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STIFTELSEN BERGTEKNISK FORSKNING
ROCK ENGINEERING RESEARCH FOUNDATION

CONTINUOUS MEASUREMENT OF THE RHEOLOGICAL PROPERTIES OF CEMENT GROUTS – DEMONSTRATOR FOR THE PUV+PD ULTRASOUND TECHNIQUE

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Cover photo: a field laboratory has been built inside a 10-foot container.

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CONTINUOUS MEASUREMENT OF THE RHEOLOGICAL PROPERTIES OF CEMENT GROUTS – DEMONSTRATOR FOR THE PUV+PD ULTRASOUND TECHNIQUE

Kontinuerlig mätning av cementbaserade injekteringsmedels reologiska egenskaper – Demonstrator för PUV+PD ultraljudsteknik

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PREFACE

BeFo has sponsored an extensive amount of research with focus on grouting of rock since the mid-1980s. Some of this research has been devoted to the rheological properties of cement-based grouts. The rationale has been that since the geometry of the conductive parts of a rock mass is complex and largely unknown in detail, it makes sense to have as much knowledge as possible of the rheology of the fluid that is going to be injected into the voids and fractures of the rock. This so at least the fluid properties can be taken out as an unknown in the complicated overall grouting process.

Grout properties are today measured at site with rather simple devices or in laboratories with some type of rheometer. Ultrasound has earlier been shown to be a feasible method of measuring the rheological properties of cement-based grouts. However, a feasibility study is often not enough to catch industry´s interest and therefore a demonstrator of the benefits is needed.

A reference group consisting of Tommy Ellison, Mats Holmberg, Rolf Christiansson, Patrik Vidstrand, Thomas Dalmalm, Tomas Bym, Staffan Hintze, Robert Melander, Johan Funehag and Per Tengborg have contributed with valuable insights and comments.The project was financed by BeFo and with in-kind contributions from Skanska and RISE.

Stockholm
Patrik Vidstrand

FÖRORD

BeFo har ända sedan 1980-talet finansierat en stor mängd forskning med fokus på injektering av berg och en del av denna forskning har behandlat de reologiska egenskaperna på främst cementbaserade injekteringsmedel. Grundfilosofin i detta arbete har varit att eftersom geometrin på sprickor i berg är mycket komplexa och omöjliga att känna till i detalj, så är det motiverat att känna till så mycket som möjligt om injekteringsmedlets egenskaper före injektering i berget. Detta så att åtminstone denna osäkerhet med medlets egenskaper kan reduceras i den övergripande komplexa injekteringsprocessen.

Injekteringsmedels egenskaper mäts idag med relativt primitiva metoder i fält och i laboratorier med någon form av reometer. Ultraljud har i tidigare forskning visat sig vara en möjlig metod för mätning av cementbaserade injekteringsmedels reologiska egenskaper. En genomförbarhetsstudie med positivt resultat räcker dock oftast inte för att fånga industrins intresse och därför behövs det även en demonstrator eller testbädd som kan påvisa fördelarna med metoden.

En referensgrupp bestående av Tommy Ellison, Mats Holmberg, Rolf Christiansson, Patrik Vidstrand, Thomas Dalmalm, Tomas Bym, Staffan Hintze, Robert Melander, Johan Funehag och Per Tengborg har bidragit med kommentarer och synpunkter.

Projektet har finansierats av BeFo och med värdefulla in-kind bidrag från Skanska och RISE.

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SUMMARY

Key words: Grouting, grout, cement, rheology, ultrasound

The rheological behavior of cement-based grouts is non-Newtonian and thixotropic and estimating these properties is not a simple task. Due to this behavior rheological measurements is not consistent when the grout is measured under different conditions and it is therefore vital to measure the rheological properties in time as close to the grouting operations as possible.

Current methods used to determine the rheological properties of grouts are primitive and measured off-line. Off-line measurements require a sample to be removed from the prevailing process and measured under non-identical conditions compared to the actual state of the fluid during processing. Furthermore, off-line measurements are time-consuming results and results cannot readily be compared. An ideal system must measure the “real-time” rheological properties in the process line and the prevailing conditions.

One technique of measuring rheological properties in-line is to simultaneously measure the pressure drop (PD) and the velocity profile in pipe flow, which is often referred to as the enhanced tube viscometer concept. The shear stress is determined from the pressure drop over a fixed distance of a pipe and the shear rate distribution is determined from the differentiation (slope) of the measured velocity profile.

Pulsed ultrasound velocimetry (PUV) is another method of obtaining an instantaneous velocity profile in fluid flow and used in combination with pressure differential (PD) measurements it forms the most suitable method for in-line flow characterization in industrial settings. The PUV+PD in-line rheometric method has been continuously optimized and evaluated for use in a wide range of complex industrial suspensions, however further investigation was required to evaluate the practical application of this technique with cement based grouts.

In order to demonstrate the effectiveness of the PUV-PD technique under field-like conditions, a transportable field laboratory was built inside a 10ft container. The laboratory contains a 1” pipe section for pumping grouts and a 6” pipe section for pumping concrete or shotcrete. The loops are fitted with various measurement devices including the Incipientus ILR in-line rheometer. Optimized ultrasound transducers and electronics were specially developed for this project with the aim to acquire improved velocity profiles in grout

mixtures. The field laboratory will also aid in continuous development and advancement of the PUV-PD method for future grouting applications.

Off-line rheological measurements were conducted with a conventional rheometer with water to cement ratios of 0.6, 0.7 and 0.8. This data was used as a baseline for the in-line measurements. The Bingham model is commonly used to describe the flow behavior of grouts, however the results from the off-line and in-line rheometer tests strongly indicate that grout rheology should be modeled using the Herschel-Bulkley model, a non-linear yield-power-law model. Grouts only exhibit Bingham behavior under very restricted conditions. Considering how error prone off-line grouting measurements are, a remarkable agreement was found between the in-line and off-line rheological measurements. In addition it was established that the optimized sensors and electronics enhanced the measured velocity profiles significantly compared to previous work.

It was demonstrated that the Incipientus ILR in-line rheometer allows true in-line flow visualization and can be used for measurement of rheological properties continuously and non-invasively during practical grouting operations. With in-line rheology it is possible to obtain more accurate results and in turn eliminate operator dependent errors.

SAMMANFATTNING

Nyckelord: Injektering, grout, cement, reologi, ultraljud

Baserat på många års erfarenhet från reologimätningar är det välkänt att cementbaserade injekteringsmedel har en komplex reologi. De är icke-Newtonska, tixotropa samt tidsberoende och på grund av detta är det ofta svårt att mäta och bestämma de reologiska egenskaperna. De mätningar som utförs i fält eller i laboratorium ger olika resultat eftersom de är utförda av olika personer, på olika sätt och under olika förhållanden. Det är därför viktigt att mäta egenskaperna så nära den verkliga processen som möjligt i fält.

Nuvarande metoder för bestämning av injekteringsmedels reologiska egenskaper är ofta primitiva och baserade på off-line mätningar, dvs utanför den rådande processen. För dessa mätningar krävs att ett prov tas bort från processen och mäts under andra förhållanden än de faktiska som vätskan är utsatt för. Off-line mätningar är dessutom tidskrävande och resultaten låter sig oftast inte jämföras eller återskapas. I ett idealiskt system måste man mäta de reologiska egenskaperna, i ”realtid”, under rådande processförhållanden.

En teknik för bestämning av de reologiska egenskaperna in-line, dvs i rådande process, är att samtidigt mäta tryckfallet och hastighetsprofilen vid rörströmning, dvs en modifiering av tub-viskometer konceptet. Skjuvspänningen bestäms av tryckfallet över en bestämd sträcka av röret och deformationshastighetens fördelning bestäms från lutningen (derivatan) på den uppmätta hastighetsprofilen.

Pulsed ultrasound velocimetry (PUV) är en metod för att erhålla en momentan ljudhastighetsprofil i ett vätskeflöde och används i kombination med tryckfallsmätningar (PD), vilket utgör den lämpligaste metoden för in-line flödes karakterisering i industriella processer. PUV + PD in-line metoden har kontinuerligt optimeras och utvärderats för användning i ett brett utbud av komplexa industriella suspensioner, emellertid krävs ytterligare utredning för att utvärdera den praktiska tillämpningen av denna teknik för cementbaserade produkter.

För att demonstrera effektiviteten av PUV+PD tekniken under fältliknande förhållanden, byggdes ett transportabelt laboratorium inuti en 10 fot container. Laboratoriet innehåller ett 1-tums rör för pumpning cementbaserade injekterimgsmedel och ett 6-tums rör för pumpning av betong och sprutbeteong. Rörslingorna är utrustade med olika mätningsenheter inklusive en Incipientus ILR in-line reometer. Optimerade ultraljudsgivare och elektronik utvecklades speciellt för detta projekt med syftet att förvärva förbättrade hastighetsprofiler för

cementbaserade injekteringsmedel. Fältlaboratoriet kommer också att användas för en kontinuerlig utveckling och provning av metoden PUV+PD i verkliga projekt.

Off-line reologiska mätningar genomfördes med en konventionell reometer med vattencementtalen 0,6, 0,7 och 0,8 och denna data användes som utgångspunkt för mätningar med in-line metoden. Bingham modellen används ofta för att beskriva reologin hos cementbaserade injekteringsmedel, men resultaten från både off-line och in-line testerna tyder starkt på att injekteringsbruks reologi bör modelleras med hjälp av Herschel-Bulkley-modellen, en icke-linjär yield-power-law modell. Injekteringsbruk uppvisar endast ett Bingham beteende under mycket begränsade förhållanden. Med tanke på hur fel benägna off-line mätningar är, har ett anmärkningsvärt samstämmigt resultat erhållits mellan off-line och in-line mätningarna. Det fastställdes dessutom att utvecklingen av optimerade sensorer och elektronik förbättrade uppmätta hastighetsprofilerna betydligt jämfört med tidigare arbete.

Det visades att Incipientus ILR in-line reometern tillåter en kontinuerlig visualisering av flödet och kan användas för mätning av de reologiska egenskaperna icke-invasivt (dvs. beröringsfritt) under praktiska förhållande. Med in-line reologi är det möjligt att få mer exakta resultat och en eliminering av operatörsberoende fel.

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1 INTRODUCTION

In many aspects, grouting is performed today in the same way and with the same equipment as it was done for more than 30 years ago. Equipment, such as pumps, mixers, agitators and cement feeders have not been developed much and the only difference today is that we now have “more of the same” (i.e. four pumps instead of two, two mixers and agitators instead of one). Measurement of pressure and flow are routinely and continuously performed, but the results are not often used as a proactive means for steering, control and modifications of the chosen initial grouting methodology. Nevertheless, important steps have been achieved during the last decade when it comes to theories for grout spread and stop criteria. The theories are based on the Bingham model for fluid flow and a detailed knowledge of the rheological properties, plastic viscosity and yield stress, are therefore necessary.

Conventional rheometer measurements of cement grouts, with commonly used water/cement ratios in practice, often result in a non-linear relationship between shear stress and shear rate. This implies that it is important to define the range of data to be used for the linear approximation using the Bingham model. Today there is no measuring standard nor any procedure available, despite the fact that both the viscosity and the yield stress are frequently specified in tender documents and technical specifications in current underground projects.
In addition, cement-based grouts are thixotropic, which means that the rheological properties are dependent on the stress and deformation history that the fluid has been subjected to. For example, different results will be achieved if the fluid has been at rest or if it has been continuously agitated (cf. thixotropic paint) before the measurement. It is therefore important to measure the properties continuously under realistic conditions, in order to detect any unwanted properties in time.

In the field, the measurement of rheological properties is today made by simple devices such as the Marsh cone and the yield-stick. The Marsh cone is a good and simple tool but the result constitutes a combination of the viscosity and the yield stress and it is not possible to distinguish one from the other. Moreover, the result is given in seconds and not in meaningful physical parameters and it is also questionable if the flow is fully developed considering the very short outlet nozzle of the cone. The yield-stick is a simple to use tool but the spread in results and the operator dependency creates uncertainty in the results.

In the laboratory, measurements are often performed with conventional concentric cylinder rheometers. It is a well-known fact that the rheological measurements of complex suspensions, such as cement grouts, implies a large spread in achieved results depending on the chosen rheometer, procedure and protocol as well as the operator. This situation is amplified for cement grouts since there is no measuring standard available today.

In summary, a problem today is that the rheological properties are either measured with primitive methods in the field or with methodology and operator dependent measurements in the laboratory. Both generating results that are difficult to use for the grouting practitioner.

The aim of this project is to address the above deficiencies by demonstrating the use of ultrasound as a superior method to measure the rheological properties of cement grouts in the field.

2 BACKGROUND

2.1 Grouting design concepts

Rheology is the science of flow and deformation of matter under the effect of an applied force and thus has a fundamental role in all theories related to fluid mechanics. To know a grout´s rheological properties are necessary to be able to estimate and predict a grouting result. The properties affect the relationship between pressure and flow as well as determines the maximum penetration length that can be achieved for a given fracture geometry (Hässler, 1991). Different requirements are stipulated for a grout depending on the stage of grouting that is referred to. The grout shall possess good penetration properties initially and it shall comply with the tightness and/or strength requirements when it is in place in the fractures, after the grouting is finalized.

The recent theory leap with respect to grouting in Sweden, known as "Real Time Grouting Control" (RTGC), is based on a characteristic (grouting) time, which in turn contains two rheological parameters from the Bingham model, viscosity and yield stress. The origin of this development for grouting under constant pressure, is an unpublished article by Gunnar Gustafson and Johan Claesson from 2005, which with the help of dimensionless parameters defines the governing factors for one-and two-dimensional flow in fractured rock. The same article was subsequently published in the Journal of Applied Mathematics (Gustafson et al., 2013) and a practical application of the above theory has been carried out by Gustafson and Stille (2005). In the latter article simplifications were made in order to obtain an analytical solution for two-dimensional flow and also different stop criteria and how they were dependent on the rheological parameters. In order to use this new theory, knowledge of the cement-based grouts rheological properties, as well as their change over time, are needed.

It is also widely known that variations in the rheological properties is a good indicator of other, wanted or unwanted, changes and can therefore provide means for continuous quality control.

2.2 Measurement of the rheological properties of cement grout - From Marsh cone to Ultrasound

a) Marsh Cone

Flow cones are widely used in various industries to primarily determine the flow properties of suspensions, like e.g. cement-based grouts. The Marsh cone, see Figure 1, originates from the oil industry where the interest is in the flow properties of drilling fluids ("drilling-muds"). The time it takes for a certain volume to flow out of the cone is measured, but this then contains a combined effect of the various rheological parameters, such as viscosity and yield stress. Therefore without further analysis, the different discharge times only provides a relative comparison of different fluids, density and concentration.

A closed form solution for flow from the cones was developed by Håkansson (1994) for Newtonian liquids and for a simplified, truncated, Bingham model. A nomogram was produced (Håkansson, 1993, 1994) and if one of the Bingham parameters, the viscosity or the yield stress, is known the other can be determined. The nomogram has recently been updated and is shown in Stille (2015) and Fransson, et al. (2016), see Figure 2.

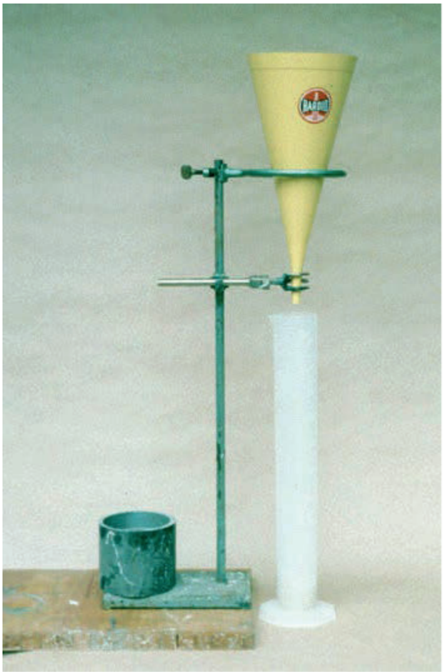
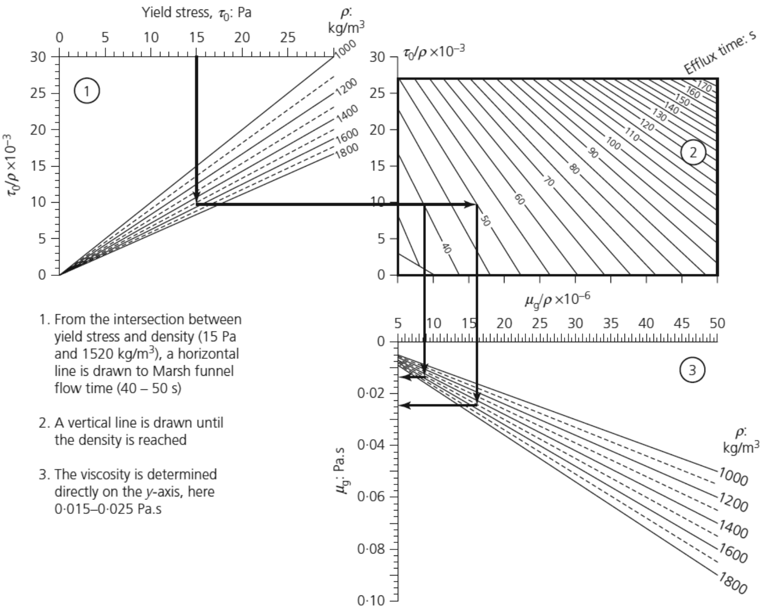


Figure 1. Marsh cone for field measurements

An advantage with the Marsh cone is that it is robust and easy to use in the field. It is also widely used in various industries and a good reference material is readily available.

A disadvantage of the Marsh cone is that it does not directly provide a measure of the rheological properties. Furthermore, it is not likely that there is fully developed flow in the short nozzle of the cone, which makes theoretical analysis difficult due to the end effects (inlet and outlet). The nozzle also has a smooth inside wall which makes the assumption of zero velocity at the wall questionable, due to slip. The discharge time is very sensitive to variations in pipe diameter and therefore it is very important that the nozzle is thoroughly cleaned between samples. Due to the above disadvantages, there is a need to develop the concept of a "cone" that is better suited for cement-based grouts.



1. From the intersection between yield stress and density (15 Pa and 1520 kg/m^3), a horizontal line is drawn to Marsh funnel flow time (40 – 50 s)

2. A vertical line is drawn until the density is reached

3. The viscosity is determined directly on the *y*-axis, here 0·015–0·025 Pa.s

Figure 2 Example of determination of viscosity using a known yield stress, density and Marsh Cone discharge time, from Håkansson (1993). In Fransson et al. (2016).

b) Plate-Cohesion Meter – PCM (Lombardi 1985)

With a PCM you measure the difference in the weight, of a plastic plate that has been dipped into a grout, and its weight in a dry state. If a fluid has a yield stress, a thin layer will stick to the plate and its weight will increase. From the difference in weight you can subsequently determine the yield stress.
An advantage of this method is that it is very simple and only requires a fluid container, a weight scale and a plate with a raw surface.

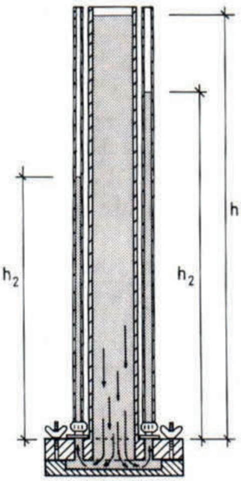
A disadvantage is that the scale is sensitive for field use and that there are not so many references despite the long time the idea has been around. Also Lombardi (1985) has developed a nomogram from which the yield stress or viscosity can be determined if one of the two parameters is known.

c) Raise-pipe (Håkansson, 1993)

The “Raise-Pipe”, see Figure 3, is a simple instrument to determine the yield stress of grouts in the field (Håkansson, 1993). The yield stress is determined by utilizing the fact that the flow ceases when the maximum shear stress, at the tube wall, equals the yield stress. The diameter of the pipe must be known as well as the grout´s density. The level inside the pipe will not rise as far as the level in the large filling pipe and by using the difference between the two levels, the yield stress can be determined by the formula shown in Figure 3.

An advantage of the Raise-Pipe is that it is easy to use and that the flow situation is similar to what is happening with grouting in the field, i.e., a flow that is high in the beginning and then ceases until the grout stops in the fracture. This is referred to as “ceasation” flow.

Disadvantages with the method are mainly related to the inside of the pipe and difficulties to clean the instrument. It is very difficult to find transparent pipes with a raw inside that is needed to minimize slip between the grout and the pipe wall. If the pipe is smooth on the inside, the level inside the pipe will rise further than what is dictated by its yield stress and the pipe diameter.



$$\tau_0 = \frac{\gamma R}{2}\left(\frac{h_1}{h_2} - 1\right)$$

Figure 3. Raise-pipe for field measurements of yield stress

d) Yield stick (Axelsson, 2005)

The “Yield-stick” (Axelsson, 2005), see Figure 4, is another simple instrument used to determine the yield stress of grouts in the field. The yield stress is determined by releasing a wooden stick into the grout. The stick has a weight at the bottom and the total weight must be known as well as the dimensions of the stick. Due to the fact that the grout has a yield stress, the stick will not sink to the bottom of the container, but stop before. By knowing the depth of penetration, the yield stress can be determined directly, see Figure 4.

An advantage of the Yield-stick is that it is easy to use and easy to clean. Disadvantages are associated with the dependency of the operator performing the test and the ability to obtain repetitive and representative results.

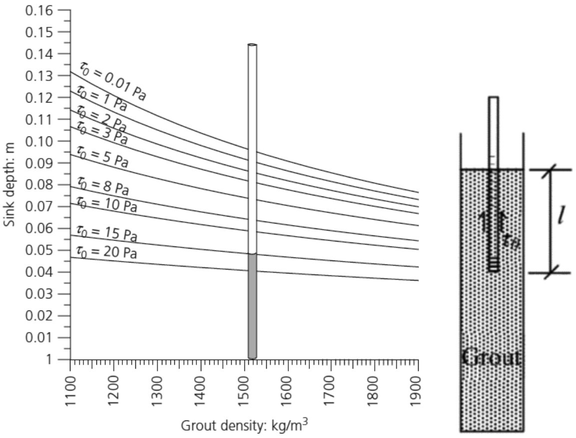


Figure 4. Yield-stick for field measurements of yield stress (Axelsson, 2005)

2.3 Measurement techniques

Measurements of the rheological properties of non-Newtonian fluids are not straightforward and complicated due to non-linear, thixotropic mechanical properties (Chabra and Richardson, 1999). The rheological properties can be measured on-site or in an off-site laboratory. Off-line measurements are most often performed by using a rheometer. In contrast, in-line measurements is performed in the industrial process in real-time, thus results are readily comparable to prevailing conditions. In-line measurements are usually non-invasive, i.e the measuring system has no contact with the fluid sample.

Definition of terms off-line, at-line, near-line, in-line and on-line
According to Roberts (2001), the terms mentioned above can be defined as:

- An off-line measurement is performed on a sample taken from the process. The sample is analyzed using an instrument located remote from the actual process e.g.in a laboratory. The instrument is thus not connected to the process line and the sample is discarded after the measurement.
- At-line or near-line measurements are performed on a sample removed from the process. The sample is analyzed in an instrument that is located near the actual process but not in physical contact with the material inside the actual process.
- In-line measurements are performed directly in the process line.
- On-line measurements are performed in a bypass loop from the main process line and may be returned to the main process line after measurement.

Already from these definitions, it is obvious that it is very desirable to be able to perform measurements directly in the process line i.e. in-line.

2.3.1 Off-line rheometry techniques

Off-line measurements are performed using different types of rheometers. Rotational rheometers are often used and the measurement geometry is chosen based on the type of suspensions. Off-line rheometry involve shearing a sample fluid in a selected geometry, from which the devices estimate the shear stress as a function of shear rate. This is referred to as the flow curve of the fluid. A direct or indirect approach can be used to estimate a flow curve. Indirect approach is referred as the extrapolation of the shear stress - shear rate data, assuming a suitable rheological model. In contrast, direct approach refers to the measurement of the rheological properties without using any assumptions of rheological models (Nguyen and Boger, 1983).

The most commonly used rheometer geometry is the concentric cylinder geometry. The advantage of this geometry is that it only requires a small sample volume. Measurements with the concentric cylinder geometry should be performed with care, since the wall slip phenomenon is inevitable when measuring a shear thinning thixotropic suspension (Barnes, 1995). The measurements can be performed in shear stress controlled (SSC) or shear rate controlled (SRC) mode. For thixotropic fluids, there is a range of shear rates below the yield stress, which are not reachable when measuring in a stress controlled mode (Moller et al. 2009). This is due to the fact that the suspension is partially sheared for an applied stress less than the yield stress and the change from visco-elastic solid to viscous behavior is abrupt.

2.3.2 Capillary or tube viscometers

Capillary or tube viscometers usually consists of a pipe where the investigated fluid is subjected to a force until it flows through a measuring section equipped with differential pressure sensors and a flow meter or a small (round) die, usually 1-2 mm in diameter. The shear rate at the capillary wall is calculated from the measured flow rate and pressure drop using the Rabinowitsch-Mooney equation. The pressure drop resulting from fully developed laminar flow through this section of known length is measured. The pressure drop is directly related to the shear stress at the wall. Alternatively, the force required to move the piston is monitored. From this data and the corresponding mass flow rate, the viscosity at a specific shear rate can be determined. Thus, the relation between shear stress and rate of shear is obtained indirectly from observations on the pressure gradient and volumetric flow rate in a straight pipe or capillary-tube. In these instruments, shear rate is not constant but varies from zero at the center of the pipe to a maximum at the wall. Consequently, interpretation of the result may not be obvious for non-Newtonian fluids. Capillary or tube viscometers can be of atmospheric or pressurized type and are very accurate at low viscosities and high shear rates. These instruments can also be used for in-line/on-line measurements, but mainly for Newtonian liquids. Some attempts have been made to use tube viscometers for non-Newtonian suspensions, but measurements must then be made in several pipes with different diameters and/or at different flow rates.

2.3.3 Rotational process viscometers and rheometers

A rotational viscometer or rheometer can also be employed as process viscometers/rheometers. They are usually installed in a vertical bypass loop and are thus considered as on-line, rather than in-line instruments. The measuring geometries are normally of the concentric cylinder or bob-in-cup type, but other geometries can also be used with these instruments on-line. A big drawback with these rheometers is that in order to convert the measured torque and rotational rate to viscosity, one assumes that the flow is laminar and that there is no secondary flow which occurs at high rotational rates with low-viscosity fluids. Since the shear rate is not uniform across the gap between the concentric cylinders, these instruments does not give a direct reading of the viscosity for non-Newtonian fluids. The Brookfield SST100 Process Rheometer has been installed on-line for some dilute suspensions, but problems with particle clogging; cleaning issues and maintenance problems have shown that it is an unsuccessful rheometer for most industrial applications.

2.3.4 Vibrational viscometers

A few commercial viscometers based on the vibrating element principle are available for in-line/on-line measurements. The working principle is that a probe is vibrating at known frequency and amplitude. The investigated fluid tends to damp the vibration in proportion to its density and viscosity. One can either measure the amplitude damping of the vibration at a constant frequency or the power required to maintain constant amplitude of vibration at a constant frequency. The instrument reading is thus a function of the product of density and viscosity in both measurement types. The viscometer is a simple device that can be placed in-line and is easy to clean. The measurement is however, dependent on the flow rate past the vibrating sphere and the obtained number is at best an oscillatory parameter and not an actual viscosity at a specific shear rate. It can thus be quite difficult to find a correlation between the oscillatory movement of the vibrating element and the actual shear rate. Since it is a single point measurement system it can only be applied to Newtonian fluids and it is therefore also difficult to correlate the obtained index numbers with off-line measurements.

2.3.5 Enhanced tube viscometers and rheometers

The optimum way to measure rheological properties in-line is to simultaneously measure the pressure drop and the velocity profile in pipe flow. This measurement methodology is often referred to as the enhanced tube viscometer concept. The shear stress is determined from the pressure drop over a fixed distance of pipe and the shear rate distribution is determined from a differentiation of the measured velocity profile. The concept was proposed already in 1980 but it has been difficult to find a technique capable of measuring a complete and instantaneous velocity profile under actual processing conditions.

Nuclear magnetic resonance imaging (NMRI or MRI) was tested in the 1990s by several research groups; see for example the review by Arola et al. (1997). However, there are several issues associated with MRI; expensive, slow, cannot provide accurate information in near wall region and that the method requires a non-metallic and non-magnetic test section, which are not be approved within most industries.

LDA or LDV (Laser Doppler Anemomentry) was invented by Yeh and Cummins in 1964. It is a non-intrusive measurement (optical) technique based on the Doppler effect (see Section 2.3.6). It is capable of measuring a velocity profile with high accuracy and often very high spatial resolution due to small measurement volumes, but it is limited to transparent fluids and e.g. Plexiglas test sections. Moreover, the sensor must be traversed to obtain a velocity profile since it can only measure the velocity at a single point. Vorwerk et al. (1994) described the method to combine LDA with a pressure difference (PD) technique to obtain rheological data in dilute shear-thinning polymer solutions. However, due to the limitations with the technique it has a very limited applicability in industrial applications.

2.3.6 The PUV+PD method for in-line rheometry

Pulsed Ultrasound Velocimetry (PUV) or Ultrasound Velocity Profiling (UVP) represents both a technique and a device for measuring an instantaneous velocity profile in liquid flow along the ultrasonic beam axis (Jensen 1996 and Takeda 1995). At every pulse repetition interval (PRI) an ultrasound burst, typically consisting of 3 to 10 sinusoidal cycles at f_t = 0.2 –10 MHz frequency, is transmitted into the fluid where it propagates with velocity, c. A particle moving at velocity v along the pipe with the flow generates an echo whose frequency, according to the Doppler effect, is affected by the shift:

$$f_D = 2f_t \frac{v}{c} \cos(\theta) \qquad \text{Eq. 1}$$

where θ is the angle between the directions of the moving particle and the ultrasound beam (Jensen 1996). Every PRI, the received echoes are sampled, demodulated in in-phase and quadrature components (I/Q) by multiplying the samples for, $\sin(2\pi f_t t)$, $\cos(2\pi f_t t)$functions, and low-pass filtered. The resulting complex samples are stored along the columns of a matrix. Adjacent columns collect data corresponding to successive PRI's. The sampling frequency is typically between 40 and 100 MHz and about 500 to 8000 samples per PRI are collected, depending on the pipe diameter. When the matrix holds enough data (e.g. 128 or 256 columns), the spectral analysis starts. Every row of the matrix, which stores the samples corresponding to the same depth in the pipe, is processed through windowing, Fast Fourier Transform (FFT), and power extraction. The resulting spectral matrix, when color-coded and displayed, shows an intuitive representation of the velocity profile of the fluid, shown in Figure 1, similar to that used in biomedical echography for investigating the blood velocity distribution in vessels (Ricci 2016). The background noise is reduced by applying a power threshold to the spectral matrix and, from each row of the de-noised data, the Doppler shift f_D at depth, d. The velocity profile is then calculated according to:

$$v = \frac{cf_D}{2f_t cos(\theta)} \qquad \text{Eq. 2}$$

In laminar pipe flow the shape of the velocity profile across the pipe diameter, depends on the rheological properties of the fluid. Newtonian fluids such as oil or water have a simple parabolic velocity profile (Figure 5, left) while complex fluids such as cement grouts develop different profile shapes. For example, shear thinning exhibits a flattened profile (Figure 5, middle) and fluids with a yield stress, such as cement grouts produce a plug flow (Figure 5, right).

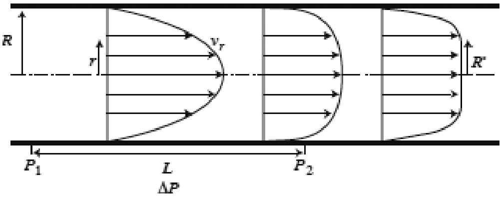


Figure 5. Three different pipe flow velocity profiles; From left to right: (1) parabolic flow profile of a Newtonian fluid (2) shear thinning fluid with flattened profile and (3) fluid with yield stress, resulting in a plug flow.

For simple Newtonian fluids, the rheology is often determined from the volumetric flowrate and the pressure difference over a fixed distance of pipe. This method is known as the tube viscometer concept and is capable of determining the shear viscosity at a single shear rate. The shear stress, τ, along the radial position r, which is zero at the centerline of a pipe with the radius R can be calculated with the pressure drop, ΔP measured over the distance L:

$$\tau(r) = \frac{\Delta P * r}{2 * L * R} \qquad \text{Eq. 3}$$

For non-Newtonian liquids, which constitute 95% of all industrial fluids, the shear viscosity must be determined as a function of shear rate. In tube viscometry, this means that the measurements must be performed in several different pipe diameters or at several different flow rates. The Pulsed Ultrasound Velocimetry (PUV) technique can be combined with pressure difference (PD) measurements to form the PUV+PD rheometric method, which stands out as the most suitable method for in-line flow characterization in industrial settings. This is possible since the measured velocity profile fully describes the shear rate distribution of the test fluid. In the PUV+PD method used in this work, the flow profile shape is used to derive the shear rate dependent viscosity at the different shear rates present in the fluid. The shear rate $\dot{\gamma}$ along the radius r is the derivative of the flow velocity, v, i.e.

$$\dot{\gamma}(r) = -\frac{dv}{dr} \qquad \text{Eq. 4}$$

Combining the shear stress determined from the measured pressure drop and the shear rate from the measured flow velocity profile it is possible to calculate the shear rate dependent viscosity $\dot{\eta}$ in real-time:

$$\dot{\eta}(r) = \frac{\tau(r)}{\dot{\gamma}(r)} \qquad \text{Eq. 5}$$

Some fluids, for example grouts, show a more solid like behavior under very low shear. A corresponding flow velocity profile is shown in Figure 5 (right). The yield stress, τ_{yield}, which is the minimum shear stress for the fluid to flow, can be determined from the plug radius R^* also measured with the flow velocity profile.

$$R^* = \frac{2 * L * \tau_{yield}}{\Delta P} \qquad \text{Eq. 6}$$

The PUV+PD method can thus determine the complete flow properties of the fluid (yield stress, flow index n and flow consistency coefficient K). Figure 6 shows the PUV+PD concept for in-line rheometry. The PUV+PD concept was first mentioned in a publication by Kowalewski (1980) and for the past two decades, the PUV+PD in-line rheometric method has been continuously optimized and evaluated for use in a wide range of complex industrial suspensions (Wiklund et al. 2007, Wiklund and Stading 2008, Windhab and Oriev 2003). The technique has been studied and developed by a number of research groups as the main tool for determining the rheometric parameters of complex industrial suspensions in-line. The main advantages with the PUV+PD method are that it allows non-invasive, continuous measurements of the velocity profile and the rheology of the investigated fluids.

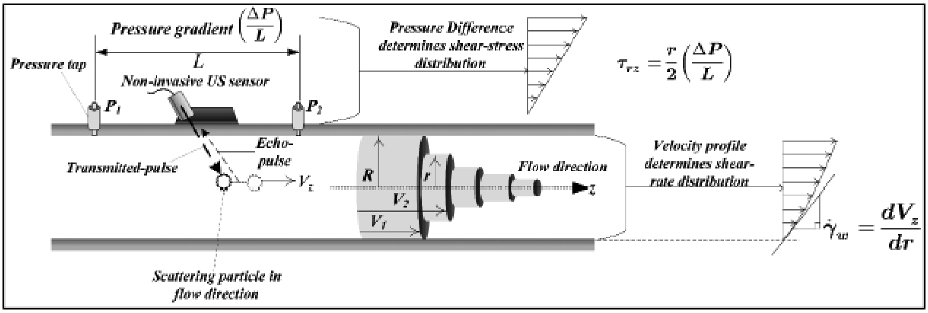


Figure 6. PUV+PD concept for in-line and instantaneous rheology measurements.

3 MATERIALS, EQUIPMENT AND METHODOLOGY

3.1 Materials and mixes

Cement based grouts are commonly used for grouting and they are non-Newtonian, thixotropic, yield stress fluids (suspensions). In this work, grout recipes were selected based on specifications for real current tunnel grouting projects.

The Swedish Road Transport Administration (Trafikverket) provided the specification shown in Table 1 for grout recipes for the Stockholm Bypass project (Förbifart Stockholm), to be used under different rock conditions.

Table 1. Required yield stress and viscosity of grout, specified by Trafikverket (Teknisk Beskrivning, FSE 403, Bergtunnlar Johannelund, 2015)

Mixture number	Penetrability criteria	Yield stress (Pa)	Viscosity (mPas)
1	b_{min}<45 μm , $b_{critical}$<75 μm	1-2	10-30
2	b_{min}<45 μm , $b_{critical}$<90 μm	2-6	10-50
3	b_{min}<90 μm , $b_{critical}$<140 μm	>8	>30

To arrive at the desired grout properties, a series of off-line rheological measurements were performed. Cementa IC 30 micro fine cement was used and a water to cement ratio of 0.6, 0.7 and 0.8 were tested. Sika i-Flow 1 was used as superplasticizer and Sika- iAcc 1 was used as an accelerator. Sika i-Flow 1 is a dispersion agent and prevents grout flocculation. Due to steric repulsion between particles during dispersion, the yield stress and viscosity of grout decreases.

In order to have a significant increase of the yield stress, a 40% solution of Sodium Silicate (water glass) was used to achieve a suitable composition for mix 3.

The following recipes, shown in Table 2, were used to test the rheological properties.

Table 2. Recipes used for the off-line rheological properties measurement of grout

Mixture No	w/c ratio	i-Flow 1 (%)	i-Acc 1 (%)
1	0.8	0,2	0,1
2	0.8	0,2	1
3	0.8	0,2	2
4	0.8	0,3	3
5	0.8	-	-
6	0.8	0,2	-
7	0.8		3
8	0.8	-	-
9	0,7	0,2	2
10	0,7	0,2	-
11	0,7	-	-
12	0,7	-	3
13	0,7	Sodium Silicate 1% (40% solution)	
14	0,7	Sodium Silicate 2% (40% solution)	
15	0.6	-	-
16	0.6	Sodium Silicate 1% (40% solution)	
17	0.6	Sodium Silicate 2% (40% solution)	

In-line tests were performed to demonstrate the real time measurement of rheological properties in the field laboratory (see below). The tests were performed for a thick (w/c 0.6) and thin (w/c 0.8) grout mix and additives were avoided. The recipes used for the in-line tests are shown in Table 3.

Table 3. Recipes used for the in-line rheological properties measurement of grout

Mix No	w/c ratio
1	0.8
2	0.6

3.2 Field laboratory

In order to demonstrate the use of the pulsed ultrasound fluids characterization technique under field-like conditions, a field laboratory has been built inside a 10 foot container, see Figure 7. Two flow loops are included inside the container as seen in Figure 9, one with a one inch and the other with a six inch pipe, with the inlet on one wall and the outlet on the opposite wall. The one inch pipe is for measuring the rheological properties of grouts and the six inch is for measuring pumped concrete. The two flow loops are fitted with Incipientus ILR in-line rheometer non-invasive sensor units for in-line fluids characterization.

Figure 7. Photos of container before (left) and after rebuild to mobile test laboratory.

3.2.1 Portable container

A standard 10 foot container was used for the mobile test laboratory, where the length, width and height are 2.8, 2.33 and 2.35 meters respectively. The container was heat insulated, which reduced the overall internal dimensions to 2.74, 2.27 and 2.29 meters. The dimensions of the container with and without insulation are depicted in Figure 8.

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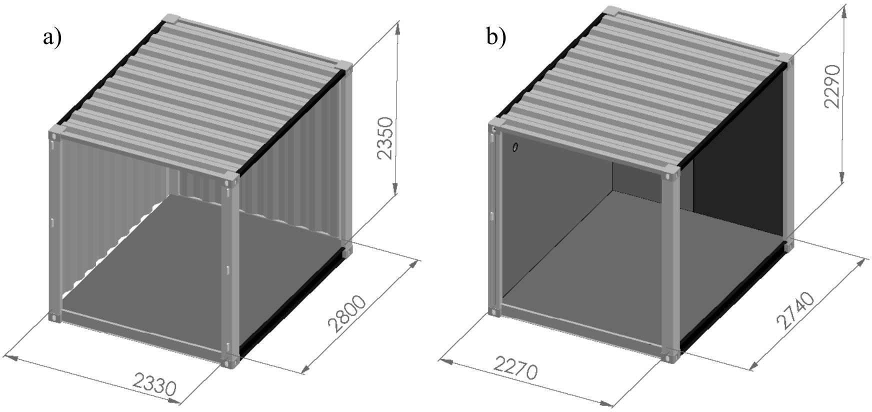

Figure 8. Container dimensions a) without insolation b) with insulation.

3.2.2 Experimental flow loop

3.2.3 Pipe loops

Two isolated horizontal pipe loops were installed along the length of the mobile test laboratory, as shown in Figure 9. The outer diameters (OD) of these two sections are 1” and 6”, respectively. The details and equipment used with the two pipe sections are discussed in more detail in the proceeding Sections.

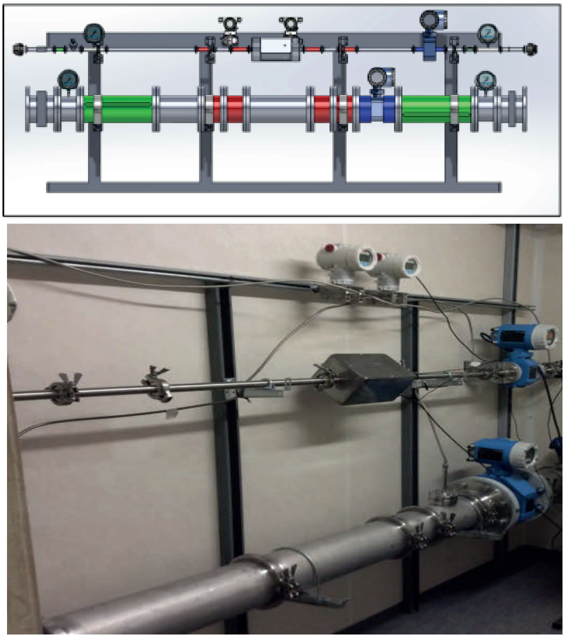

Figure 9. Stainless steel flow loops, 1" top and 6" bottom pipe sections

3.2.4 One inch pipe loop

This pipe loop consists of 316L stainless steel pipe sections interconnected to various measurement instrumentation, as shown in Figure 10. The intermittent sections were connected using tri-clamps. The pipe dimensions are given in Table 4 and the total length of this section is 3280 mm.

Table 4. 1" pipe dimensions

Inner Diameter (ID)	25,1 mm
Outer Diameter (OD)	22,1 mm
Wall thickness	1,65 mm

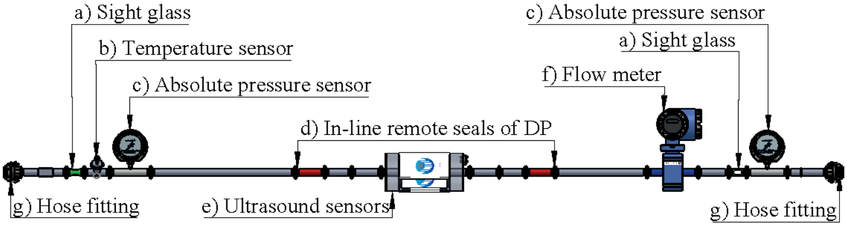

Figure 10. 1" pipe section

The measurement instrumentation is discussed in the subsequent sections.

e) Sight glass

Two sight glasses (Staitech, model HSG02), were installed at the inlet and outlet of the pipe loop. This allows the operator to visually see if the fluid is well mixed and fully developed in the pipe. The maximum pressure of these site glasses are 10 Bar and can only be installed when the expected pressure will not exceed this specification.

f) Temperature sensor

A surface mount PT100 temperature sensor (Pentronic, model 7907000), depicted in Figure 11, is used to acquire the temperature of the fluid in pipe. Since the sensor is mounted on the outside of the pipe it can be mounted on any location along the pipe. The temperature data is acquired by the Incipientus ILR in-line rheometer.

Figure 11. Pentronic PT100 temperature sensor, model 7907000

g) Absolute pressure sensor

The inlet and outlet of the pipe loop also contains two absolute pressure sensors (Wika, model UPT-21), represented in Figure 12, with a measurement range between 0 and 40 Bar. These point pressure sensors function as safety devices to insure that the pressure does not exceed the maximum pressure of the pipe.

Figure 12. Wika absolute pressure sensor, model UPT-20

a) Differential pressure sensor

The differential pressure sensor in Figure 13 (ABB 266MRT Sensor code N) is used for monitoring the pressure difference (measuring range is ±20 Bar) across the ultrasound sensors. This information is used to estimate the shear stress distribution across the pipe diameter. The remote seals for the 1” pipe differential pressure sensor make use of in-line technology, where the diaphragm is installed in the pipe wall. With in-line technology no disruption to the flow field is introduced, as there is no cavities and allows for more accurate measurements.

Figure 13. ABB 266MRT Sensor code N (20 Bar)

a) Ultrasound sensors

The latest generation non-invasive ultrasound transducers, depicted in Figure 14, are used to obtain velocity profiles in the 1” pipe. These transducers have a narrow focused beam, which allow more sound energy to travel through and in the pipe. This results in a higher acoustic penetration depth due to less signal attenuation. These transducers use a modified frequency, which was specially optimized for grouting applications. Since these transducers are installed non- invasively it does not disrupt the flow field of the liquid and do not add any extra pressure losses, which is important in order to obtain good grouting results.

Figure 14. Incipientus ILR in-line rheometer 1” standard non-invasive ultrasound transducer

h) Flow meter

An electronic flow meter shown in Figure 15 (Endress and Hauser Promag 55S) was installed on the pipe loop to monitor the flow rate. Monitoring the flow rate is important, because the flow rate is used as input to the Incipientus ILR in-line rheometer to optimize the velocity resolution.

Figure 15. Endress and Hauser Promag 55S flow meter

i) Hose fitting

House couplers as depicted in Figure 16 are used to connect the pump to the inlet and outlet of the pipe section in container.

Figure 16. Hose coupler

3.2.5 6 inch pipe loop

The 6 inch test loop was constructed from stainless steel. The pipe sections and instrumentation were connected using tri-clamps (PN30), as shown in Figure 17, however the flow meter was connected with flanges using the EN10921-1 (DIN150,PN16) standard. The maximum pressure of this loop is approximately 16 Bar. The pipe dimensions are given in Table 5 and the total length of this section is 3280 mm.

Table 5. 6" pipe dimensions

Inner Diameter (ID)	146.86 mm
Outer Diameter (OD)	152.4 mm
Wall thickness	2.77 mm

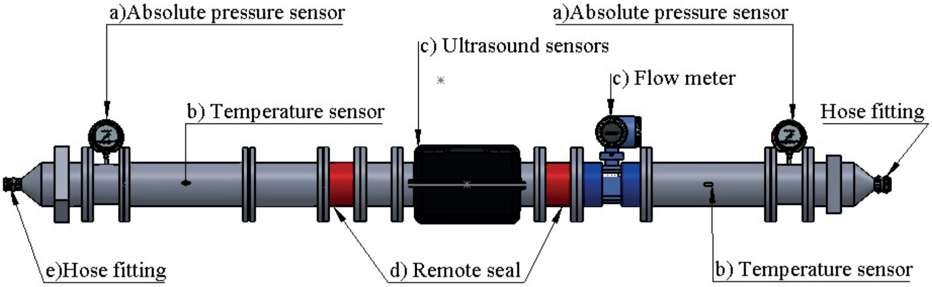

Figure 17. 6" pipe section

The measurement instrumentation is discussed in the subsequent sections.

a) Absolute pressure sensor

Absolute pressure sensor (Wika, model UPT-20) were installed at the inlet and outlet of the 6" pipe section to ensure pressure in loop does not exceed safe working limits.

b) Temperature sensor

The same model PT100 temperature sensor (Pentronic, model 7907000) used on the 1” pipe is used to obtain the temperature in the 6” pipe. The Incipientus ILR in-line rheometer RheoFlow system reads and stores the temperature data.

c) Ultrasound sensors

New low frequency transducers, in Figure 18 were developed and optimized for grouting applications in 6” pipes. These transducers are also installed non-invasively onto the pipe. The low frequency and narrow ultrasound beam allows for a very high penetration depth.

Figure 18. Incipientus ILR in-line rheometer 6” non-invasive ultrasound transducer

d) Flow meter

The flow meter is the same make and model as the 1” pipe flow meter.

e) Differential pressure sensor

The differential pressure sensor (ABB 266MRT Sensor code L) used with the 6” pipe does not use in-line technology and the measuring range is ±2.5 Bar. In-line remote seals are not available for this size pipe; however the diaphragm was installed close to the pipe wall to minimize the effect of the cavity on the flow field.

f) Hose fitting

Hose couplers where installed on reducers at the inlet and outlet of the 6“ pipe.

3.2.6 Container furniture and equipment

The portable rig was equipped with an air condition unit, work bench, cupboard, first aid kit, power distribution board and Incipientus ILR in-line rheometer. Some of the equipment is discussed in the following sections.

3.2.7 Power distribution board

The Power distribution board, in Figure 19 receives power from an external alternating current (AC) 230 Volt -50 Hertz power source. The AC power is distributed in the container via circuit breakers to wall sockets for electrical safety. The flow meters and Incipientus ILR in-line rheometer receives power from these wall sockets. The distribution board also contains a 24 Volt direct current (DC) power supply, which is used to power the absolute pressure sensors. The DP transmitters are powered by the Incipientus ILR in-line rheometer.

Figure 19. Power distribution board

3.3 Incipientus ILR in-line industrial rheometer

Incipientus ILR in-line rheometer is a complete commercially available and patented in-line fluids characterization system based on pulsed ultrasound velocimetry in combination with pressure difference measurements. The instrument measures rheological properties non-invasively through industrial grade stainless steel pipes and outputs complete rheograms and the yield stress. Unlike other commercially available process viscometers, the instrument is designed specifically for non-Newtonian industrial fluids, such as cement-based grouts. The PUV+PD based in-line fluids characterization system, is the only commercial and patented in-line rheometric system that has been developed for industrial implementation and that meets the industrial requirement. International Patent has been granted in USA, EU, South Grouting 2017, Hawaii Topic: Real-Time Monitoring – 6 – Rev. 12/2016 Africa and Japan. The development is described in detail in a number of publications (Wiklund et al. 2014, Håkansson and Wiklund 2016, Wiklund et al. 2016a, Kotzé et al. 2016, Shamu et al. 2016) and an overview of the system and its successful application in a highly concentrated suspension is demonstrated in Håkansson et al. 2016) and is this work. The instrument consists of an operator's panel and a remote sensor unit and it is shown in Figure 20.

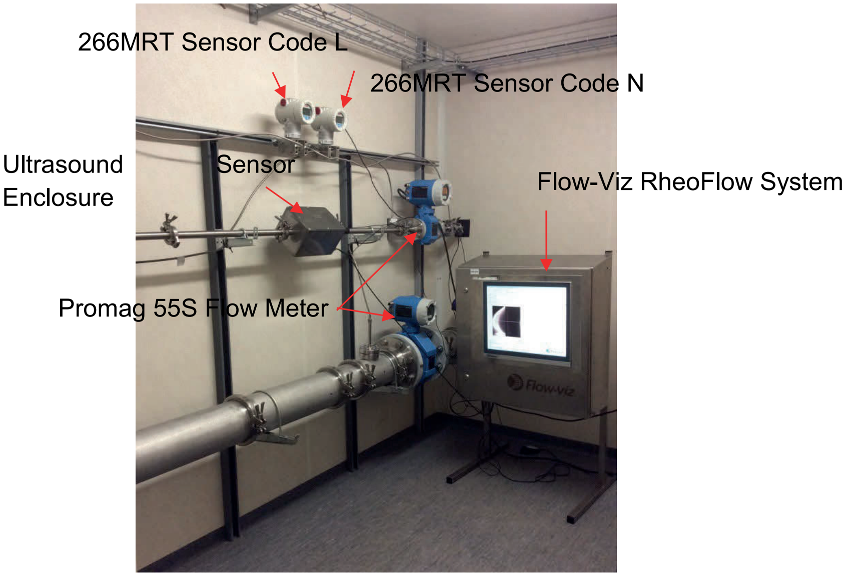

Figure 20. Incipientus ILR in-line rheometer.

The Operators' panel, shown in Figure 20 (right), houses all the electronics necessary for processing and conditioning the data and graphical user interface software that displays the acquired data on a touch screen monitor. The software running is used to set the working parameters and can display the raw echo data, the demodulated data, the spectral matrices or the final normalized frequency profiles as well as rheograms and rheological parameters in real time.

3.4 In-line measurement of the rheological properties of cement grout

The combination of measured velocity profiles and pressure difference data is used to determine shear viscosities and rheological model parameters. The developed PUV+PD methodology is based on a non-model signal processing approach where the shear rate vector is obtained from direct differentiation of the measured velocity profile. The shear stress vector is determined from a pressure drop measurement over a fixed distance of pipe. Specific measured parameters set by the user can be linked to an output analog signal and fed back in order to control and optimize a specific process. For more details about the PUV+PD methodology, see Wiklund et al. 2012, 2014, 2016a and 2016b.

3.5 Optimization of in-line sensor technology for cement grout

The Incipientus ILR in-line rheometer non-invasive sensor technology was first introduced and tested with cement grouts in a PhD project in 2012. It was demonstrated that the new sensors could be used to successfully measure radial velocity profiles in cement grouts with water cement ratios ranging from 0.6-1.0. However, data obtained in the near wall pipe region was found to have a wider velocity spread compared to data obtained closer to the pipe center. The penetration depth was found to be acceptable for determining grout rheology but the conclusion was that it could probably be improved by optimization of the acoustic beam; see Rahman (2015) and Shamu (2016) for more details.

In this work, acoustic characterization of acoustic beam propagation through high-grade stainless steel pipes were made in order to modify the sensor design for improved pulsed ultrasound velocimetry measurements in complex industrial fluids, such as drilling muds and grouts. A specially designed Incipientus ILR in-line rheometer XYZ-mechanical scanning system was used for the acoustic beam characterization and the setup is shown in Figure 21 below.

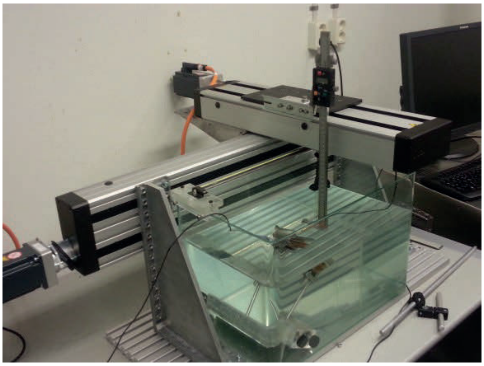

Figure 21. Incipientus ILR in-line rheometer XYZ-scanning system used for the acoustic beam characterization

The following equipment was used for the acoustic characterization tests in addition to the XYZ-mechanical scanner; 1 mm needle hydrophone, 50 Ohms, NI USB 5133 oscilloscope (100MHz sampling rate), Ultratek and V3 pulser-receiver electronics and the emission voltage for all tests was 100V and the emission pulse length: 500-784 ns for 0.7-3MHz. Tap water at room temperature (20oC) was used as continuous medium and the clamp-on sensors were installed on 1”, 1.5” and 2” pipes that were cut in half. The sensor set-up used for the acoustic beam characterization is shown in Figure 22 below. See results section for more details about the optimization of the non-invasive sensor design.

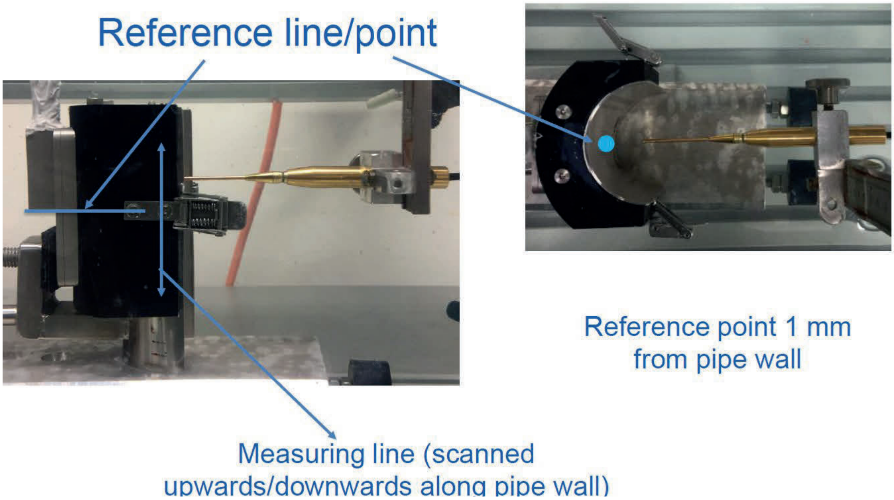

Figure 22. Incipientus ILR in-line rheometer sensor set-up used for the acoustic beam characterization

3.6 Conventional off-line rheometry

3.6.1 Equipment and measuring

A TA instrument AR 2000 EX concentric cylinder rheometer was used for the measurements. The rheometer was operated in a controlled shear rate (CSR) mode and a DIN type (DIN 53019-1:2008) geometry of the inner rotating cylinder (rotor) was used to perform rate ramp tests (i.e. increasing and decreasing shear rate). The radius of the rotor was 13,96 mm and the inner radius of the stator (cup) was 15 mm, creating a gap of 1,04 mm. The immersed height was 42.25 mm and the distance between the conical cylinder end and the bottom of the cup was 5.92 mm. The surface of the cylinder was smooth but the wall of the cup was serrated (grooved). The measured radius or diameter of the cup is taken from the top of the serrated cup asperities.

Controlled shear rate (CSR) ramp tests were performed to measure the rheological properties. The rotor was immersed in to the sample and rotated at an applied shear rate range from 0.1 s-1 to 300 s-1, first increasing (up-curve) and subsequently decreasing (down-curve).

3.6.2 Determination of the yield stress and viscosity

The Bingham model linear curve fit was used to determine the yield stress and viscosity of the grouts, by a linear curve fit to produced data over a certain range of shear rate. The Bingham model was chosen due to its simplicity and acceptance in the grouting community. In addition, it was the model specified in the tender documents for the Stockholm Bypass project (Förbifart Stockholm).

It is a well-known fact that the real yield stress is over-estimated when the Bingham model is used, depending on the range of the shear rate (Håkansson, 1993). In order to minimize this error, it is appropriate to use the shear rate range which will be prevailing during the spread of grout in the rock fractures.

The estimation of the shear rate was performed based on the nomogram introduced by Rahman (2015), and shown in Figure 23. The nomogram was developed based on the Real Time Grouting Control (RTGC) theory.

To estimate the shear rate, the required input parameters are the pressure (ΔP), yield stress (τ_o), viscosity (μ_B) and aperture (B). The required pressure depending on the overburden rock is specified by the Trafikverket, for the Stockholm Bypass project and shown in Table 6.

Table 6. Specification for grouting pressure (Teknisk Beskrivning, FSE 403, Bergtunnlar Johannelund, 2015)

Over burden rock (m)	Pressure (MPa)
<5	0,5
5-15	1,0
15-50	2,5
50-75	3,5

The grouting time is specified to be 15 minutes for mix 1 and depending on the input parameters, the relative grouting time (t_D) can be determined. Using the nomogram, the dimensionless shear rate for the corresponding (I_D) is achieved, as shown in Figure 23. To determine the actual shear rate, the dimensionless shear rate needs to be multiplied by the corresponding ${}^{\tau_o}/_{\mu_B}$ factor.

Following the above procedure the shear rate was estimated for the ultimate conditions, i.e., highest and the lowest pressure, rheological properties etc. as shown in Table 7.

Table 7. Estimated shear rate for different parameters

Yield stress (Pa)	Viscosity (mPas)	Pressure (MPa)	$\frac{\tau_o}{\mu_B}$ (s^{-1})	t_0 (s)	Shear rate (s^{-1})
2	10	3,5	200	52000	~500
2	10	0,5	200	7500	~100
6	50	3,5	120	30000	~200
6	50	0,5	120	4100	~90

The estimation of the shear rate is performed in order to get a general idea of the actual shear rate, following the provided specification. To estimate the shear rate for a specific case, certain input parameters, e.g., pressure, aperture, rheological properties have to be introduced. In Table 7, the shear rate range is between 100 s^{-1} and 500 s^{-1} for mixture 1 and between 90 s^{-1} and 200 s^{-1} for mixture 3. The shear rate 500 s^{-1} is the highest, estimated for the smallest value of the rheological properties and maximum pressure. Therefore, the majority of the shear rate range, which would take place in practice, is expected to be less than that.

Based on the above approach, the Bingham model fitting was performed over a shear rate range of 0 to 200 s^{-1}.

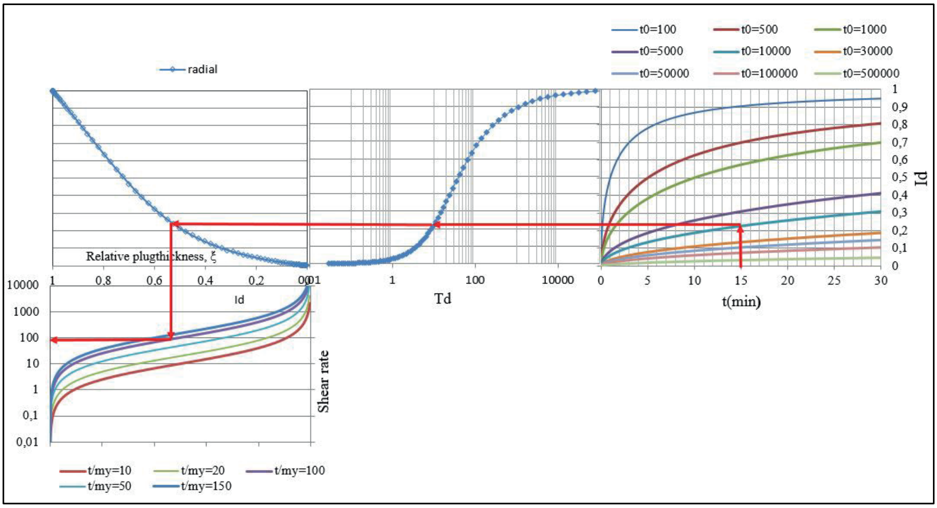

Figure 23. Nomogram for the estimation of the shear rate

3.6.3 Applied shear history

Due to the thixotropic nature of cement grouts, the rheological properties are shear history dependent. Therefore, a similar shear history was applied to all the samples, tests were performed for a duration of 30 minutes and shear rates were applied to get a range covering 200 s^{-1}, as discussed above. The shear history and measurement protocol which was applied to all the tests are shown in Figure 24.

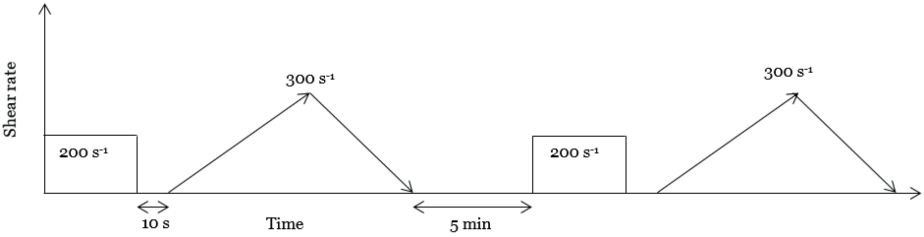

Figure 24. Measurement protocol for the determination of the rheological properties

The procedure can be summarized as follows:

- A pre-shearing of 200 s^{-1} for 30 s was applied after the grout was placed into the cup, prior to the measurements. This was applied in order to avoid any buildup between the cement particles and to achieve a reproducible result in all samples.
- A resting period of 10 s was followed after the pre-shearing to avoid any secondary motion effect during the measurement, i.e. to start from a stand still.
- The shear rate was increased from zero to a 300 s^{-1} for the up curve and subsequently decreased to zero, in the down curve. The corresponding torque was measured and the shear stress was determined.
- A resting period of 5 minutes was applied in order to achieve a prolonged test to see any early effects of cement hydration, i.e. changes with time.
- The above steps were repeated until reaching a total test period of 30 minutes.

4 RESULTS

4.1 Real time measurement of the rheological properties in field laboratory

The initial plan was to use a commercial grouting rig for field use to prepare the grout mixtures and to circulate the grouts in a closed circulation system. A commercial grouting rig is shown in Figure 25.

Figure 25. Commercial grouting rig for field use.

The commercial grouting rig for field use however require a large volume of grout and it was therefore decided to use a smaller grouting pump and mixer for the feasibility test. The small grouting pump and mixer used in this work is shown in Figure 26. A closed circulation system was assembled at Skanska Maskin in Upplands Väsby consisting of a smaller grouting pump and mixer, 1” black rubber hoses, commonly used for grouting operations in the field and the straight 1” stainless steel measuring section, equipped with the Incipientus ILR in-line rheometer including non-invasive sensors mounted inside the container. A ball valve was installed after the container in order to produce a back pressure.

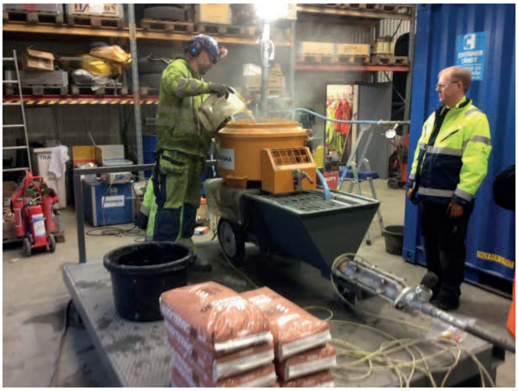

Figure 26. Portable commercial grouting pump and mixer for field use.

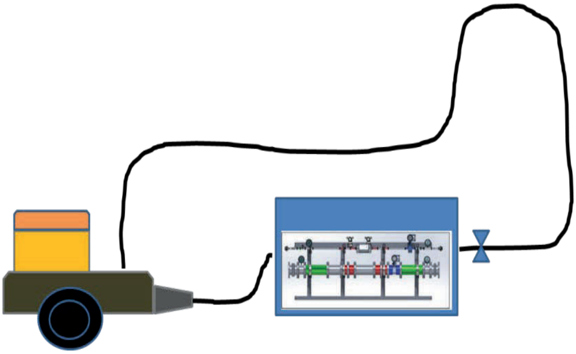

Figure 27. Closed grouting circulation system used for the tests including grouting pump and mixer, portable field laboratory with Incipientus ILR in-line rheometer instrumentation and 1" hoses.

4.1 Optimization of non-invasive in-line sensors for grout

From previous work on grouts we concluded that the non-invasive sensor design could be improved for grouts. The main problems with the existing design were that the data in the near wall region was noisy and the penetration depth was lower than expected. Acoustic characterization tests were therefore made using the standard Incipientus ILR in-line rheometer sensors and the acoustic map is shown in Figure 28 below. Our results showed that the focal point is located at a non-ideal position; it is shifted away from the liquid-wall interface, see area inside the green circle in the Figure 28. Moreover, the focal zone is too short, thus resulting in too low energy level, see area indicated by the purple line in the Figure 28.

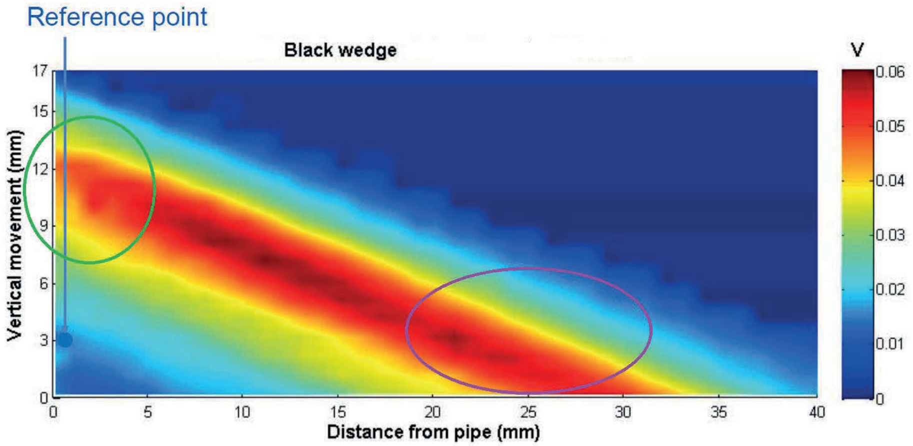

Figure 28. Acoustic characterization of standard Incipientus ILR in-line rheometer sensor.

A new sensor design was therefore proposed in order to overcome the problems highlighted in the text above. The beam focusing was improved in order to move the focal point closer to the pipe wall and to extend the focal zone so that it covers the pipe diameter. An acoustic characterization test result using the improved Incipientus ILR in-line rheometer sensors is shown in Figure 29 below.

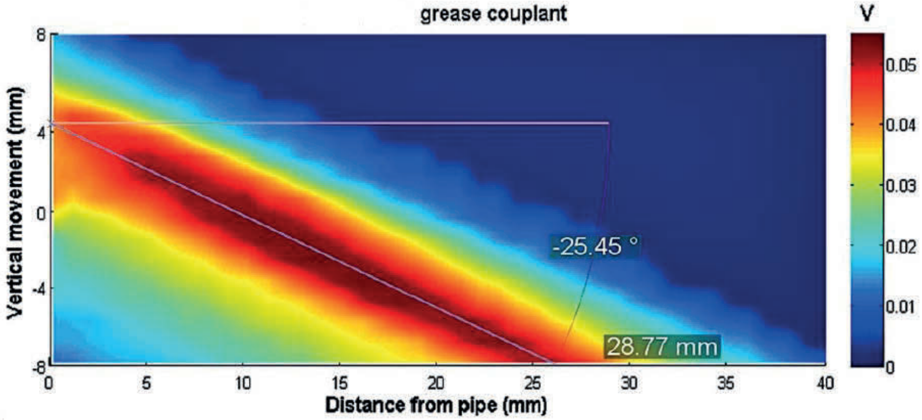

Figure 29. Acoustic characterization of Incipientus ILR in-line rheometer sensor designed for grouts.

This new design configuration results in a narrower beam with higher energy and an extended focal zone. Satisfactory results were obtained when this sensor was used for in-line measurements of velocity profiles and rheology of grouts. The new and improved sensor design used in this work is shown in Figure 30 below.

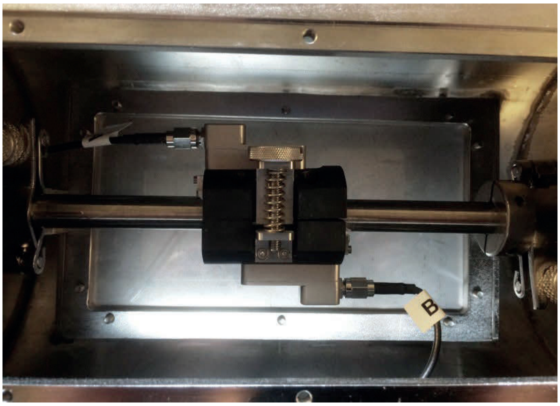

Figure 30. New Incipientus ILR in-line rheometer sensor (one pair) designed for grouts.

However, even with the new improved beam focusing and updated sensor clamp design it was found difficult to find an ideal coupling material to be used between the sensor and the pipe so that optimum acoustic coupling is ensured for grouts. This is because grouts have complex acoustic properties and it results in the coupling material shifting the start of the focal zone a few millimeters away from the pipe wall, even if the beam focusing is correct. The most optimized coupling configuration for pipe tests with grouts was seen to be that which incorporates a semi-solid coupling at the wedge-to-pipe interface. Improvements for next revision include higher energy output and improved focusing for extended (longer) focal zone, but better acoustic coupling is required for placing the focal point directly at the liquid-wall interface and minimizing energy loss.

4.2 In-line measurement of velocity profiles

4.2.1 Doppler spectra of w/c = 0.8

Figure 31 shows the measured Doppler spectra of a grout with w/c = 0.8 obtained at a flow rate of 3.3 L/min, which was the lowest flow rate that could be obtained with the grouting rig used. There is a sharp velocity gradient at the wall, followed by a characteristic smooth curvature, which is characteristic for shear-thinning fluids. A pronounced plug region is observed in the center of the pipe thus indicating a yield stress. The Doppler spectra only shows limited spectral broadening, which indicates that the grout suspension was well mixed and that the flow rate was constant at around 3.3 L/min and that it therefore gives an accurate "picture" of the velocity distribution in the pipe.

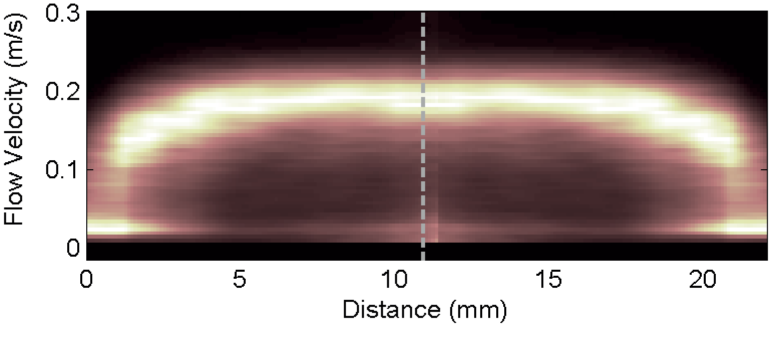

Figure 31. Spectral image of a grout with w/c = 0.8 obtained at a flow rate of 3 L/min.

Figure 32 shows the corresponding measured Doppler spectra of a grout with w/c = 0.8 obtained at a flow rate of 4.4 L/min. This measured spectra is very similar to the one shown in Figure 31 but the velocity gradient shows more curvature in the shearing region, which is characteristic for shear-thinning fluids. The plug region is observed in the centre of the pipe thus indicating a yield stress. It is however important to point out that the yield stress is linked to the pressure drop and the plug radius. A smaller measured plug does thus not necessarily mean a lower yield stress. The Doppler spectra is less noisy in the near wall region compared to at the lower flow rate. This indicates that the grout flow is more homogenous and better mixed when the flow rate is increased.

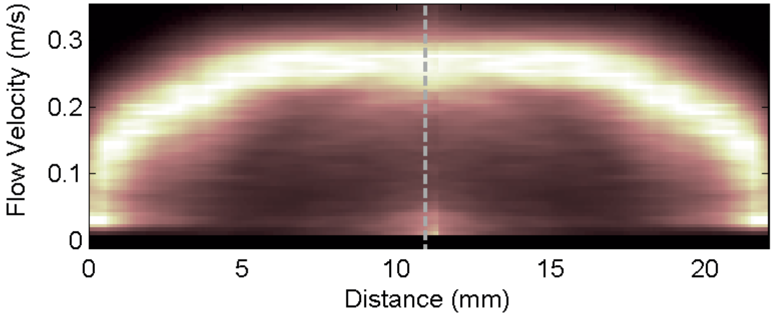

Figure 32. Spectral image of a grout with w/c = 0.8 obtained at a flow rate of 4.4 L/min.

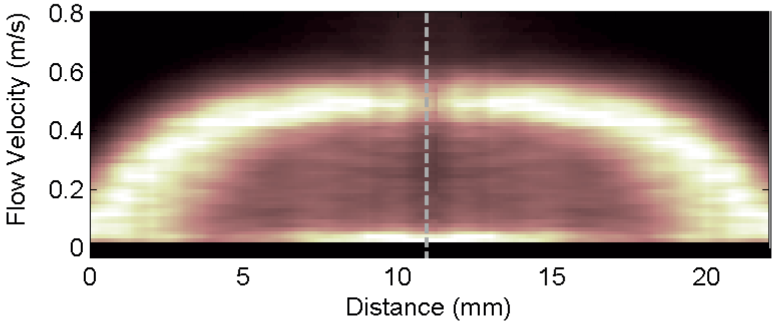

Figure 33. Spectral image of a grout with w/c = 0.8 obtained at a flow rate of 7 L/min.

Figure 33 shows the corresponding measured Doppler spectra of a grout with w/c = 0.8 obtained at a flow rate of 7 L/min. This measured spectra is again very similar to the one shown in Figure 32 but more low frequency noise can be observed near 0 m/s. The noise was caused by mechanical vibrations of the hoses and pipes used. The hose after the outlet was elevated in order to create a more stable and less pulsating flow and thereby minimize this low-frequency noise. The velocity gradient shows the same curvature in the shearing region, which is characteristic for shear-thinning fluids.

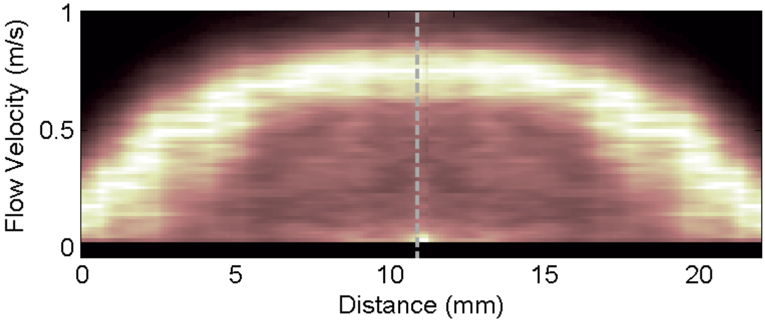

Figure 34. Spectral image of a grout with w/c = 0.8 obtained at a flow rate of 10 L/min.

Figure 34 shows the corresponding measured Doppler spectra of a grout with w/c = 0.8 obtained at the highest flow rate of 10 L/min. The Doppler spectra is slightly more noisy and shows more broadening in the measured velocities. However, the quality of the measured spectra is good and it clearly shows a smaller plug and a lower yield stress for the highest flow rate. The observed noise was caused by poor initial mixing of the grout since the spectra corresponds to one of the first measurements that were made in this test trial. The grout equipment used was equipped with a low-rpm mixed, which was not ideal for micro cement grouts and it therefore took some time before the grout was properly mixed. The quality of the measured profiles improved after pumping the grout through the closed circulation system for a few minutes and after the elevation of the hose at the container outlet.

Figure 35 shows the measured normalized velocity profiles of a grout with w/c = 0.8 at different flow rates, from 3.3 L/min up to 10 L/min. A non-zero velocity at the wall was observed but this is expected due to a gradual overlap between the measuring volume at the pipe wall interface, which leads to a smoothing of velocities in this region. Low frequency noise, such as mechanical vibrations, also leads to low velocities being filtered out in the data processing steps by the use of a clutter filter. It is sometimes possible to reduce the clutter filter setting in order to determine velocities at the wall but this will affect the overall spectra and the measured data may then need to be processed twice with two different filter settings. The measured profile shape clearly shows a pronounced plug in the center of the pipe at the lowest flow rate. The corresponding yield stress was found to be in the range between 3.5-4 Pa. The plug region was found to decrease with increasing flow rate, which was expected since the grout was then subjected to higher shear rates. At the highest flow rate the grout is in a broken-down state and the yield stress was then found to be approximately 2 Pa, which corresponds to a 50% reduction in yield stress. The measured profile shape was found to be more parabolic at behavior. A possible and presumed Bingham behavior was only observed at a volumetric flow rate of 7 L/min. The highest flow rate thus corresponding to a less shear-thinning or i.e. more Newtonian like behavior. It is interesting to note that based on the measured profile shapes the grouts were found to exhibit more Herschel-Bulkley type of flow behavior and not the presumed Bingham

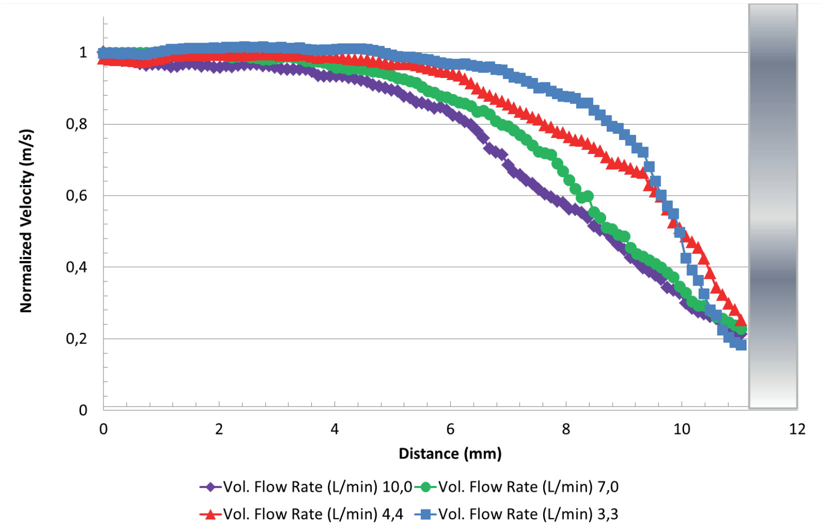

Figure 35. Normalized velocity profiles of a grout with w/c = 0.8 at different flow rates.

4.2.2 Spectral images of w/c = 0.6

Figure 36 shows the spectral image of a grout with w/c = 0.6 obtained at a flow rate of 2.5 L/min using sensor A. Figure 37 shows the corresponding spectra obtained at the same flow rate but using sensor B. As can be seen from the two spectra's there is a good agreement between the data obtained using sensor A and sensor B but a big difference between the profile shape measured for a grout with w/c = 0.8 and the grout with w/c = 0.6. The grout with w/c = 0.6 shows a much more pronounced shear-thinning behavior and with a more distinct shearing region. The transition between the plug and the shearing region is also much sharper thus indicating a distinct Herschel-Bulkley type of flow behavior. The measured pressure drop was higher for the grout with w/c = 0.6 and this in combination with the distinct plug results in a yield stress of 10-11 Pa, which is about three times higher than for the grout with w/c = 0.8 at approximately the same flow rate.

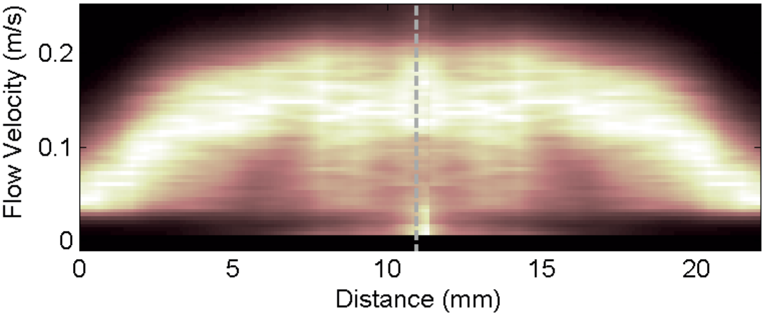

Figure 36. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 2.5 L/min.

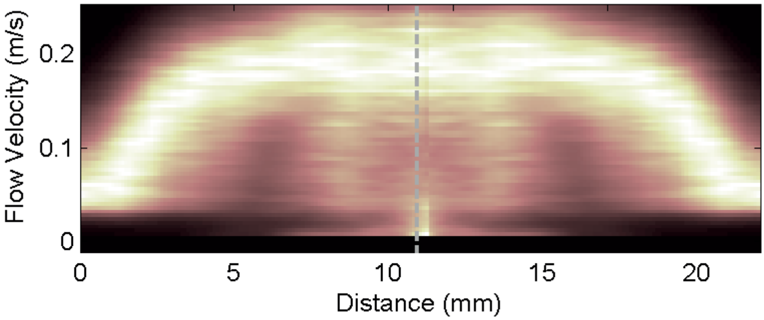

Figure 37. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 2.5

Figure 38 shows the corresponding measured Doppler spectra of a grout with w/c = 0.6 obtained at the second lowest flow rate of 3 L/min. It is interesting to note here that the measured yield stress had an almost identical value but the Doppler spectra shows already here a slightly smoother transition, i.e. more curvature, between the plug and the shearing region. This indicates that the re-circulation of the grout in the flow loop and the longer mixing, both in the mixer and in-line due to pumping, resulted in a grout that is in a more broken down state. The Incipientus ILR in-line rheometer was thus shown capable of detecting even small changes in the profile shape and thus also in changes in grout rheology caused by increased shearing of the grout. This information is useful when monitoring a grouting operation in the field so that the grout properties can be adjusted in real time in order to obtain good grouting results.

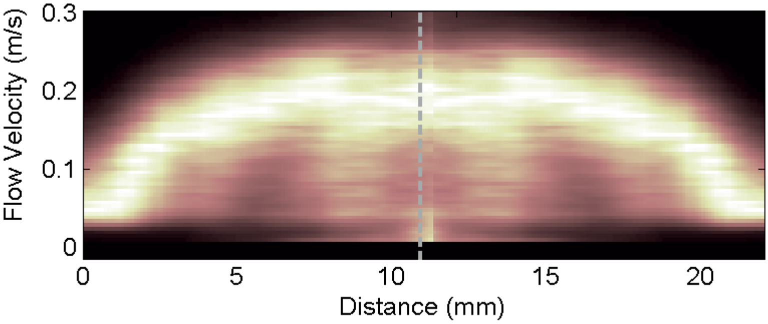

Figure 38. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 3 L/min.

Figure 39 shows the measured Doppler spectra of a grout with w/c = 0.6 obtained at the second highest flow rate of 7 L/min. The Doppler spectra here show a much more curvature, between the plug and the shearing region thus indicating a less shear-thinning behavior and more Bingham-like behavior. The Doppler spectra obtained for grout with w/c = 0.6 was in general more noisy compared to w/c=0.8 and shows more broadening in the measured velocities. As discussed above, the observed noise was caused by poor initial mixing of the grout since the low-rpm mixer used was not ideal for mixing micro cement grouts and it therefore took some time before the grout was properly mixed.

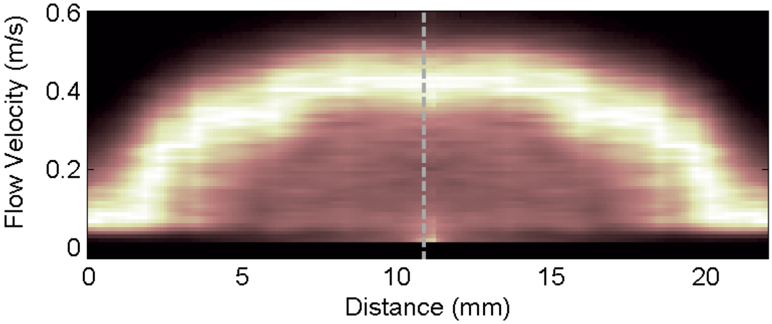

Figure 39. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 7 L/min.

Figure 40 and Figure 41 shows the corresponding measured Doppler spectra of a grout with w/c = 0.6 obtained at the two highest flow rates of 8.5 and 9 L/min. The Doppler spectra in Figure 40 was measured with sensor A and the Doppler spectra in Figure 41 was measured with sensor B. From the two measured spectra it is clear that the plug region is smaller, that the velocity gradient has a more or less linear distribution from the pipe wall to where the plug region starts and this leads to the conclusion that the grout is here in a broken down state. The shape of the measured velocity profiles suggests a Herschel-Bulkley type of flow behavior and not the commonly presumed Bingham behavior. The probability of a grout to exhibit true Bingham behavior tends to decrease with decreasing w/c ratio and this claim is supported by all of our experimental data obtained also in previous work. See for example: Håkansson et al. 2012, Rahman 2015, Rahman et al. 2016. This observation is important since modern grouting designs are often based on the Bingham model simply because it is a two parameter model that is convenient to use. However, our experimental data suggest that grouts, at least with low w/c ratio, only exhibit Bingham behavior under very specific conditions and the use of this model in practical grouting applications may therefore lead to errors and poor grouting results.

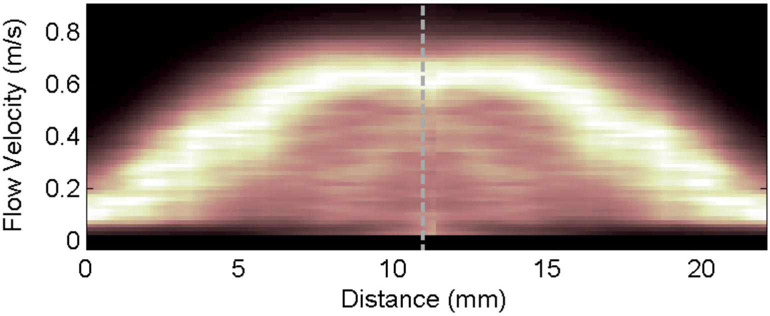

Figure 40. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 8.5 L/min.

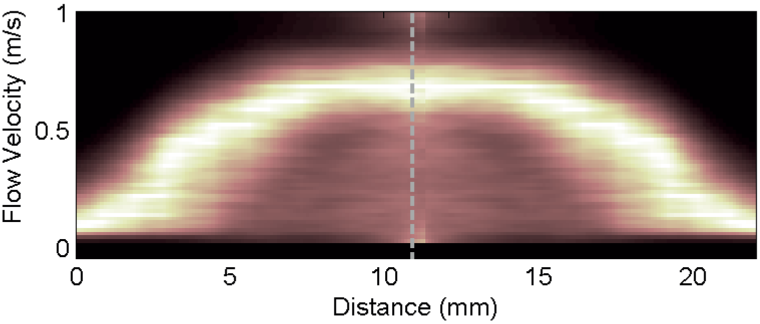

Figure 41. Spectral image of a grout with w/c = 0.6 obtained at a flow rate of 9 L/min.

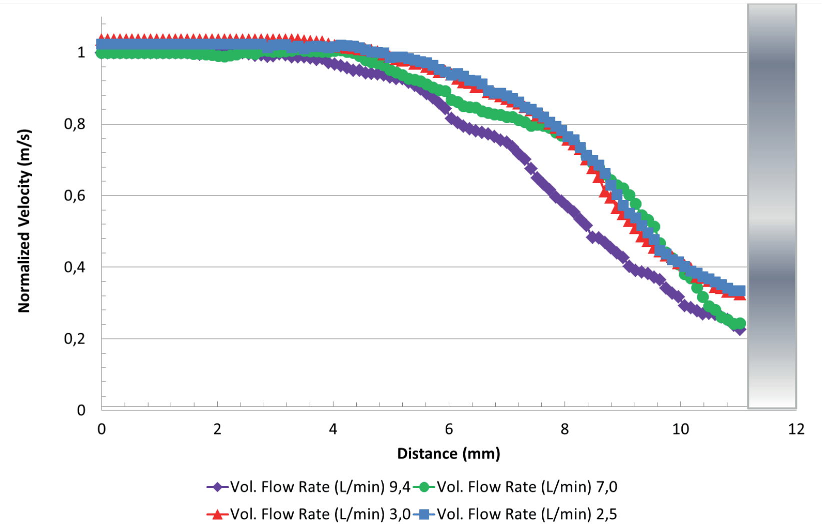

Figure 42. Normalized velocity profiles of a grout with w/c = 0.6 at different flow rates.

Figure 42 shows the measured normalized velocity profiles of a grout with w/c = 0.6 at different flow rates, from 2.5 L/min up to 9.4 L/min. The measured profile shapes clearly shows a pronounced plug in the center of the pipe for all flow rates. The corresponding yield stress was found to be in the range 8-11 Pa, which is significantly higher compared to w/c = 0.8. The plug region was again found to decrease with increasing flow rate, which was expected since the grout was then subjected to higher shear rates.

A non-zero velocity at the wall was observed and this could be caused by slip at the wall, which is common for concentrated suspensions. However, as discussed above a non-zero velocity at the wall is also expected in both pulsed ultrasound velocimetry and laser Doppler anemometry due to a gradual overlap between the measuring volume at the pipe wall interface, which leads to a smoothing of velocities in this region. Low frequency noise, such as mechanical vibrations also leads to low velocities being filtered out in the data processing steps by the use of a clutter filter. At the highest flow rate the grout is in a broken down state but the yield stress is not decreased that much, compared to w/c = 0.8 and was then found to be approximately 8 Pa. The measured profile shapes suggests that the grouts exhibit a pronounced Herschel-Bulkley type of flow behavior.

4.3 In-line measurement of yield stress and viscosity of grout

The yield stress is an important control parameter for rock grouting. The yield stress describes the critical stress that must be exceeded in order to create flow and to be able to inject the grout into the rock fractures. When the pressure drops below the critical stress the grout will form an internal rigid structure that will seal the fracture and prevent the grout from leaking out before it sets. It is therefore extremely important to adjust the properties of the grout injected into the fractures for the rock conditions on the grouting site.

In this work a comparison was made between the yield stress measurements for w/c = 0.8 and w/c = 0.6 using two instruments, a rotational off-line rheometer and the Incipientus ILR in-line rheometer. The Bingham model approximation was used to determine the yield stress from the obtained rheograms using the off-line rheometer. One major advantage with the Incipientus ILR in-line rheometer is that the yield stress could be obtained directly from the measured velocity profiles using the determined plug radius and the corresponding pressure drop measured over a fixed length of pipe.

Table 8 shows a comparison between yield stress values measured with a rotational off-line rheometer and the Incipientus ILR in-line rheometer for w/c = 0.8. The agreement between the measured values was found to be very good for all flow rates except for the highest one. The yield stress measured in-line was significantly lower at 11 L/min compared to the off-line rheometer. The difference in the measured values at the highest flow rate is most likely due to the higher degree of shearing of the grouts in the flow loop compared to the unrealistic shearing in small couette cylinder geometry.

Table 8. Comparison between yield stress measurements for w/c, 0.8

In-line Incipientus ILR in-line rheometer			Off-line rheometer	
Flow Rate (L/min)	Yield stress (Pa)	Pressure Drop (Pa/m)	Time (min)	Yield stress (Pa)
3	3,6	1170	0	3,5
4,4	3,9	1290	5	3,2
7	2,4	1360	10	2,9
11	1,4	1570	15	2,9
-	-	-	20	2,9

Table 9 shows the corresponding comparison between yield stress values measured with a rotational off-line rheometer and the Incipientus ILR in-line rheometer for w/c = 0.6. The agreement between the measured values was found to be very good for all flow rates and it was found to be more or less constant at around 10-11 Pa for all flow rates except for the highest one. Based on previous work and from the measurements we can conclude that the yield stress is an important parameter for grouting control and that this parameter can now accurately be measured directly in-line, without having to use for example a Bingham approximation. This is an important observation since it can significantly improve the robustness of a grouting control system and eliminate the need for operator dependent results obtained using an off-line rheometer and unrealistic testing conditions in small couette cylinder geometries.

Table 9. Comparison between yield stress measurements for w/c, 0.6

In-line Incipientus ILR in-line rheometer rheometer			Off-line rheometer	
Flow Rate (L/min)	Yield stress (Pa)	Pressure Drop (Pa/m)	Time (min)	Yield stress (Pa)
2,5	10,3	5750	0	11
3	10.6	6040	5	10
7	11,4	6330	10	9
9	8,1	7440	15	8
-	-		20	7,7

Figure 43 shows the rheograms for w/c = 0.8 measured in-line at different flow rates using the Incipientus ILR in-line rheometer. All rheograms shows a yield stress at zero shear rate followed by an initial seemingly Newtonian region and then a characteristic shear-thinning region. Note that the shear rate ranges are determined by the flow rates used in the experimental trials and not the in-line instrument. The observed flow behavior is typical for so-called structured fluids. This again supports the claim that grouts should be modeled using a Herschel-Bulkley type of model and not the Bingham model.

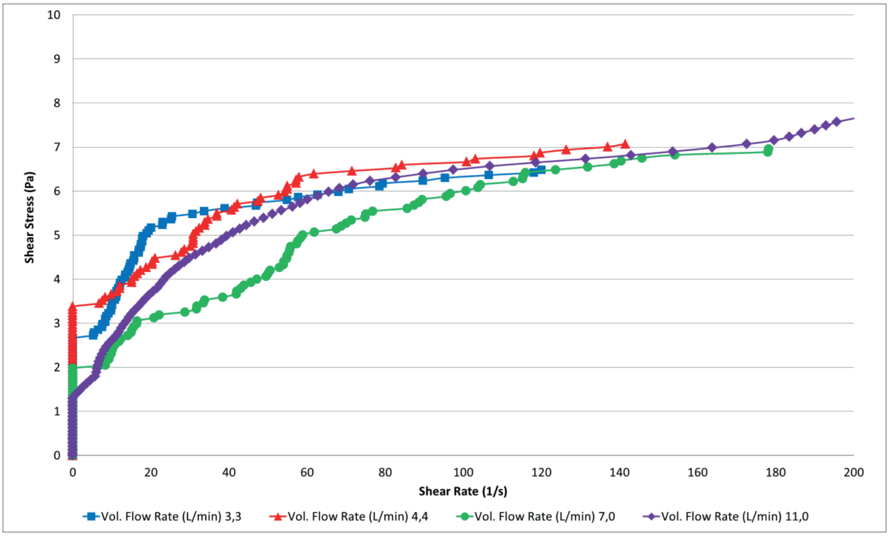

Figure 43. Rheograms measured in-line at different flow rates for w/c 0.8

Figure 44 shows the corresponding rheograms for w/c = 0.6 measured in-line at different flow rates using the Incipientus ILR in-line rheometer. The rheograms follows the same trends as for w/c = 0.8 but with higher yield stress values. The difference in maximum shear rate is also more noticeable due to the higher viscosity of grouts with lower w/c ratio. The viscosity was found to be a bit higher than expected for the highest flow rate but this could be attributed to poor initial mixing of the sample, which resulted in pulsating flow and comparatively higher pressure drop measurements. As discussed above, the flow pulsations was minimized by the addition of a Saunders valve and by raising the hose at the outlet to create more back pressure prior to the measurements at lower flow rates.

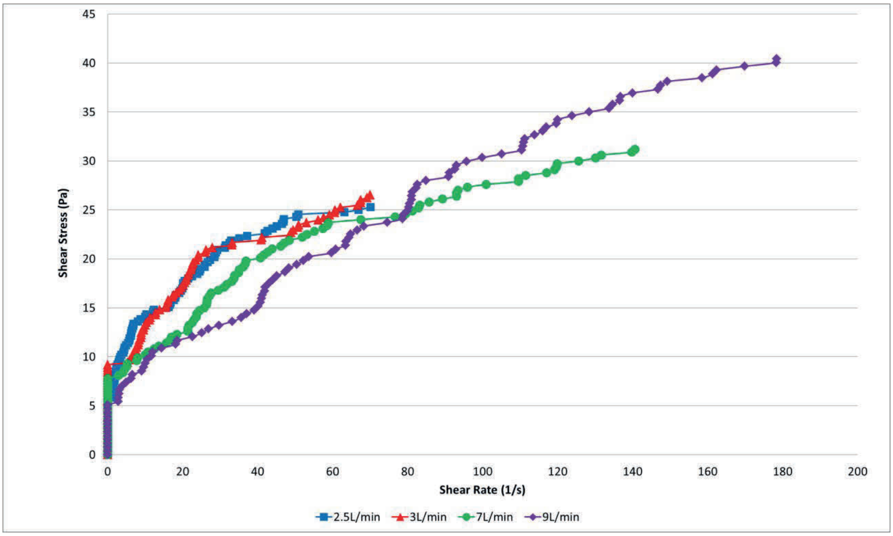

Figure 44. Rheograms measured in-line at different flow rates for w/c 0.6

Figure 45 shows a comparison between rheograms measured in-line and off-line for w/c 0.8. The off-line data was obtained directly after mixing and the grout was thus in a broken down state. The rheogram obtained using a rotational off-line rheometer show more curvature at low shear rates and slightly less shear-thinning behavior at the highest shear rates but the overall agreement between the two methods was in this case found to be surprisingly good. The measuring conditions differ significantly between the two cases and it is often difficult to mimic actual field conditions in small concentric cylinder geometry.

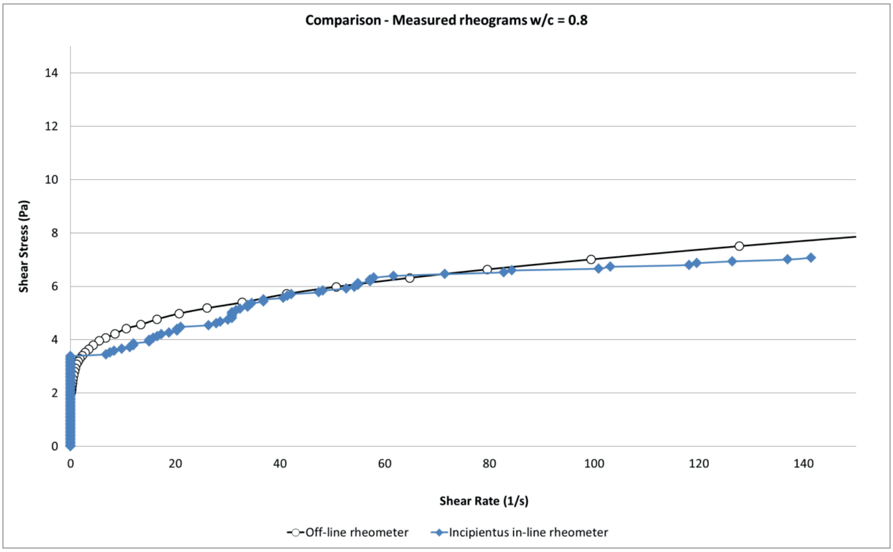

Figure 45. Comparison between rheograms measured in-line and off-line for w/c 0.8

Figure 46 shows a comparison between rheograms measured in-line and off-line for w/c 0.6. The off-line data was also in this case obtained directly after mixing when the grout was in a broken down state. The rheogram obtained using a rotational off-line rheometer show approximately the same degree of shear-thinning behavior over a wide range of shear rates and the overall agreement between the two methods was also in this case found to be good. The pre-shearing of the sample in the rheometer plays an important role and can lead to large measurement errors with non-Newtonian fluids if the sample is pre-sheared for too long or at a high shear rate prior to the measurement. The measured rheograms from both instruments clearly suggest a Herschel-Bulkley type of flow behavior and this behavior is more pronounced at lower w/c ratios.

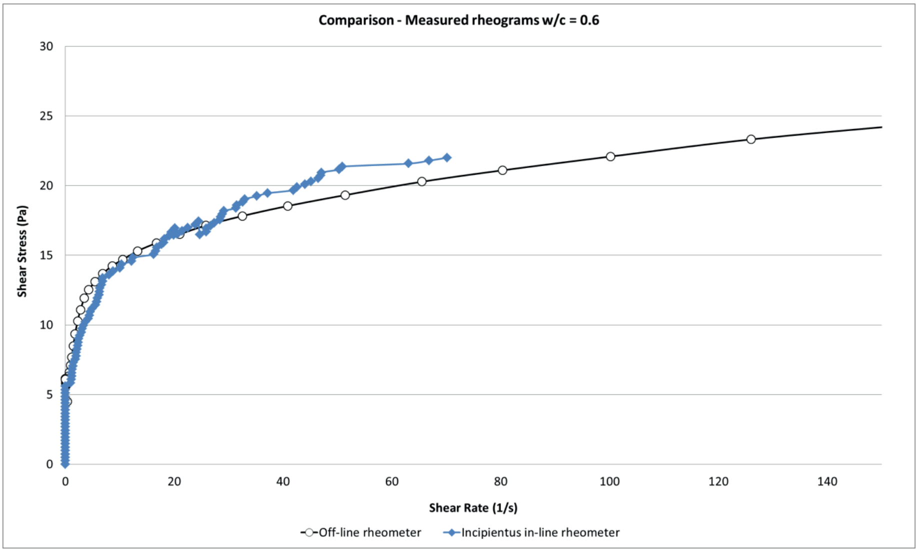

Figure 46. Comparison between rheograms measured in-line and off-line for w/c 0.6

Figure 47 and Figure 48 shows a comparison between the flow curves measured in-line and off-line at different flow rates for w/c = 0.8 and w/c = 0.6. For w/c = 0.8 there is a good agreement between the two methods over a wide range of shear rates at the lowest flow rates. As the flow rate and thus also the shear rates was increased the viscosity of the grouts dropped as expected. The sudden drop in viscosity with increasing shear rates is more noticeable in the lower shear rate region.

The viscosities were found to be higher for w/c 0.6 compared to w/c =0.8, which is again the expected behavior due to a higher solids concentration at lower w/c ratios. The shape of the measured flow curves did not change significantly between the two w/c ratios. The deviation at the highest shear rate between the off-line and in-line data in Figure 48 suggests that the shearing conditions differ between the concentric cylinder geometry used for the off-line tests and the actual flow conditions in the test flow loop.

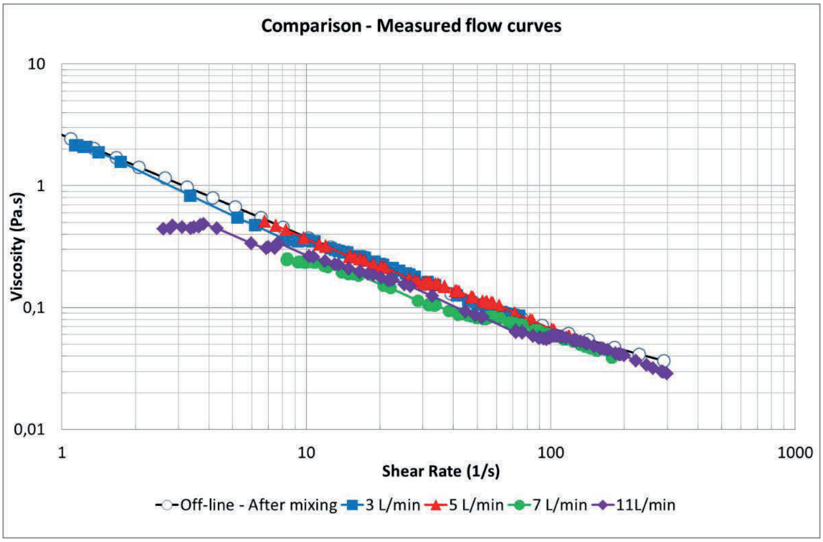

Figure 47. Flow curves measured in-line at different flow rates for w/c 0.8

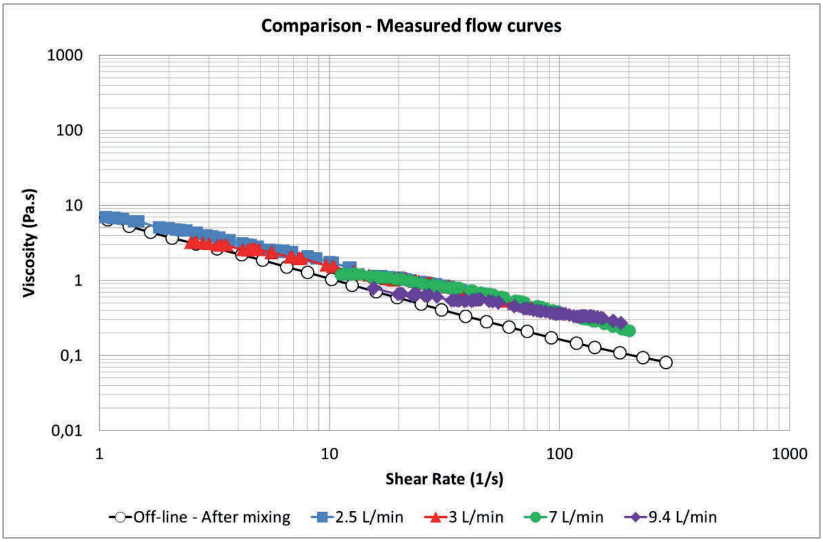

Figure 48. Flow curves measured in-line at different flow rates for w/c 0.8

4.4 Off-line measurement of the rheological properties

The yield stress was determined using the linear Bingham model for w/c ratio 0.8, 0.7 and 0.6, respectively. The results are shown in Figure 49, Figure 50 and Figure 51.

For w/c ratio 0.8, tests were made with different combinations of i-Flow 1 and I-Acc 1. As understood, the yield stress decreases due to steric repulsion when i-Flow 1 is used. i-Acc 1 decreases the setting time, however, as shown in Figure 49, it does not have any significant effect until after 25 minutes. The yield stress is higher than the required value for mixture 1 when no super plasticizer is used. A rapid increase of the yield stress was observed when only the accelerator i-Acc 1 was used.
As shown in Figure 49, w/c ratio 0.8 with 0,2% i-Flow 1 and 2 % i-Acc 1 and w/c ratio 0.8 with 0,2% i-Flow 1 and 1 % i-Acc 1, w/c ratio 0.8 with 0,2% i-Flow 1 satisfy the requirement for yield stress for mixture 1.

For w/c ratio 0.7, tests were made with and without additives.
As shown in Figure 50, w/c ratio 0,7 with 0,2% i-Flow 1 and 2 % i-Acc and w/c ratio 0,7 with 0,2% i-Flow 1 both satisfy the requirement of yield stress for mixture 2.

For w/c ratio 0.6, tests were made without any additive and with Sodium Silicate (water glass, SiO2). For w/c ratio 0.6 without any additives, the determined yield stress was less than the requirement, as shown in Figure 51; therefore, this mixture did not qualify as recipe 3. This indicated a need for an additive which could increase the yield stress immediately after mixing. Therefore, a 40% solution of Sodium Silicate was used following an earlier work by Håkansson (1993). For w/c ratio 0.6 with 1% water glass solution, an immediate increase of yield stress was observed. For 2% water glass, the determined yield stress was comparatively higher and would require a high pumping capacity. To reduce the usage of cement and cost, w/c ratio 0.7 was tested with 1% and 2% water glass solution and as shown in Figure 50, w/c ratio 0.7 with 2% water glass satisfy the requirement of yield stress for mixture 3.

The viscosity was determined using the linear Bingham model for w/c ratio 0.8, 0.7 and 0.6, respectively. The results are shown in Figure 52, Figure 53 and Figure 54. As can be seen in the figures, the viscosity increased after 25 minutes when i-Acc 1 is used.
As can be seen in Figure 52, w/c ratio 0.8 with 0,2% i-Flow 1 and 2 % i-Acc 1, w/c ratio 0.8 with 0,2% i-Flow 1 and 1 % i-Acc 1 and w/c ratio 0.8 with 0,2% i-Flow 1 satisfy the requirement for viscosity for mixture 1. As shown in Figure 53, w/c ratio 0.7 with 0,2% i-

Flow 1 and 2 % i-Acc, w/c ratio 0.7 with 0,2% i-Flow 1, w/c ratio 0.7 with 2% water glass and w/c 0.7 without any additives satisfy the requirement of yield stress for mixture 2.

For w/c ratio 0.6, the determined viscosity was very high, as shown in Figure 54. Even though this satisfies the requirement for mixture 3, a comparatively lower viscosity will increase the spread of grout, with time.

From the results presented, the following results can be summarized

- w/c ratio 0.8 with 0.2% i-Flow satisfies the requirement for mixture 1
- w/c ratio 0.7 with 0.2% i-Flow satisfies the requirement for mixture 2
- w/c ratio 0.6 with 1% Sodium Silicate (40% solution) and w/c ratio 0.7 with 2% Sodium Silicate (40% solution) satisfy the requirement for mixture 3.
- i-Acc 1 does not have significant effects in first 25 minutes and can therefore be omitted.

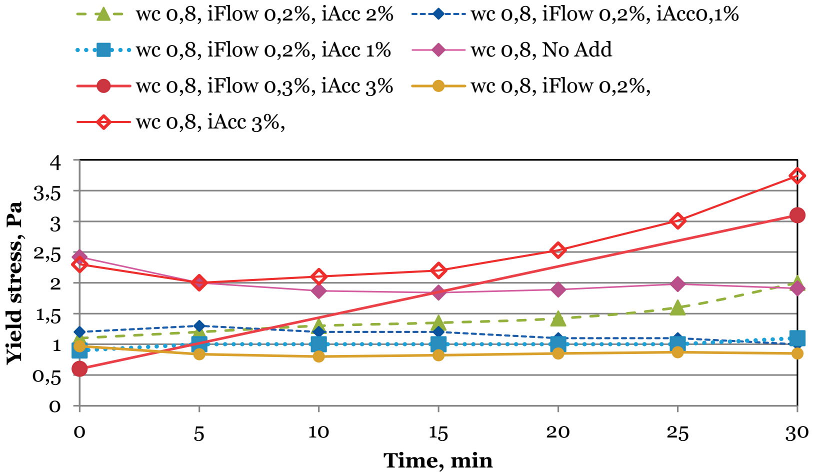

Figure 49. Yield stress determined by Bingham model for w/c 0.8

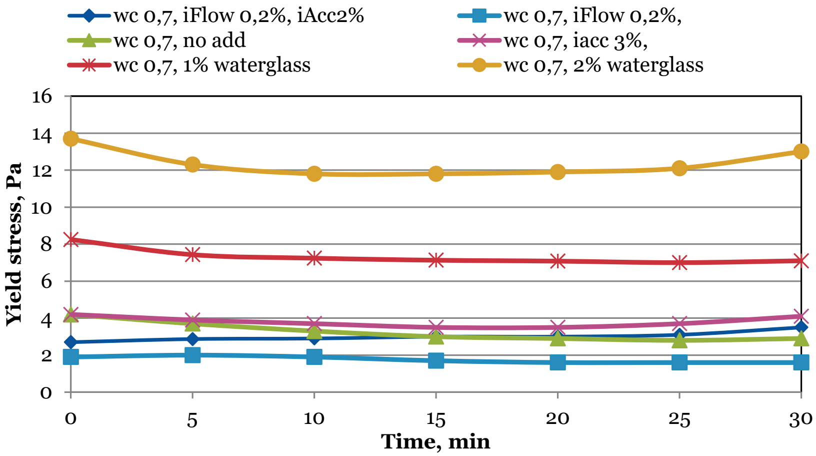

Figure 50. Yield stress determined by Bingham model for w/c 0,7

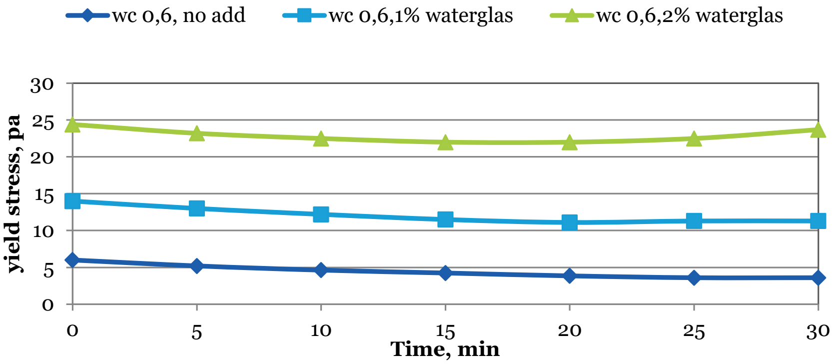

Figure 51. Yield stress determined by Bingham model for w/c 0.6

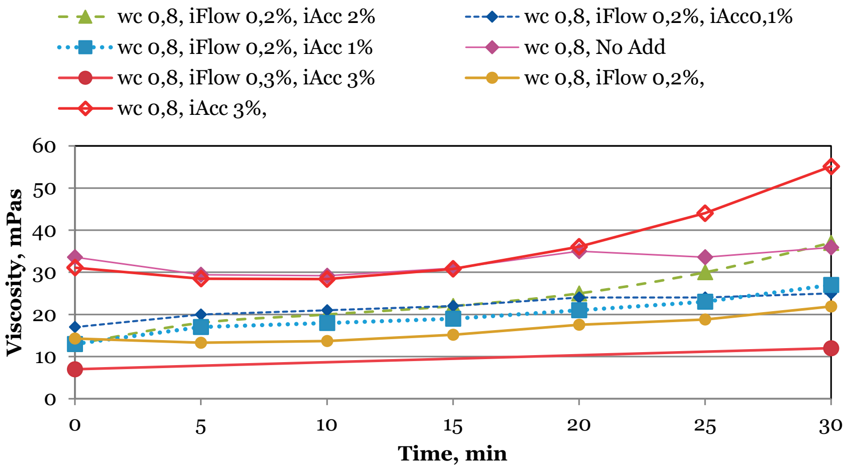

Figure 52. Viscosity determined by Bingham model for w/c 0.8

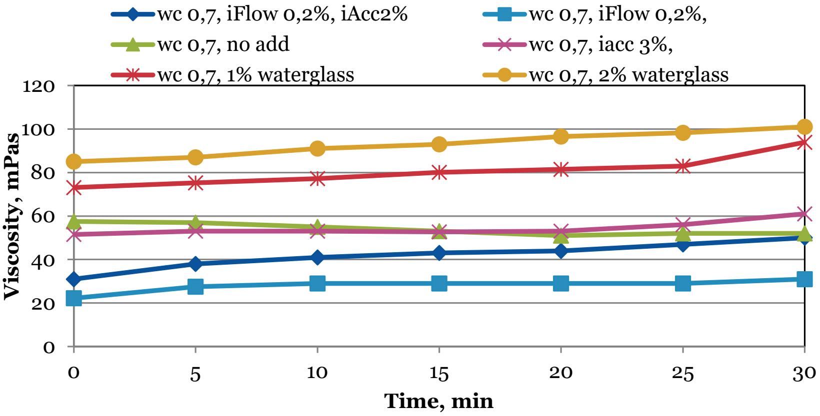

Figure 53. Viscosity determined by Bingham model for w/c 0,7

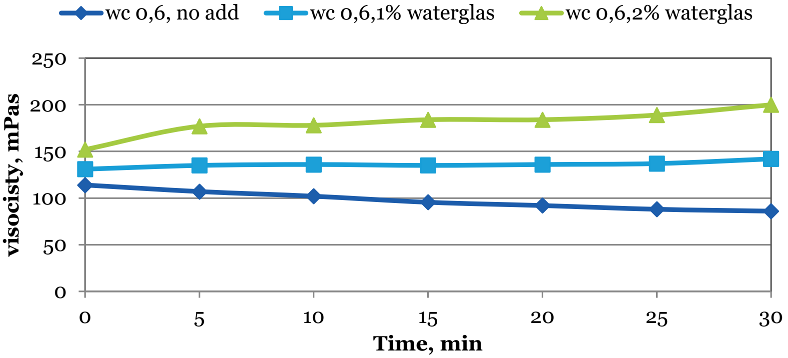

Figure 54. Viscosity determined by Bingham model for w/c 0.6

Off-line rheograms, for w/c ratio 0.8 and 0.6 without any additives, are shown in Figure 55 for reference. The measurements are made directly after mixing. As can be seen, the used cement grouts are typical yield stress, shear thinning, fluids, fitting well to the Herschel-Bulkley model.

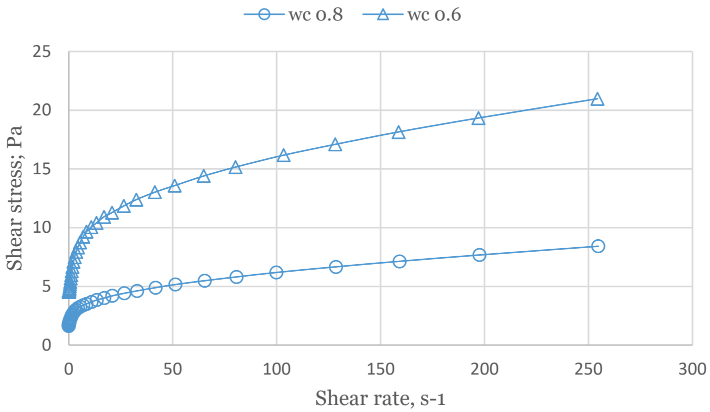

Figure 55. Rheograms for w/c ratio 0.8 and 0.6 without any additives, directly after mixing

4.5 Discussion on in-line results

4.5.1 Mixing of grout, pumping of grout etc.

The results from the field tests showed that it is important to select and use a suitable high-rpm mixer for the Cementa IC 30 micro fine cement used in this work. Mixing was not an issue for the small scale laboratory tests but it was more difficult to prepare larger batches for the field test for w/c = 0.6. The initial measurements at the highest flow rate resulted in slightly more noisy Doppler spectra but the situation was rapidly improved due to in-line mixing when the grout was recirculated in the flow loop. The grouting rig used was equipped with a screw-pump, which normally produces a more stable flow with less pulsation compared to a traditional piston-pump setup. However, it was found that the field laboratory flow loop should be equipped with a valve at the outlet that can be used to control the back pressure and to produce a more stable flow. Moreover, the field laboratory should also be fitted with a safety release valve that can open at a specified pressure in case of pipe blockage.

4.5.2 Optimized sensor design

It was found that the new sensor design with improved beam focusing resulted in less distortions in the near wall region and that it works well for measuring grout rheology in field conditions. This is important since the velocity gradient is the highest in this region and this is where the shearing of the sample has the highest impact on the grout rheology. In addition, the optimized beam characteristics with a smaller divergence angle and extended focal zone resulted in a sustained penetration depth compared to the sensors used in previous work. However, the sensor design can be further improved, for example be made even more robust if subjected to mechanical forces, moisture, temperature variations, vibrations etc. in the field. The sensor cover can either be reinforced or alternatively a small enclosure can be added to protect the sensors from the harsh conditions in the field. An attempt will be made for the next sensor revision to further extend the focal zone to cover the full pipe diameter. This is not a requirement for accurate flow and rheology measurements but it will result in a better visual experience for the operator.

The results from both laboratory and field tests showed that the measured Doppler spectra and resulting velocity profiles has improved significantly compared to our previous work after optimization of electronics and data processing algorithms. The Doppler spectra and the corresponding velocity profiles are now processed directly on the digital board, which offers much better control of the data processing. Moreover, several parameters, such as analog and digital gain can be auto-tuned by the board, which results in fewer problems with saturation of the ADC electronics or too low amplification of weak signals in highly attenuating media. The new optimized firmware also features a dual-PRF mode, which allows the instrument to overcome the traditional velocity-range limitation (due to the Nyquist sampling limit, which results in aliasing. More information about the new firmware and the optimized Incipientus electronics is presented in Ricci et al. 2016, 2017b and Wiklund et al. 2016a.

4.5.3 Wall slip

Wall slip is the term given when fluids violate the classic no-slip boundary conditions of fluid mechanics and do actually slip over solid surfaces when the shear stress exceeds a critical value. It is generally observed and accepted that concentrated suspensions exhibit wall slip at specific conditions but it is practically very hard to determine if slip occurs or not. A non-zero velocity at the wall is often observed when velocimetry techniques such as PUV are used to determine velocity profiles in concentrated suspensions. A non-zero velocity was observed in our field tests for w/c = 0.6 but this is also expected due to a gradual overlap between the measuring volume at the pipe wall interface, which leads to a smoothing of velocities in this region. Moreover, the concentration of solids in the near wall region may also be lower than closer to the pipe center due to shear induced radial migration effects. The thickness of the slip layer is generally much smaller than the size of the measuring volume. It is therefore difficult to determine the presence of wall slip and the actual slip velocity but PUV technology may be used to give an indication if slip is present or not. Low frequency noise, such as mechanical vibrations, also leads to low velocities being filtered out in the data processing steps by the use of a clutter filter. It is sometimes possible to reduce the clutter filter setting in order to determine velocities at the wall but this will affect the overall spectra and the measured data may then need to be processed twice with two different filter settings.

4.5.4 Modelling grout rheology

As previously discussed in the preceding chapters modern grouting theory is often based on the assumption that grouts exhibit Bingham flow behavior. The Bingham model is a two parameter model in which the yield stress and the plastic viscosity are the two parameters used to describe the flow behavior of grouts. However, the results from our laboratory and field tests strongly suggests that grout rheology should be modeled using e.g. the Herschel-Bulkley model and that grouts only exhibit Bingham behavior under very restricted conditions. There are also several different ways to perform the linear Bingham approximation when using a conventional off-line rheometer, which makes the determination of the yield stress and plastic viscosity operator dependent. This can lead to large errors and may result in a poor grouting result if the wrong parameter set was used as a design basis for a grouting job. In contrast, with the Incipientus ILR in-line rheometer the grouting control parameters can be determined directly from the measure velocity profile and the pressure drop, without assuming that the grout under investigation will exhibit a specific flow behavior. If grout rheology is determined directly in the field under actual conditions using the ILR in-line rheometer it is possible to obtain more accurate results and to eliminate operator dependent measurement errors.

4.5.5 Robust In-line rheometry vs off-line rheometry

An impressive correlation between the off-line and in-line rheometry was found, taking in to account the difficulties associated with off-line rheometry. Off-line rheometry used in field is usually not of high quality and is time consuming. The measurements are also very dependent on the conditions, operator and equipment, however with in-line rheometry results are available in "real-time" and with less uncertainties

Conclusions and future outlook
This work presented a new container-based field laboratory, designed for in-line measurements of rheological properties of cement-based grouts under field-like conditions. The field laboratory is equipped with the new commercially available Incipientus ILR in-line rheometer.

The new portable container-based field laboratory equipped with the Incipientus ILR in-line rheometer is considered a useful rheological characterization tool for field use, pilot plant installations and academic research. This in turn makes it possible to omit cumbersome pre-testing, change the grouting procedure on the basis of the results and also makes quality control easy to perform during operations. The main challenge with the system is that it provides continuous data that allows the operator to make rapid adjustments of the processing parameters in the field. However, there is currently no grouting practice that supports the continuous feed of such quality control parameters during grouting operations.

Our results showed that the technology has now been optimized for cement-based grouts measured under field-like conditions. The new electronics features simultaneous acquisition and on-board processing of ultrasound, pressure and temperature signals for real-time velocity estimation and rheological characterization. The results show that the new system combined with the non-invasive sensor technology allows true in-line flow visualization and can be used for measurement of the rheological properties continuously and non-invasively in-line during a practical grouting operation.

The next step is to perform in-line rheological measurements of cement based grouts using the developed 10ft container laboratory equipped with the Incipientus ILR in-line rheometer under realistic field conditions in a real tunnel, during grouting operations.

The following aspects should be implemented for future tests:

- All the measurement equipment should be recorded with a data acquisition device (DAQ). This information should be displayed clearly on a wall mounted tablet.
- The point pressure output must trigger an emergency relay that switches of the pump and activates a warning alarm, when it measures some predefined over pressure. This is an important safety feature.
- A safety valve (pressure relieve valve) must always be installed at the outlet of the pump to prevent accidents in case of pipe blockages.
- An extra valve is required on the outside of the container on the return line in order to apply some back pressure and to obtain stable flow.

In this work, the Incipientus ILR in-line rheometer successfully measured even a very small change of the shape of the velocity profiles. For w/c ratio 0.6, a relatively more shear thinning behavior was observed in comparison with w/c ratio 0.8. This indicated that cement grout might behave as a Herschel-Bulkley fluid at a decreased w/c ratios and the change of the velocity gradient is dependent on the pressure. However this needs to be investigated further and following works can be performed:

- Velocity profiles can be measured for a standard Herschel-Bulkley fluid and Bingham fluid and compared with the cement grout for different w/c ratios.
- The change of the plug radius, pressure drop can be measured and the flow index and consistency index can be determined for different w/c ratios and compared, to investigate the change of the shear thinning behavior of grout at different concentrations. Moreover, these parameters should also be compared for different flow rates.
- The measurements can be performed in a larger diameter pipe to perform detail investigation on the change of the shape of the velocity profiles.

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