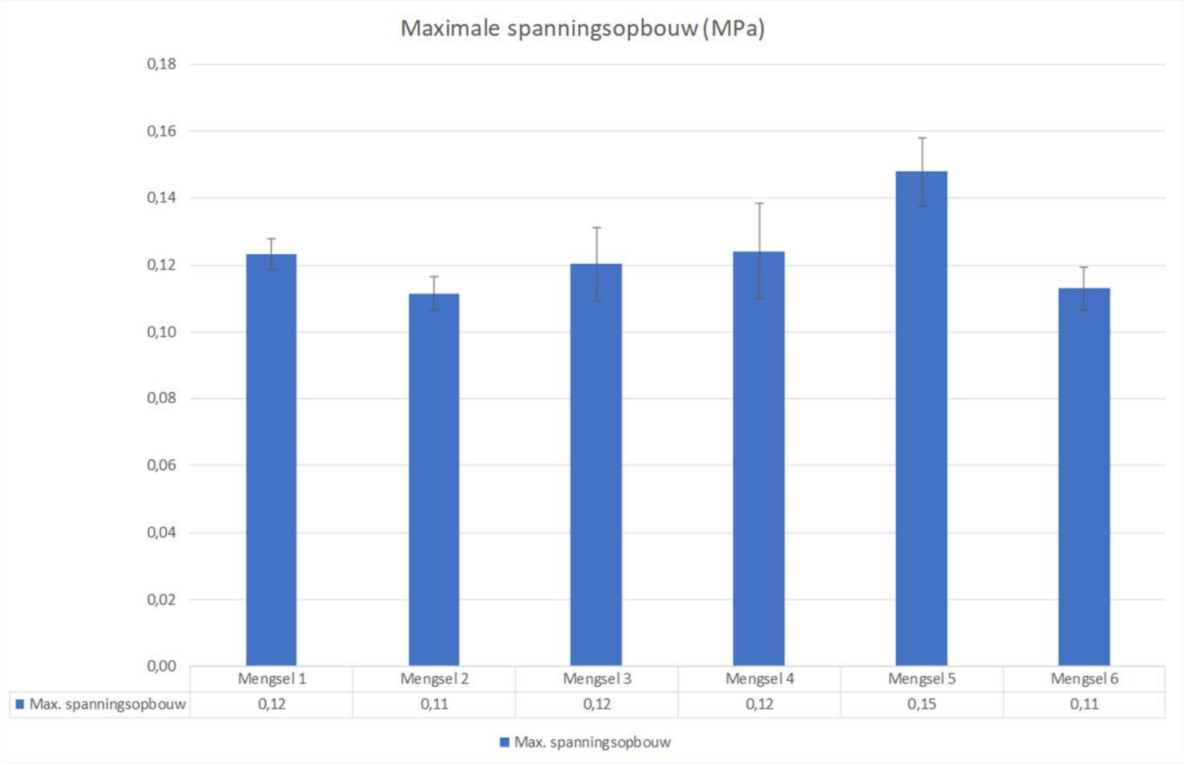
Prismatic specimen

Stress development (MPa) during TSRST



- The stress buildup profile per mixture is the average of three tests
- The stress build-up for M1 to M4 and M6-R2 is comparable.
- M5-R1 with 160/220 bitumen shows a higher maximum stress with limited degree of relaxation.
- The rheology confirms that the M5 binder is stiffer.
- Normalized graph shows that M1 to M4 reach approximately 80% of the maximum stress of M5-R1.

Maximum stress build-up during TSRST



- Maximum stress build-up (MPa) during TSRST for M1 to M6. The stress build-up per mixture is the average of three tests

Summary

- Objective was to fingerprint rejuvenators and evaluate their effectiveness, at initial state and after ageing.
- Provides data on chemical, molecular, thermal, rheological and mechanical properties, a comprehensive assessment of rejuvenators, and potential to restore properties.

Fingerprinting of rejuvenators:

- Each rejuvenator can be identified based on their unique chemical properties.

Effectiveness of rejuvenators:

Initial Properties

- 4 systems, M1 to M4 and reference M6 show comparable effectiveness in restoring rheological properties of PR binders.
- Assessment at the asphalt level: freeze-thaw and relaxation behaviors are comparable between the 4 systems and M6

Effect of ageing

- Based on the BSDB, the PR-binder indicates ~15 to 17 years aged asphalt.
- After artificial ageing of 21 days, which should simulate 10 years of field aging, show properties similar to PR bitumen.
- The mixtures show higher ageing-sensitivity (with 60% RAP) compared to standard asphalt mixtures with only fresh components.
- A combination of techniques is useful for comprehensive assessment of rejuvenator- systems.

Recommendations

- As a control mixture system, one mixture with **PR+70/100 bitumen** will be insightful specially for the chemical evaluation.
- A mixture with fresh **70/100 bitumen** without PR can provide insights on the effectiveness of the rejuvenated systems.
- For the fingerprinting of rejuvenators, the combination of chemical and thermal tools are recommended- FTIR, SARA, GPC and DSC.

Approach for evaluating the effectiveness of rejuvenators:

1. To identify and fingerprint the rejuvenator (**FTIR**, SARA, **GPC** and DSC)
2. – Artificially age the rejuvenated mixture to simulate field-ageing

   – To determine rheological properties of the recovered binder from the **unaged and artificially aged** mixture and compare with the benchmark,

   – For a comprehensive evaluation, include characterization techniques used in fingerprinting

   (FTIR, SARA, GPC and DSC).
3. To determine mechanical properties of the rejuvenated asphalt mixture (rapid evaluation ITSR; extended freeze-thaw evaluation; TSRST).