Wide-Gap Rheometer Step-Profile Analysis for Thixotropy Assessment and Numerical Criteria for Equilibrium and Flow Model Choice

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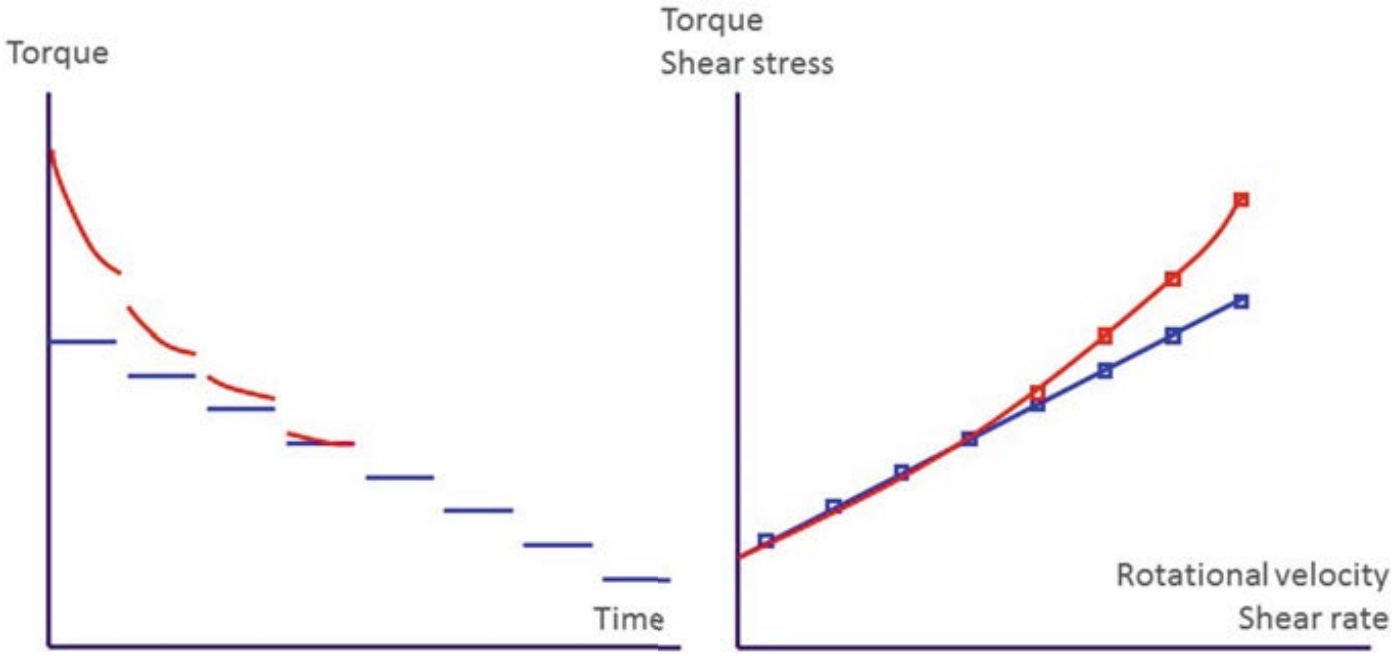
Equilibrium



Who is determining equilibrium by visual inspection?

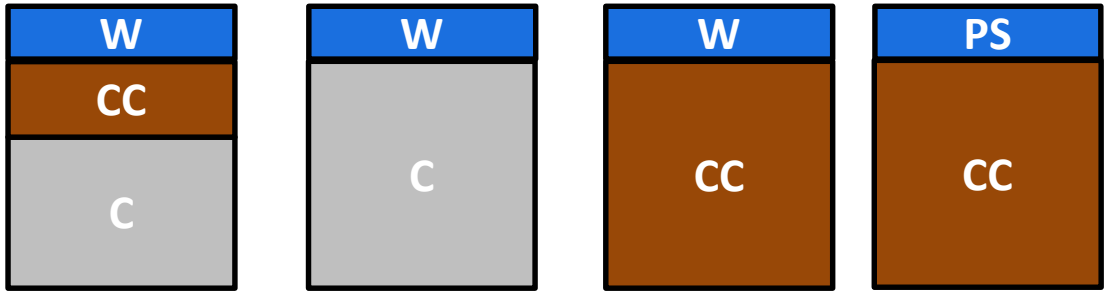
(Feys & Wallevik, 2024)
(Petit, et al. 2006)
(Geiker et al. 2002)

Methods

Data set

~275 rheometer runs

Different ratios of



PS pore solutions

W water

CC calcined clay

C cement

water/binder ratio: 0.4-1.2

w/ and w/o

At different time intervals

Rstudio

Equilibrium

Rheometer profile suggestions

pre-shear at highest shear rate

downward ramp

~30s per step

State of the Art Report

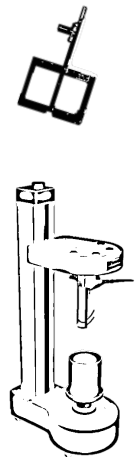
Measuring Rheological Properties of Cement-based Materials: State-of-the-Art Report of the RILEM Technical Committee 266-MRP

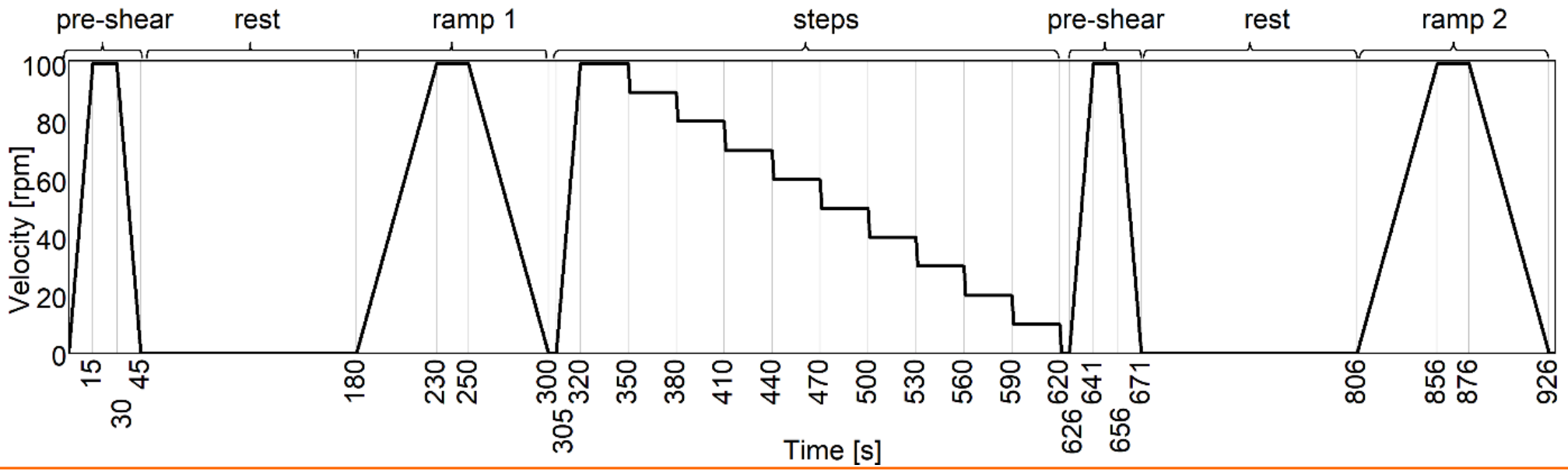
methods

rheometer – Viskomat NT

Sampling frequency ~ 9.9125 Hz

All data apparent rheological values (viscosity, yield stress etc!!)





Equilibrium

Integrity of data

Sedimentation

Plug flow

…

…

…

Viskomat NT

Viskomat XL

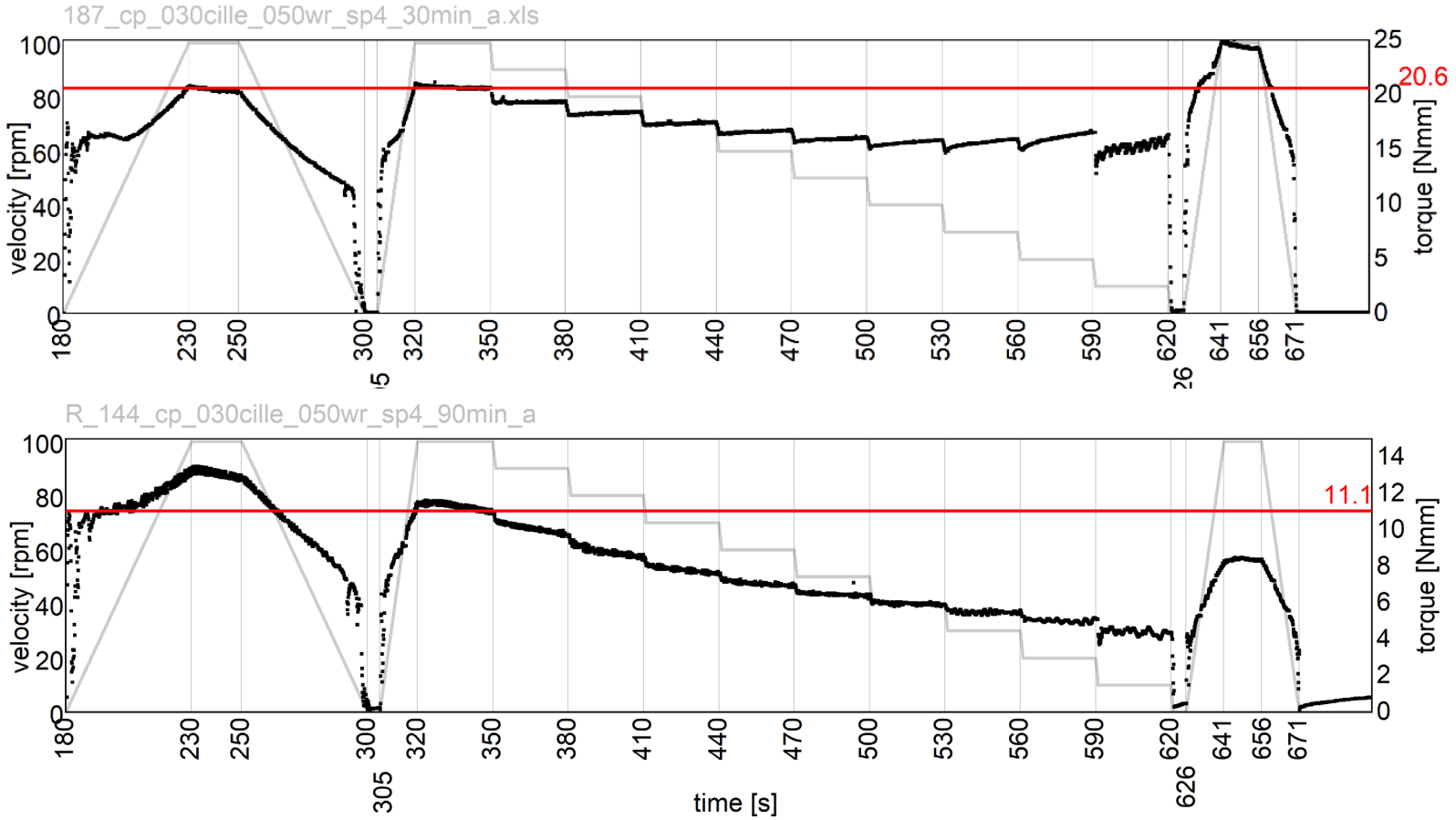
Schau in den Topf

Look in the cup

Equilibrium

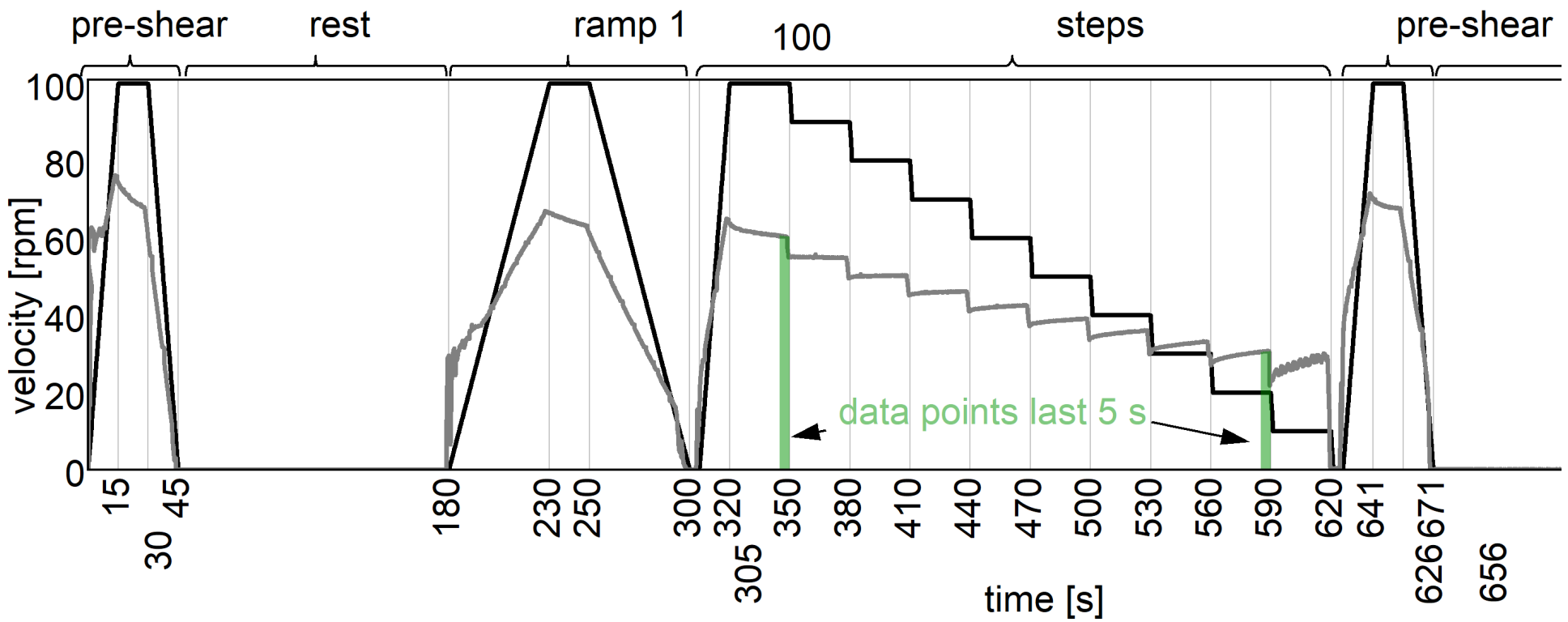
Max RPM factor

0.9<x<1.1 -> ~ 30 measurements



Equilibrium

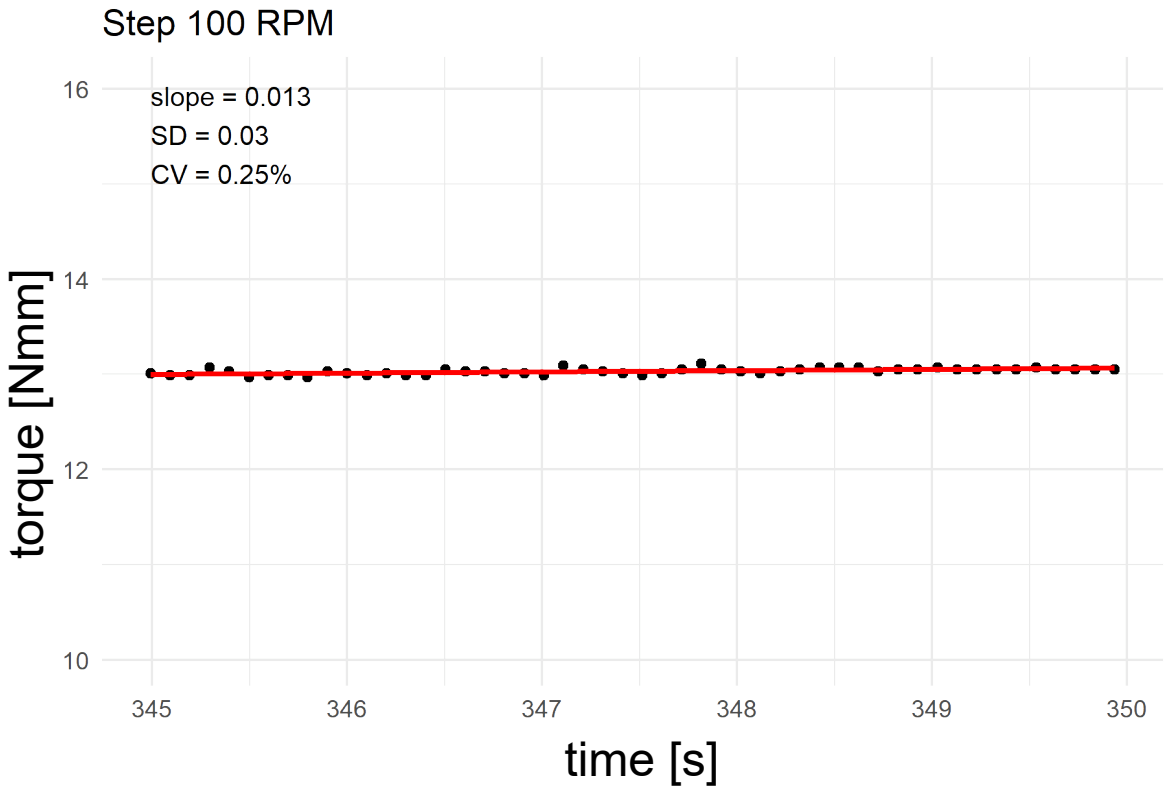
Step analysis



Equilibrium

Step analysis

$$1\ \% < \text{Coeffecient of variation} = \frac{\text{standard deviation}}{\text{mean average (ME)}} \qquad |0.05|\ \frac{\text{Nmm}}{\text{s}} < \text{slope incline}$$



Equilibrium

Step analysis

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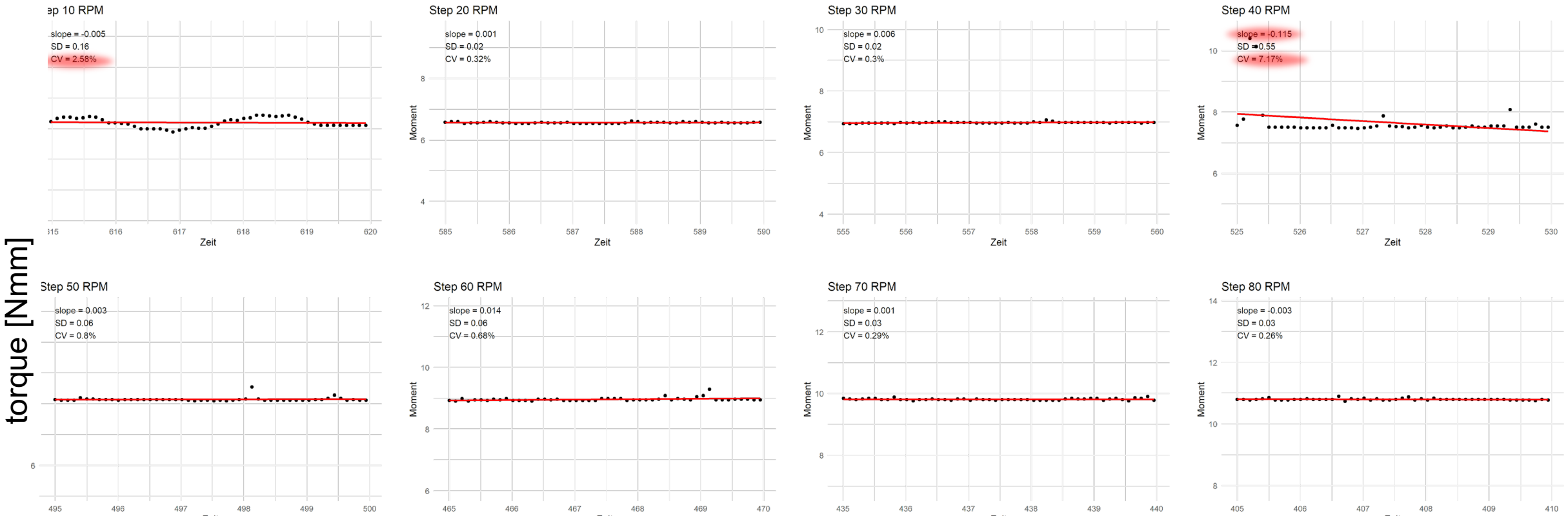
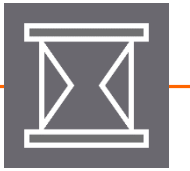


Summary equilibrium



Choice of profile

Integrity of data (particle migration)

Max RPM check

Slope incline / variation coefficient

} need to be fine tuned to the data set

Minimum of 5 data points

Flow model choice

Flow models used

Bingham (BH)	linear	$\tau = \tau_0 + \mu_p \dot{\gamma}$
Modified Bingham (MB)	quadratic	$\tau = \tau_0 + \mu_p \dot{\gamma} + c^* \dot{\gamma}^2$
Herschel-Bulkley (HB)	non-linear power-law	$\tau = \tau_0 + K \dot{\gamma}^n$

τ_0	yield stress	c	MB factor nonlinearity (shear thinning <0<shear thickening)
μ_p	viscosity	K	HB viscosity factor
$\dot{\gamma}$	shear rate	n	HB Flow behavior index (shear thinning < 1 < shear thickening)

Flow model choice

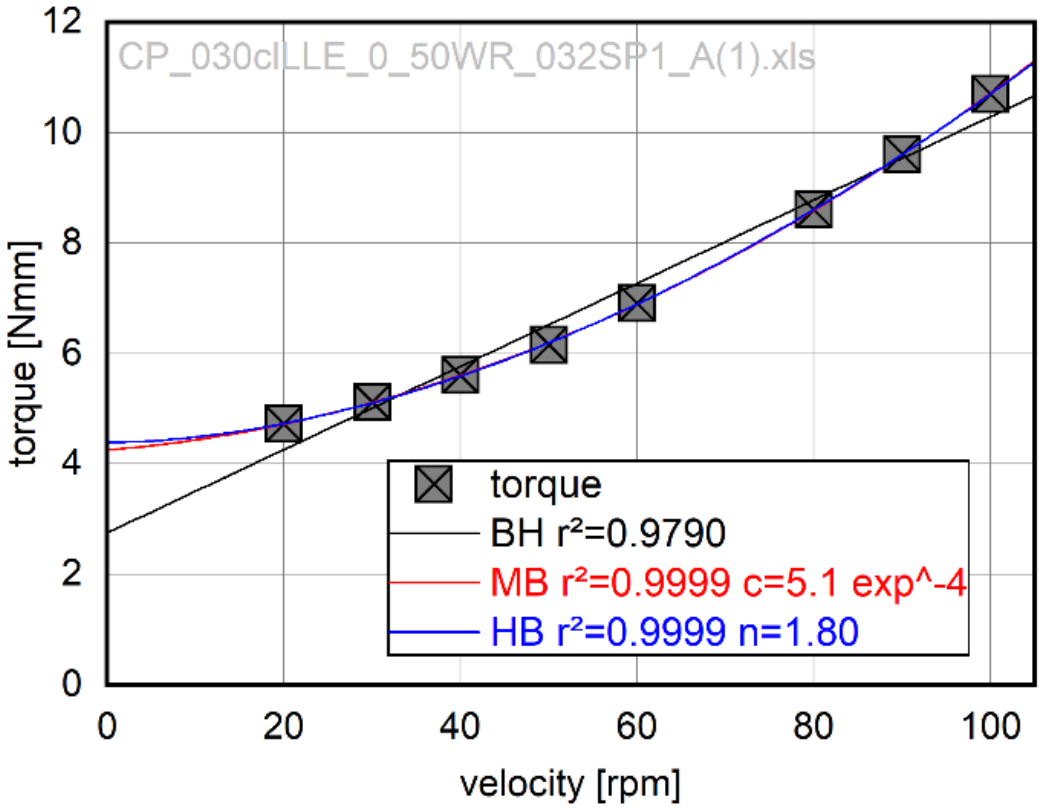
Herschel-Bulkley conditions

$$\tau = \tau_0 + K\dot{\gamma}^n$$

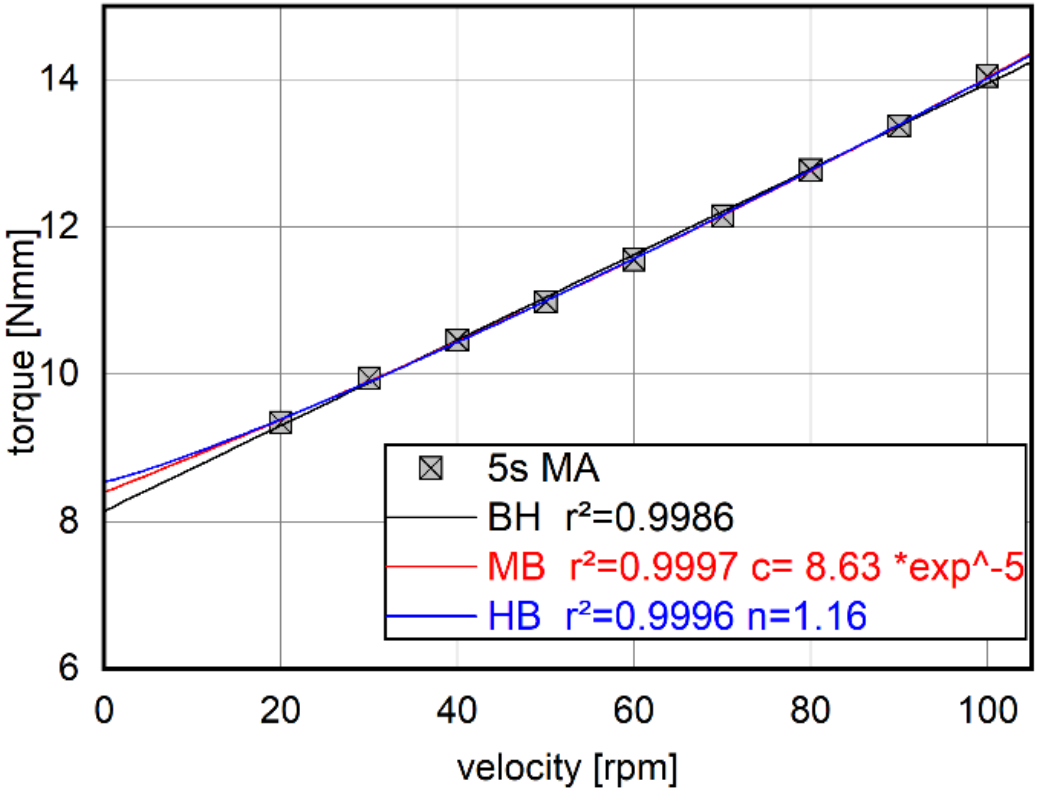
Boundaries

$\tau_0 > 0$	Seeding value for τ_0 =Bingham τ_0
$K > 0$	Seeding value for K = Bingham μ_p
$0.5 \leq n \leq 2.3$	Seeding values for n [0.7; 0.9; 1.0; 1.1; 1.5]

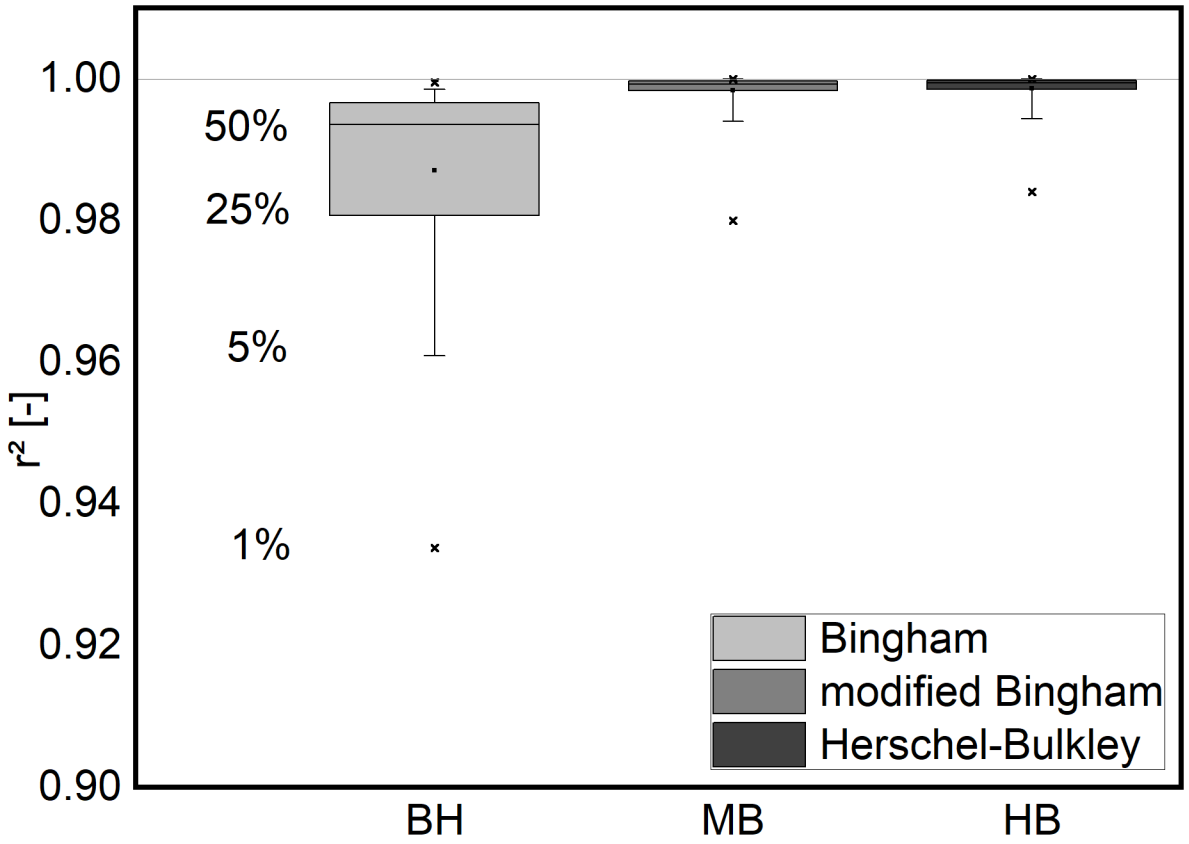
Flow Model Choice
by hand – Example 1



Flow Model Choice
by hand – Example 2



Flow model choice

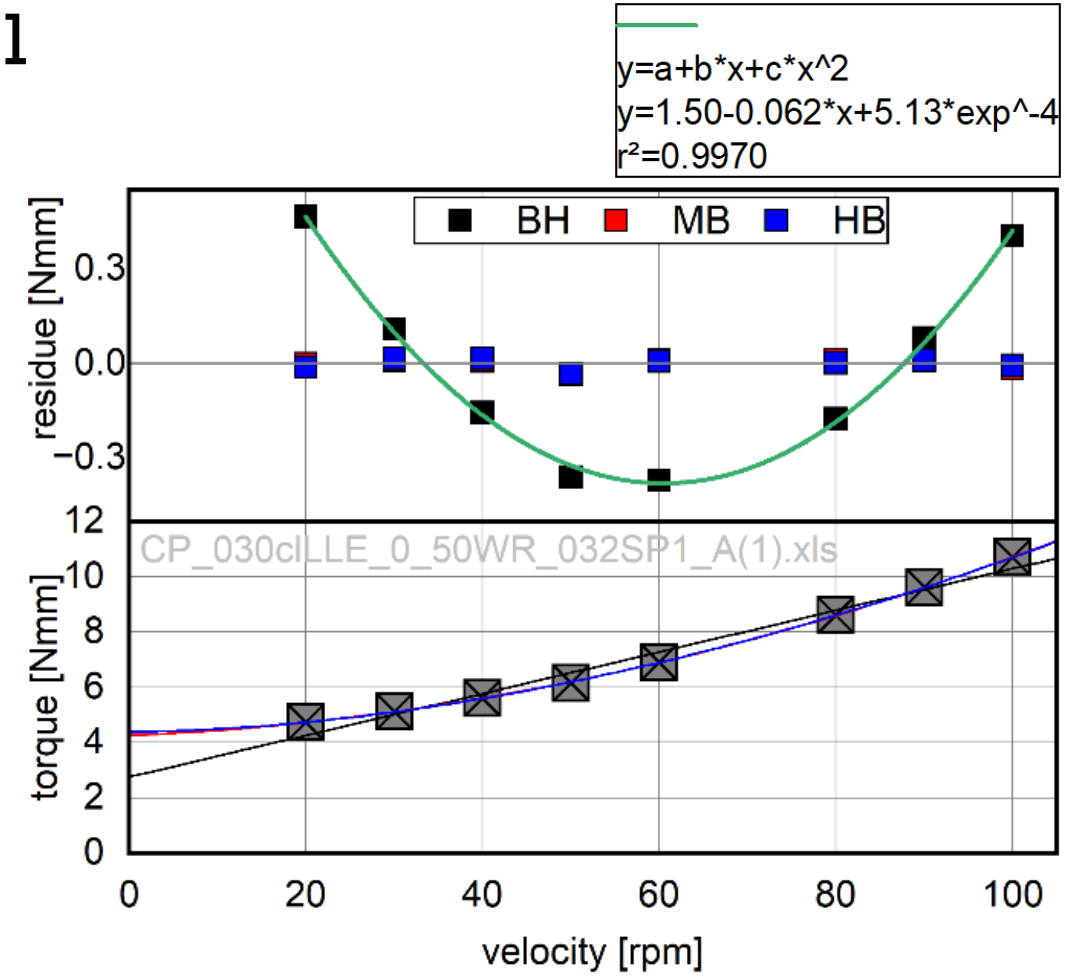


~77 data sets have n > 1.5

BH r^2 ~ 0.95

Flow model choice

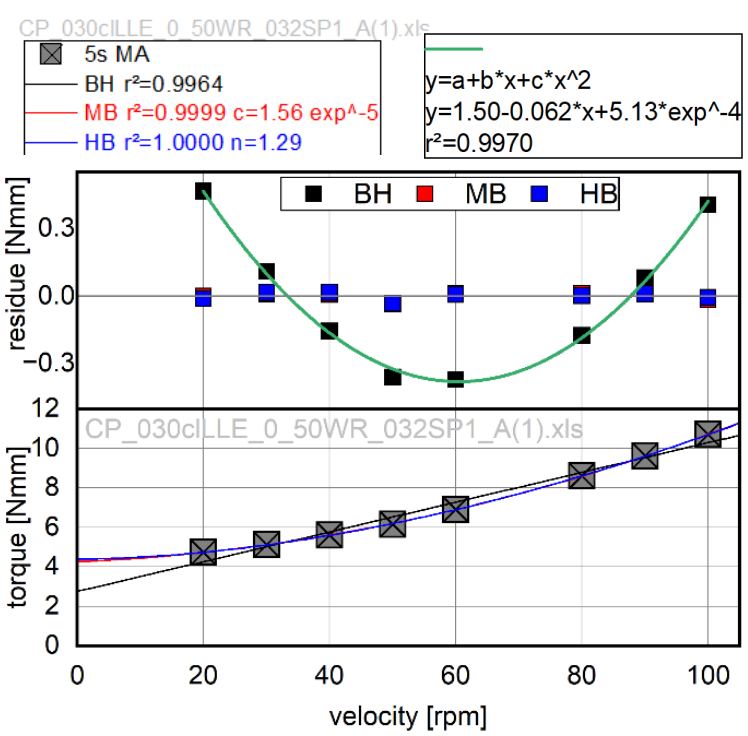
Residue analysis 1



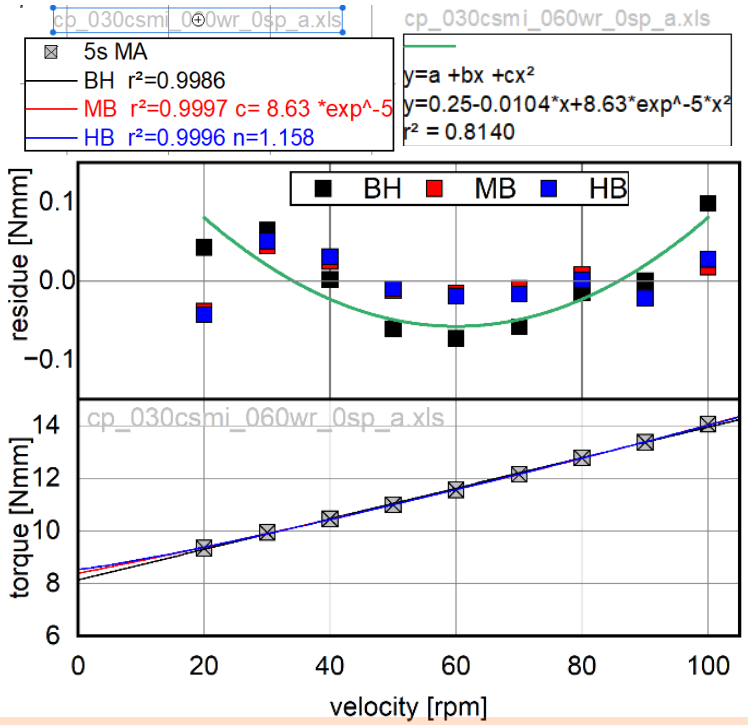
Flow Model Choice

Residue

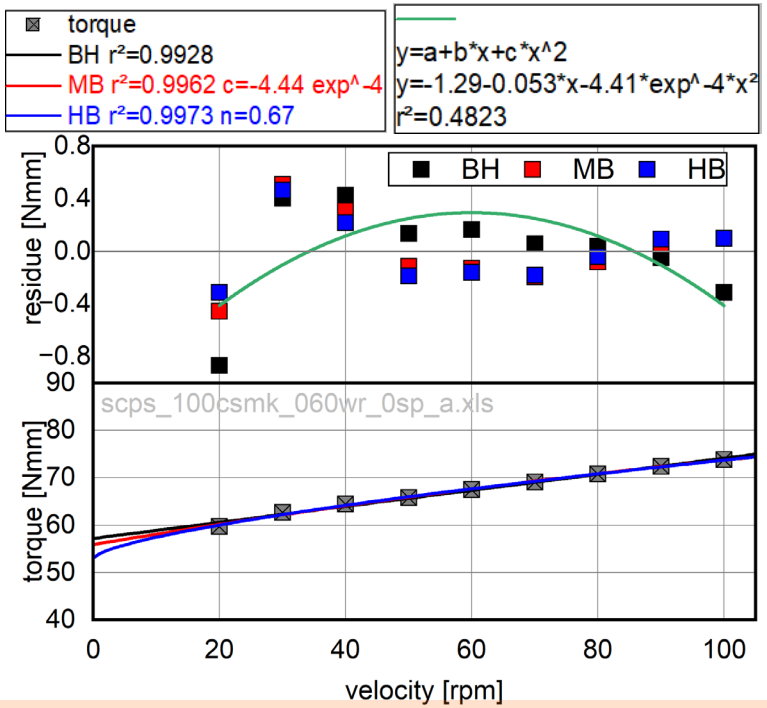
Example 1



Example 2

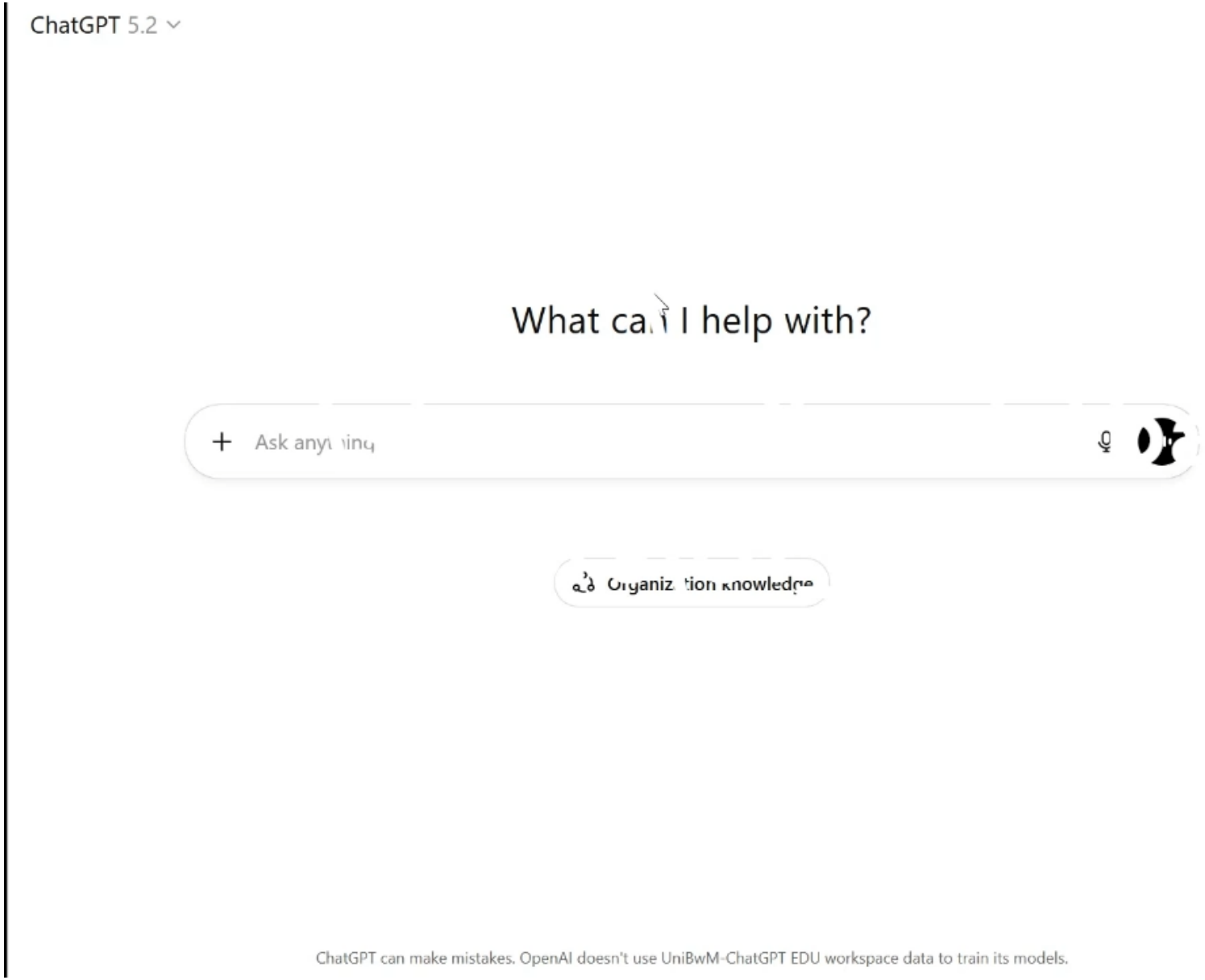


Example 3



2nd degree polynomal fit on bingham residue.

linear fit<r²=0.5<non linear fit

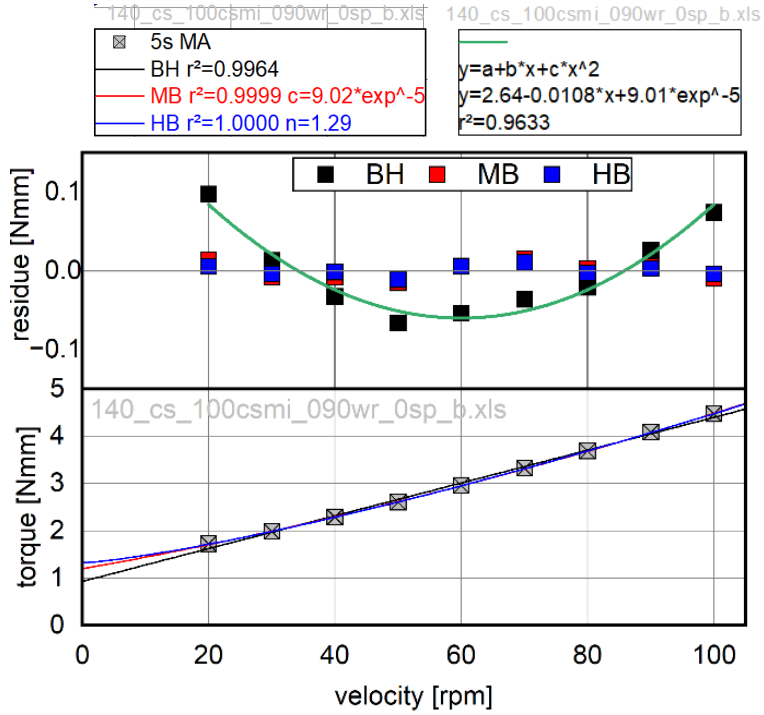


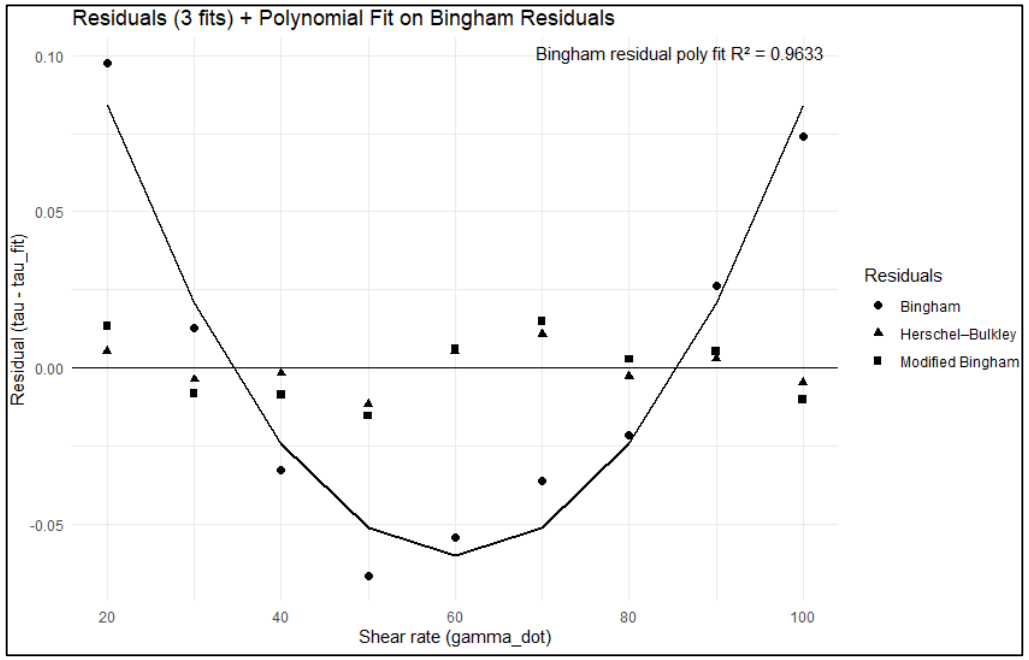
Good morning chatgpt

its fine. thank you for asking. how are you?

I need some help with rheology data. I have an excel sheet with the following data format: and i need an anylsis on that. 1. i need bingham, mod bingham and herschel bulkley fit on these values. I need residues on the previous three fits. and i need a second degree polynomal fit only on the bingham residues with the according r^2. this needs to be displayed in two graphs. 1 the three fits with their values and raw data and second the three residues plus polynomal fit with r^2 please write me an r studio script. the excel sheet name is "schleibi_conference_140"

Flow model choice

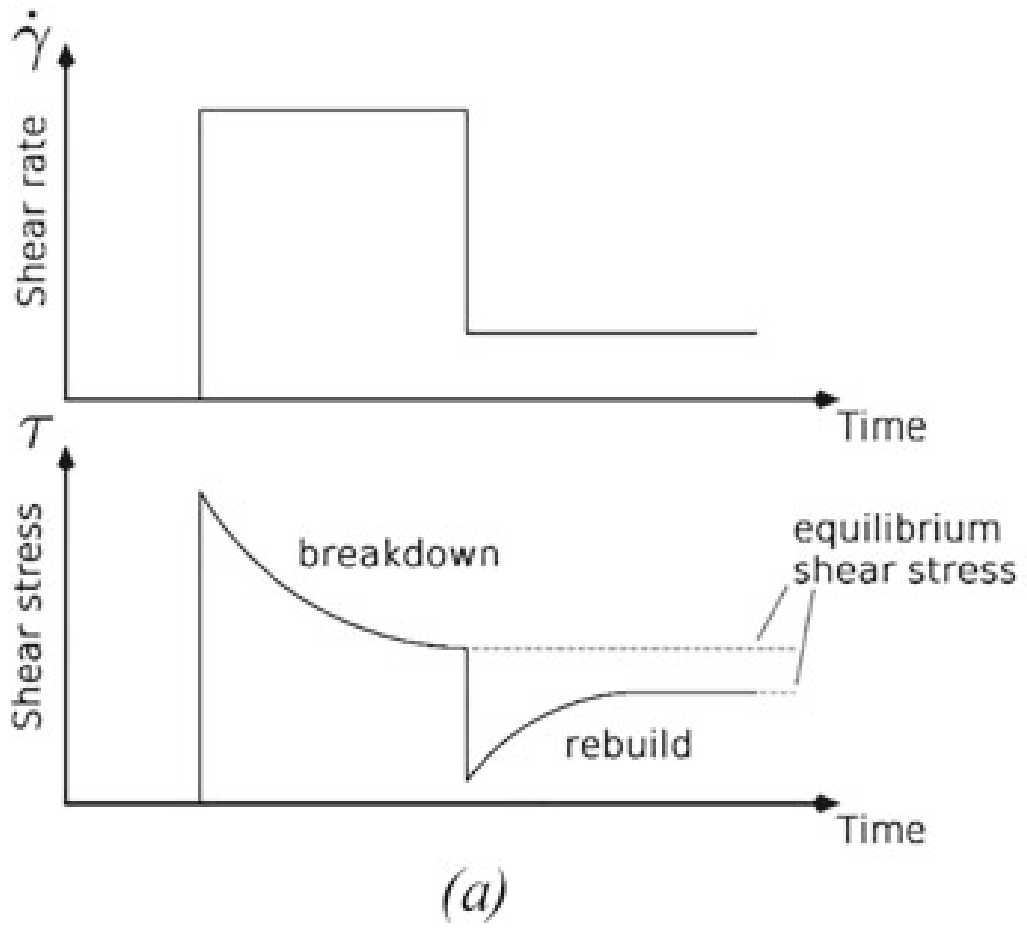




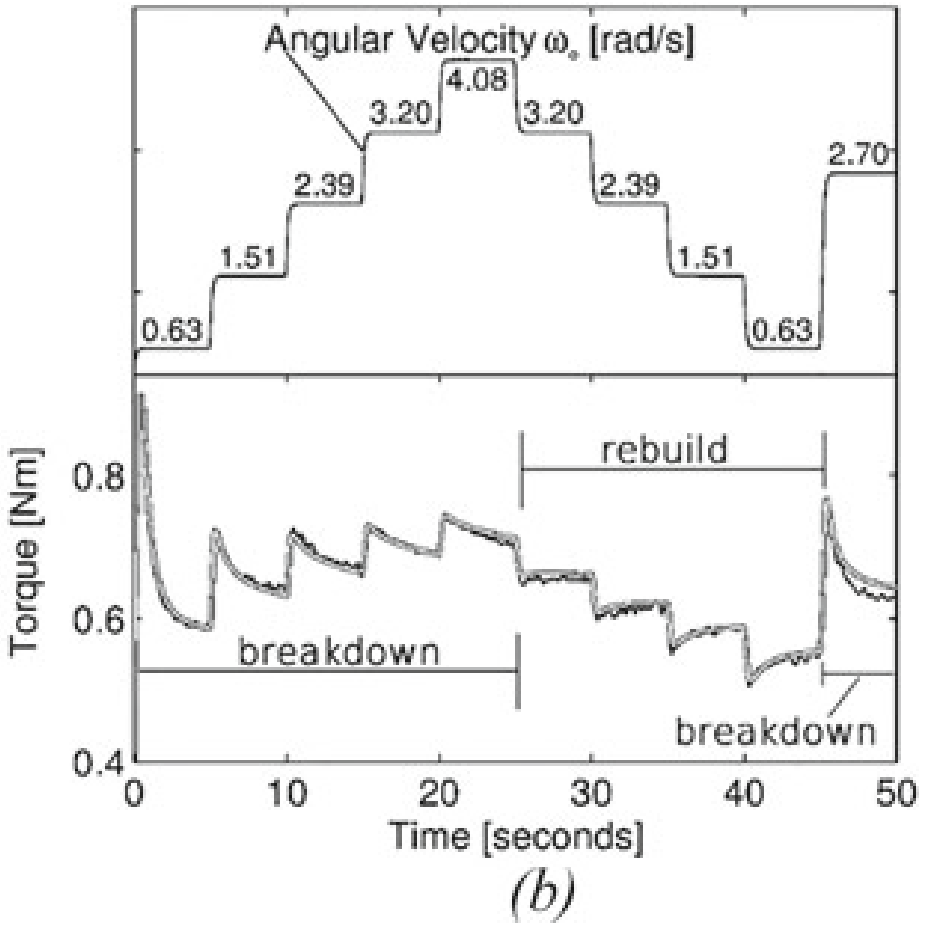
$$BH = 0.9336 + 0.0346 * x$$
$$MB = 1.1982 + 0.0239 * x + 9.01 * 10^{-5} * x^2$$
$$HB = 1.3257 + 0.00817 * x^{1.293}$$

```
===== FIT RESULTS =====
Bingham: tau = 0.933684 + 0.034689 * gamma
Modified Bingham: tau = 1.198150 + 0.023870 * gamma + 0.000090 * gamma^2
Herschel-Bulkley: tau = 1.325735 + 0.008177 * gamma^(1.293244)
Polynomial on Bingham residuals: R^2 = 0.963343
```

Thixotropy on a step profile



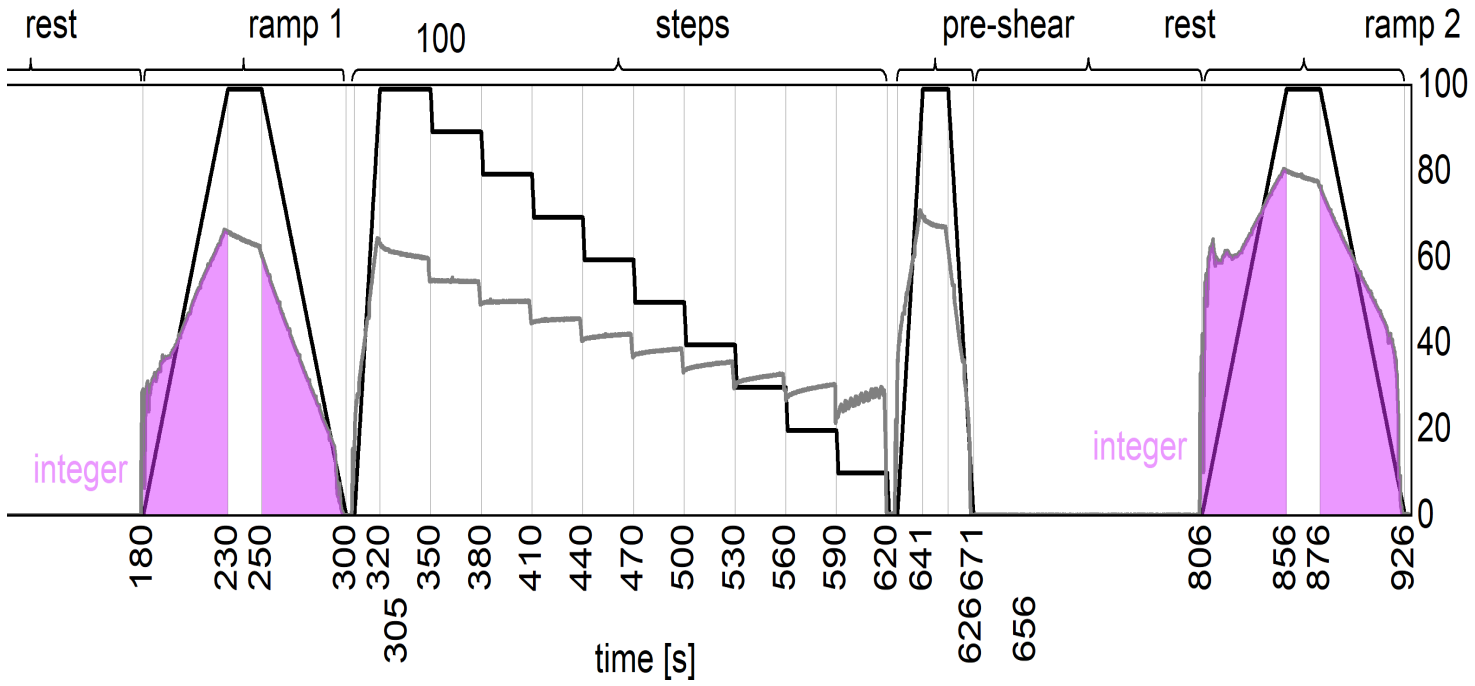
(Sonebi & Feys, 2024)

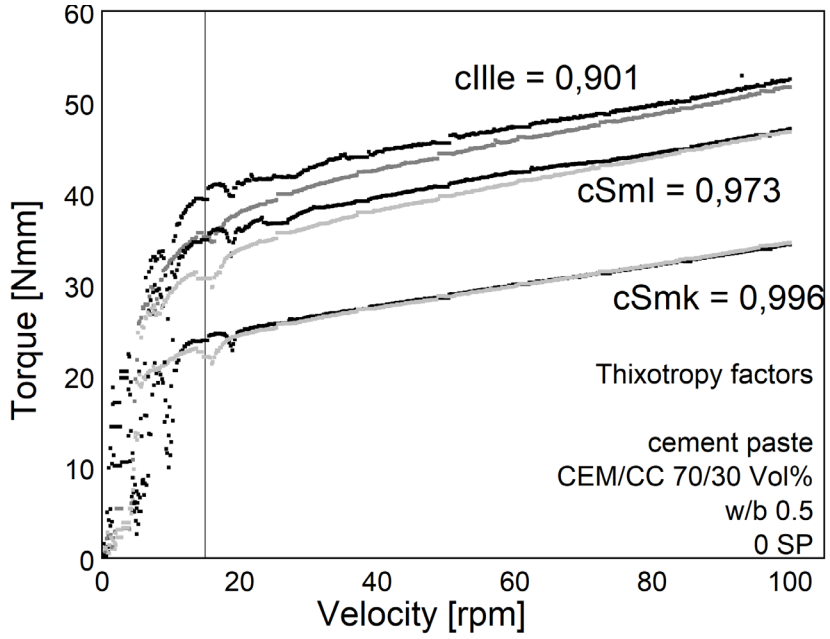


(Wallevik, 2009)

Thixotropy on a step profile

hysteresis



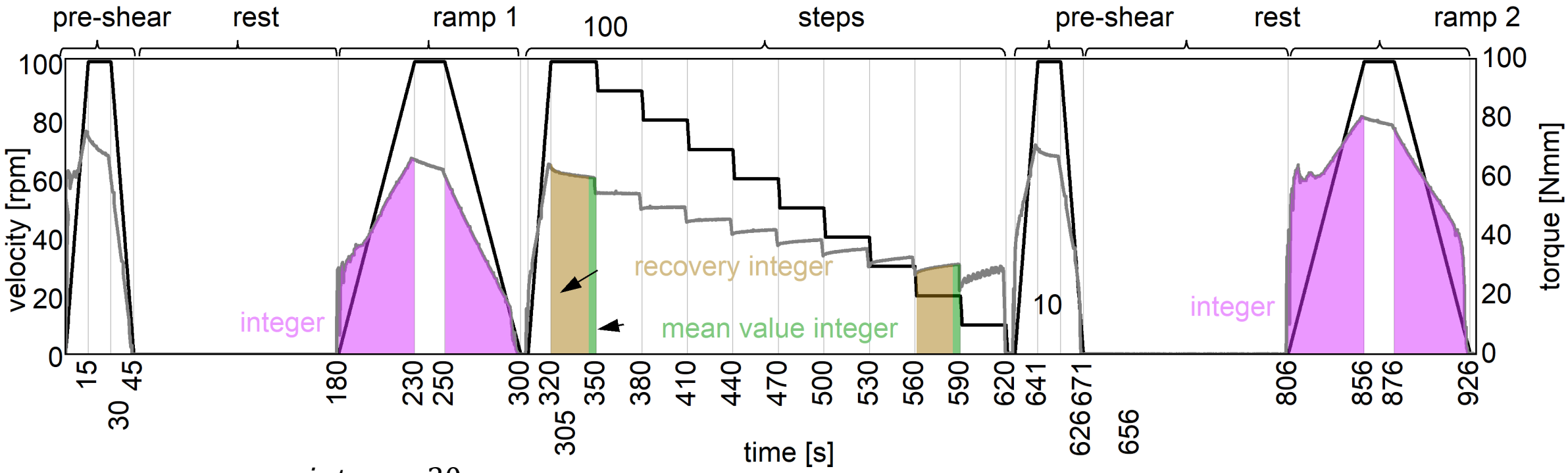


Thixotropy factor 1 (Thix 1)

Thixotropy factor 2 (Thix 2)

Thixotropy on a step profile

Inertia and recovery value



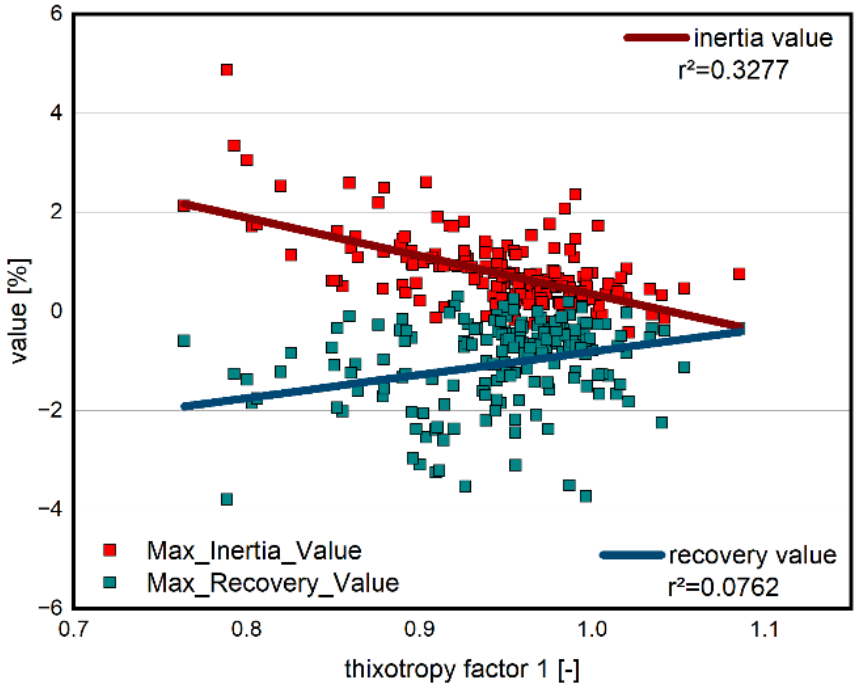
$$value = \frac{recovery\ integer: 30s}{mean\ value\ integer: 5s} * 100 - 100\ [\%]$$

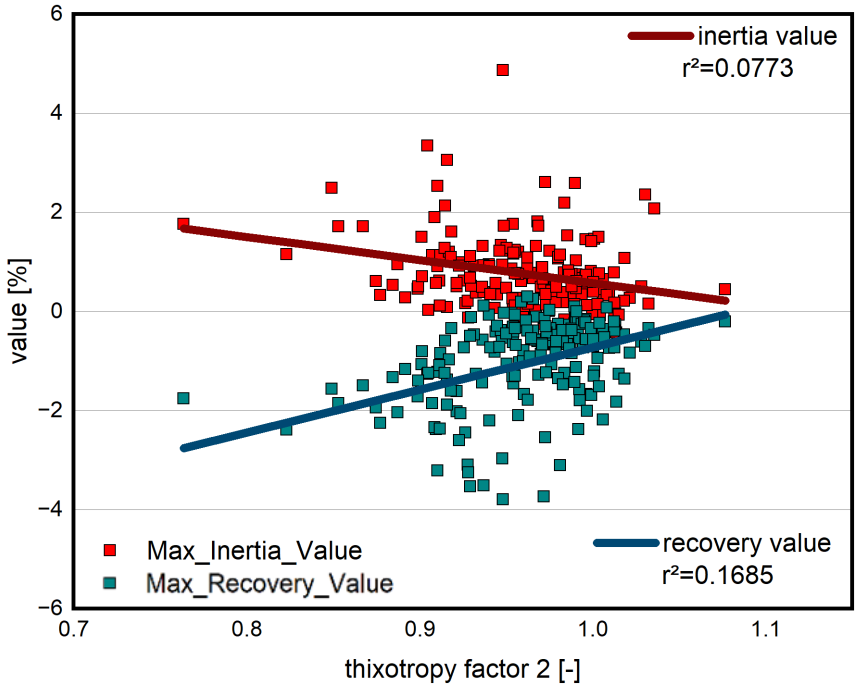
Maximum (values[10steps])

value > 0 -> inertia value
value < 0 -> recovery value

Thixotropy on a step profile

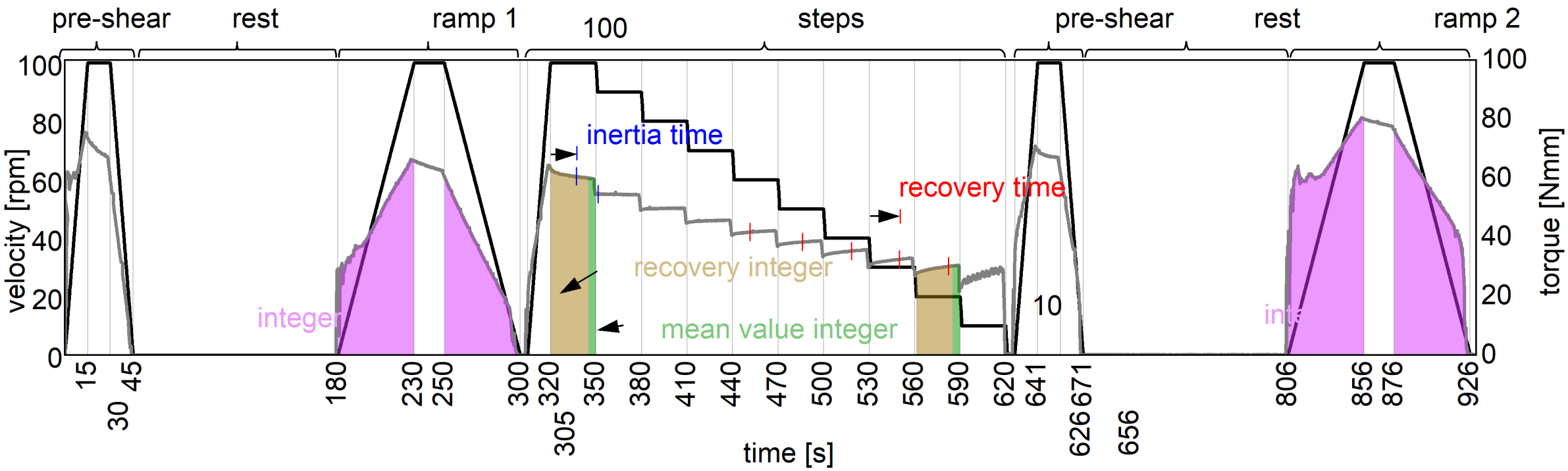
Inertia and recovery value





Thixotropy on a step profile

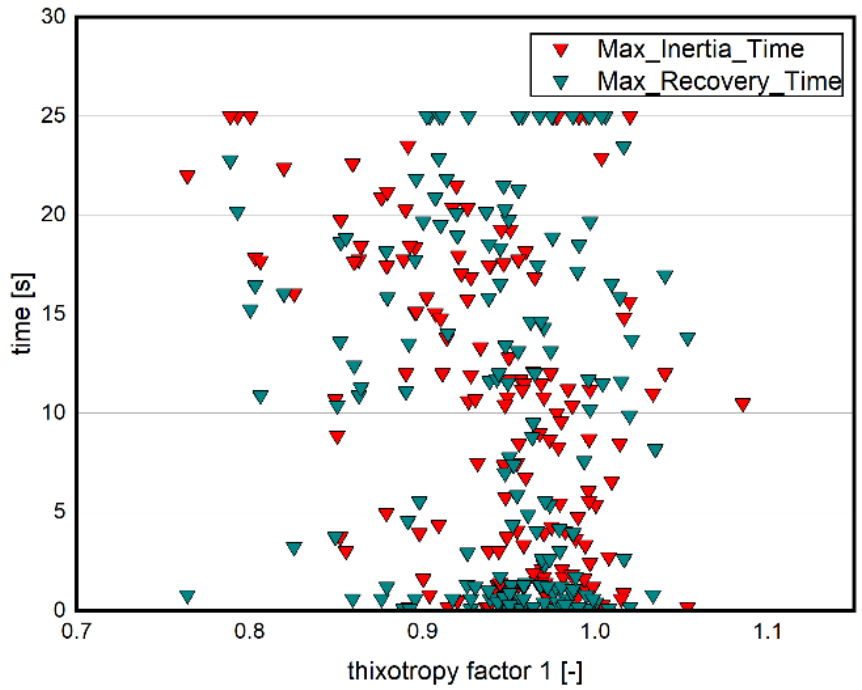
Hystereis loop

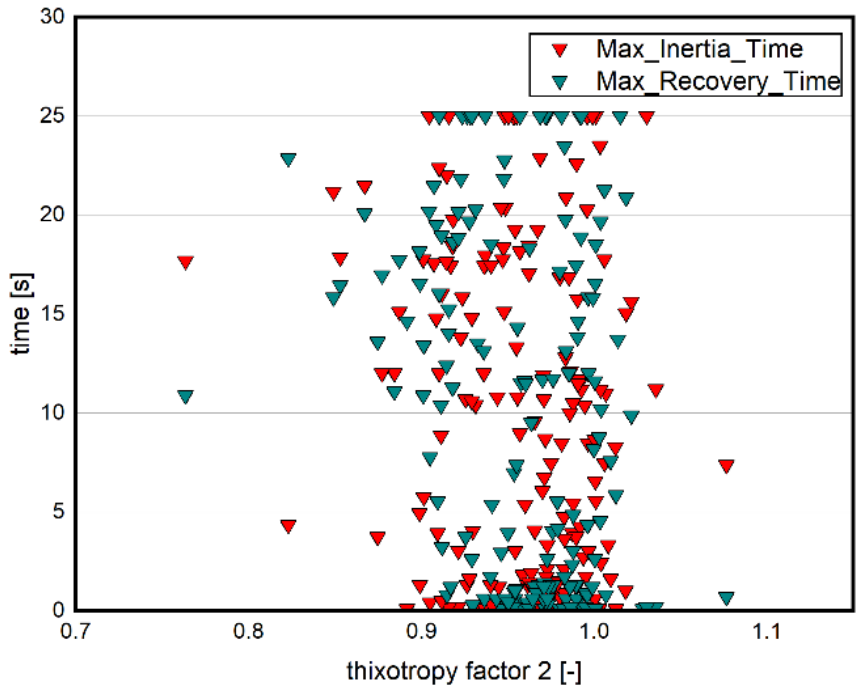


Inertia / recovery time: how fast does torque move towards mean value.
Threshold 1%
Minimum time within threshold: 1sec (~10data points)

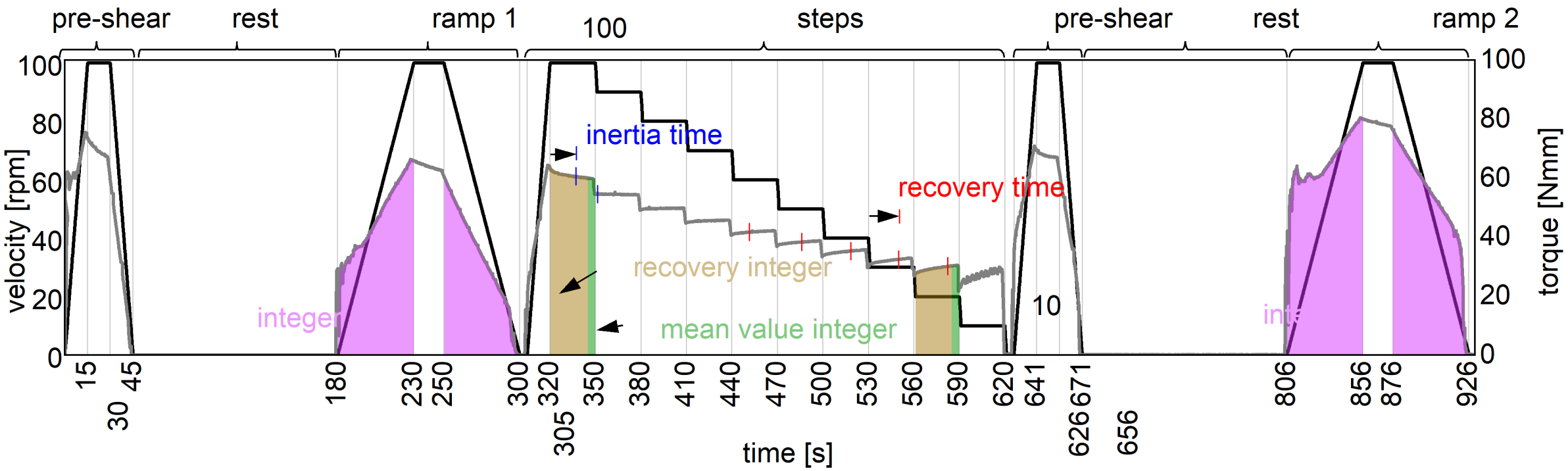
Thixotropy on a step profile

Inertia and recovery time





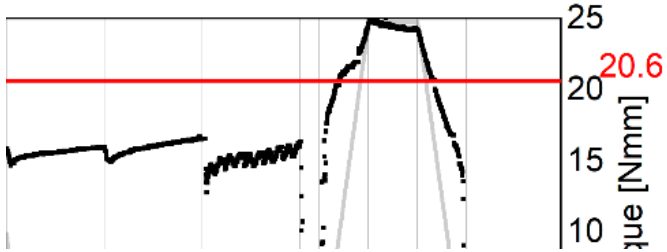
Thixotropy on a step profile

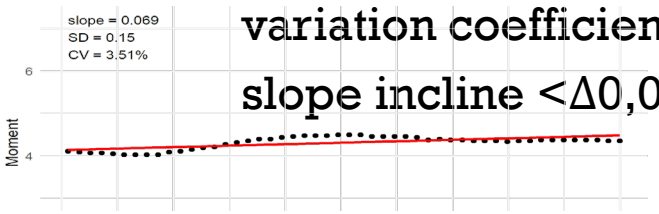


Ramp before steps
Step changes

Take Home Messages

max RPM after steps check for data integrity





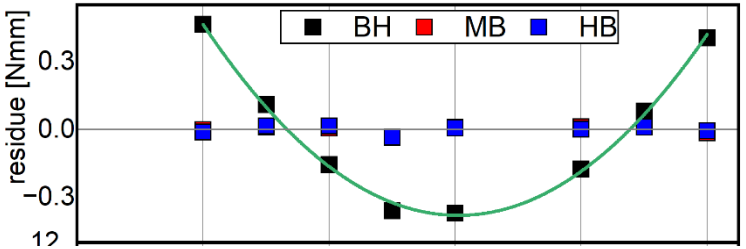
variation coefficient < 1 %

slope incline <Δ0,05 Nmm/s

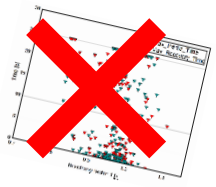
Equilibrium conditions via slope and variation coefficient

Flow model choice with Bingham residuum fit

$r^2 = 0.50$ > linear < nonlinear



$r^2 = 0.00$



Thixotropy on step analysis as a "sidekick" unreliable

Thanks for your attention

www.unibw.de/werkstoffe-en