Application Note
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# Rheological Properties of Fresh Building Materials

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## Abstract

The properties of building materials during application and after hardening highly depend on their flow behaviour at the fresh state. Due to the wide spread use of rheology modifying additives the currently common empirical test methods are no longer sufficient to describe the rheological properties of fresh building materials. Using rheological testing devices such as the HAAKE RheoStress 600 allows for a comprehensive description of the flow behaviour of such materials.

This report will give a short overview over the various problems regarding the description of the rheological behaviour of freshly prepared building materials. Further the test methods for the determination of these properties and results, which were acquired during a project on the development of pumpable self-compacting lightweight concrete (SCLC) will be presented.

## Introduction

Both the mechanical properties as well as the durability of the hardened building materials strongly depend on the properties of these materials at the fresh state. For optimum hardened properties the minimization of the porosity of the final product and the ability to influence the pore structure are of great importance. These two properties are both highly dependent on the rheological properties of the freshly prepared building material.

Today it is easily possible to intentionally influence the rheological properties of building materials based e. g. on cement by adding organic or inorganic additives. On the over hand, the determination of the rheological properties of these materials still is a very demanding task. In the past empirical methods have been developed to describe the material's properties during certain applications. In many cases these were flow experiments, during which a certain flow distance or, less frequent, a flow speed were determined.

However, the empirical methods are usually not suitable for a scientific approach. Rheological measurements on the other hand allow a precise and separate determination of different parameters influencing the flow behaviour. With a better knowledge about the effects certain additives have on these properties, a targeted approach is possible.

Compared to standardized methods for other materials, the physical and chemical heterogeneity of fresh building materials has to be taken into account when developing a rheological method for their characterization. First of all the particle size between e. g. 0.2 and 2000 µm for a usual mortar makes it impossible to use the classical plate/plate and cone/plate geometries. Since these materials are dispersions with up to 70 vol.% solids content, they show a strong tendency to form a surface layer of water and fine particles leading to slippage of the measuring system.

Mortars and other materials with fine particles can still be characterized using specials measuring systems, which usually have in common that the sample will not be sheared by a defined surface-area. Absolute, physically sound figures for the rheological properties can therefore only be acquired by comparison with calibration measurements. If a comparison between different products is sufficient, the relative values can directly be used.

Further, the constituents of building materials like e.g. cement have a high reactivity in the presence of water, which causes additional problems during the attempt to measure their rheological properties. After the addition of water these materials show a time dependency in their flow behaviour, an effect which must be taken into account during the design of the measuring methods and the evaluation of the data measured.

This report will explain some basic parameters and methods relevant for the measurement of the rheological properties of building materials. Afterwards some techniques to determine these parameters and the necessary analytical tools will be introduced. The evaluation of measuring results and the determination of certain properties will be shown. Finally various results of investigations performed at the Institute of Concrete Structures and Building Materials using a HAAKE RheoStress 600 (Thermo Scientific) will be presented.

## Background and Methods

Modern mortars are used in a wide variety of applications e. g. as adhesives for tiles or matrix of concretes for construction. Subsequently the composition of these materials varies over a wide range. All compositions however do have in common, that organic and inorganic additives are used in increasing amounts to influence the pastes or mortars fresh and final properties. It is therefore not possible to simply describe the properties of the fresh materials based on their composition. At the same time test methods based on flow tests, spreading tests or compression tests give only an incomplete picture of the material's properties.

This background explains the need for new test methods yielding further information necessary for the control of the fresh material properties. Despite the great range of applications and demands, this broad bandwidth can be sufficiently described by a limited number of physical characteristics.

This can be traced back to the fact, that fresh construction materials partially exhibit the characteristics of an elastic solid and partially those of a viscous liquid. Using rheological methods the elastic and viscous properties can be detected separately. It is therefore possible e. g. to avoid the slippage of freshly glued tiles by only increasing the elastic properties of the grout. Alternatively it is possible to get a soft concrete consistency by drastically lowering the elastic properties of the mortar, which forms the matrix for this material.

The viscous properties of a given material can be depicted using the so-called „flow curve“ (Fig. 1). It shows the relation between the **shear stress $\tau$** applied and the resulting **shear rate** $\dot{\gamma}$. The slope of the flow curve in any given point is the **dynamic viscosity $\eta$** related to a certain shear stress and shear rate. While so-called Newtonian fluids like water or mineral oils have a constant viscosity, i. e. the flow curve runs through the origin and has a constant slope, disperse construction materials show a totally different behaviour. Initially a freshly prepared concrete or mortar shows an elastic deformation when a defined stress is applied. Only if the applied stress exceeds a critical value, the so-called **yield stress $\tau_0$**, the behaviour of the material becomes predominantly viscous. The majority of concrete-based materials are so-called **Bingham Materials,** i. e. they show a yield stress and have a linear flow curve at higher stresses. The slope of the flow curve is the **plastic viscosity $\mu$** of a given mixture. As an example, Fig. 1 shows the flow curves of a Newtonian fluid, an ideal Bingham material and a real cement paste. Compared to the ideal Bingham behaviour, the real material shows a pronounced increase in shear stress at lower shear rates. This behaviour is important especially for the segregation-stability of a mixture.

Since for disperse construction materials the measurement of their rheological properties is possible only by using special geometries without a well defined surface area, instead of using shear stress and shear rate the torque M and the rotational speed Ω are being used as relative characteristics. The derived expressions viscosity and yield stress subsequently have the units mNm/(1/min) and mNm respectively.

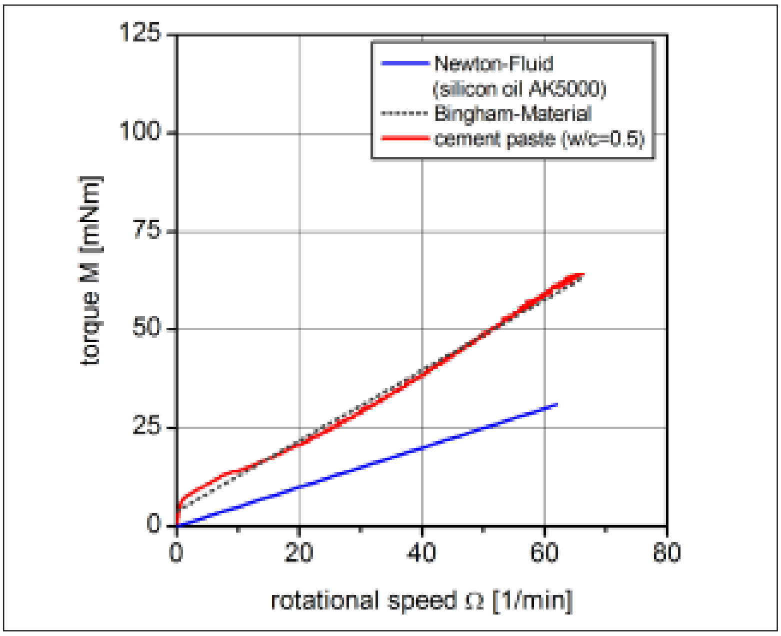

*Fig 1: Flow curves of a Newtonian fluid, of an ideal Bingham-material and a real cement adhesive*

Another speciality regarding the rheology of fresh construction materials is the pronounced dependency of their rheological properties on the shear history and age. In other words, the rheological properties change with time and due to the imposed flow itself. In the usual case the viscosity of construction materials drops with increasing shear time and recovers after the material is allowed to rest again. In case of a fully reversible process, this behaviour is called **thixotropy.** The degree of thixotropic behaviour has a mayor influence on the properties during application as well as on the stability and homogeneity of the material during and after placing. The determination of thixotropic behaviour and the time depending material properties is only possible using a highly developed rheometer like e. g. the HAAKE RheoStress 600.

Regarding the wide variation of particle size distribution of different mixtures, a cylindrical cup with a diameter of 83 mm in combination with a paddle-shaped rotor has proven to be useful (Fig. 2). Because of the special shape of the rotor slippage due to the formation of a superficial film is avoided and the homogeneity of the mixture is guaranteed.

After filling the cup with a defined sample amount of 375 cm$^3$ it is put into the rheometer and the measurement is started. The HAAKE RheoStress 600 allows a wide variety of possible tests. The rheometer can be operated in CS mode where a defined shear stress is applied and the resulting shear rate is measured or in CR mode where the shear rate is set and the necessary shear stress is measured. Especially the measurements in CS mode allow test programmes, which closely reflect real life conditions e. g. the behaviour of a fresh mortar under the influence of gravity.

Apart from rotational tests, oscillatory test are very useful for the characterization of the rheological properties of disperse construction materials, especially for the investigation of the elastic properties without destroying the internal structure of the sample.

Since the rheological properties of fresh concrete-based materials strongly depend on time and rheological history the whole measurement including the sample preparation has to follow an exactly defined procedure. This includes storing the starting materials at constant temperature, defining a mixing procedure using a mixer according to DIN EN 196-1, as well as the determining the sample’s density after preparation. It might be helpful to compact the samples on a shaker. The time between adding the water and starting the measurement should be kept constant to get comparable results.

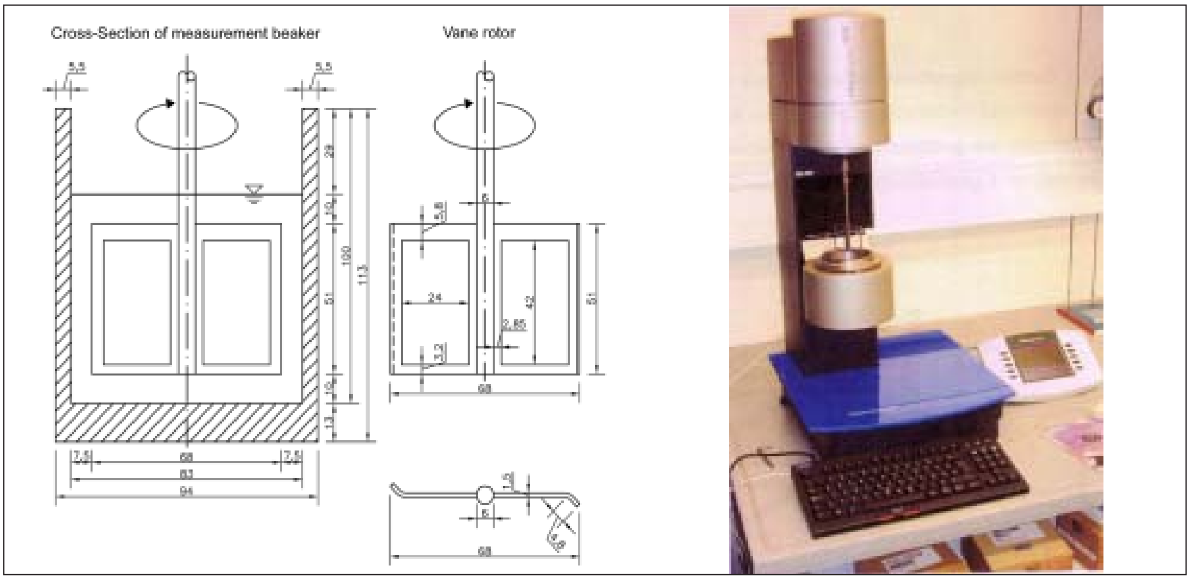

*Fig. 2: Measuring geometry for the determination of the rheological properties of pastes and mortars with HAAKE RheoStress 600*

## Measurements and Data Evaluation

As mentioned previously, pastes and mortars can usually be characterized by the Bingham-model. This behaviour can be described using two independent parameters, the plastic viscosity μ and the yield stress $\tau_0$. The simplest approach to determine these parameters is to submit the sample to a certain shear profile. The shear rate is continuously increased from zero to a given end value or it is decreased from a certain start value to zero. The measured shear stress is plotted over the shear rate and the parameters μ and $\tau_0$ are taken from alinear regression of this data (Fig. 1).

While this approach is suitable for the determination of the plastic viscosity, the regression for the yield stress leads to a major error. The main reason for this is the fact, that the flow behaviour of fresh construction materials deviates strongly from the linear Bingham model at low shear rates. On top of that, determining a yield stress with a shear rate controlled measurement (CR) does not reflect the real conditions during the application, where the gravitational force controlls the flow. Using the CS-mode already mentioned above the real material behaviour can be simulated by applying a shear stress gradually increasing from zero up to an individually defined value. When $\tau_0$ is exceeded the rotational speed of the vane rotor increases instantaneously. This can be seen e. g. when plotting the dynamic viscosity η - the shear stress τ applied divided by the resulting shear rate $\dot{\gamma}$. (or torque divided by rotational speed) - against the shear stress. Initially the dynamic viscosity increases strongly and goes through a maximum when reaching the yield stress $\tau_0$ (Fig. 3). Another option is to determine the yield stress from an amplitude sweep i. e. in oscillation mode. This will be covered in a separate application note.

As mentioned above, the rheological behaviour of pastes and mortars shows a strong time dependency. Such a thixotropic behaviour can be characterized with a shear rate jump. First a very low shear rate (rotational speed Ω = 1 rpm) is applied to the sample for 180 s. Afterwards the speed is increased instantaneously to 140 rpm and kept at this value for at least 300 s before returning to 1 rpm for further 180 s. During this sequence the resulting torque or shear stress are recorded.

The results prove that shearing the sample at a constant high shear rate leads to a strong drop of the torque reaching a constant value after a certain time. During the recovery phase i. e. the third segment at a low shear rate the sample rebuilds its structure, which leads to an increase of the torque over time. The degree of structural breakdown and the subsequent recovery is a measure for the thixotropy of the material. When performing such a measurement it must be kept in mind, that the shear history prior to the measurement itself strongly influences the result.

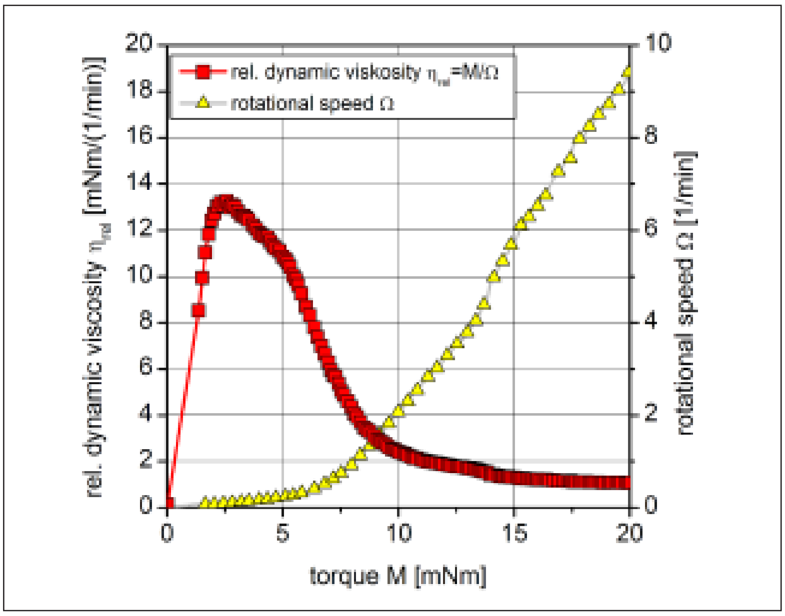

*Fig. 3: Determination of the relative yield stress $\tau_{0,rel}$ from the maximum of the relative viscosity curve, measured using a CS-ramp*

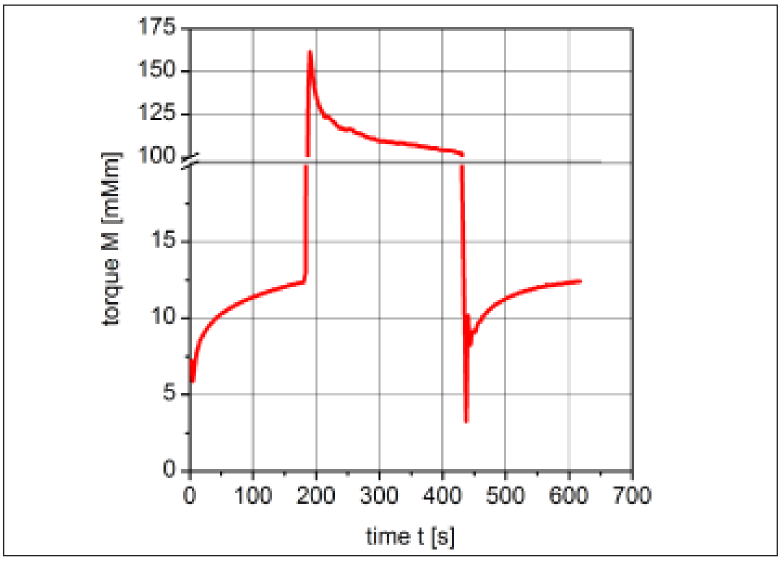

*Fig. 4: Structure recovery, breakdown and recovery during rest (4 rpm), constant shear (140 rpm) and rest (4 rpm) again.*

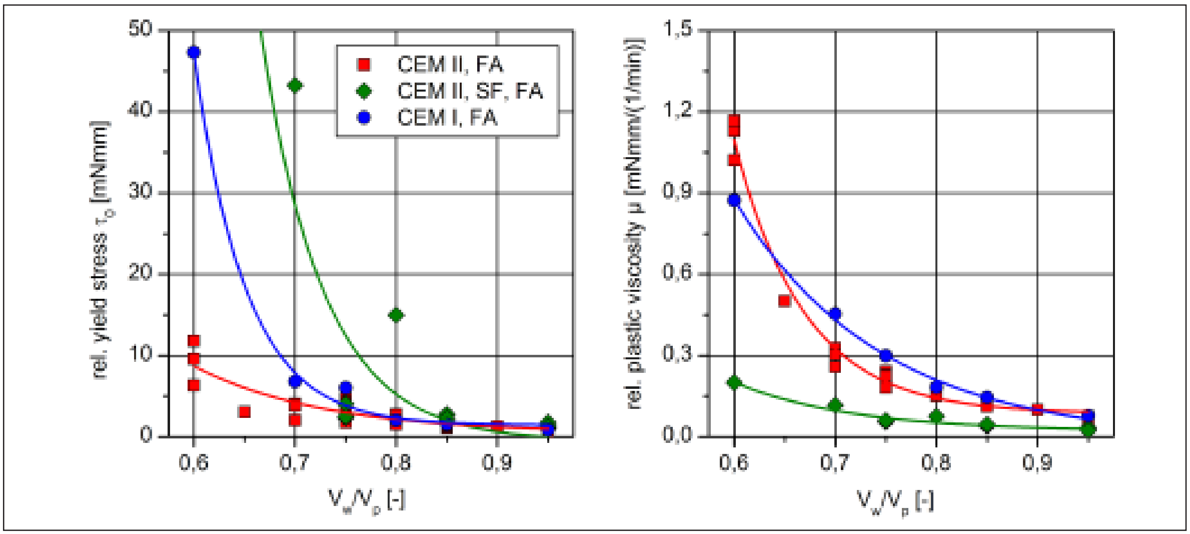

*Fig. 5: relative yield stress $\tau_0$ (left) and relative plastic viscosity µ (right) as a function of the mixture's $V_w/V_p$ -value of paste with CEM II/A-LL 32.5 R (CEM II), coal fly ash (FA), silica-fume (SF) or CEM I 32.5 R (CEM I)*

## Investigations of pastes and mortars for self-compacting lightweight concrete

All of the above named testing methods were applied during an extensive research program focussing on the development of self-compacting lightweight concrete conducted at the Institute of Concrete Structures and Building Materials at the University of Karlsruhe (TH), Germany. In this context a large number of different pastes – i. e. mixtures of cement, fly ash and as the case may be other materials and additives with a particle size below 0.125 mm – and mortars – mixtures with biggest particles of 2 mm – were investigated.

Self-compacting lightweight concretes are usually prepared using porous lightweight aggregates. Due to the increased pressure during pumping and depending on the porous structure of the aggregates a mixture of water and fines particles will be pressed into the grains. The speed and the degree of this absorption process influence the pumping behaviour of the concrete significantly. Since this absorption process is flow dominated, the above named investigations focussed on the question if and to what degree good pumping properties of the concrete can be guaranteed by adjusting the rheological properties of the paste matrix. First the volume ratio of water to powder, the so called $V_w/V_p$-ratio was varied. In a next step, various kinds of raw-materials and additives were used.

Fig. 5 shows the influence of the $V_w/V_p$-Value on the yield stress $\tau_0$ (left) and the plastic viscosity µ (right) of the pastes. Both parameters strongly increase with decreasing water content. In addition significant differences between pastes with different composition can be seen.

Based on these results, the absorption behaviour of various lightweight aggregates, which are used in the production of standard lightweight or self-compacting lightweight concrete, has been investigated under an isotropic pressure of 30 bar.

Within this project the authors could show that the total absorption and the absorption speed increase strongly with decreasing plastic viscosity of the pastes (Fig. 6). Based on this knowledge it was possible to develop lightweight concretes, which could be applied by pumping without any problems [1].

Another application for rheological measurements is the evaluation of an additive's effect on the flow behaviour of fresh concrete. As an example, Fig. 7 shows the influence of a stabilizing additive on the yield stress and the plastic viscosity of a paste (ref [2]).

## Summary

The quality of construction materials like e. g. concrete or mortar is significantly influenced by their properties at the fresh state. It is possible to adjust these properties by using organic or inorganic additives. For a systematic approach in research or development but also in quality control, the currently existing, mostly empirical test methods do not provide the relevant information. Modern rheometry on the other hand allows an in-depth characterization of the elastic and viscous properties of a freshly prepared building materials

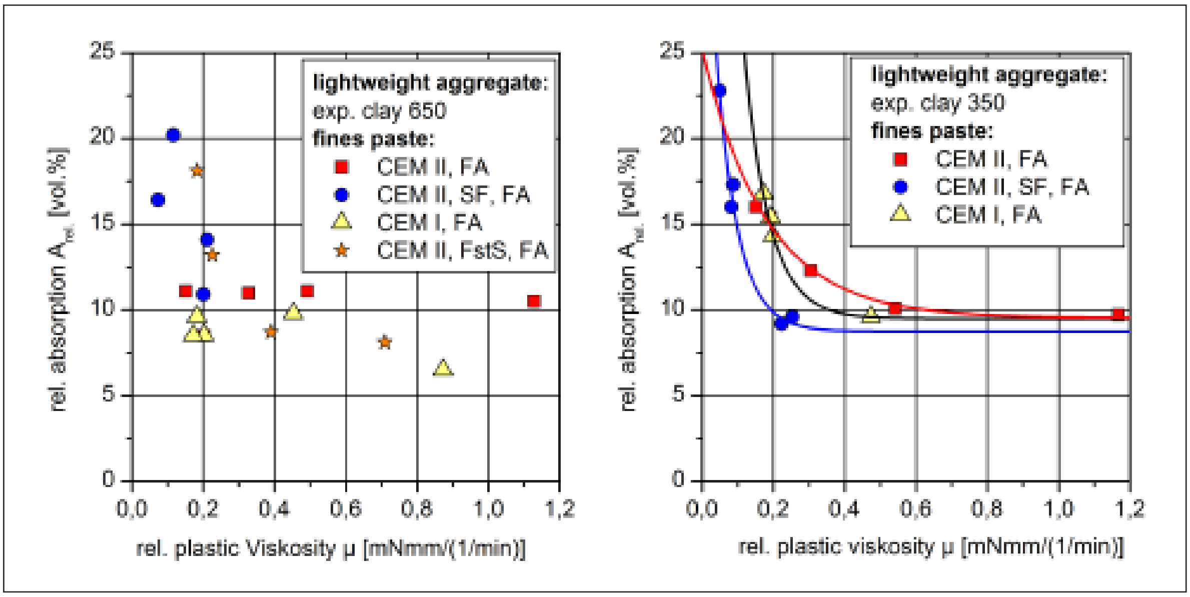

*Fig. 6: Influence of paste viscosity μ on the relative absorption $A_{rel.}$ of porous lightweight aggregates with a mean bulk density of 350 kg/m for the preparation of self-compacting lightweight concrete.*

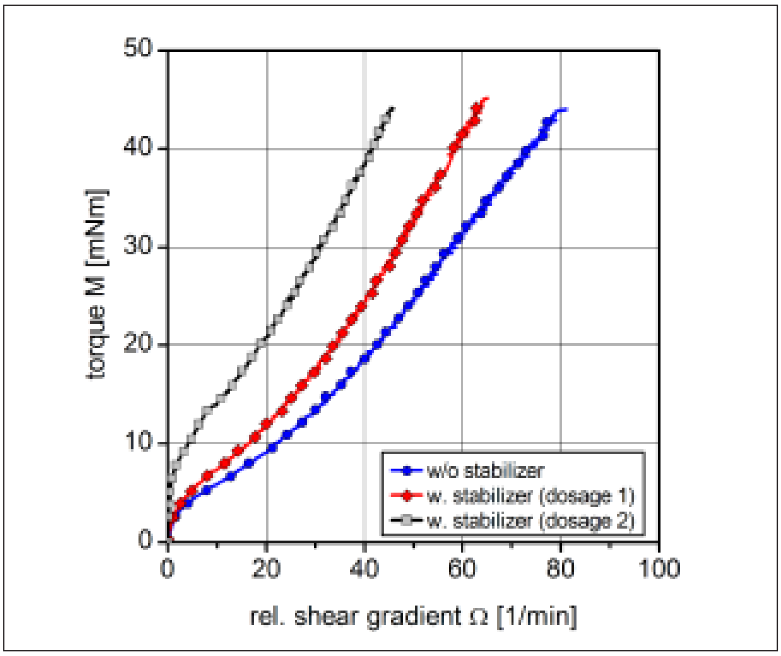

*Fig. 7: Flow curve of pastes with cement CEM I 42, 5 R, fly ash, superplastiziser and a $V_w/V_p$-value of 0.6 for different levels of stabilizer added.*

suspension. The methods introduced in this report represent only a small fraction of the capabilities of a modern rheometer. For further information concerning this topic please contact the authors (contact info see below).

## Literature

*[1] Müller, H. S., Haist, M.: Bauwerksertüchtigung mit selbstverdichtendem pumpbarem Leichtbeton. Abschlussbericht zum Forschungsprojekt im Auftrag des Bundesamtes für Bauwesen und Raumordnung, Institut für Massivbau und Baustoff technologie, Universität Karlsruhe (TH), 2004*

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