Effect of sample preparation and testing methods on the measured rheological parameters of bitumen using a Dynamic Shear Rheometer

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NABin-seminar
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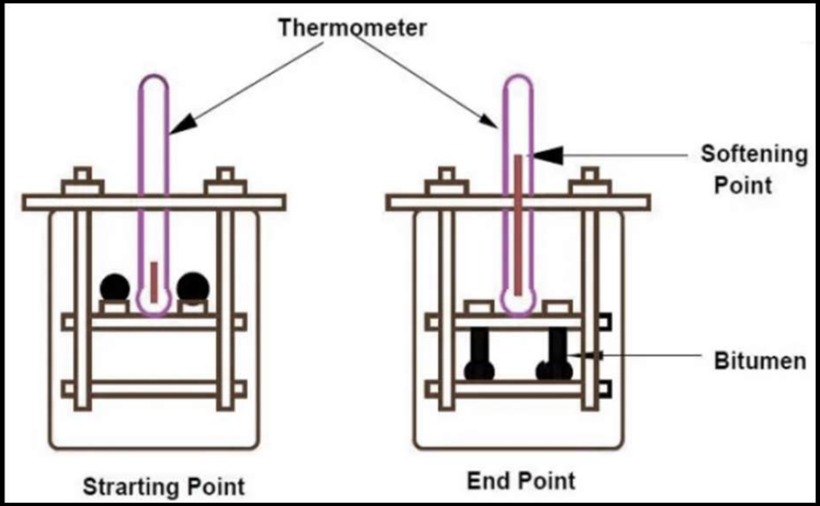
Today's Agenda

- Background
- Purpose of the Project and my Findings
- Future research

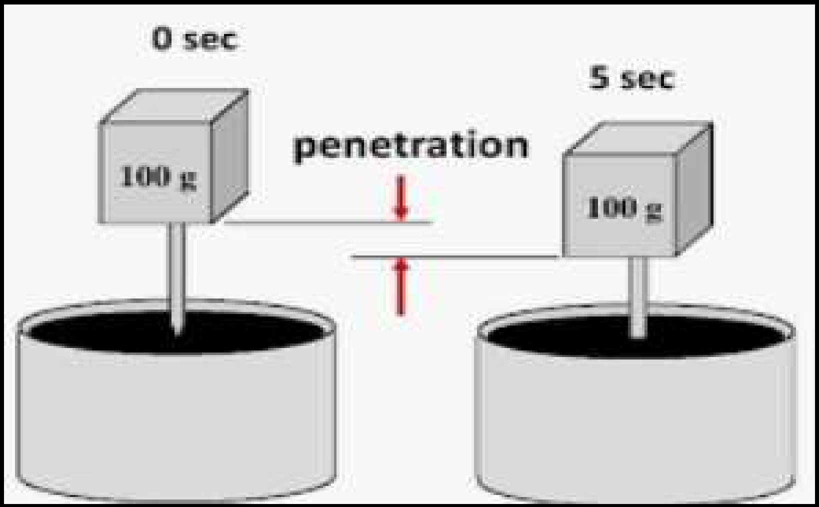

The roads aren’t ready for climate change ?!
Fewer, but heavier vehicles on the road?
East Cambridgeshire_2018
Northeast of Athens_2018

- Pavement engineering also can apply the sustainability notion
- From *Empirical* Bitumen tests to Fundamental Rheological tests



Softening Point (EN 1427)

Test the behavior of bitumen at elevated service temperatures



Needle Penetration (EN 1426)

Test the behavior of bitumen at intermediate service temperatures

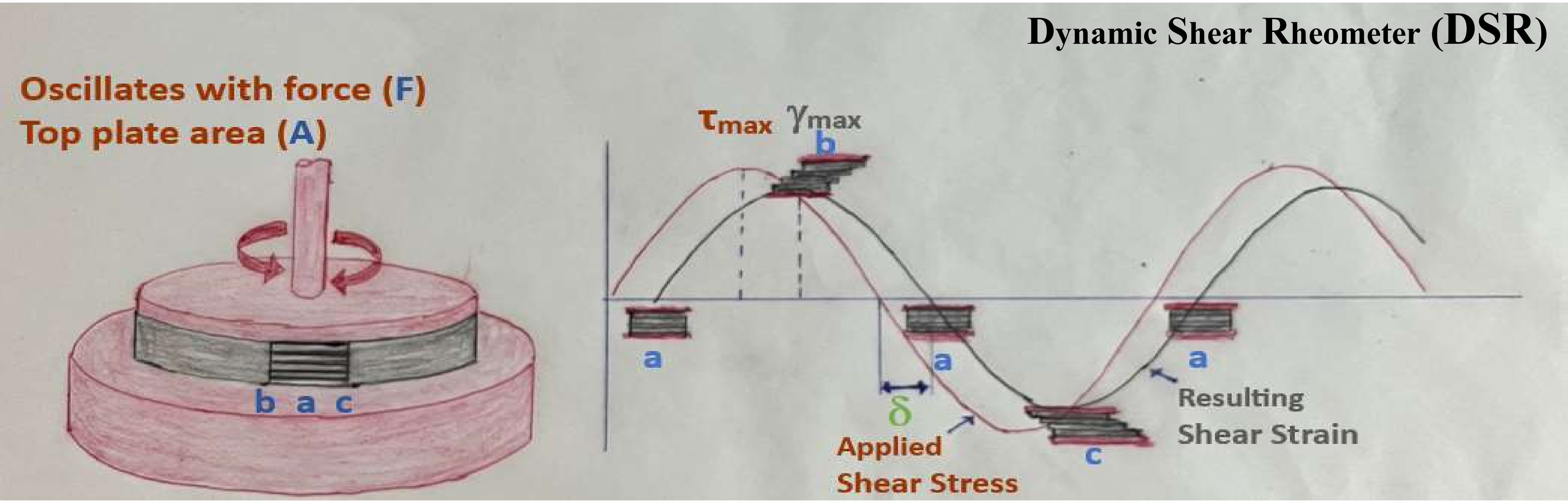
Tabell 651.3 Krav til polymermodifisert bitumen *(Handbook N200 Road Construction)*

	Enhet	Provings-metode NS-EN	Gradering				
			65/105-60	40/100-75	90/150-60	75/130-80	25/55-75
Penetrasjon ved 25 °C	0,1 mm	1426	65-105	40-100	90/150	75-130	25-55
Mykningspunkt	°C	1427	≥ 60	≥ 75	≥ 60	≥ 80	≥ 75
Kohesjon målt med kraftduktilitet[1)]	J/cm²	13589	≥ 1 ved 10 °C	≥ 2 ved 10 °C	≥ 0,5 ved 10 °C	≥ 2 ved 10 °C	≥ 3 ved 10 °C
Fraass bruddpunkt	°C	12593	≤ -12	≤ -12	≤ -18	≤ -20	≤ -10
Elastisk tilbakegang ved 10 °C[1)]	%	13398	≥ 50	≥ 75	≥ 75	≥ 75	≥ 50
Flammepunkt	°C	ISO 2592	≥ 220	≥ 220	≥ 220	≥ 220	≥ 220
Lagringsstabilitet 72 timer ved 180 °C		13399	Krav til lagringsstabilitet				
Forskjell i mykningspunkt	°C	1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Forskjell i penetrasjon	0,1 mm	1426	≤ 9	≤ 9	≤ 9	≤ 9	≤ 9
			Krav til gjenværende egenskaper etter korttidsaldring				
Motstand mot oppherding, RTFOT ved 163 °C		12607-1[3)]					
Masseendring	%	12607-1[3)]	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5
Gjenværende penetrasjon	%	1426	≥ 60	≥ 60	≥ 60	≥ 60	≥ 60
Økning i mykningspunkt	°C	1427	≤ 10	≤ 10	≤ 10	≤ 10	≤ 10
Fall i mykningspunkt[2)]	°C	1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Ytelsesrelaterte tilleggskrav Gjelder ikke generelt, kun der dette er spesifisert i kontrakten.							
Motstand mot oppherding, RTFOT ved 163 °C		12607-1[3)]	Krav til gjenværende egenskaper etter korttidsaldring				
MSCRT Jnr3,2 kPa ved 60 °C	kPa^{-1}	16659	≤ 0,5	≤ 0,2	≤ 1,0	≤ 0,2	≤ 0,1
Temperatursensitivitet T for G*=15 kPa @ 1,59 Hz	°C	14770 med 25 mm plate	≥ 50	≥ 55	≥ 40	≥ 55	≥ 60
Temperatursensitivitet T for G*=5000kPa @ 1,59 Hz	°C	14770 med 8 mm plate	≤ 20	≤ 25	≤ 15	≤ 25	≤ 30
RTFOT ved 163 °C etterfulgt av PAV ved T=100 °C i 20 timer		12607-1[3)] + 14769	Krav til gjenværende egenskaper etter kort- og langtidsaldring				
BBR etter langtidsaldring T (S=300 MPa)	°C	14771	≤ -15	≤ -15	≤ -21	≤ -24	≤ -12

Dynamic Shear Rheometer (EN 14770)

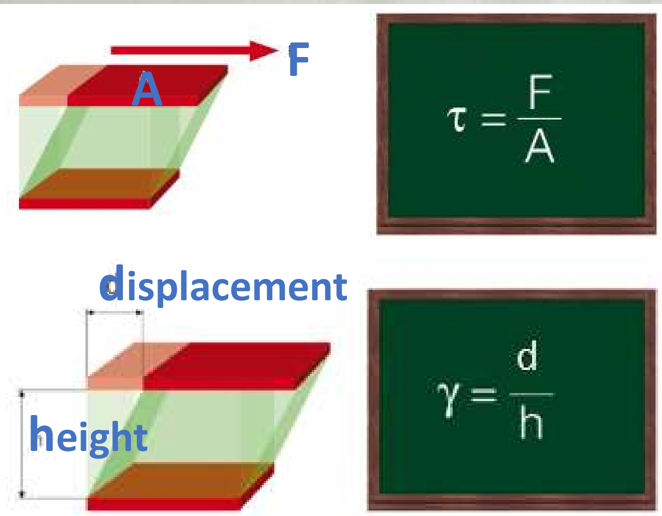
Measurements and predictions of bitumen performance must furthermore include a wider range of *test temperatures* and *loading time*

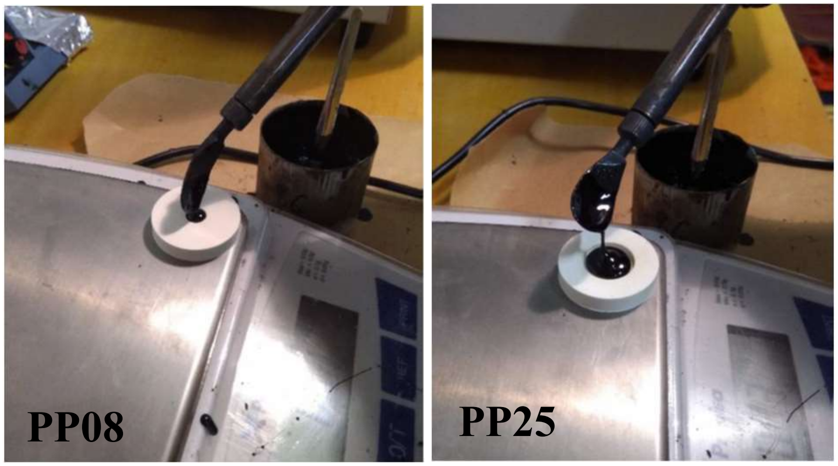
Norway leads the way!



$$G^* = \frac{\tau_{max}}{\gamma_{max}} \frac{(\mathrm{Stress}_{max})}{(\mathrm{Strain}_{max})}$$

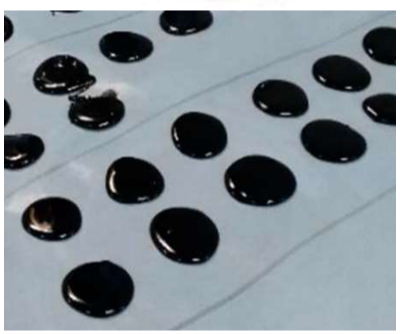
δ = time lag

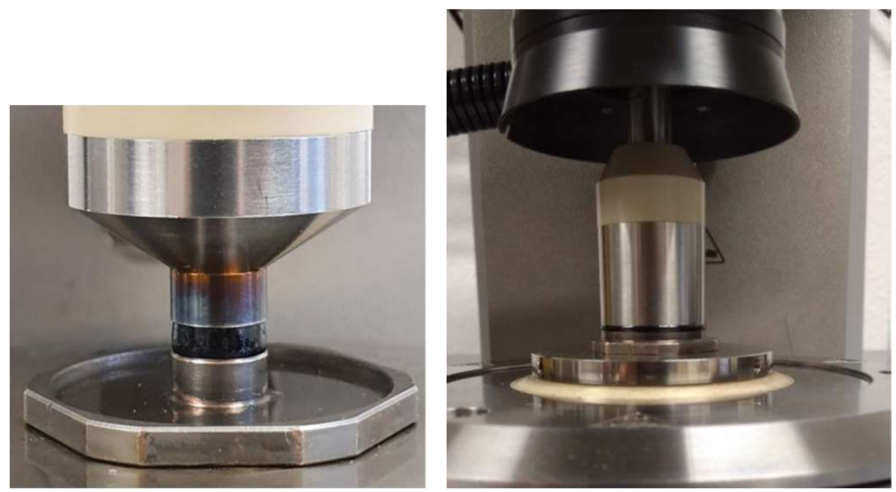




Pour onto mould

Pour onto sheet





Study I: Sample preparation techniques on DSR testing: round-robin tests on bitumen

https://doi.org/10.1080/14680629.2023.2213775

Petroleum Standardization Bureau

(Bureau de Normalisation du Pétrole)

Study I. Aim

Identify the sample preparation phases that are crucial to achieving consistent results and underline the importance of employing the same sample preparation and test conditioning approaches because of the possible impact on the outcome.

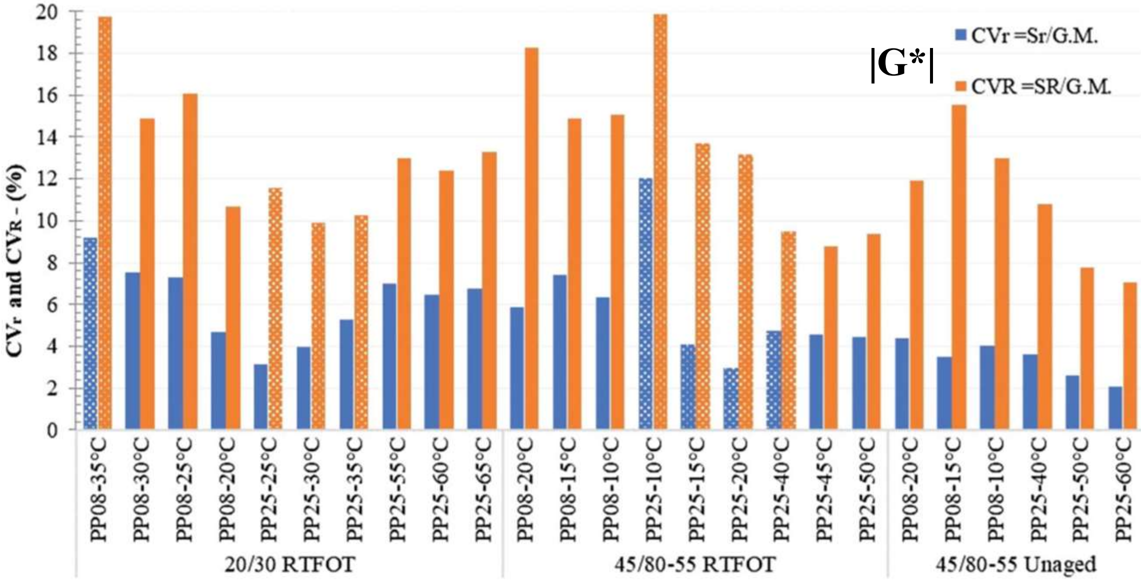
Study I. Material

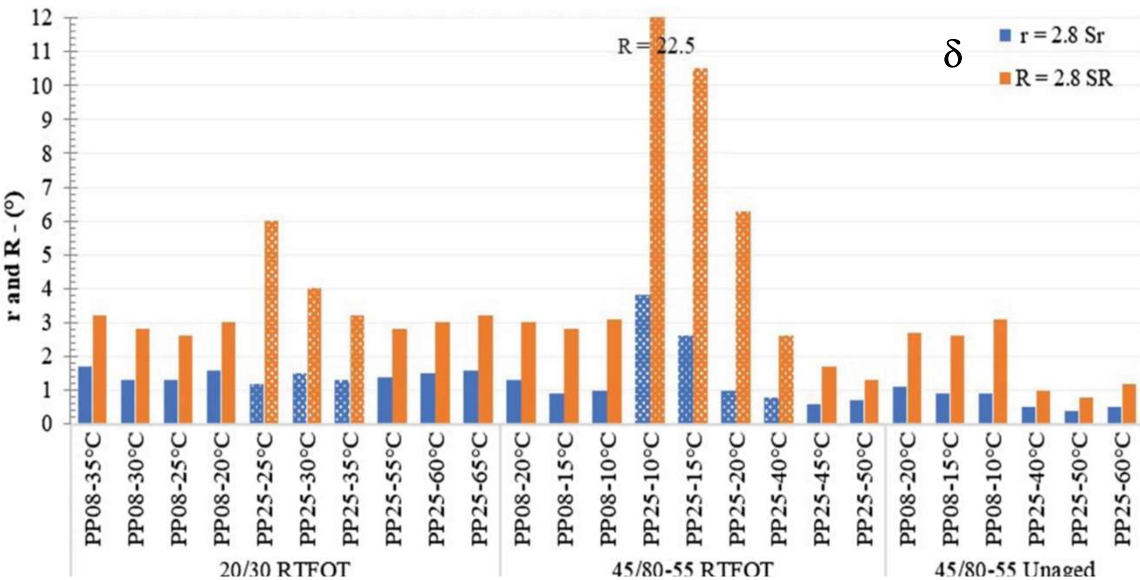
Material type	PEN (0.1mm) EN 1426	SP (°C) EN 1427	Test Temperatures (°C) and parallel plate dimensions (PPmm)
50/70	58	54	15, 20 and 25°C (PP08) 40, 45 and 50°C (PP25)
20/30	31	60.4	35, 30, 25 and 20°C (PP08) 20, 25, 30,35, 55, 60, and 65°C (PP25)
45/80-55	51	58.4	20, 15 and 10°C (PP08) 10, 15, 20, 40, 45 and 50°C (PP25)
45/80-55	49	61	20, 15 and 10°C (PP08) 40, 50 and 60°C (PP25)

Study I: Results and Findings

The investigated steps of DSR test method

- *Repeatability and reproducibility* for |G*| and δ
- *Equipment*
- *Sample manufacturing*
- *Waiting time between mfg. of sample and test start (storage time)*
- ***Sample bonding (mounting) temperatures***
- ***Pre-heating time and temperature for manufacturing sample***
- *Duration of Equilibrium time*





<u>Reproducibility</u> coefficient of variation

G*: 10% and δ: 5% (14770:**2012**)

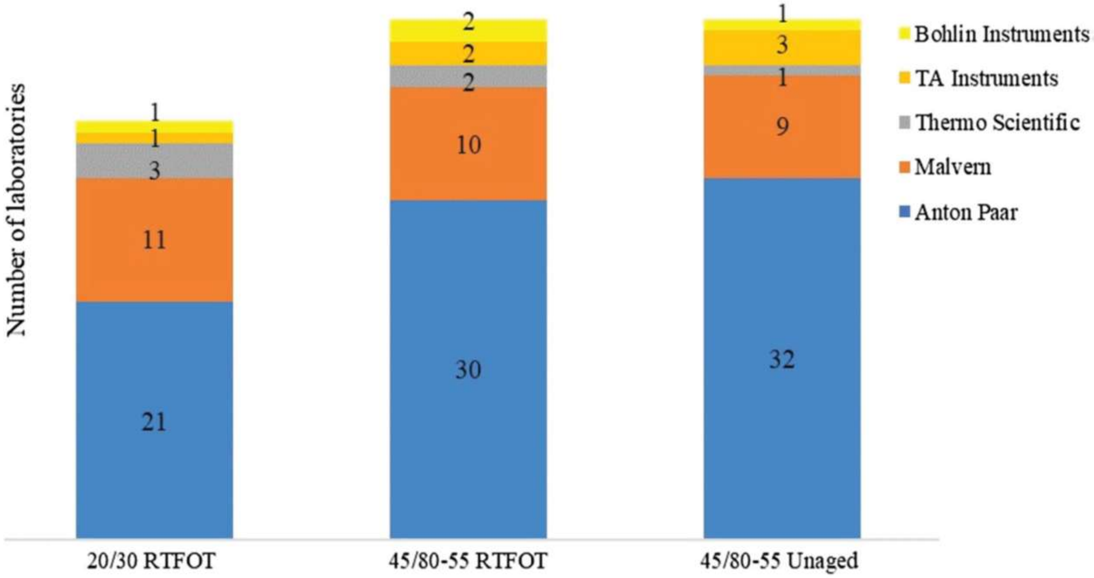
G*: 30% and δ: 4° (14770:**2023**)

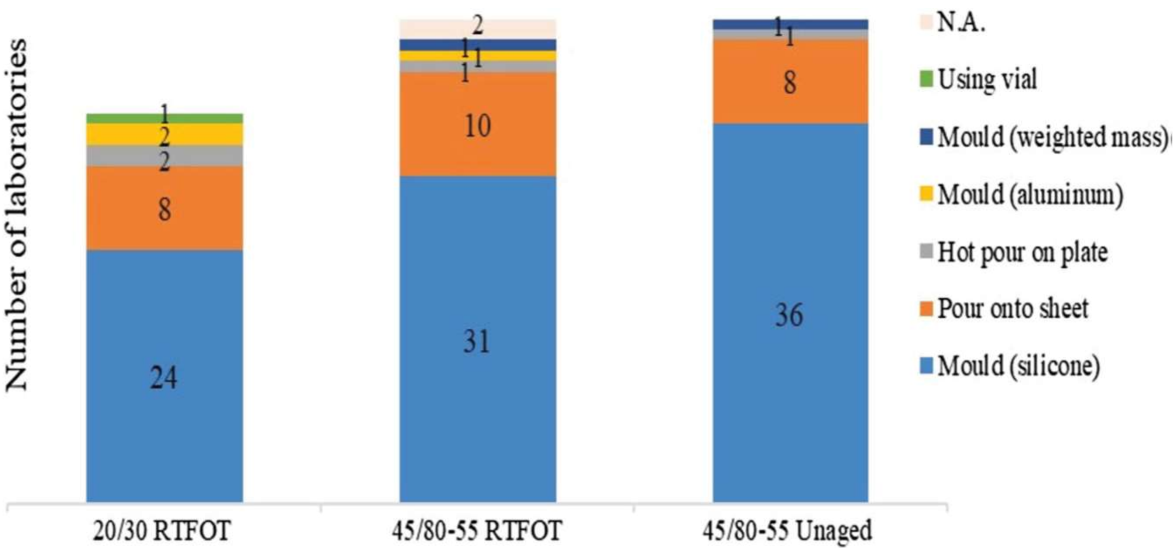
<u>Reproducibility</u> coefficient of variation

G*: (7-20%) and δ: (1-2)°

Repeatability coefficient of variation

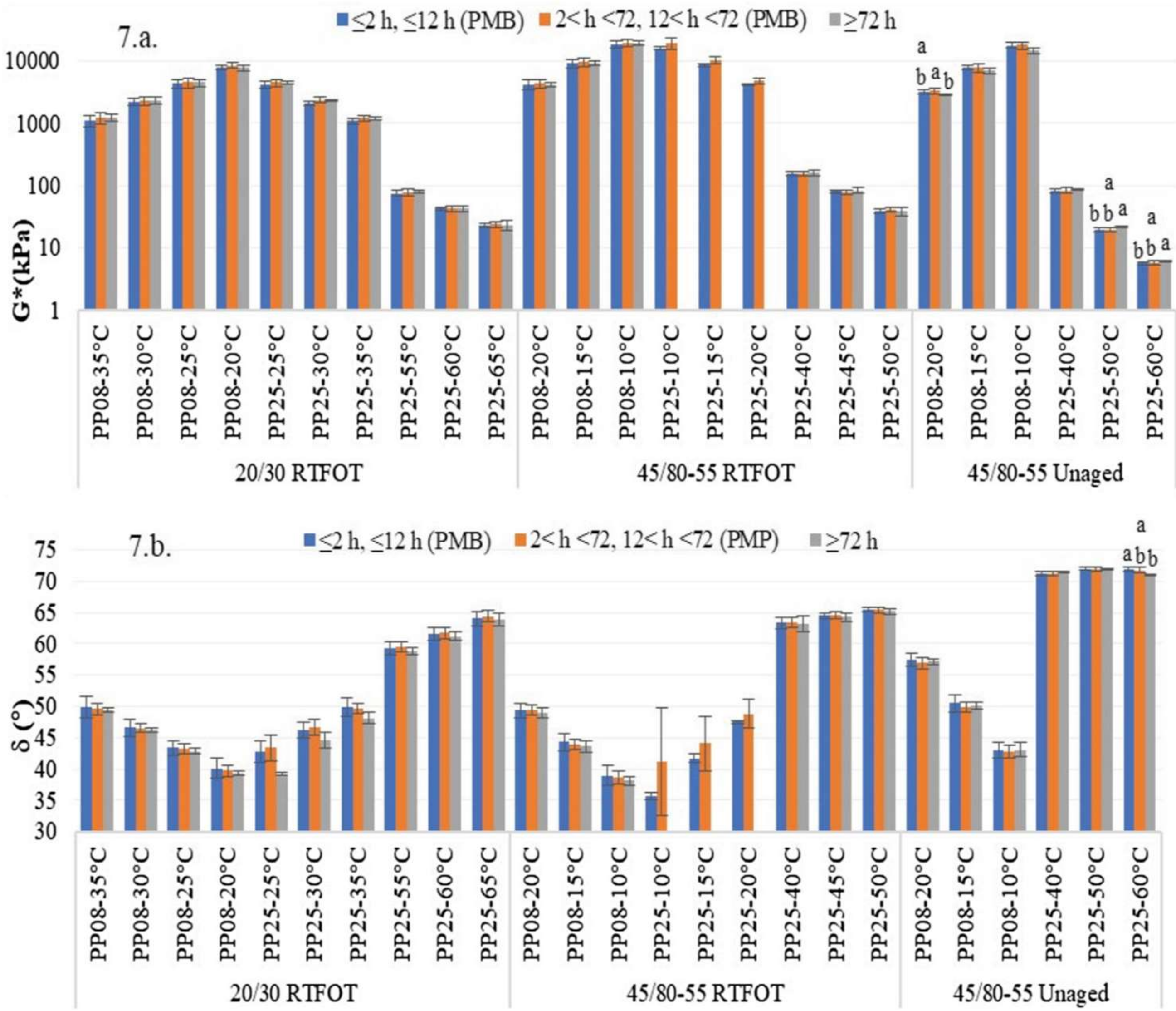
G*: (2-12%) and δ: (1-3)°





In most test combinations, the **equipment brand** and **manufacturing method** had negligible impact on the results;

a ***higher G∗*** with ***Anton Paar*** & ***sheet***, while a higher δ with Malvern & mould.



None of the studied bitumen was significantly affected by **waiting times** of less than 2 h or longer than 72 h;

waiting time ≥ 72 h
(a higher G∗ & a lower δ value)

waiting time ≤ 2h
(a lower G∗ & a higher δ value)

- **Heating temperature** of (SP+100°C) for PMB, yielded more accurate results in terms of coefficient of variation and standard deviation for G* and δ, respectively.

- The **bonding temperature** and sample manufacturing temperature had a significant association in more test combinations than other sample preparation processes.

- PP08: Equilibrium durations of 5–**15** min (a lower G*)

PP25: Equilibrium durations of **15**–30 min (a lower G*)

In most test conditions, δ value increased by equilibrium durationthe

Study II:

Effects of Various DSR Testing Methods on the Measured Rheological Properties of Bitumen

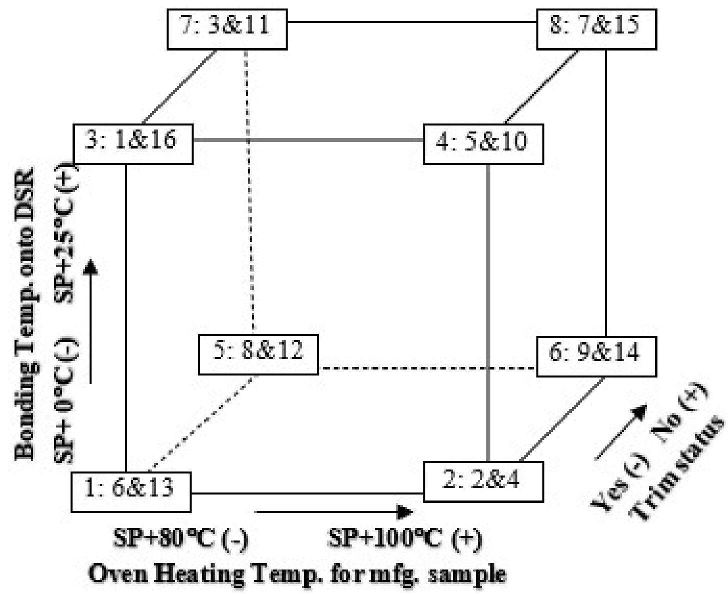
https://doi.org/10.3390/ma16072745

Study II. Aim

Determine whether and under what conditions the heating temperature (HT) for sample manufacturing, bonding temperature (BT), and radial trimming (Trim) of the sample on DSR testing have a significant impact on the results.

Study II. Material and Method

Material type	PEN (0.1mm) EN 1426	SP (°C) EN 1427	Density kg/m³
50/70	61	48.4	1030
70/100	77	46.0	1022
160/220_I	160	41.2	1000
160/220_II	161	39.5	1013



Aim | Material & Method | Results & Findings

Study II: Results and Findings

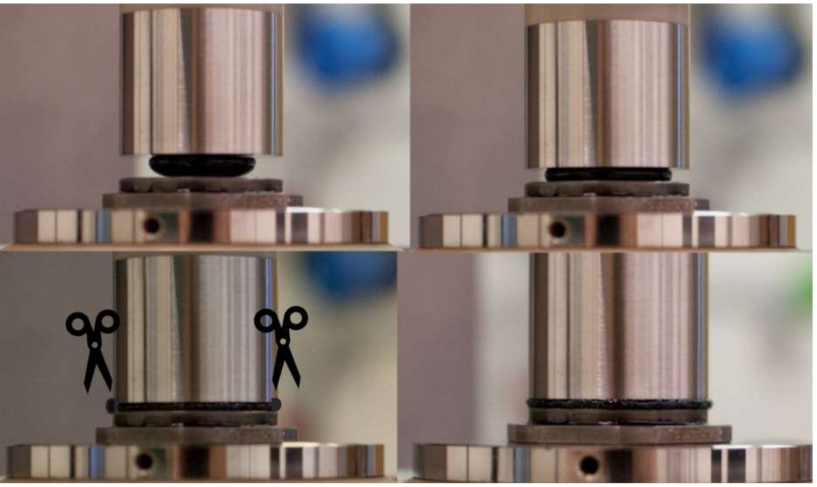
The main and interaction effect of three selected factors

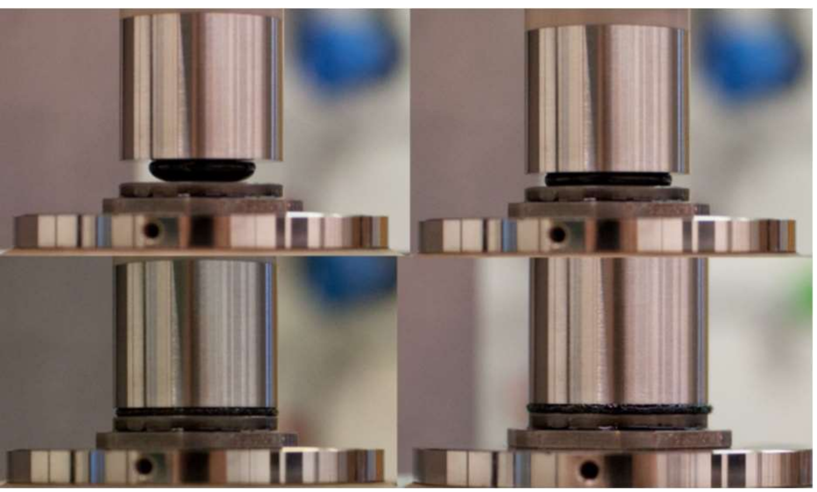
Main Effects
Trim
BT
HT
Two-factor interactions
Trim:HT
Trim:BT
BT:HT
Three-factor interactions
Trim:BT:HT

1. ***Trim***

*2. temperature at which bond sample onto rheometer **(BT)***

*3. pre-heating temperature for manufacturing sample **(HT)***





- A significant difference in **trim**med and un**trim**med samples when PP08 is applied.
- The **BT** and **HT** take on varying degrees of significance depending on the materials and tested temperatures. The variation in **HT** strongly affected the results of the 160/220_I contrary to all other studied materials.
- For G*, the two-way interaction **Trim:BT** has the strongest effect on almost all tested materials and temperatures
- For δ, the two-way interaction **Trim:BT** has the strongest effect for 70/100 and 160/220_II
- The study showed that G* and δ have been affected by studied factors, most for 160/220.

Study III:

Assessing the effect of specimen preparation methods on DSR testing of bitumen using factorial design analysis

Material type	PEN (0.1mm) EN 1426	SP (°C) EN 1427	Density kg/m^3
50/70	61	46.8	1025
100/160	160	41.2	1000
70/100	85	45.6	1019
70/100+ 4% SBS	39	67.2	1015
70/100+ 4% Wax	33	79.0	1014

Study findings underline

the importance of employing the same sample preparation and test conditioning approaches because of the possible impact on the outcome.

Bitumen Future?

Future research?