

# How to Run a Round-Robin Test for Concrete Rheometers?

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# The Idea

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Somewhere in an obscure meeting room at the DTU Campus in Copenhagen on Sunday, August 21<sup>st</sup>, 2016...

... after 3 hours of discussions on the State-of-the-Art Report in RILEM TC 266: Measuring Rheological Properties of Cement-based Materials...

... Yannick Vanhove introduces a new business item:

“Why don’t we organize a new campaign to compare the output of different rheometers?”

# A Round-Robin Test.

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A Round-Robin test is an interlaboratory test performed independently several times.

A couple of examples:

- RRT on chloride analysis (RILEM TC 178, 2001)
- RRT on particle size distribution of Portland Cement (NIST, 2002)

Typically: send around a sample of material and have different people analyze the sample.

For fresh concrete, sending around a sample is a challenge...

# A Round-Robin Test.

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For fresh concrete, sending around a sample is a challenge...

Reproducing a sample around the world is also a challenge due to the influence of an extensive list of factors on rheology:

- Constituent Materials
- Mixing Energy
- Climate Conditions

So, when you can't bring the concrete to the equipment, you bring the equipment to the concrete...

We did this on May 29 and 30<sup>th</sup>, 2018 at the Universite d'Artois in Bethune, France



# The Organization

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Ideally, this is organized at a location which possesses a good amount of concrete rheometers:

U. Artois: ICAR, RheoCad (with vane and helix), ConTec 4SCC  
(with mixer and Mk-II), Tribometer

And others come from close or far away with more equipment or tools:

Schleibinger Gerate: Viskomat-XL, eBT, Sliper

EQIOM: ICAR

U. Bretagne-Sud: Plate test

U. Clermont-Auvergne: ICAR

Ghent University: ICAR

Missouri S&T: Interface rheometer tool



# The Organization

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Ideally, this is also organized at a location which has a lot of space and hands to help:

Apart from all collaborators (17), we also had the help from 1 technical staff member, and 7 students!

# The Organization

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To organize a RRT on concrete rheology, with the special demands the team put forward, you need a concrete supplier who is a leader in formulating the right concrete for each application.

EQIOM Concrete, under the lead of Faber Fabbris, was exemplary in meeting the complicated demands put forward by team: five concrete and three mortar mixtures, with some requests formulated an hour before delivery.

# The Organization

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## Financial Support

- French Group of Rheology
- Artois agglomeration community
- French National Federation of Public Works
- Structure & Rehabilitation Company
- Universite d'Artois
- Civil and Geo-Environmental Engineering Laboratory



# The Planning: Mixtures

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Compared to early 2000s:

- More flowable concrete
- Better knowledge about what can go wrong during a rheological measurement

Goal of the RRT: Maximize results output

- Minimize measurements that are incorrect or invalid
- Perform as many measurements as possible on the same mixture
- Perform different types of measurements

# The Planning: Mixtures

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Incorrect or invalid measurements:

Related to mixtures:

- Friction (avoid too many aggregates)
- Particle sedimentation (stable mixture, avoid low viscosity and large aggregates)
- Particle migration (avoid low viscosity and large aggregates)
- Extensive plug flow (avoid high yield stress and low viscosity)

Related to measurement and analysis:

- Non-equilibrium (appropriate duration of pre-shear)
- Plug flow (can be corrected if not extensive)
- Particle migration (short measurement duration)

# The Planning: Mixtures

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Flowable mixtures without high aggregate contents were desired.

No conventional vibrated concrete was tested.

Three mortars were evaluated intending to reduce torque fluctuations during measurements and minimizing shear-induced particle migration.

# The Planning: Mixtures

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Concrete 1: SCC	Reference	SF = 600 mm / VF = 20 s
Concrete 2: SCC	Lower Yield Stress	SF = 700 mm / VF = 5 s
Concrete 3: SCC	Higher Viscosity	SF = 550 mm / VF > 30 s
Concrete 4: Flowable	Less Powder	SF = 600 mm / VF = 20 s
Concrete 5: Flowable	Higher Yield Stress	SF = 400 mm / Slump = 230mm
Mortar 1:	Reference	SF = 750 mm / VF = 3 s
Mortar 2:	Higher Viscosity	SF = 650 mm / VF = 5 s
Mortar 3:	Higher Yield Stress	SF = 550 mm / VF = 3 s



*\*No conventional mixtures were evaluated*

# The Planning: Mixtures

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All mixtures were prepared in a nearby central batching plant and delivered on-site in one truck (no batch-to-batch variability).

For some mixtures, on-site adjustments with chemical admixtures were made to modify the workability towards the intended target.

Six wheelbarrows of concrete were kept in the laboratory for testing with all rheometers.

# The Planning: Measurements

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Flow curves: Measure dynamic yield stress and viscosity.

ICAR (4), Viskomat-XL, eBT-V, RheoCad Vane: fundamental units  
RheoCad Helix, 4SCC Rheometer: Relative units

Structural build-up: Measure static yield stress (stress growth curve)  
ICAR (2), Viskomat-XL, eBT-V, RheoCad Vane

Interface properties:

Related to pumping: SLIPER, Interface tool on ICAR

Related to friction: Tribometer



# The Planning: Measurements

	Flow Curves	Structural Buildup	Interface / Friction
0 min	Flow Curve 1		
10 min		Initial Static Yield Stress	
20 min			Interface / Friction 1
40 min		Final Static Yield Stress	
50 min	Flow Curve 2		
60 min			Interface 2
80 min	Flow Curve 3		

# The Planning: Measurements

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Flow curves: Pre-shear at  $N_{\max}$  of 20 s for concrete, 30 s for mortar to assure equilibrium. Decrease in 7 steps of 5 s each to  $N_{\min}$ .

However, what is  $N_{\max}$ ?

- Limitations of rheometers
- Limitations on shear-induced particle migration
- Do we impose same  $N_{\max}$  for each rheometer?

What is the fundamental parameter governing shear-dependency of rheological properties? Rotational velocity of shear rate?



# The Planning: Measurements

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Start with most restrictive rheometer (ICAR), impose  $N_{\max} = 0.5$  rps  
Assume a virtual concrete with  $\tau_0$  of 50 Pa and  $\mu_p$  of 20 Pa s

Reiner-Riwlin in the opposite sense

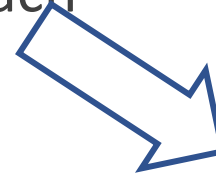
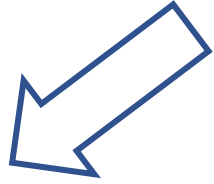
$$G_x = \frac{4\pi h_x \ln\left(\frac{R_{o,x}}{R_{i,x}}\right)}{\left(\frac{1}{R_{i,x}^2} - \frac{1}{R_{o,x}^2}\right)} \tau_0$$

$$H_x = \frac{8\pi^2 h_x}{\left(\frac{1}{R_{i,x}^2} - \frac{1}{R_{o,x}^2}\right)} \mu_p$$

# The Planning: Measurements

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Calculate G and H for each rheometer



$$T_{\max} = G + H \times 0.5$$

$$N_{\max} = (T_{\max} - G) / H$$



ICAR

$$\tau_{\max} = \frac{\left( \frac{1}{R_{i,x}^2} - \frac{1}{R_{o,x}^2} \right)}{4\pi h_x \ln \left( \frac{R_{o,x}}{R_{i,x}} \right)} T_{\max}$$

$$T_{\max} = \frac{4\pi h_x \ln \left( \frac{R_{o,x}}{R_{i,x}} \right)}{\left( \frac{1}{R_{i,x}^2} - \frac{1}{R_{o,x}^2} \right)} \tau_{\max}$$

Other Rheometers

# The Planning: Measurements

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	$N_{\max}$	$N_{\min}$
ICAR 1-3	0.500	0.025
Viskomat XL	0.540	0.027
eBT-V	0.529	0.026
Rheocad	0.570	0.028
4SCC Rheometer	0.210	0.010

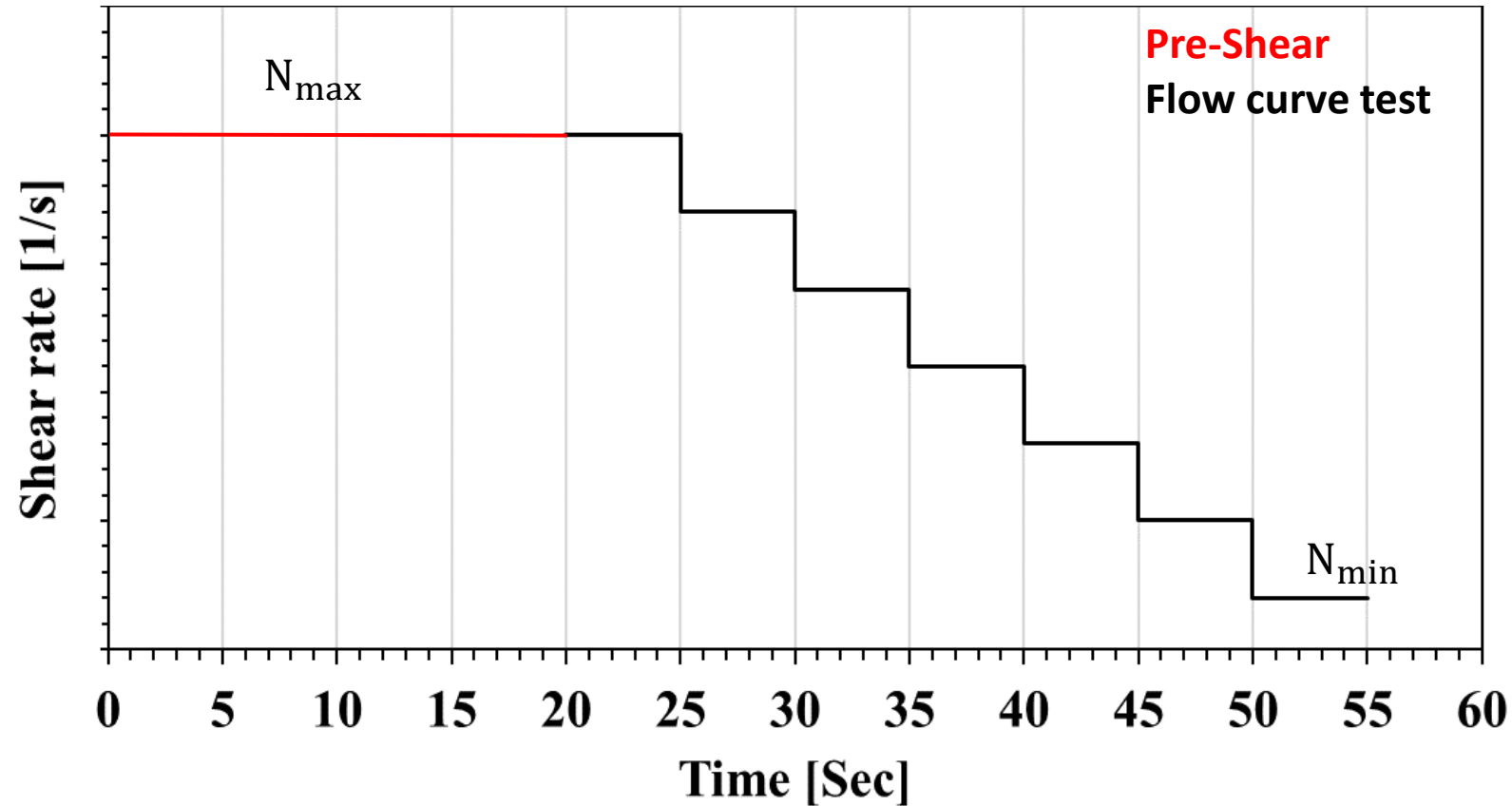
No fundamental units for RheoCad Helix, so used same as RheoCad Vane.



4SCC Rheometer: arbitrary values.

# The Planning: Measurements

Perform empty measurement: eliminate residual torque / set reference value



*One ICAR followed different procedure: discussed in next presentation*

# Lessons Learned Afterwards

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Need calibration material (e.g. silicon oil). Some rheometers delivered systematically higher values, which could have been avoided with a calibration material.

We did have a sample of the NIST reference material, but:

- Volume insufficient to cover the largest rheometers
- When recycling the material for a new test in a new device, paste and small beads are lost more than large beads, changing the concentration.

# Lessons Learned Afterwards

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Need more communication ahead of time on mixtures and procedures.

Most items were discussed / decided on the day before the tests, accompanied by a lot of fatigue and a lot of coffee...

# Summary

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Organizing a RRT for concrete rheometers is a large undertaking, requiring a lot of attention to details (batch homogeneity, uniform procedures, finetuning mixtures, timing of tests, etc.).

Five concrete and three mortar mixtures were evaluated to maximize output of valid results in different tests.

The contributions of the industrial partners were paramount for the success.

# Acknowledgments

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**Organizers: *Universite d' Artois*:** Y. Vanhove, C. Djelal, L. Libessart and the entire technical staff, undergrad and grad students

**Concrete and Mortar: *EQIOM*:** F. Fabbris and his team

***U. Clermont-Auvergne*:** S. Amziane,

***TU Dresden*:** E. Secrieru, S. Fataei, I. Ivanova, V. Mechtcherine

***Ghent U.*:** K. ElCheikh, C. Chibulu

***Schleibinger Gerate*:** M. Greim, H. Keller

***Ponificia U. Catolica de Chile*:** I. Navarrete

***U. Bretagne Sud*:** A. Perrot

***Queen's U. Belfast*:** M. Sonebi

***Missouri S&T*:** K. Khayat, D. Feys

