



## Yield stress of cement grouts



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### ABSTRACT

The rheology of cement grout is complex due to its thixotropic nature and the presence of a yield stress. Despite the importance of the yield stress for grouting design, no standard methods are yet available to determine the yield stress. Most common methods are based on using conventional rheometers, but the results are subjective due to the measurement techniques, applied shear history and hydration. In this work, measurement of the yield stress of cement grout was performed with different measurement techniques using a conventional rheometer. In addition, in-line measurements using an ultrasound based technique were made in order to visualize the flow profile and perform a direct measurement of the yield stress. Two ranges of yield stress, static and dynamic yield stress, were measured. These results should be used for design purposes depending on the prevailing shear rate. The ultrasound based Flow Viz industrial rheometer was found capable of performing direct in-line measurement of the yield stress and providing a detailed visualization of the velocity profile of cement grout.

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## 1. Introduction

In the construction industry, grouting is frequently used to alter or improve the natural properties of a geological formation, such as soil and rock. The aim is often to make the material stronger, stiffer or less permeable by pumping cement based suspensions, i.e. grouts, into voids or fractures of the formation. In tunneling and hydro-power dam foundations, so called permeation grouting is used to reduce the permeability and thereby the groundwater flow in the fractures of the rock mass. Permeation grouting implies that the grout is pumped into the formation from drilled boreholes by exerting a pressure that is higher than the prevailing groundwater pressure but not so high that it creates new fractures or displaces the rock mass (Stille et al., 2012). Micro-cement grouts, with a maximum particle size below 50  $\mu\text{m}$ , are commonly used in permeation grouting due to their relatively low environmental impact and cost as well as ease of preparation and use. In order to improve the grout's flow properties and penetration, especially at low water/cement ratios, admixtures such as plasticizers are frequently used. Typical water/cement ratios used in practice are in the range of 0.6–1.0.

Suspensions in general and cement grouts in particular have a complex rheology, and the rheological properties are difficult to measure and determine (Banfill, 2006; Håkansson et al., 1992). Interaction between the cement particles creates a network structure that leads to a non-linear relationship between shear stress vs shear rate, a thixotropic behavior, and introduces a yield stress that must be overcome in order to initiate flow (Håkansson et al., 1992). Due to chemical reactions when water is added, cement grouts also change their properties with time during hydration (Banfill, 2006). The rheological data for cement grouts used in practice often fit well to the Herschel-Bulkley model, but the linear Bingham model is often used because of its simplicity, especially with respect to theoretical development (Gustafson et al., 2013; Hässler, 1991; Lombardi, 1985). Test results for micro-cement grouts with low water cement ratios, e.g. 0.6, are usually non-linear while high water cement ratios, e.g. above 1.0, can be linear (Yang et al., 2011; Rosquoet et al., 2003).

Despite the importance of the yield stress as a design parameter for estimating the spread of grout, no standard method is available to determine this parameter. The most common technique is to measure the shear stress vs shear rate flow curve and extrapolate to the yield stress at zero shear rate. However, a very accurate measurement is required at low shear rates and this is often difficult to achieve due to slip at the wall of the measuring device (Liddel and Boger, 1996). The vane method offers superiority in

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avoiding a slip layer at the wall, provided that it is operated at a low rotating speed to eliminate the effect of secondary flow between the blades (Nguyen and Boger, 1983). Different measurement techniques have been compared for different model fluids and good agreement was found (Yoshimura, 1987). However, as cement grout is a thixotropic material, the yield stress depends on the shear history, and this generates different results depending on the measuring protocol, measurement geometry and other conditions (Barnes and Nguyen, 2001; Nguyen and Boger, 1992; Nguyen et al., 2006). Even though the above issues are well known, engineers often have problems defining the yield stress as a constant material property. To solve this issue, two yield stresses, static and dynamic yield stress, were introduced (Håkansson, 1993; James et al., 1987). The static yield stress was defined as the yield stress after the material has been at rest, and the dynamic yield stress was defined as the yield stress when the material was subjected to shear and was in a fully broken down state.

In recent work, researchers have used various methods to be able to visualize the actual velocity profiles (Jarny et al., 2005; Rahman et al., 2015). The advantage of using this type of measurement technique is that the yield stress can be determined directly from the measured plug radius instead of using curve fitting to rheological models. Recent grouting theories are based on the estimation of spread of grout and the yield stress is required as an input value for the calculations (Gustafson and Stille, 2005). However, the yield stress is used as a single numerical value without considering different phenomena, such as, the applied shear rate, thixotropy, and hydration. The purpose of this work is to measure the yield stress of cement grout, including the effect of thixotropy and hydration, by means of different techniques.

## 2. Yield stress

The yield stress of a grout will influence the relationship between pressure and flow and will set a maximum distance that the grout can penetrate into voids or fractures of the formation that is being grouted. As the grout enters into e.g. a fracture, the shear stress and the shear rate will decrease continuously and, at some point in time, the shear stress will be below the yield stress and the grout penetration will stop (Hässler, 1991). This situation, termed ‘cessation’ of flow, is not seen in many other industrial applications where the flow is often steady state and the yield stress will mainly influence the flow rate at a given pressure (Chatzimina et al., 2005). Needless to say, the grout can also stop because the particles, or flocs of particles, are blocking the fractures as they are larger in size than the fracture opening. However, this phenomenon is not the subject of this paper. Apart from implying a limitation to the penetration distance, the yield stress also plays an important role in the recent theoretical development of the real time grouting concept (RTGC) (Gustafson and Stille, 2005; Gustafson et al., 2013).

The yield stress notion is controversial. Some researchers claim that it is only a concept, that yield stress does not exist and only appears to be present due to limitations in the rheometers that are used (Barnes and Walters, 1985). Others claim that, even if yield stress does not exist, it makes sense to use an “apparent” yield stress for engineering applications, as long as the application generates the same range of shear rates that have been measured with the rheometers (Hartnett and Hu, 1989; Schurz, 1990). Researchers showed that a yield stress indeed exists and, when the fluid is thixotropic, the yield stress depends on a build-up (aging) and brake-down (shear rejuvenation) of the material during shearing. In order to properly evaluate rheometric data, it is necessary to distinguish between two types of yield stress fluids – thixotropic and non-thixotropic (simple). A thixotropic fluid is

one for which the rheological behavior is dependent on the shear history of the sample (Møller et al., 2009a). The rheological behavior is influenced by the competition between a build-up of some microstructure at rest and its break-down by flow (Fall et al., 2010). In this work, the yield stress concept is accepted as an engineering reality, demarking the transition between visco-elastic and fluid behavior. In addition, it is suggested here that different yield stress values should be used for grouting design purposes depending on the prevailing shear rate of grout.

## 3. Materials and methods

### 3.1. Materials

Cementa IC30 micro cement was used for the experimental work. IC30 has a particle distribution where more than 95% of the cement particles are less than 30  $\mu\text{m}$ , which has been shown to be the optimum particle size to avoid clogging inside rock fractures. Cementa IC30 is a sulphate resistant, chromate reduced and low alkaline injection cement, with a compact density of approximately 3100–3200  $\text{kg}/\text{m}^3$  and a bulk density of 800–1500  $\text{kg}/\text{m}^3$ . The chemical composition includes MgO (max 5% by weight),  $\text{SO}_3$  (max 3% by weight) and Cl (max 0.1% by weight).

All the tests in this work were based on a cement grout with a water to cement ratio of 0.7 without any additives or admixtures. This water cement ratio was chosen because of its typical usage in field applications. The mixing was done using a high speed dissolver DISPERMAT CV 3-PLUS (VMA GETZMANN GMBH) and rotational speed of 2500 rpm was applied during mixing for 4 min.

A commercial hair gel (Gatsby Water Gloss/Wet Look Soft) was used as a model fluid following earlier work (Møller et al., 2009b).

### 3.2. Off-line rheometry

#### 3.2.1. Equipment

A TA instrument AR 2000 EX rheometer was used to perform the rheological measurements. The AR 2000 EX rheometer can be operated in stress controlled, rate controlled and strain controlled mode. The concentric cylinder and vane geometries were used to perform the different tests. Controlled shear stress (CSS) was used to perform the stress ramp tests, and controlled shear rate (CSR) was used to measure the critical shear rate and rate ramp tests. Detail regarding the CSS and CSR measurement technique can be found in Stokes and Telford, (2004); Barnes and Bell, (2003); Møller et al., (2009a).

Standard DIN concentric cylinder geometry was used to perform stress ramp tests. The stator inner and rotor outer radius were 15 mm and 14.65 mm, respectively. The immersed height was 42.25 mm and the distance to the bottom of the stator was 5.912 mm. The cylinder surface was roughened with fine sand to eliminate slip at the inner cylinder wall and the stator was serrated.

The vane rotor geometry was used with wide gap to perform the stress ramp tests, constant rate tests and creep tests. The stator inner and rotor outer radius were 15 mm and 7 mm, respectively. The immersed height was 38 mm and the distance to the bottom of the stator was 4 mm.

A Brookfield DV-II Pro viscometer was also used to perform the stress relaxation test due to the advantage associated with the spring mechanism, which enables the spindle to rotate. This viscometer can only be operated in a shear rate controlled mode. The maximum applicable speed is 200 rpm, and the measurable viscosity range is 0.015–6000 Pa s. Two types of spindles, SC4-31 and SC4-34, were used for the tests.

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