

FROM CEMENT GROUT TO CONCRETE SCALE: A STUDY OF SUPERPLASTICIZER-DESIGN-CONTROLLED THIXOTROPY TO MATCH SCC APPLICATION REQUIREMENTS

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ABSTRACT

Self compacting concrete is now widespread in precast applications due to its ability to fill even complex formworks without vibration. This study shows how properties like long distance self levelling, segregation or stickiness may be influenced by superplasticizers through the interaction with thixotropy.

Low shear rate rheological measurements were performed on concrete-equivalent grouts to monitor the influence of polycarboxylate superplasticizers on structure buildup rates. The thixotropy description initially developed by N. Roussel was applied to steady state flow curves in order to model the influence of admixtures.

Specifically designed concrete channel and box-flow tests were developed in order to assess workability and formwork-filling retention while applying as little energy as possible to the material. The trends observed at the rheometer scale were confirmed at larger scale showing that structure build-up kinetics have a major influence on concrete placing and that superplasticizers may partly control it. A new superplasticizer allowing faster and more homogeneous formwork-filling properties was developed and successfully tested on a full scale.

Keywords: Superplasticizer, thixotropy, rheology, channel flow test

INTRODUCTION

In the last decade self compacting concretes have spread widely in the precast market owing to the ability to cast without vibration. This aspect enabled to reduce human operations and exposition to noise and other health-related risks. Superplasticizers are essential to the production of SCCs [1, 2] but nowadays they should not only reduce water content, but also bring more complex flow characteristics, such as speeding up the filling of intricate geometries or dense rebar networks. Such features require careful control of rheological properties, such as yield stress and plastic viscosity [3].

Early on during this project it appeared that common laboratory tools such as Abrams slump cone, V-Funnel flow time, J-Ring flow or even L-Box failed to distinguish subtle differences. A rheological approach was then developed for a more precise insight on the flow properties and specific tools were developed to further differentiate concrete properties at lab scale, according to customer feedback.

A close look at casting operations led us to consider the behaviour at low shear rates as most significant. Low shear rate flow was shown to be largely influenced by suspension structure build-up kinetics, often summed-up under the term ‘thixotropy’ [4]. This aspect is the key topic of this multi scale study from grout scale modelling of flow in the presence of several superplasticizers, to a laboratory scale concrete validations.

THIXOTROPY MODELLING BACKGROUND

Roussel [5] proposed a model for taking thixotropy into account in the flow curves of a material. It is based on the assumption that viscosity is linked to a parameter λ which measures the ‘structure degree’ of the suspension (which is described in Eq. (1)) and obeys a relaxation law Eq. (2) :

$$\eta = \eta_{\infty} (1 + \lambda^n) \quad (1)$$

$$\frac{d\lambda}{dt} = \frac{1}{\theta} - \alpha \lambda \dot{\gamma} \quad (2)$$

In Eq. (1) η is the apparent viscosity, η_{∞} is the viscosity plateau at high shear rate and n quantifies the influence of λ on the viscosity. In the differential equation θ is a time constant for the structure build-up rate and α describes the efficiency of shear in breaking up the structure. At the equilibrium state under a constant shear rate Eq. (1) may be rewritten under the form of a steady state flow curve:

$$\eta_{eq} = \eta_{\infty} (1 + (\alpha \theta \dot{\gamma})^{-n}) \quad (3)$$

As will be seen in the experimental data, a shear-thickening behavior often appears beyond a given shear rate. This led us to modify Eq. (3) by adding a hydrodynamic dissipation term $\beta\dot{\gamma}$, following the approach by Hot and Roussel [6]:

$$\eta_{eq} = \eta_{\infty} \left(1 + (\alpha\theta\dot{\gamma})^{-n} \right) + \beta\dot{\gamma} \quad (4)$$

GROUT RHEOLOGY MODELLING –EXPERIMENTAL RESULTS

MATERIALS AND EXPERIMENTAL METHODS

The water and admixtures were weighted in a Krups YY8506FD mixer bowl, the dry powders (composition in *Table 11*) were added during the first 30 seconds of mixing at speed 1 with a leaf-shaped blade. The mixing speed was then increased to speed 7 for 1 minute, then stopped for 30 seconds (to scrape the sides of the bowl) before applying a last speed 7 – mixing stage.

Table 11. Grout composition (g)

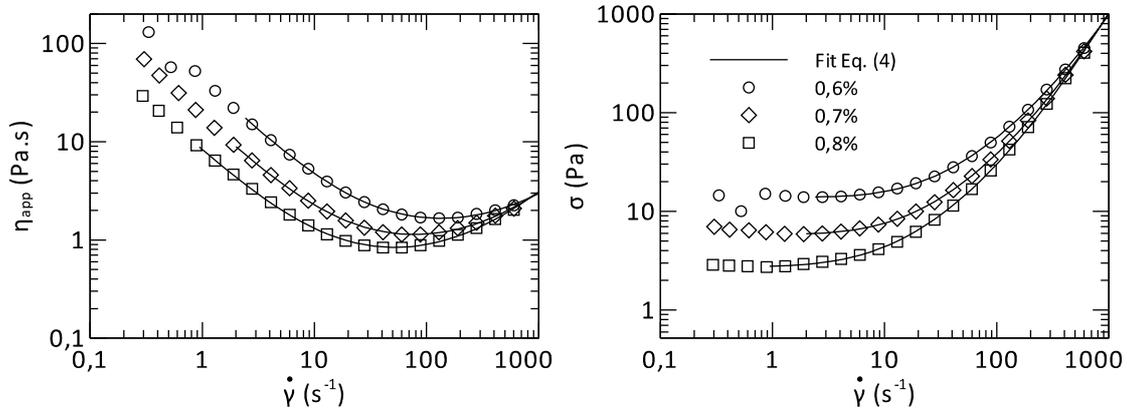
Cement (CEM I 52,5 R)	Limestone Filler	Sand 0/0.160	Sand 0/0.315	Water
196.1	89.6	97.8	20.7	93.6

The sample was loaded on the lower plate of a Kinexus rheometer (Malvern Instruments, U.K.) equipped with a serrated parallel plate geometry (1 mm gap). The procedure starts five minutes after the beginning of mixing with a pre-shear at 200 s^{-1} during one minute, followed by logarithmic shear rate steps from 200 to 0.01 s^{-1} . Each stress data point was sampled at the steady state whenever possible in order to build the flow curve. At the same time minislump tests at 5 minutes were performed (cone dimensions : upper diameter 18mm, lower diameter 36mm, height 54mm).

RESULTS

In order to check the model response and determine the most relevant parameters, Eq. (4) was fitted to the obtained experimental data with an increasing dosage of superplasticizer. *Figure 19* shows flow curves for increasing dosages of a standard acrylic polycarboxylate superplasticizer. As expected, apparent viscosity and yield stress decreases at all shear rates when dosage increases.

Figure 19. Flow curves for three different dosages of superplasticizer. Left: Apparent viscosity, right: Shear stress.



The left plot stresses out the need of a hydrodynamic term in order to better simulate the increase in apparent viscosity beyond a critical shear rate. The right plot shows a minimum shear stress occurring at a critical shear rate below which no steady-state may be achieved; consequently Eq. (4) was not fitted to these data according to the approach described in [5].

Figure 20. Model parameters as a function of the dosage of superplasticizer. Error bars are based on a χ^2 fit with a 95% confidence interval

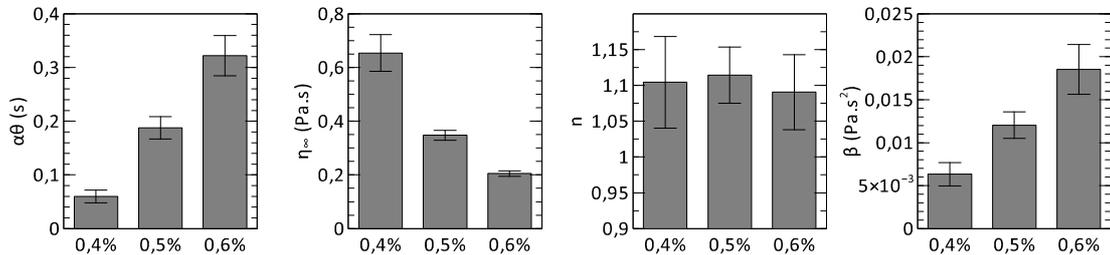
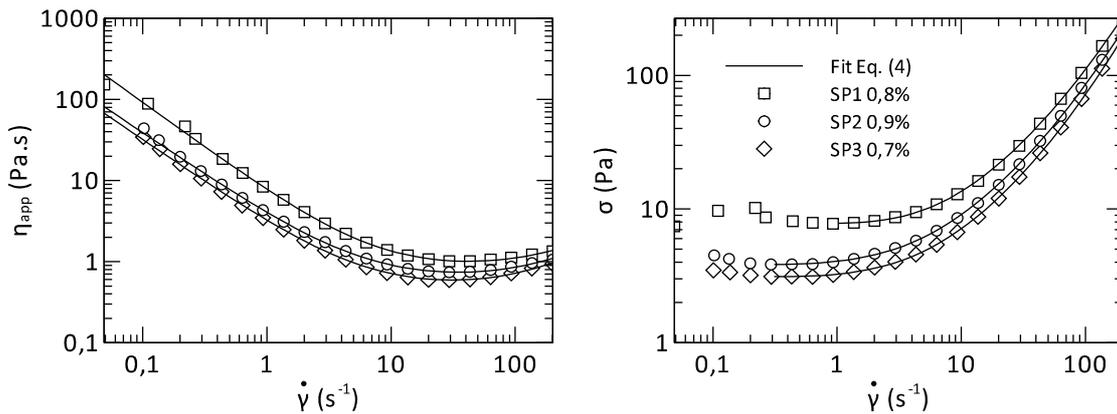


Figure 20 displays the parameter values obtained by the modelling of the flow curves according to Eq. (3). Increasing the dosage leads to an increase in the product $\alpha\theta$ which means that either structure build-up is slower (θ increases) or shear break-up efficiency is higher (α increases). η_∞ decreases when superplasticizer dosage increases which implies that the high shear rate state of the suspension is more deflocculated. The parameter n does not seem to have a meaningful dependence on the superplasticizer content. β increases with dosage meaning that the hydrodynamic dissipation increases according to the deflocculation degree.

This preliminary study allowed us to better understand the physical meaning of the different parameters and to determine that $\alpha\theta$ and η_∞ are most relevant for the description of low-shear rate flow. For a lesser influence of thixotropy on flow one would look for a higher $\alpha\theta$ and a lower η_∞ .

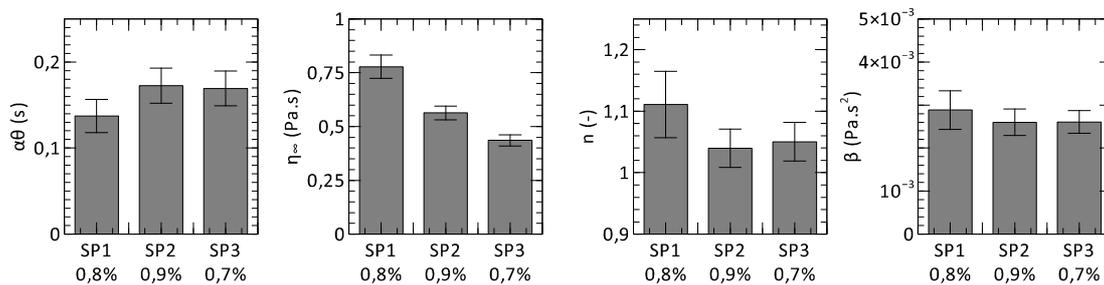
The method described above was used to select the most efficient superplasticizers before testing at the concrete scale. Dosages were adjusted in order to obtain a target mini-slump flow of 105 ± 5 mm, a target value determined from the slump flow of a reference concrete. Three examples from the study are displayed in *Figure 21*, SP1 being a common precast superplasticizer and SP2 and SP3 being two products specifically designed for a reduction of thixotropy.

Figure 21. Flow curves as a function of shear rate of grouts at the same slump flow. Left: apparent viscosity, right: Shear stress



Despite the grouts being prepared at equal minislump flow, slight yield stress differences may be observed on the right plot. This was quite unexpected since the general idea is that flow spread is directly linked to yield stress [7, 8]. Here it may show the influence of thixotropy on the slump test itself, which is possibly magnified by a high paste volume fraction compared to concrete testing. Model parameters fitted according to Eq. (3) are shown in *Figure 22*.

Figure 22. Parameter values for the three selected superplasticizers



SP2 and SP3 seem to induce higher values of $\alpha\theta$, which corresponds to a slower structure build-up or a higher shear efficiency. They also have a similar influence on n while SP1 yields a slightly higher n value. η_∞ shows the same trend as the apparent yield stress and SP3 seems to be the most efficient superplasticizer to decrease this parameter which confirms its deflocculating efficiency. The hydrodynamic dissipation term β seems to be equivalent for the three measurements.

This methodology was systematically used for qualifying polymer structures in the aim to reducing thixotropy. Concrete scale tests of SP1, SP2 and SP3 will be presented in the next paragraph as they perfectly illustrate the best performance in the series.

CONCRETE SCALE TRIALS

MATERIALS AND EXPERIMENTAL METHODS

The concrete tested in this study represents a typical composition of a self compacting concrete for prestressed applications.

Table 12. Concrete composition (kg/m³)

Material	Cement CEM I 52,5 R	Limestone Filler	Sand 0/4	Crushed Gravel 4/10	Riverbed Gravel 8/16	Effective Water
Amount	350	160	642	859	114	176

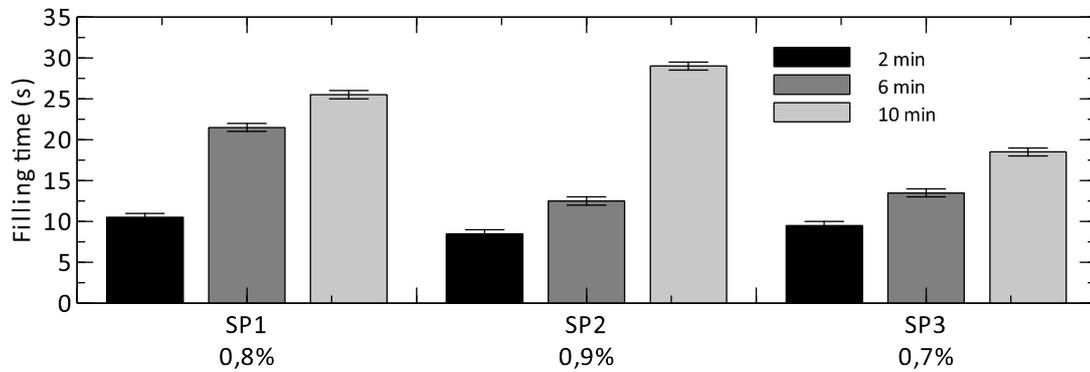
In a laboratory SKAKO horizontal mixer the sand and gravel were blended and pre-humidified for 5 minutes with a fraction of the batching water. The binders were then added in the dry state. The admixtures were dosed in a pail with the remaining water, subtracting the water brought by the superplasticizers from the total amount of liquid, and then incorporating the aqueous part with the rest of the blend into the mixer for 3 minutes and 30 seconds. Given the need for differentiating thixotropy-induced flowability variations at low shear rate a robust and simple tool was designed to evaluate such behaviour on concrete. The CHRYSOBox (as nicknamed) is a rectangular container separated in two parts of equal size by a removable gate. One compartment (side 1) is filled with concrete right after the end of mixing and the inner wall is then lifted 2, 6 or 10 minutes later. The time taken by the concrete to reach the opposite end (side 2) of the box is measured at each resting time and gives a practical evaluation of structure build-up during the considered time interval.

RESULTS

SCCs were prepared with the three superplasticizers selected after the rheological study. The dosages were determined in order to achieve a 700-720mm slump flow with the Abrams cone. CHRYSOBox filling times are displayed in Figure 23. For a 2 minute resting time all three products are comparable. For a 6 minute resting time SP1 seems less efficient in slowing down structure build-up. For a 10 minute resting time

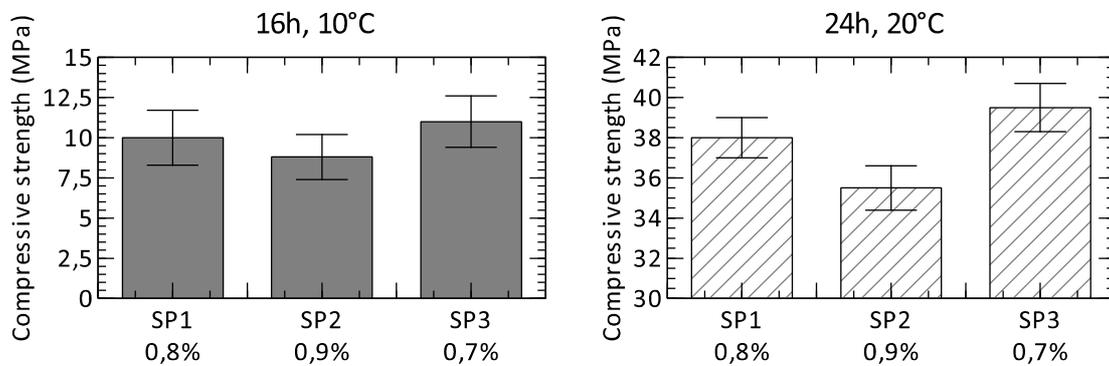
SP3 shows a significantly faster filling after wall lifting, which confirms it is the best admixture for limiting thixotropic effects.

Figure 23. CHRYSO box filling times after different aging periods



Compressive strengths were checked on 15x15x15 cm³ cubes cured at 10°C during 16h or 20°C for 24h. Figure 24 shows that the flow improvement brought by SP3 does not induce a decrease in strength, even in the harsh conditions of 16h, curing at 10°C. In the same conditions SP2 induces a strength decrease due to both its slightly higher dosage and its polymer architecture.

Figure 24. Compressive strengths in two different conditions



INDUSTRIAL APPLICATION FEEDBACK

SP3 was tested versus the current superplasticizer of the factory in a typical prestressed beam application. Results are shown in Table 13.

Table 13 Field trial results

Cement (kg/m ³)	Water (l/m ³)	Admixture	Dosage	Flow (mm)	Comp str 14h (MPa)	Comp. str. 16h (MPa)
433	205	SP3	1.00%	680	47.7	51.3
		Current superplasticizer	1.25%	680	41.8	50.1

For the same target slump flow of 680 mm the required SP3 dosage was 1% by weight of total binder versus 1.25% for the current superplasticizer. Despite the lower dosage faster form filling was obtained as observed by the customer. Moreover the decrease in dosage led to lower risks of collapsing and allowed a 6 MPa gain at 14h, thus an earlier stress release and a faster manufacturing cycle. Surface finishing was noticeably improved as well as facing quality (no pin holes).

CONCLUSIONS

In this work rheological measurements were correlated to concrete-scale tests which allowed us to facilitate the development of a new superplasticizer, since most of the less efficient experimental admixtures were ruled out at the grout scale. The thixotropy model by Roussel proved quite convenient for the modelling of steady state flow curves and allowed to better understand the interaction between superplasticizer polymer architecture and suspension structure build and break-up under shear. These results were confirmed at the concrete scale with specifically designed tests for the assessment of formwork-filling efficiency and flow restart after rest. Field trials at customer sites were consistent with the observed trend, leading to significantly shorter casting times. SP3 was launched earlier this year as CHRYSO®Fluid Premia 500.

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