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# An overview of RILEM TC MRP round-robin testing of concrete and mortar rheology in Bethune, France, May 2018

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**Abstract**

Rheology of cement-based materials has been receiving a steadily growing interest. Seen the complexity of the material, several custom-made tools have been developed to assess rheological properties of these materials. However, different concrete rheometers can deliver different results, as demonstrated from two testing campaigns held in the early 2000s. A new campaign was performed in May 2018 in Bethune, France, and included new rheological devices, such as the ICAR, Viskomat XL, eBT-V, Rheocad, BML 4 SCC rheometers, as well as the plate test, Sliper, interface rheometer and tribometer instruments. The measurements were performed on three SCC, two highly flowable concrete and three mortar mixtures. Testing involved the evaluation of flow curves, structural build-up and interface properties and their evolution with time. Sources of errors were taken into consideration before and during measurements, and during data analysis.

**Keywords:** Concrete; flow curve; interface; rheology; structural build-up.

## 1. Introduction

Research on rheology of cement-based materials has been ongoing for over half a century [1], but it has known a more recent boom with the introduction of self-consolidating concrete (SCC) and other highly flowable mixtures, and more recently 3D printing. Concrete rheometers before roughly the year 2000 were instruments with complicated flow

patterns where particle sedimentation and shear-induced particle migration constituted major artefacts that can affect the assessment of rheological properties [1]. Since the introduction of SCC, rheometer design has been simplified with “simple geometries” used in new instruments, inspired from conventional rheometry.
In the early 2000s, two experimental campaigns were undertaken to compare different concrete rheometers: the first in Nantes, France and the other in Cleveland, OH, United States [2,3]. These campaigns focused on different concrete types, including a limited number of SCC mixtures, evaluated in commonly used rheometers at that time. However, since then, many new devices have been developed and refined. In addition, knowledge and awareness of measurement errors, such as structural breakdown, plug flow and shear-induced particle migration, has increased.
The RILEM TC MRP (Measuring Rheological Properties of Cement-based Materials) organized a new campaign in Bethune, France in May 2018 to evaluate the rheological properties of different mortar and concrete mixtures. This paper describes the various rheometers employed during the testing, the mixtures, the testing procedures and the data analysis.

## 2. Rheometers

The various devices employed during the Round-Robin tests are shown in Figure 1. A short description of each rheometer is provided below.

### 2.1. ICAR

The ICAR rheometer is a portable device, composed of a container, a driver, a four-blade vane and a frame [4]. The container is equipped with vertically protruding ribs to prevent slippage. Two types of tests can be performed: a stress growth test and a flow curve test. In the stress growth test, the vane is rotated at a constant slow speed and the maximum torque measured during this test is used to calculate the static yield stress. For flow curves, the torque is plotted versus rotational velocity of the vane, allowing the calculation of the Bingham parameters (dynamic yield stress and plastic viscosity).

### 2.2. Viskomat XL

The Viskomat XL rheometer is designed for stationary measurements of fresh mortar and concrete with maximum particle size of up to 16 mm. It is based on the Couette principle and can be operated with probes with different geometries. The measurement profiles can be freely defined using the wide dynamic speed range from 0.0001 to 80 rpm. Due to the high resolution and a broad torque range from 0 to 10 Nm, fresh concrete with different rheological properties can be characterized. For the investigation of other materials properties, such as visco-elastic properties of the material, Shear Stress Control mode and an Oscillation Mode can be used [5,6]. For the round-robin test measurements, a six-bladed vane probe was used.

*a) ICAR Rheometer* *b) Viskomat XL Rheometer* *c) eBT-V Rheometer*

*d) Rheocad Rheometer* *e) BML Rheometer 4SCC* *f) Plate Test Method*

*g) SLIPER Rheometer* *h) Interface tool for ICAR* *i) Plane/Plane Tribometer*

**Figure 1.** Rheometers evaluated during the round-robin testing program.

### 2.3. eBT-V

The mobile rheometer eBT-V from Schleibinger Geraete GmbH is a compact battery driven rheometer specially designed to determine the rheological properties of fresh mortar and concretes with maximum particle size of up to 32 mm [6-8]. The eBT-V has two built-in sensors and can be operated in two different methods: V-mode for modern concretes such as SCC, or ultrahigh performance concrete (UHPC); and P-mode for conventional vibrated and very stiff or no-slump concrete. The measurement profiles are freely programmable and can be adjusted to the rheometer configuration used and the mixture to be measured. For the measurements, the eBT-V in V-mode with the six-bladed vane geometry was used. The vane probe is attached to the drive shaft of the rheometer and was

immersed into the concrete together with the device holder containing rods to prevent the wall slippage during the measurement.

### 2.4. Rheocad

The RheoCAD500 device imposes a rotation speed to an inner cylinder tool, submerged into a container with the sample, and measures the resulting torque. During this study, two tools were used: the helical screw and the vane. The concrete is placed in a cylindrical container of 300 mm of diameter and 350 mm of height. This container contains a cage to prevent any slipping. The torque sensor has a capacity of 1000 Ncm and the speed can reach 250 rpm. For the vane tool, the Reiner-Riwlin equations allow to determine yield stress and plastic viscosity in Pa and Pas (assuming Bingham behaviour), respectively. For the helical tool, the device gives the variation of the torque (in N.cm) according to the rotational velocity.

### 2.5. BML Rheometer 4SCC

The Rheometer-4SCC is a portable rheometer for SCC developed by OH Wallevik at the Icelandic Building Research Institute (IBRI) [9,10]. It is capable of determining the yield stress and plastic viscosity of concrete mixtures with yield stresses ranging from 5 Pa to 120 Pa, plastic viscosities ranging from 5 Pas to 120 Pas, and maximum aggregate sizes of up to 22 mm. The Rheometer-4SCC consists of a control box and measurement unit. The control box and measurement unit are each less than 25 kg, which makes the device suitable for quality-control measurements at both building sites and in laboratories [11]. Two geometries were employed: a mixer-type of device and a cylinder inspired by the Tattersall Mk-II rheometer. Due to the lack of equations to obtain fundamental units, the ConTec Rheometer 4SCC software provides flow resistance results (driver electric intensity that can be linked to the torque) in mA.

### 2.6. Plate test

The plate test consists of measuring the mass variation of a rough tool (plate or cylinder) immersed in a fresh cement-based material with time [12] and was adapted for SCC [13,14]. This device was initially developed to enable a simple measurement of the evolution of yield stress with time or the structuration rate. The apparent mass of the immersed tool or the apparent mass of the sample is continuously monitored (recorded every 5 seconds during one hour). During the tests, the mortar or concrete was placed in a 1.5 m high and 200 mm in diameter column. The tool was a steel rebar (d = 150 mm, l = 130 mm) for the concrete mixtures and a screw (d = 14 mm, l = 80 mm) for the mortar mixtures, in order to adjust the tool's roughness to the size of the largest particles [14].

### 2.7. Sliper (Sliding Pipe Rheometer)

The Sliper was developed by Kasten [15] and is produced by Schleibinger Geräte GmbH. It allows a quick assessment of the pumping characteristics of concrete and other materials in the laboratory and on-site. The device enables to establish a relationship between

pressure (P) and flow rate (Q) results. Although the formation of the lubrication layer is not measured directly, it influences the P-Q results and hence can be indirectly measured by the Sliper. The Sliper was successfully applied for both stiff and highly flowable concretes [16,17]. Further details on the measuring procedure and limitations are available in [17]. In the study at hand, the prediction of P–Q relation was compared with the analytical approach based on the tribometer measurements according to [18].

### 2.8. Interface rheometer on ICAR

To evaluate the interface properties of concrete near a smooth surface, the vane of the ICAR rheometer was replaced by a smooth cylinder, 125 mm in diameter and 200 mm in height. The bottom has a conical shape to facilitate insertion in the concrete. This interface device enables the calculation of the lubrication layer properties yield stress and viscous constant, according to the procedure described in [19], which can be used to analytically predict pressure with the equations in [18].

### 2.9. Tribometer

The tribometer aims to mimic the friction between concrete and formwork. The principle is as follows [20]: an auger coupled to a motor moves, at various speeds, an interchangeable plate between two concrete samples. The concrete samples are pressurized between 50 kPa
and 1000 kPa. The sample holders are cylinders fitted with a hatch for introducing the concrete. A tension sensor tied to the plate measures the tangential friction stresses [21]. Tests were conducted on a plate cut from a formwork wall with roughness value $R_a$ = 1.07 µm. The plate displacement speed of 3 m/h (0.84 mm/s) corresponds to the casting speed of concrete in a formwork. The contact pressure of the selected jack corresponds to the concrete pressures exerted at the formwork base for a given height. Pressure values between 50 kPa to 150 kPa have been evaluated, corresponding to concrete heights between 2 m and 6 m.

## 3. Mixtures

Over the two-day testing campaign, eight mixtures were evaluated. All mixtures were prepared in a nearby concrete batching plant. In the case of some mortar mixtures, admixture dosages were adjusted on-site to ensure that the workability of the mixture was within the requested target range. Five wheelbarrows were sampled for rheological and workability testing.

### 3.1. Concrete mixtures

The first day consisted of measuring the properties of three SCC mixtures and two highly flowable concrete mixtures intended for foundation construction. Concrete 1 was a reference SCC mixture containing a Type III/A cement (EN 197) and limestone filler as powder materials, and a maximum aggregate size of 12 mm. The target slump flow of SCC

1 was 550-650 mm. Concrete 2 had the same mixture proportioning as concrete 1, except for a higher HRWRA content to increase the target slump flow to 650-750 mm. Concrete 3 was a highly viscous SCC, containing 50 l/m$^3$ less water than concrete 1, with a target slump flow of 550-650 mm. The cement content in the powder was increased, and the maximum aggregate size was 20 mm for concrete 3. Concretes 4 and 5 are typical mixtures for concrete foundations that have lower powder contents compared to the three SCC mixtures. These were produced with a maximum aggregate size of 12 mm. The admixture combinations were altered between mixtures 4 and 5 to create a less flowable and more cohesive nature for the latter mixture. It should be noted that the team chose not to evaluate any conventional concrete mixtures to avoid the issue of extensive plug flow.

### 3.2. Mortar mixtures

On the second day of the campaign, three mortar mixtures were evaluated. The idea behind testing mortar mixtures in concrete rheometers is to reduce the influence of shear-induced particle migration. However, the mixtures had to be sufficiently stiff or sticky to ensure adequate response of the load cells in the rheometers. The mortars were produced with a Type I 52.5 cement and a limestone filler. Mortar 1 was a reference mixture, mortar 2 was a mixture with high viscosity and low yield stress, while mortar 3 was intended to have a (relatively) high yield stress and low viscosity. None of the mixtures were air-entrained.

### 3.3. Reference material

During the course of the testing campaign, the ICAR rheometers, BML with modified Mk-II configuration and the Viskomat XL were used to evaluate the rheological properties of the NIST reference material. This material consists of a mixture of corn syrup, limestone filler and water to mimic a Bingham-like paste. Glass beads with 1 mm diameter were used as fine aggregate to simulate mortar, and other beads with 10 mm diameter were added to create an artificial concrete. The reference material was only used to evaluate flow curves.

## 4. Testing methodology

In contrast to the previous comparison campaigns, different properties were evaluated with different devices. Flow curves, static yield stress values and interface properties were evaluated at each time. The following sections describe how each of these properties were determined.

### 4.1. Flow curves

Flow curves were determined by decreasing the rotational velocity, stepwise, from the maximum to the minimum imposed rotational velocities. Each flow curve contained seven or eight steps of 5 s duration each (except for one ICAR rheometer). Each flow curve was preceded by a 20 s (concrete) or 30 s (mortar) preshear period at the highest rotational velocity (except for one ICAR, where the pre-shear was 60 s). However, the rotational velocities were different for each rheometer. Although this sounds counter-intuitive for a

comparison, one of the reasons why different rheometers may give different results is the applied shear rate. As geometries are different, the shear rate in each rheometer will be different if the same rotational velocity is imposed. Instead, the rotational velocities were adjusted theoretically to ensure that an approximately constant shear rate was imposed across the different rheometers in use.

### 4.2. Static yield stress

Static yield stress values were determined by evaluating the peak torque at a low rotational velocity in each rheometer. Static yield stress tests were executed with a 30-min interval to determine and compare the structural build-up of each mixture. The computed values of structural build-up are compared with the results obtained using the plate test.

### 4.3. Interface properties

The interface properties were calculated according to the procedures developed for each device. No procedure could be standardized, as all three rheometers have different modus operandi. The comparison between the devices can be made by predicting the pressure-flow rate relationship in a pipe with the results from each equipment separately.

### 4.4. Fresh concrete tests

The fresh concrete mixtures were evaluated for temperature, slump or slump flow, T50 (if possible), air content, density, L-box, J-Ring and V-Funnel flow time. In addition to the standard slump cone, several smaller cones were utilized for the mortar flow testing.

### 4.5. Timeline

Table 1 shows the timeline of tests for each mixture. All times are relative to the first tests executed. All rheometer tests were started at exactly the same moment. In between tests, the rheometers were emptied, cleaned and prepared for the next tests. When static yield stress tests were executed, the sample was left undisturbed until after the 40 min test.

**Table 1.** Timeline of tests. FC = flow curve, YS = static yield stress, IF = interface properties.

| | | 0 min | 10 min | 20 min | 40 min | 50 min | 60 min | 80 min |
|---|---|---|---|---|---|---|---|---|
| **ICAR 1** | Vane | FC | YS | | YS | FC | | FC |
| **ICAR 2** | Vane | FC | YS | | YS | FC | | FC |
| **ICAR 3** | Vane | FC | YS | | YS | FC | | FC |
| **ICAR 4** | Vane | FC | | | | FC | | FC |
| | Cylinder | | | IF | | | IF | |
| **eBT-V** | Vane | FC | YS | | YS | FC | | FC |
| **Viskomat XL** | Vane | FC | YS | | YS | FC | | FC |
| **Sliper** | | | | IF | | | IF | |
| **Rheocad** | Vane | FC | YS | | YS | | | FC |
| | Helix | | | | | FC | | |
| **BML 4SCC** | Mod. MK-II | | | | | FC | | |
| | Mixer | FC | | | | | | FC |
| **Tribometer** | | | | IF | | | | |

| | | | |
|---|---|---|---|
| **Plate test** | Continuous structural build-up measurement | | |
| **Fresh concrete tests** | Yes | Yes | Yes |

## 5. Analysis steps and challenges

At the writing of this paper, data analysis and comparison of the results of the different rheometers are still underway. However, to ensure that all data can be compared, the following analysis steps were enforced, as described below.

### 5.1. Flow curves

The flow curves are perhaps the most critical to evaluate, as many measurement errors need to be taken into consideration. For each test, the following procedure was followed:

- Zero measurement to evaluate the residual torque before the introduction of the test material.
- Determination of equilibrium at each rotational velocity step. Steps with decreasing torque trends during the measurement interval or extreme fluctuations of data are not considered in the analysis.
- Preliminary calculation of yield stress and plastic viscosity by means of the Reiner-Riwlin equation, if the rheometer configuration can be approached as concentric cylinders (vane geometry).
- Verification of plug flow, and iterative calculation of yield stress and viscosity by taking reductions in the sheared domain into consideration.
- Estimation of the thickness of the sheared zone: if this zone is smaller than the maximum aggregate size, the measurement is certainly invalid.

This analysis procedure ensures that measurements errors caused by structural breakdown and plug flow are eliminated from the measurements. Shear-induced particle migration is more difficult to assess, but mixtures with very small sheared zones have probably undergone shear-induced particle migration. However, it is uncertain whether no shear-induced particle migration is present in each of the tests.

The expected outcome of this analysis step are the yield stress and viscosity values for each rheometer tests. Ongoing analysis evaluates the correlation between each rheometer and the average, as well as an identification of outliers and any reason why certain points could be outliers.

### 5.2. Static yield stress tests

For the static yield stress tests, the analysis is somewhat less complicated. The zero-measurement remains important to correct measured torque values. The static yield stress values are determined by dividing the peak torque value by $2 \pi R_i^2 h$, in which $R_i$ and h are the radius and height of the inner cylinder in the rheometer, respectively, in case of a rotational rheometer. It can be noted that the shear surface located underneath the vane tool is neglected. Based on the 10 and 40 min data, thixotropic indices can be calculated, according to different strategies in literature [e.g. 22]. The analysis will be focused on the calculated indices, rather than the individual static yield stresses.

### 5.3. Interface tests

Each interface rheometer has its own procedure to analyze the data, so no uniform strategy was established. However, each device enables the calculation of a pressure-flow rate relationship for flow inside a pipe. This strategy will be employed to compare the different interface devices.

## 6. Concluding remarks and outlook

A new testing campaign compared the output of different concrete rheometers, being several ICAR rheometers, Viskomat XL, eBT-V, Rheocad, BML 4SCC, Sliper, plate test, and tribology tests. The tests were carried out on three SCC mixtures, two highly flowable concrete mixtures for foundation construction, and three mortars. Significant variations in yield stress, viscosity and thixotropy were induced by variations in the mix designs.
The testing procedures were adjusted to ensure that each rheometer imposed approximately the same shear rate on the materials. Flow curves, structural build-up, and interface properties were evaluated. Attention was paid to potential sources of errors in the analysis. With the results, the research team will draw comparisons between rheometers, analyze outliers, investigate the validity of the measurements and potentially identify critical combinations of mix design parameters and rheological properties that can compromise the correct assessment of the characteristics of the materials.

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