



# IN-LINE RHEOLOGICAL MEASUREMENTS OF CEMENT BASED GROUTS USING THE UVP-PD METHOD

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# IN-LINE RHEOLOGICAL MEASUREMENTS OF CEMENT BASED GROUTS USING THE UVP-PD METHOD -

A Pre-Study

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# Förord

Forskning om injektering har under trettio år varit ett prioriterat område inom undermarksbyggandet. Under denna tid har injekteringstekniken utvecklats från experimentell verksamhet till ingenjörsvetenskap. Tillämpningen har dock gått långsammare till följd av att nyckelfrågor förblivit olösta. En sådan fråga är styrningen och kontrollen av materialets reologiska egenskaper som påverkas av en rad olika faktorer och avgör strömningsegenskaper. De reologiska egenskaperna förändras dessutom över tid under injektering vilket man idag saknar möjlighet att kontinuerligt kontrollera.

Reologiska egenskaper kan mätas med ultraljud, och sådana tekniker har framgångsrikt tillämpats inom bl. a. livsmedelsindustri och sjukvård. I detta projektområde där målet är att kunna styra injekteringsprocessen i realtid har samverkan initierats med livsmedelsinstitutet (SIK) som är världsledande på området. SIK tillhör SP som har samarbetsavtal med KTH vilket passar väl in för att utnyttja SP:s resurser inom högskoleforskningen.

Möjligheten att mäta reologiska egenskaper "in-line" i injekteringsprocessen, utan kontakt med cementsuspensionen, efterlystes redan i Ulf Håkanssons doktorsavhandling "*Rheology of Fresh Cement-Based Grouts*" KTH 1993 (och SveBeFo rapport 15 1994) och ser nu ut att kunna förverkligas. Det blir då möjligt att styra bruket bättre, ändra dess egenskaper och mer effektivt kvalitetssäkra det använda bruket. En av de saknade nycklarna inom forskningsområdet (RTGC) "*real time grouting control*" ser därmed ut att kunna lösas och skapa praktiska möjligheter för effektivare tätning av berg under mark.

Denna rapport, den första i BeFo:s rapportserie skriven på engelska med svensk sammanfattning, är författad av doktoranden Md. Mashuqur Rahman & Professor Ulf Håkansson, båda på KTH.

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Mikael Hellsten

#### SUMMARY

In underground construction grouting is used to seal tunnels and caverns against excessive water inflow and to limit the lowering of the surrounding groundwater table. Rheological properties of the grout used, such as viscosity and yield strength, play a fundamental role in design and execution, but no method has yet been developed to measure these properties in-line during field work. For the first time, the in-line rheometry method combining the Ultrasound Velocity Profiling (UVP) technique with Pressure Difference (PD) measurements, known as "UVP-PD", was tested successfully for continuous in-line measurements of concentrated micro cement-based grouts. The test set-up consisted of a combination of an experimental "flow loop" and a conventional field grouting rig – UNIGROUT, from Atlas Copco. The velocity profiles were measured directly in-line, and the obtained rheological properties were subsequently compared with off-line measurements using a conventional rotational rheometer. In this work, the UVP-PD method was demonstrated to be a promising new in-line tool for the determination of rheological properties of commonly used cement-based grouts.

KEY WORDS: Grouting, Rheology, In-line rheometry, Cement grouts, Cement suspensions, UVP-PD method Ultrasound velocity profiling, Flow profiling

#### SAMMANFATTNING

Injektering används inom undermarkabyggande för att täta tunnlar och bergrum mot stora vattenflöden eller för att begränsa avsänkningen av grundvattennivåer för att förhindra sättningar. Reologiska parametrar, såsom viskositet och flytgräns, har en fundamental betydelse för injekteringsdesign och utförande, men ännu har inte någon metod utvecklats för att kunna mäta dessa egenskaper kontinuerligt "in-line", under pågående injektering. För första gången har nu en metod som kombinerar hastighetsprofiler, uppmätta med ultraljud, med mätning av tryckfall – den sk. "Ultrasound Velocity Profiling" (UVP) metoden med "Pressure Difference" (PD) använts. Lyckade försök har utförts på en cementsuspesion baserat på mikro cement. Försöksutrustningen har bestått av en "flödes slinga" för ultraljudsmätning samt en konventionell injekteringsutrustning, UNIGROUT, från Atlas Copco. De reologiska parametrarna uppmätes direkt "in-line" och jämfördes sedan med mätningar utförda med en vanlig rotationsviskometer. Resultaten visar tydligt att detta är en lovande metod för att direkt bestämma de reologiska parametrarna för cement baserade injekteringsmedel i samband med en verklig injektering i fält.

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#### 1. INTRODUCTION

In underground construction, grouting is used to seal tunnels and caverns against excessive water inflow or to limit the lowering of the groundwater table, by injecting a grout into joints and fractures in the rock. A lowered groundwater level can have adverse environmental impact or it can increase the risk of settlement to adjacent buildings. Most infrastructure projects today are located in urban areas and the cost to avoid groundwater related problems can constitute a large portion of the total project cost.

Performed grouting work is often based on rules of thumb and despite the fact that extensive research has been conducted for the last 25 years in Sweden, practical implementation of the result has so far been rather limited.

The rheological properties, such as viscosity and yield strength, of the used grouts play a fundamental role in grouting and difficulties in the measurement of the rheological properties of cement based suspensions, are well known (Håkansson, 1993). However, testing methods that deliver a continuous in-line measurement of the properties as well as their change with time are still lacking. Measurements are today made with rather primitive methods, developed many years ago, mainly in order to verify and fulfil stipulated quality criteria. The activity itself often implies a stop and disruption of the ongoing grouting and it is frequently difficult to achieve quality assured results.

Modern grouting rigs are today equipped with monitoring devices for continuous measurement of flow and pressure and the output constitutes an important means for quality control and steering of a grouting operation. However, as pointed out in his thesis, Håkansson (1993) concluded that future improvements of grouting equipment should also involve continuous in-line monitoring of the rheological properties. This holds true even more today since the latest development regarding design and steering of a grouting operation necessitates an accurate and reliable determination of the rheological properties, as well as their change with time. In recent work regarding design and steering of a grouting operation (Gustafson and Stille, 2005) it is shown that an accurate and reliable determination of the rheological properties, as well as their change with time is important. The authors showed that the characteristics grouting time depends on the pressure, viscosity and yield stress and that it is also independent of the joint aperture, according to following equation

$$t_0 = \frac{6 \cdot \mu_g \cdot \Delta p}{{\tau_0}^2}$$

This means that the designer can choose the grouting time scale at his own choice and that the necessity of continuously in-line monitoring of the rheological properties therefore becomes important. It is interesting to note that the equation above includes the viscosity in the nominator and the yield stress in the denominator, to the power of two, emphasising the importance of the rheological properties.

A promising contribution towards in-line measurements of cement-based grouts is the so-called "Ultrasound Velocity Profiling (UVP) - Pressure Difference (PD)" method, used by various other industries (Takeda, 1986). The UVP-PD method has recently been successfully used on other industrial suspensions, such as food, paper pulp and mine tailings (Ouriev, 2000, Kotzé, 2007, Birkhofer, 2007 and Wiklund, 2007). One of the major advantages, compared to conventional rotational or capillary rheometers, is that the measurement is conducted continuously in-line and that it is non-invasive, i.e. there is no measuring device in contact with the suspension itself. Output from the device is an image of the actual velocity profile from which either the flow curve, i.e. shear stress vs. shear rate, can be found directly or the rheological model determined by various curve-fitting procedures. Once the velocity profile is found, the flow rate can be determined by integration and subsequently compared with the measurements from ordinary flow rate instruments. An interesting feature of this method is that parameters, such as the yield strength and the shear rate, can be measured directly from the image of the velocity profile. The shear stress, however, must be estimated from the pressure drop over a certain distance of the pipe by using conventional pressure transducers.

The development of an in-line measurement procedure, based on ultra-sound, implies that it can be possible to control the properties and early determine if the grout is starting to hydrate or if it is by some other mechanism achieving unwanted flow characteristics. It is envisaged that the monitoring device in the future can be mounted on one of the pipes on the grouting rig for continuous measurements of the properties throughout the entire grouting operation. The results can be stored digitally, the same way as pressure and flow is stored today, and used to steer the grouting operation as well as quality assure the used grouts.

The applicability of the in-line UVP-PD method for the measurement of rheological properties of grouts has been reported previously in a pre-feasibility study (Håkansson and Rahman, 2009).

# 2. OBJECTIVES AND LIMITATIONS

#### 2.1 Objectives

The main objective of this study was to verify the feasibility of the ultrasound velocity profiling – pressure difference (UVP-PD) method for measuring the velocity profiles of commonly used cement based grouts directly in-line. The UVP-PD data was subsequently used to evaluate how accurate and effective it is in determining the rheological properties of grouts with different water-cement ratios. A standard cement grout mixing equipment, UNIGROUT E22H and LOGAC flow meter, was used to keep the conditions similar to the ones in the field. Secondary objectives of the investigation were to determine the following:

- 1. Velocity of sound using the customized flow loop.
- 2. Rheological properties by curve fitting to the velocity data.
- 3. Shear rate, yield stress and viscosity directly from the velocity profiles by the gradient method.
- 4. Flow curves, i.e. shear stress vs. shear rate, by the two different methods given above.
- 5. Volumetric flow rate by integrating the velocity profiles and comparing the results with a conventional flow meter (LOGAC).
- 6. Rheological properties from off-line measurements by a conventional rheometer.

#### 2.2 Limitations

This was the first time the UVP-PD method was used for measuring the rheological properties of cement based grouts. The set up was also different from previous measurements and studies using the UVP-PD application. Limitations involved in the current work are described below.

In order to have accurate measurements of velocity profiles and subsequent determination of rheological properties, it is very important to have a stable flow through the flow loop. The UNIGROUT E22H is equipped with a piston type of pump and as a consequence, the movement of the piston and variations of pressure creates high fluctuations in the flow rate which, was observed in all the measured velocity profiles.

Ball valves mounted on the UNIGROUT E22H were used to change the flow rate. The ball valves are simple and crude, making it difficult to control the flow rate accurately enough for this type of measurements.

In the measurements, only 4 MHz transducers were used and this creates limitations with respect to signal penetration when using dense grouts, i.e. low w/c ratios.

Cement grouts were tested both with and without SetControl II. However, since the main objective was to determine the feasibility of the UVP-PD method, no actual comparisons were within the scope of this work. The sampling time and other conditions were not identical, which means that the results with or without SetControl II cannot readily be compared.

Off-line measurements were performed by using a rotational rheometer. However, it was out of the scope of work to thoroughly compare the results between in-line and off-line measurements and to observe the change of the rheological properties over time. As most of the sampling time and other conditions were not identical between the two methods, the results of the off-line measurements are only indicative and are shown as a separate result for convenience.

#### **3 ULTRASOUND PHYSICS**

#### 3.1 Introduction

Sound waves propagate through air or water by mechanical vibration, i.e. by the transmission of energy through the molecules of a medium. Every molecule transfers the energy to other molecules but remains in the same position after transferring the energy. The energy passes in the form of a sound wave, which can propagate transversely or longitudinally. Transverse waves are also called shear waves and they are the most easily propagated waveform in nature. Sound waves can propagate both longitudinally and transversely in solids but fluids only support longitudinal propagation.

Ultrasound is defined as any sound wave with frequencies higher than the human hearing threshold, i.e. in the range 20 kHz. The amplitude of a sound wave comes from the change of air pressure in the wave and it is a degree of motion of the air molecules. The frequency of a sound wave is the number of the waves passing through a point in one second. Intensity is the average rate of flow of energy per unit area perpendicular to the direction of propagation. Wavelength is the distance between the two successive peaks of the wave. The sound velocity can be determined from the product of the frequency and the wavelength by the following equation

 $c = f \times \lambda$ 

.....3.1

where,

c = Acoustic velocity in the medium (m/s)

 $\lambda =$  Acoustic wavelength (m)

f = Frequency (Hz)

#### 3.2 Sound velocity in fluids

Sound velocity in gases can be measured very accurately by the molecular theory. Due to the higher compressibility, the density of the gas varies in the same way as the change of pressure. But in case of solid the density remains partially constant. The pressure and the density are the same throughout the entire volume of a gas in equilibrium state. If the pressure of the gas is distributed the pressure gradient increases and a volume element gains some motion. Adiabatic linear bulk modulus of elasticity is used to show the change of pressure per unit change of density in gases. It can be expressed as

where

 $\kappa_{AD}$  = adiabatic linear bulk modulus of elasticity (N/m<sup>2</sup>)  $\rho_0$  = equilibrium density (kg/m<sup>3</sup>)

The adiabatic linear bulk modulus of elasticity indicates the stiffness of the gas medium and their changes in volume when distributed at a constant temperature. The compressibility of liquid is lower than gases but still this theory can be used with a modified volumetric modulus of elasticity. The propagation of ultrasound in a liquid is nearly an adiabatic process. The compressed regions possess more heat than the relaxed zones in the liquid where sound is propagating. The inverse of the adiabatic linear bulk modulus of elasticity is known as the compression coefficient and can be expressed as

/ \

The velocity of sound wave propagating in liquids can be expressed as

$$c = \sqrt{\frac{\kappa_{AD}}{\rho_0}} = \sqrt{\frac{1}{\chi_{AD} \cdot \rho_0}} \qquad \dots 3.4$$

Where

c = Sound velocity in liquid (m/s)

#### 3.2.1 Attenuation of ultrasound waves

The ultrasound waves are attenuated as some of the energies are refracted or scattered. If we know the wave amplitude for a propagating distance, the attenuation of an ultrasound wave can be determined. The damping of an ultrasound wave propagating in a certain distance can be characterized by the spatial attenuation coefficient,  $\alpha_0$ , expressed as

where

 $A_{Max}$  = wave amplitude of the transmitted ultrasound wave

The amplitude of an ultrasound wave reduces following an exponential law which is expressed as

$$A_{Max} = A_{Max0} \cdot e^{-\alpha_0 \cdot c \cdot t}$$

and is shown in Figure 3.1.



*Figure 3.1 Attenuation of an Ultrasound Wave (Ouriev, 2000) Figur 3.1 Dämpning av ultraljudvågen (Ouriev, 2000)* 

The attenuation coefficient depends on the material which is important in choosing the pipe materials for the UVP-PD test. Materials with a high attenuation coefficient should be avoided.

#### 3.2.2 Acoustic impedance

Acoustic impedance is a physical property of a material and an important factor for optimizing the energy transfer from one medium to another. It determines the refraction and transmission of energy at the boundary between two successive media.

The acoustic impedance can be expressed as

Z=ρ×c .....3.7

.....3.6

where

7

Z = Acoustic impedance (kg/m<sup>2</sup>s = Ray)

$$c =$$
 sound velocity (m/s)

 $\rho$ = density of transmitting medium (kg/m<sup>3</sup>)

It is possible to obtain the non-invasive measurements through a pipe wall if the acoustic impedance of the wall material is greater than the acoustic impedance of the liquids used.

### 3.2.3 Propagation of ultrasound through the boundary between two layers

Reflection and refraction occurs when ultrasound waves pass through the boundary of two layers. While passing through the boundary, the ultrasound waves will reflect and refract at different angles. The angles depend on the acoustic properties of the two materials. A correlation between the incident angle and refractive angle is established by Snell's equation, which was established for optics but found valid for all wave propagation and expressed as

where

 $\theta_1$  = angle of incidence  $c_1$  = sound velocity in medium 1  $\theta_2$  = angle of refraction

 $c_2$ = sound velocity in medium 2

The equation is explained by Figure 3.2.



Figure 3.2 Ultrasound wave propagating through two materials Figur 3.2 Ultraljudvåg genom två olika material

The ultrasound propagates from layer 1 to layer 2 with an incidence angle  $\theta_1$  and it is refracted in layer 2 with a refraction angle of  $\theta_2$ . From Snell's law we can say that the higher the sound velocity in layer 2 the larger the refraction angle  $\theta_2$  will be.

While propagating through the boundary layers the ultrasound wave will lose some energy due to the acoustic impedance of the materials. In Figure 3.2 the incidence intensity of the ultrasound wave at layer 2 is  $I_1$  and transmitted intensity at layer 2 is  $I_2$ . The reflective intensity at layer 1 is  $I'_1$  and the initial intensity of the ultrasound wave can be expressed as

A reflection coefficient can be expressed as

A transmission coefficient can be written as

The total acoustic energy at the boundary of two layers consists of the incidence intensity, transmitted intensity and reflective intensity expressed as

The reflection coefficient can be shown as the function of the material acoustic impedance and the corresponding angles and can be expressed as

From equation 3.12 and 3.13 it can be shown that

The incident wave will be fully reflected when  $\theta_2 = 90^{\circ}$ . The corresponding  $\theta_1$  is known as the critical angle. According to Snell's law when the wave is propagating from a lower wave velocity medium to higher wave velocity medium, the critical angle can be achieved. The maximum angle of incidence can be expressed as

The critical angle is an important factor for non-invasive measurements through the pipe wall. The critical angle varies intensively in different materials and the value of critical angle must be check for testing materials before the experiments.

#### 3.2.4 Ultrasound transducer

Transducers are used to emit ultrasound signals and transducers developed by Met Flow, SA has been used by most of the studied authors (Wiklund 2007, Birkhofer, 2007, Kotze 2007). Transducer frequencies are available for 0.5, 1, 2, 4 and 8 MHz. The ultrasound generated by the transducer is divided into two fields- near field and far field. In the near field, measurements are avoided because ultrasound measurements are unstable in this region. The acoustic sound field is irregular here and acoustic waves

oscillate along the axis of the propagation. The near field region starts in front of the transducer and continues until the maximum acoustic intensity. The near field distance can be expressed as

where

N = near field distance (m) D = active element diameter (m)  $f_0$  = basic ultrasound frequency (Hz) c = sound velocity (m/s)

The acoustic wave propagation from an ultrasound transducer is shown in Figure 3.3



Figure 3.3 Schematic diagram of the sound wave generation from an ultrasound transducer (Met-Flow, 2000)

*Figur 3.3 Schematiskt diagram av ljudvåg från en ultraljuds givare (Met-Flow, 2000)* The near field distance, N, is the natural focal point of the transducer.

Transducers are fixed in the flow adapter in order to measure the pipe flow with as low interference as possible. Transducers can be set with direct contact with the liquid material. Different kinds of transducer setups for non-invasive measurements used in different publications are as follows (Wiklund, 2007).

Several ultrasound transducers, 1-3 can be used and the data's are combined. For noninvasive measurement an acoustic coupling material has to be used. Small errors in doppler angle determination can lead to a large error when all transducer's data's are combined. Transducers can be mounted in the pipe through small as shown in Figure 3.4. They are installed in such a manner that the near field region is equal to the distance between the transducer and the pipe wall. This is done to avoid measurements in near field zone where it is irregular and not fully developed. If the transducer is mounted inside the pipe wall then there will be loss of ultrasound energy and the doppler angle will be incorrect due to the refraction of ultrasound wave in the pipe wall. It has found that it is possible to achieve good results when the transducer is in direct contact with the fluid. Two transducers are used in the opposite direction to also perform acoustic measurements in addition to the velocity profiles.

A thin wall membrane, e.g. polymethyl methacrylate (PMMA), can be used in the near field region to separate the transducers from the fluid inside the pipe.



Figure 3.4 Schematic diagram of flow adapter cell with two transducers Figur 3.4 Schematiskt diagram av flödes adapter med två givare

### 4. PRINCIPLES OF ULTRASOUND VELOCITY PROFILING (UVP)

#### 4.1 Ultrasound doppler theory

Using sound waves for measuring the distance between two points was invented by mankind due to their own need for measuring distances and this method has been continuously developed since the last century. Bats use sound waves for their navigation and to measure the distances to other flying objects. This method involves transmitting sound waves in a medium and measuring the time required to propagate and coming back from the reflecting surface. This type of phenomenon is referred as 'sonar'. The idea of using acoustic measurements first arose for detecting icebergs after the tragic accident of Titanic in 1912. During the first and second world war lots of military and naval applications of ultrasonic wave and electromagnetic waves, which also use the same principle, were found. A powerful ultrasonic echo sounding device 'hydrophone' was introduces and it was the base of medical pulse-echo sonar. Radar was developed by using the radio detection and ranging of electromagnetic waves. Invention of the methodology of non-destructive material testing based on 'sonar' is presently one of the most frequently used applications (Wiklund, 2003).

In the medical field for blood flow monitoring an instrument was commercially marketed by Novamed, SA, Switzerland. It was possible to record and analyze a large number of signals along the measuring axis and hence instantaneous velocity profiles could be plotted. Takeda used this type of instrument to find out the possibility for general flow measurement of fluids. He found it very promising and the principles are in detail given by Takeda (1986). The limitation with this instrument was that only a limited number of channel/gates were available in which the received echo signal could be stored and analyzed. Consequently, the resolution of the velocity profiles was not good enough. Further work was performed by Takeda and this led to the development of the ultrasound velocity profile monitor (UVP) and the windowing function patented by Takeda (Takeda, 1989, 1991). The UVP monitor is for example, marketed by Met-Flow, Switzerland who also has made further developments on the equipment.

#### The Doppler Effect

The Doppler effect is named after an Austrian physicist, Christian J. Doppler, who first noticed it and gave a lecture on this phenomenon in 1842. When an observer and the source from where the sound wave is originating are in motion with respect to each other, the frequency of the wave to the observer is different than the source. This phenomenon is said to be Doppler shifted and known as the 'Doppler effect'. It is valid not only for sound waves but also for electromagnetic waves, microwaves, radio waves and visible light. The change of frequency with motion of the source and observer can

be illustrated easily by the whistle of a train and an observer. If the train and the observer both are static then the driver and the observer both will hear the sound with same frequency and no Doppler shift will occur. But if the train and the observer are in relative motion to each other then a different frequency will be observed and the sound intensity will be changed. The basic frequency of a ultrasonic wave can be expressed by the following equation

$$f = \frac{c}{\lambda}$$
 .....4.1

where

f = emitted frequency (Hz) c = velocity of sound wave in the medium (m/s)  $\lambda =$  wavelength of the emitted ultrasound wave (m)

The general Doppler equation when the source and observer are both moving in the direction of wave propagation can be expressed by the following equation

$$f_{observer} = f_{source} \left[ \frac{c - v_{observer}}{c - v_{source}} \right]$$
 .....4.2

where

 $v_{observer}$  and  $v_{source}$  are the velocity of the observer and source in the direction of wave propagation, c is the velocity of sound wave in that medium. If the source and observer are moving in the same direction then the values of  $v_{observer}$  and  $v_{source}$  are positive and they are moving in opposite direction then the values are negative.

#### 4.2 Ultrasound doppler velocimetry (UDV)

#### 4.2.1 General principles

The ultrasound pulsed Doppler velocity profiling technique was originally developed in medical engineering to measure the flow of blood in the human body (Takeda, 1985). The ultrasound is reflected from the surface of the blood particles and by using the principle of the Doppler effect, the particle distribution as well as the velocity profile in a blood vessel can be determined. This method was subsequently extended by Takeda for application in other fields of engineering and the principles are in detail discussed in his literatures (Takeda 1985, 1990, 1995).

The principles of ultrasound doppler velocimetry are shown in Figure 4.1. Here the ultrasound transducer is placed at an angle  $\theta$  with respect to the pipe wall. It emits ultrasound waves and also works as a receiver. When the ultrasound waves hit a particle, some portion of the ultrasound energy scatters and the echoes come back to the transducer.



Position :  $x = \frac{c\tau}{2}$  Velocity:  $v_x = f_D c/2f_0$ 

Figure 4.1 Principles of ultrasound doppler velocimetry (a) transducer emitting ultrasound wave (b) received ultrasound signal (c) velocity profile (Takeda, 1995)

Figur 4.1 Principer för doppler ultraljudsmätning (a) givare för ultraljud (b) erhållen ultraljudssignal (c) hastighetsprofil (Takeda, 1995)

In Figure 4.2, the transmission and reflection of an ultrasound wave from the moving particles inside the acoustic beam are shown.



Figure 4.2 Transmission and reflection from a moving particle inside the acoustic beam (Ouriev, 2000)

# Figure 4.2 Överföring och reflektion från en partikel i rörelse inom en akusisk signal (Ouriev, 2000)

The transducer is static with respect to the reflector particle and if the particle is moving with non-zero velocity into the acoustic wave, then doppler shift occurs. The received signal frequency is doppler shifted and can be expressed as

where

 $f_r$  is received frequency,  $f_e$  (or  $f_0$ ) is the emitted frequency and  $v_{TP}$  is the velocity of the target particle. In Figure 4.1, the ultrasound wave transmission and the reflected echo receiving is shown. After The second doppler shift the velocity of the moving reflector particles can be expressed as

Here the doppler shifted frequency  $f_d$  is the difference between the emitted frequency and the received frequency. The time interval between two consecutive echoes is measured. If the time interval is t between the emission of the pulse and the reception of the backscattered echo, then the distance of the moving particle from the transducer, x along the measuring axis can be given as

The echoes are amplified to come over the attenuation of ultrasound energy loss due to the pipe wall and the liquid materials. The doppler shift frequencies are measured continuously and a velocity profile with velocity v can be obtained as a function of x.

Several doppler shifted echoes will be received in a certain time period. This very small time period should be set before the experiment and is known as a 'gate'. Each gate gives a single value of velocity which is measured from the mean value of total doppler energy of that certain time. The gate is closed after each transmission and the next pulse is transmitted after the previous one has reached its maximum depth. This is known as pulse repetition frequency and the process is repeated until enough gates are available for a complete velocity profile. Windowing function is used to fix the measurement volume and the distance from each other. To obtain a good resolution a large number of gates are desirable. In Figure 4.3, a schematic diagram of a measuring window is shown. In UVP-DUO-MX from Met-Flow SA, Switzerland it is possible to choose upto 2048 gates, as required.



Figure 4.3 Diagram of measuring window in ultrasound beam (Ouriev, 2000) Figur 4.3 Diagram over "mätfönstret" in en ultraljudssignal (Ouriev, 2000)

The sampling frequency and the aliasing

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Analog data is received in pulsed doppler ultrasound instruments and the analog signal is converted to digital signal by sampling the signal in certain points. The sampling frequency is the sampling rate per unit time and it is determined from the sampling interval, which is the time between two successive pulse emissions. It is also known as the pulse repetition frequency,  $F_{prf}$  (Wiklund, 2003). The maximum measurable frequency is determined by the theorem of 'Nyquist frequency'. According to this theorem the maximum measurable frequency is less than or equal to the half of the sampling frequency, expressed as

Here  $f_{max}$  is the Nyquist frequency. If the measured frequency is higher than the Nyquist frequency then the lower frequency regions will be overlapped and it will cause distortion. This effect is called aliasing.

#### Maximum depth and maximum velocity

The maximum depth of the ultrasound wave is limited by pulse repeated frequency,  $F_{prf}$  and signal to noise ratio (SNR). The ultrasound echo has to be reflected and then come back to the transducer before emitting a new pulse. The maximum measurable depth is expressed as

Here  $P_{max}$  is the maximum measurable depth, c is the velocity of transmitted pulse in the fluid medium. Consequently for a larger depth, lower frequencies should be used.

Since the maximum measurable doppler shift is limited by Nyquist theorem, the maximum velocity is also limited. The maximum detectable velocity is expressed as

Subsequently the limiting condition can be obtained from equation 4.7 and 4.8, expressed as

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We can see that for constant pulse velocity and emitted frequencies, the maximum depth and velocity is dependent on each other (Takeda, 1991). In higher velocities the penetration depth will reduce and vice versa. However higher velocities can be achieved with the same penetration depth by reducing the transducer frequency.

# Doppler shifted frequency or time-phase lag

The UVP method does not use the doppler shifted frequency  $(f_d)$  but measure the timephase lag for the reflection of the emitted US wave for each particle. The pulse repetition frequency  $(F_{prf})$  is used to determine the velocity of the particles. There is an ongoing debate that if it should be called a doppler shifted method.

# Doppler angle

The Doppler angle is the angle between the ultrasound beam and the direction of the flowing particles inside the pipe. As the ultrasound doppler instruments measure velocities along the ultrasound beam axis, it should be multiplied by a factor  $1/\cos\theta$  in order to achieve the velocity in the direction of the main flow. An illustration of the doppler angle is shown in Figure 4.4 where both particles are moving with the same velocity, implying that the travelled distance  $\Delta s$  is the same for both cases.  $\Delta d_1$  and  $\Delta d_2$  are the distance travelled, relative to the beam direction. If the doppler angle is lower, as we can see in Figure 4.4,  $\theta_1 < \theta_2$ , the perpendicular distance  $h_1$  is larger than  $h_2$ . A longer perpendicular distance will cause a velocity spread for all the particles inside the beam and generate a spectrum of doppler shift frequencies.



Figure 4.4 Variation of the velocity spread of the particles with respect to the doppler angle  $\theta_1 \& \theta_2$  (Ouriev, 2000)

Figur 4.4 Variation av hastighetsspridning på partiklarna I förhållande till Doppler vinklarna  $\theta_1 \& \theta_2$ (Ouriev, 2000)

The accuracy of the measured velocity is dependent on the pulse doppler spectrum and a narrow spectrum is desirable. Based on available literature, a doppler angle of  $60^{0}$ - $80^{0}$  is optimum for the accuracy of velocity estimation (Birkhofer, 2007).

# 4.2.2 Velocity estimation using time/frequency domain based signal processing

Doppler shift frequencies are obtained continuously in order to obtain instantaneous velocity profiles. The change of phase between two consecutive pulses is measured to determine the velocity. Multiple vectors are acquired along a single scan line while the transducer is stationary and the change in phase at every channel along the scan line is calculated. Most commercially available ultrasound equipments are using time domain method. The mean frequency of each channel can be estimated in time domain using the cross correlation of two consecutive pulse emissions.

The advantage of the time domain is that it does not require very fast electronics. There are some difficulties in detecting the doppler shift from a single echo for the instruments based on pulse doppler method. As lots of echo receptions are required to obtain the full profile, the time resolution is limited to 10 ms. Time domain algorithms are superior to frequency domain but does not provide the whole spectra. Not so much information about the quality of the measurements are available in time domain which means that the time domain method is found disadvantageous for real time and high time resolution equipments. From frequency domain one can have the spectral distribution of the velocity profile implying that it is actually known in what volume the velocity profiles are measured and the quality of the measurement.

In frequency domain, frequency spectrum is obtained from demodulated echo amplitude (DMEA) using fast Fourier transformation (FFT). The average doppler shift frequency at each radial point is calculated by weighted averaging of the frequency spectrum. The velocity in the direction of the flow is obtained from the doppler shifted frequencies as shown in equation 4.4.

# 4.2.3 UVP monitor

The UVP monitor is used to measure the velocity profiles continuously during the fluid flow. Ultrasound waves are emitted and received by the transducers and the integrated data acquisition and processing softwares of the UVP monitor delivers the result. It is based on the ultrasound pulse doppler velocimetry principle. Wiklund (2007), Birkhofer (2007), Kotze (2007) used the UVP-Duo instrument of Met-Flow, SA which can emit pulses of 1 to 32 cycles with a base frequency of 0.5 MHz, 1 MHz, 2 MHz, 4 MHz and 8 MHz respectively. The emitted voltage can be 30, 60, 90 and 150 V and the maximum pulse repetition frequency is 1024 Hz. It is possible to use up to 2048 gates per channels in order to obtain a good resolution of the velocity profile. The UVP monitor adjusts the attenuation of the received echo signal as it increases with increased depth. This is possible by using an amplification procedure which follows the exponential law. A detail description of the UVP monitor can be found in Birkhofer (2007) and Met-Flow, 2002.

#### 5. UVP-PD METHOD

#### 5.1 Introduction

The term UVP – PD represents Ultrasound Velocity Profiling using Pressure Difference. It consists of the continuous measurement of velocity profiles using ultrasound pulsed doppler velocity profiling and the pressure difference between two certain points. The concept of combining UVP with pressure difference was implemented around 1993 when the Met-Flow UVP monitor became commercially available. The concept is described by Muller (1997) and it is discussed in other literatures, such as Brunn et al (1993), Muller et al (1997) etc. A large number of publications were presented by Ouriev (2000) and recent developments were performed by Wiklund (2007) and Birkhofer (2007).

#### 5.2 Principles of the UVP-PD method

The basic principles of the UVP-PD method come from the capillary viscometry concept originating from a force balance over a cylindrical fluid element in fully developed, steady state, laminar pipe flow. The relationship between the velocity distribution, shear stress, shear rate and (shear rate dependent) viscosity can be shown by Figure 5.1



Figure 5.1 Link between velocity distribution, shear stress, shear rate and viscosity for a Newtonian fluid (n=1) and shear thinning fluid (n<1)

Figur 5.1 Sambandmellan hastighetsfördelning, skjuvspänning, deformationshastighet och viskositet för en Newtonsk vätska (n=1) och en skjuvförtunnande vätska (n<1)

Examples of a Newtonian and a shear thinning fluid are shown in the Figure 5.1 for five moving particles (reflectors) inside a pipe. In the first segment we can see the velocity distribution, as measured by UVP over the diameter of the pipe. In the second segment

the distribution of the shear stress, as measured by the pressure difference (PD) and independent of the velocity profiling is shown. The third segment shows the distribution of the shear rate accross the pipe diameter which is dependent on the first derivative (dv/dr) of the velocity profiles. In the fourth segment the viscosity is shown which is derived from the flow curve, i.e. shear stress vs shear rate.

# 5.3 Data acquisition and software

The velocity in different points of the pipe is obtained from the velocity profile and the pressure difference, measured by the pressure sensors. By using, e.g. rheological models, the flow curve and other rheological parameters are determined. In general, the UVP-PD setup consists of an UVP DUO monitor, flow adapter with ultrasound transducers, highly accurate pressure sensors for measuring the pressure difference, digital oscilloscope, mass flow meter, volumetric flow meters, water tank and a pump. A pc with data acquisition software is connected and the data acquisition and processing steps are shown in Figure 5.2. The data acquisition steps and the UVP-PD setup varies depending on the type of experiment. A MATLAB (Math Works, Natic, MA, USA) based application with Graphical User Interface (GUI) has been developed by Wiklund et al (2007) for the data acquisition and processing. The tasks that can be performed by this software as shown in Figure 5.2:



Figure 5.2 Data acquisition and processing flowchart of the UVP-PD method (Wiklund et al, 2007)

Figur 5.2 Flödesschema för data insamling och bearbetning med UVP-PD metoden (Wiklund et al, 2007) The data is acquired and stored in the memory as a MATLAB file. It is possible to read a saved MATLAB file associated with the UVP monitor application or raw binary data for post processing. The velocity profile is determined from the power spectra of the baseband signal and the flow curve can be found by using rheological models such as power law, Herschel Bulkley etc or by using the gradient method. The following results can be obtained:

• Power spectra of a single profile or the average of several profiles including the velocity determination

- Velocity profile from time domain
- Rheological properties such as viscosity, yield stress, consistency index such as n, k and plug radius
- Flow curve, i.e. shear stress vs shear rate
- Velocity of sound
- Temperature and flow rate
- Animations of the velocity profile over time

### 5.4 Acoustic characterization

The velocity of sound is an important parameter when using the UVP-PD method and If the measured velocity is incorrect, the velocity of the fluid particles will also be incorrect. Individual methods and flow adapters have been developed to measure the sound velocity both off-line and in-line. In Figure 5.3 a schematic diagram of the setup of ultrasound transducers for measuring acoustic properties off-line is shown. Here two transducers are used, one for emitting ultrasound waves and the other working as a receiver.



Figure 5.3 Schematic diagram of the transducer setup for the acoustic measurements off-line(Wiklund 2007)

# Figur 5.3 Schematiskt diagram över givarkonfigurationen för akustiska mätningar offline (Wiklund 2007)

This technique is known as 'pulse echo time of flight' or 'acoustic time of flight measurement'. The ultrasound pulse emitting transducer is connected to a UVP monitor and the receiver transducer is connected to an oscilloscope and a master PC. The sound velocity is determined from the measured time of flight ( $\Delta t$ ) and the fixed distance between the transducers. The attenuation is measured as peak-peak voltage of transmitted ultrasound and it increases with an increasing concentration of solids in the fluid. Distilled water is used as a reference sample to measure the variation of attenuation and peak-peak voltage. The velocity of sound is temperature dependent and increases approximately 50m/s over the temperature range of 25-50<sup>o</sup>C.The acoustic measurements for the UVP-PD setup is in detail described by Wiklund (2007).

# 5.5 Previous studies based on UVP-PD method

This section presents a brief overview of various published research work based on the UVP-PD method, see Table 5.1.
Research Group	Tested Material / Type of work	Published by
University	Aqueous Hydroxypropyl Aqueous Polyacrylamide	Brunn et al, 1993
Erlangen	4000 ppm Aqueous Polyacrylamide	Muller et al, 1997
Nurnberg, Germany	Flow process of Polyacrylamide solution	Wunderlich and Brunn, 1999
	Body lotion	Brunn et al, 2004
	Feasibility studies of UVP-PD method, Semester Work	Cantz, 1994
ETH Zurich	Feasibility studies of UVP-PD method, Diploma Work	Drost and Wagner, 1994
	PhD Thesis	Ouriev, 2000
	Chocolate crystallization process	Ouriev and Windhab, 1999
	Fat crystallization	Ouriev et al, 1999
	Corn starch in glucose syrup diluted by water (Shear thickening fluid)	Ouriev, 2002
	Time averaged flow mapping of shear thinning and shear thickening suspensions	Ouriev and Windhab, 2003
	Transient flow and pressure driven shear flow of highly concentrated suspension	Ouriev and Windhab, 2002,2003
	Chocolate suspension including cocoa butter with 60% solid in pre-crystallization process	Ouriev et al, 2004
	PhD Thesis on model and cocoa butter suspension	Birkhofer, 2007
	Fat crystallization process of cocoa butter	Birkhofer et al, 2007

Table 5.1 Previous studies based on the UVP-PD methodTabell 5.1 Tidigare studier baserade på UVP-PD metoden

		Г
	Shear thinning surfactant solutions and	Johansson and Wiklund, 2001.
	cellulose suspensions	Wiklund et al, 2001
	Application of UVP-PD in the food industry	Wiklund, 2003
SIK, Sweden	Pulp suspensions	Wiklund et al, 2004
CPUT, South	r uip suspensions	Wiklund et al, 2006
Africa	Food and industrial suspensions	Wiklund and Stading, 2008
		Wiklund et al, 2010
	Methodology of UVP-PD technique	Wiklund et al, 2007
	Mineral suspensions	Kotze <sup>2</sup> , 2007
	Carboxy Methyl Cellulose	Kotze´ et al, 2010
	Microcrystalline cellulose gel, Xantham gum solution, starch gels, polymer melt	Powell et al, 2006
	Diced tomatoes suspended in tomato slurries	Dogan et al, 2002
	Flow of tomato concentrated with three different amount of solid content	Dogan et al, 2003
UC Davis, USA	6% (w/w) acid thinned and native corn starch including gel	Dogan et al, 2005a
	Polymer melt suspensions	Dogan et al, 2005b
	65.7 <sup>°</sup> Brix corn syrup and 4.3 <sup>°</sup> Brix tomato juice	Choi et al, 2002
	Matlab based graphical user interface program implemented for UDV based viscometry	Choi et al, 2005

### 6. MATERIALS

Due to ease of preparation and use, wide availability and a relative low cost, cementbased materials are the most commonly used grouts for permeation grouting. Cement can be divided into two main groups, Portland cement and slag cement, with different chemical composition. Depending on the fineness and maximum particle size, cement can be distinguished as standard or micro-fine cement. For grouting purposes, the trend has been directed to achieving as fine cement particles as possible. However, it has been found that very fine cement are difficult to handle and disperse due to the increased interaction between the particles that comes with increasing specific surface.

### 6.1 Micro cement

In this work, a relatively fine cement has been used, Cementa Injektering 30. It has been found that this has superior characteristics with respect to penetrability compared to more fine cements (Draganovic, 2010).

# Particle size distribution

Cementa Injektering 30 has a particle size distribution where 95 percent of the cement particles are less than 30  $\mu$ m in size. The particle size distribution is shown in Figure 6.1.



Figure 6.1 Particle size distribution of Cementa Injektering 30 Figur 6.1 Partikelfördelning för Injektering 30

Properties of micro cement Injektering 30 are shown in Table 6.1

Table 6.1 Properties of Injektering 30 from CementaTabell 6.1 Egenskaper för Injektering 30 från Cementa

Properties	Description
Physical properties	Compact density approximately 3100-3200 kg/m <sup>3</sup> . Bulk density 800-1500 kg/m <sup>3</sup> .
Chemical properties	MgO maximum 5% by weight. $SO_3$ maximum 3.5% by weight and chloride maximum 0.1% by weight.
Setting time	100 minutes

### 6.2 Additive

Cementa SetControl II (SC) is a high performance setting time regulator and dispersing additive that is especially suitable for grouts based on Cementa Injektering 30, Ultrafin 16 and Ultrafin 12. It is based on sulphonated naphthalene polymers and nitrate. The

color of the SetControl II is yellowish brown and its density is 1476 kg/m<sup>3</sup>, pH value approximately 6 and dry content 45%.

# 6.3 Sample preparation

A total number of 8 batches were made using w/c ratio 0.8 and 06 with and without SetControl II (SC). Sample preparations for the different batches are shown in Table 6.2. The mixing time in the high speed mixer was 4 minutes.

Batch Number	w/c ratio	SetControl II (% by weight)
1	0.8	-
2	0.6	2
3	0.8	-
4	0.6	-
5	0.8	-
6	0.8	2
7	0.6	2
8	0.8	2

Table 6.2 Sample preparation for different w/c ratioTabell 6.2 Provdata för olika w/c tal

# 7. EXPERIMENTAL SET-UP

To investigate the feasibility of the UVP-PD method under practical grouting conditions, it has been important to use as much standard grouting equipment as possible. In order to achieve field like conditions, an ordinary grouting rig has been used, including normal pressure hoses. The grouting rig was placed outside on a parking lot and connected to the UVP-PD equipment, placed under a garage roof, forming a closed experimental flow loop.

# 7.1 Flow loop characteristics

The experimental flow loop consists of the following parts:

- UNIGROUT E22H, grouting rig
- LOGAC, pressure and flow meter
- UVP flow adapter
- Pressure sensors
- Temperature sensor
- Grouting hoses
- Stainless steel pipes

A schematic diagram of the flow loop is shown in Figure 7.1. The pressure sensors were mounted on each side of the 4 MHz transducers and separated by a distance of 1.3 meters. Two 10 m and one 2 m grouting hose pipe of 25 mm inner diameter was used. The inner diameter of the stainless steel pipe for the flow adapter was 22.5 mm. All the measurements were performed in ambient temperature of approximately 21  $^{0}$  C. One ball valve was added close to the agitator to control the flow of the grout mixture when it was returning back to the agitator tank. A lever, was integrated with the mixer to control the flow from the mixer to the agitator. Another ball valve was placed after the pump to control the flow from the agitator to the UVP-PD test section.



Figure 7.1 Schematic diagram of the flow loop Figur 7.1 Schematiskt diagram over försöksuppsättningen

# 7.1.1 UNIGROUT E22H

The UNIGROUT E22H is a complete grouting rig, manufactured by Atlas Copco, consisting of a mixer, agitator, pump, control unit and the necessary hoses. The rig was used to produce a grout with the same properties as would be the case under field conditions.

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# Grout mixer, Cemix 203H

Cemix 203H is a high speed colloidal mixer consisting of a container and impeller. After mixing, the grout is pumped in to the agitator. The volume of the mixer is 200 L and the mixing capacity 0-3  $m^3/h$ .

# Grout agitator, Cemag 402H

Cemag 402H is a slow running agitator consisting of a cylindrical container with angular base and an inclined mixer shaft fitted with two pair of blades. The volume of the agitator is 400L and the rotational speed of the agitator shaft is 60-70 rpm.

# Grout pump, Pumpac

Pumpac is a hydraulic piston pump based on the double acting pump principle. The pump cylinder diameter is 110 mm and the grout flow capacity is up to 0-120 l/min. Two grout pressure setting levels are available, a low pressure range operated at 2-10 bars and a high pressure range at 8-100 bars. In the presented test, the low pressure range was always used as the transducer connection could only sustain up to 20 bar.

The UNIGROUT E22H is shown in Figure 7.2



Figure 7.2 UNIGROUT E22H used for the experimental work Figur 7.2 UNIGROUT E22H som använts för experimenten

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### 7.1.2 LOGAC

An Atlas Copco LOGAC 4000 was used, consisting of a computer based recording system for storing and sampling data during a grouting operation. The parameters that can be logged and stored on a PC card are flow, pressure, volume, time and real time, recorded on the card at every 10<sup>th</sup> seconds. The CFP meter unit consists of an electromagnetic flow meter and a pressure meter. The flow meter operates in a range of 0-200 l/min with a maximum allowed pressure of 40 bar. The LOGAC device was used as a reference, to compare the flow rate with the one achieved by the UVP method. In the presented case the flow rate was always maintained within 15-30 l/min. The LOGAC experiment is shown in Figure 7.3.



Figure 7.3 LOGAC used for the experimental work Figur 7.3 LOGAC som använts i experimenten

### 7.1.3. UVP-PD instruments

### Flow adapter with ultrasound transducer

In this experiment two 4 MHz ultrasound transducers (TR0405LH-X; Signal-Processing SA, Savigny, Switzerland), high temperature, were fitted with the flow adapter. The flow adapter was made out of stainless steel with an inner diameter of 22.5 mm and

with a pressure limit of 20 bars. The transducers were mounted through cavities in the pipe wall, with a diameter equal to the housing diameter of the transducers. The transducers were installed at a distance equal to the near field distance to avoid this region, where the ultrasound field is highly irregular. The flow adapter and transducer installation used for this experiment is shown in Figure 7.4.

These transducers allow measurements directly from the transducer front, implying that more or less zero velocity at the wall can be recorded. The active and outer diameters of the transducers are 5 mm and 8 mm respectively. The transducers were fixed inside the flow adapter in a horizontal plane, to minimize the sedimentation effect, opposite to each other with a doppler angle (between the flow direction and the transducer axis)



Figure 7.4 Flow adapter with 4MHz transducers used for the experiment Figur 7.4 Flödesadapter med 4 MHz givare som använts i experimenten of 70<sup>0</sup> and 110<sup>0</sup> respectively. The near field distance for these transducers are 7 mm.

### UVP DUO

The velocity profile measurements were performed with a pulser/receiver instrument, UVP-DUO MX (Met-Flow, SA, Lusanne, Switzerland) model with a multiplexer. The instrument firmware and driver software were modified to allow access to the demodulated echo amplitude data (DMEA; raw data which is not possible to obtain using the standard instruments). The UVP DUO instrument and other hardware devices were connected to a master PC via Ethernet and a DAQ card (National Instruments, ABB). A MatLab based software with graphical user interface (Rheoflow) was used to control all hardware devices for data acquisition, signal processing, visualization of the data and real time monitoring of the rheological properties. UVP data acquisition was implemented using an active X library (Met-Flow, SA). A high speed digitizer card (Agilent Acquiris) was used as an integral part of the data acquisition scheme, enabling simultaneous measurements of the velocity profiles and acoustic properties.

### Pressure sensors

Two differential pressure sensors (ABB 256DS, ETP80,ABB Automation Technology Products AB, Sollentuna, Sweden), 45V DC, 20 mA, PS 40 bar were used to measure the pressure difference over a distance of 1.3.

### UVP-PD experimental parameters

The UVP-PD experimental parameters used for this experiment are shown in Table 7.1.

# Table 7.1 Experimental parameters for UVP-PDTabell 7.1 Experiment parametrar för UVP-PD

4 2-4
2-4
50-150
5
0.37-0.74
128-512
104-115

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Doppler angle ( <sup>0</sup> )	70/110
Number of Time domain profiles (steady state flow)	500
Number of FFT profiles (steady state flow)	30
Sampling time for pressure difference (ms)	100
Temperature ( <sup>0</sup> C)	16-20
Length of pipe between pressure sensors (m)	1.3
Volumetric flow rate range (l/min)	15-30

# 7.2 Experimental Procedure

The experiments were performed in the following steps:

1. The mixer was filled with water and the whole system was operated with water for several minutes in order to calibrate the distance between the transducers.

2. The mixer was filled with the required amount of water for a certain water cement ratio. Cement was added after the high speed mixer was switched on.

3. The mixing was performed for 4 minutes. SetControl II, if used, was added after mixing for 2 minutes.

4. The mixture was shifted from the high speed mixer to the agitator.

5. The pressure level was set for the pump. For these experiments a low range of 1-4 bar pressure was always used.

6. The mixture was pumped through the system including the LOGAC and the UVP-PD flow adapter. A ball valve was used to regulate the flow rate and keep it between 15-30 l/min.

7. The pressure and flow rate was continuously recorded by the LOGAC at every 10 seconds'.

After starting, the UVP settings were altered and tuned to get the best result. Maximum penetration depth and frequency was optimized to change the pulse repetitions frequency and to measure the prevailing flow rate.

### 7.3 Off-line measurement instrument

#### 7.3.1. ARES-G2 rheometer

An ARES-G2 rheometer was used to verify the in-line measurements. It is based on the deformation controlled design, where deformation or shear rate are applied to the same sample via rotating outer cylinder or plate at the bottom. ARES G2 from TA Instruments is a controlled stress, direct strain and controlled shear rate rheometer. ARES G2 uses smart swap geometries with automatic detection including an integrated magnetic cylinder that stores unique geometry information. The information is automatically read and the software is configured with appropriate parameters (type, dimension, material, etc).

Experimental parameters are shown in Table 7.2

Table 7.2 Experimental parameters for ARES-G2 Rheometer	
Tabell 7.2 Försöksparametrar för ARES-G2 Rheometer	

Parameter	Remarks
Geometry	27mm DIN, Concentric cylinder
Temperature( <sup>0</sup> C)	20
Soak time (s)	10
Number of flow sweeps	2 (low to high shear rate and vice versa)
Shear rate(1/s)	0.1-1000
Points per decade	10
Equilibration time(s)	5
Averaging time (s)	5

### 7.3.2. Brookfield DV-II+ pro (LV) viscometer

Brookfield  $DV-II^+$  pro (LV) viscometer from Brookfield Engineering was used for offline measurements. This experiment was performed at the laboratory of KTH. The experimental parameters are shown in Table 7.3. Measurements were done after mixing the sample for 4 minutes and after agitating for 2 hours respectively. Results of Brookfield viscometer are shown in Appendix A.

Parameter	Remarks
Geometry	Concentric cylinder
Spindle	SC4-31
Temperature( <sup>0</sup> C)	25-27
Shear rate (1/s)	0.1-200
Speed (rpm)	5-200

Table 7.3 Experimental parameters for Brookfield viscometerTabell 7.3 Försöksparametrar för Brookfield viscometer

### 8. RESULTS

In this chapter in-line measured velocity profiles and the derived rheological parameters for cement grouts, with water cement ratio 0.6 and 0.8, are presented. As this is a feasibility study, the objective was to find out whether the UVP-PD method works for cement grouts under field like conditions. The primary objective was to measure the velocity profiles of cement grouts directly in-line. Other objectives were to determine the rheological parameters, using mathematical models and the gradient method and to determine the volumetric flow rate. Off-line measurements were also performed using conventional rheometers. In order to keep the conditions same as in the field, a standard grouting rig UNIGROUT E22H and LOGAC flow/pressure meter was used. 4 MHz ultrasound transducers were used as a trial for this setup as it has been found optimum for food suspensions previously used by other researchers. For controlling the flow, ball valves were used in this setup which was found very rough and it was difficult to change the flow rate in small increments. Acoustic off-line characterization measurements were performed to study the pre-feasibility of UVP-PD method for cement based grots prior to the UNIGROUT trials.

The results are shown in the order the tests were performed. As the accuracy of the velocity profiles and derived rheological parameters depend on the accuracy of the velocity of sound in cement grouts, this is presented first. Subsequently the velocity profiles, flow curves and rheological properties (viscosity, yield stress) are shown. Results obtained from the off-line rotational viscometers are also presented for comparison.

### 8.1 Acoustic measurements

An acoustic measurement is the first step to study the feasibility of ultrasound velocity profiling for a new fluid. As most of the mathematical derivations (discussed in chapter 4) are based on the velocity of sound in the sample fluid, it is important to know whether it is possible to measure the velocity of sound in the cement grout. It is also important to study the attenuation of the signal (absorption of energy in the suspension), penetration depth and to make sure that the signal is not distorted. A pre-feasibility study of acoustic characterization with water cement ratio 0.6 and 0.8 was performed using the off-line flow adapter cell to measure the sound velocity in the cement grout (Wiklund, 2009). The results are shown in Figure 8.1 and 8.2 and summarized in Table 8.1. The ultrasound transducer acting as a pulser can emit high voltage electrical pulses and generate high frequency ultrasonic energy. The sound energy is propagated through the cement grouts and received by another transducer which acts as a receiver. In Figure 8.1 and 8.2 the amplitude of the received ultrasound wave signal strength is shown as a function of data point which represents the distance from the transducer.



Figure 8.1 Received waveform pulse for w/c ratio 0.8 (Wiklund, 2009) Figur 8.1 Erhållen signal för w/c tal 0.8 (Wiklund, 2009)



Figure 8.2 Received waveform pulse for w/c ratio 0.6 (Wiklund, 2009) Figur 8.2 Erhållen signal för w/c tal 0.6 (Wiklund, 2009)

Sample	Cement grout	Cement grout	Distilled water	Cement
	(off-line)	(off-line)	(off-line)	grout
				(in-line)
w/c ratio (by	0.8	0.6	N/A	0.8
cement weight)				
Frequency (MHz)	4	4	4	4
Voltage (V)	150	150	150	150
Gain (-)	5-7	5-7	5-7	5-7
Cycles (#)	4	4	4	3
Sound Velocity	1525.6	1540.1	1485.4	1487.3
(m/s)				
Time-of-flight	0.093	0.092	0.095	0.018
(ms)				
Transducer	14.19	14.19	14.19	27.77
Distance (mm)				
Peak-Peak voltage	0.102	0.042	35.31	0.053
(V)				
Temperature ( <sup>0</sup> C)	24.8	25.5	20.3	21.1

Table 8.1 Acoustic parameters for off-line and in-line measurementsTabell 8.1 Akustiska parametrar för off-line och in-line mätningar

The sound velocity of distilled water was also measured in the flow adapter cell to compare with the results obtained for the cement grouts. The waveforms were captured without noticeable distortions. During off-line acoustic characterization the maximum ultrasound baseband frequency for cement grouts used were found to be 4 MHz. Penetration depths were found less than half of the pipe using baseband frequencies more than 4 MHz. The optimum voltage was 150V due to strong attenuation of the signal. Both for the cement grouts and distilled water the complete waveform was captured after propagating a distance of 14.19 mm, calibrated with micrometer precision which was the distance between the transducers inside the off-line cell. The peak-peak voltage, i.e. the difference in amplitude, for cement grout was very low compared with distilled water which means that the amplitude of the received ultrasound wave through the cement grout is much more attenuating for distilled water. High attenuation in low w/c ratio was caused due to increased multiple scattering of ultrasound with increasing concentration of solids. The velocity of sound was lower for in-line measurement than the off-line measurement. During off-line measurements, sedimentation occurs in the flow cell so the velocity of sound is higher than the in-line measurements.

Within the scope of this work, acoustic measurements were also performed directly inline to measure the sound velocity in the cement grout. First the distance between the transducer is calibrated by using tap water as the sound velocity in water is known. Then the grout mixture was circulated through the pipe system and the velocity of sound was measured. During in-line measurements the transducers were moved and the flow adapter was shaken to avoid flocculation of the cement grouts. It was not possible to calibrate the transducer distance with distilled water as cement grout was circulated in the flow loop. The transducer distance was not correct all the time and it caused lower velocity of sound during the in-line measurements. The different acoustic measurement parameters and the results are summarized in Table 8.1. The performed acoustic measurements show that it is possible to measure the velocity of sound in cement grouts.

Figure 8.3 shows received waveform pulse for w/c ratio 0.8. The resulting peak-peak voltage was found to be ~0.053 V and the measured sound velocity was 1487.3 m/s. The complete wave form was captured without noticeable distortion. The velocity of sound changes with the temperature. The properties of the cement grout also changes with time due to hydration. The complete waveform was achieved over a distance of 27.77 mm. The diameter of the flow cell was 22.5 mm and the distance between the transducer was 27.77 mm calibrated with micrometer precision. Both of the measurements are showing the same waveform and there is only little distortion near the far wall. That means velocity of sound did not change much within time of the measurements and it was possible to obtain the sound velocity directly in-line. The lower peak- peak voltage indicates the highly attenuating behavior of the cement grout.



Figure 8.3 Received wave form pulse for W/C ratio 0.8 Figur 8.3 Erhållen signal för w/c tal 0.8

In case of in-line measurements the tone burst was achieved at 4 cycles/pulse but with a lower spatial resolution. The tone burst is generated to transmit burst of acoustic energy in to a test section, receive the resulting signals and alter and analyze the received signals. More than 4 cycles/pulse was not tried due to overlapping of the channels as the channel volume increases with increasing number of cycles/pulse. At 2 cycles/pulse the penetration depth was lower than half of the pipe. With decreasing number of cycles/pulse, the measured volume was smaller but the penetration depth was decreased. At 3 cycles/pulse undistorted signal were achieved with a penetration depth of at least half of the pipe which was sufficient for the analysis of the velocity profiles and the rheological parameters. It was thus concluded that 3 cycles/pulse were optimum for the 4MHz transducers. The performed in-line acoustic measurements show that it is also possible to measure the velocity of sound in cement grouts under field conditions.

### 8.2 Velocity profiles measured by UVP

In this section, velocity profiles measured by the ultrasound velocity profiling (UVP) techniques are shown. The velocity profiles were measured for cement grouts with a water cement ratio of 0.6 and 0.8, both with and without SetControl II as additive. SetControl II was used as a high performance setting time regulator and dispersing additive which is suitable for grouts based on Cementa Injektering 30. SetControl II dosages of 2% of the cement weight were used as it is commonly used in field applications. The velocity profiles were captured within a time period of 1-3 hours from

mixing of the grouts. The main objective was to investigate whether it is feasible to achieve the velocity profiles for different w/c ratios under field conditions and it was not in the scope of work to compare the measurements with respect to time and additives. These profiles were measured for different samples and in different timings so there were no fixed time intervals of data collection for each measurement. The samples and time after mixing used for velocity profile measurements and corresponding determination of rheological parameters are shown in Table 8.2. Firstly, all the velocity profiles obtained over a certain period of time are shown in Figure 8.4 and Figure 8.6 and subsequently, the average of the profiles for that certain time period is determined and presented in Figure 8.5 and Figure 8.7.

Table 8.2 Samples used for measuring velocity profile and rheological parametersTabell 8.2 Prov för mätning av hastihetsprofil och reologiska parametrar

w/c ratio of cement grout	SetControl II used by cement weight (%)	Time after cement mixing (min)
0.8	-	30
0.8	2	150
0.6	-	60
0.6	2	30

By UVP the instantaneous doppler shift frequencies are measured at various times after the emission of the initial pulse and in this way a complete velocity profile is obtained as the local velocity is a function of the position of the individual cement particle. The velocity profiles are presented with respect to channel number which represents an individual measuring volume as shown in Figure 4.2. The returning doppler shifted echo signals are received in a small time interval which is known as 'channel'. According to the desired time/depth the time period for each channel is specified. Consequently from these channels the radial position of the cement particle inside the pipe is determined.

Examples of velocity profiles, obtained by the Ultrasound Velocity profiling (UVP) method, are shown in Figure 8.4 and 8.6 for water cement ratio 0.8 and 0.6, respectively. In Figure 8.4, 1024 captured velocity profiles are shown for a time period of 3 minutes. These velocity profiles show the actual velocity distribution over the radial distance of the pipe for a sampling period of 169 ms and by using 4 MHz transducers with 4 cycles and 512 pulse repetition frequency and a voltage of 150. The profile near the far wall was distorted due to the loss of signal but up to half of the profile a very good signal was achieved and due to pipe symmetry, this was enough to

determine the required properties. Here it can be seen that the average velocity changed from 0.15 m/s to nearly 0.8 m/s.

As a piston pump was used, a sudden change in flow rate due to the pulsation of the pump is observed. Due to the fluctuation of the flow rate, the velocity profiles were randomly averaged into 3 regions up to 0.3 m/s, from 0.3-0.5 m/s and above 0.5 m/s in order to facilitate the presentation. It can also be seen that different shapes of the velocity profiles prevail, depending on the pump characteristics.



Figure 8.4 Captured velocity profiles for w/c ratio 0.8, 90 minutes after mixing Figur 8.4 Erhållna hastighetsprofiler för w/c tal 0.8, 90 minuter efter blandning

As shown in Figure 8.5, a large number of the velocity profiles were in the range above 0.5 m/s, which implies that the total average was close to the average of the  $3^{rd}$  region of 'above 0.5 m/s'.



Figure 8.5 Average velocity profiles for w/c ratio 0.8, 90 minutes after mixing

Figur 8.5 Medelhastighetsprofiler för w/c tal 0.8, 90 minuter efter blandning

In Figure 8.6, 256 captured velocity profiles are shown for a time period of 30 seconds. These velocity profiles represent the actual velocity distribution over the radial distance of the pipe for a sampling period of 117 ms and by using 4 MHz transducers with 2 cycles and 512 Hz pulse repetition frequency.



Figure 8.6 Captured velocity profiles for w/c ratio 0.6, 60 minutes after mixing Figur 8.6 Erhållna hastighetsprofiler för w/c tal 0.6, 60 minuter efter blandning

As shown in Figure 8.7, a large number of the velocity profiles were in the range above 1.2 m/s, which implies that the total average was close to the average of the  $3^{rd}$  region of 'above 1.2 m/s'.



# Figure 8.7 Average velocity profiles for w/c ratio 0.6, 60 minutes after mixing Figur 8.7 Medelhastighetsprofil för w/c tal 0.6, 60 minuter efter blandning

From Figure 8.6 it can be seen that a good signal was achieved up to half of the pipe which was sufficient to obtain the rheological parameters. Fluctuations of the peak velocities were observed from 0.4 m/s to 1.6 m/s. The effect of attenuation was clearly visible here. Signal was lost after the half of the pipe hence the apparent velocity drop in the graph. This is expected for the thicker grout. The velocity profiles were divided into 3 regions and shown in Figure 8.7.

From the Figure 8.4 and 8.6 it can be seen that a high volumetric flow rate was achieved for the thicker grout. The reason for this was that a higher pump pressure was used during the measurement of the w/c ratio 0.6.

In all measurements the velocities were generally unstable, due to the dual-piston pump action. If the shape of the velocity profile was changing that means the shear rate was also changing. From the velocity profiles a plug flow situation can be directly identified and the radius of the plug at the middle of the pipe can be determined.

# 8.2.1 In-line velocity profiles fitted with Herschel-Bulkley model

In order to obtain rheological data, measured velocity profiles and pressure drop data were fitted to the H-B model. In all cases the velocity profile and pressure drop was measured, averaged over a specific time interval and then fitted to the H-B model. It was possible to obtain a very good fit for most of the data. The radial position zero indicates the middle of the pipe where the velocity was maximum and the zero velocity indicates the wall of the pipe. The flow index n, consistency index k, plug radius  $R_0$  and the coefficient of determination  $R^2$  is also shown with the fitted profiles. Figure 8.8 shows the measured velocity profile and the fitted H-B velocity profile for w/c ratio 0.8 without SetControl II. Here the fitted in-line profiles are presented according to the time after mixing shown in Table 8.2, i.e., the measurement was taken 30 minutes after mixing the cement grout.



Figure 8.8 Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.8 without SetControl II, 30 minutes after mixing the cement grout

# Figur 8.8 Uppmätt hastighetsprofil och kurvanpassning till H-B modellen för w/c tal 0.8 utan SetControl II, 30 minuter efter blandning

Figure 8.9 shows the measured velocity profile and H-B fitted velocity profile for w/c ratio 0.8 with SetControl. The measurement was taken 150 minutes after mixing of the cement grout.

From Figure 8.8 and 8.9, it can be seen that the flow index, n for w/c ratio 0.8 with SetControl II was lower which means it was showing a more shear thinning behavior. The plug radius was larger in case of w/c ratio 0.8 with SetControl, which means it had a higher yield stress. The reason for this was that the profile was taken 150 minutes after the mixing of the cement grouts which means that the yield stress probably had increased due to hydration of the cement. The profile shape was a little bit distorted in some regions. As cement grout was circulated inside the pipe for a longer period, some cement particles were flocculated inside the pipe and sometimes there were air bubbles. The pipe was shaken randomly to get rid of these air bubbles.



Figure 8.9 Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.8 with SetControl II, 150 minutes after mixing the cement grout

Figur 8.9 Uppmätt hastighetsprofil och kurvanpassning till H-B modellen för w/c tal 0.8 med SetCcontrol II, 150 minuter efter blandning

Figure 8.10 and 8.11 show velocity profiles for w/c ratio 0.6 with and without SetControl, respectively. In Figure 8.10, for w/c ratio 0.6 without SetControl II the velocity was higher than the samples of 0.8 which was due to the application of a higher pressure. Signal quality was dropping near the center of the pipe due to attenuation. A good fit was obtained as the coefficient of determination was 0.993. The measurement was taken 60 minutes after mixing of the cement grout.



Figure 8.10 Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.6 without SetControl II, 60 minutes after mixing the cement grout

Figur 8.10 Uppmätt hastighetsprofil och kurvanpassning till H-B modellen för w/c tal 0.6 utan SetControl II, 60 minuter efter blandning

Figure 8.11 shows the H-B fitted velocity profile for w/c ratio 0.6 with SetControl II. The measurement was taken 30 minutes after mixing of the cement grout. The little distortion in some regions of the measured profile of Figure 8.10 and 8.11 were because of the high fluctuations of the velocity profiles.



*Figure 8.11 Measured velocity profile and H-B fitted profile for w/c ratio 0.6with SetControl II, 30 minutes after mixing the cement grout.* 

Figur 8.11 Uppmätt hastighetsprofil och kurvanpassning till H-B modellen för w/c tal 0.6 med SetControl II, 30 minuter efter blandning

It was slightly more difficult to obtain a good signal with the higher concentration, i.e. lower w/c ratio, due to increased attenuation of the ultrasound signal. The signal was dropping near the center of the pipe in case of w/c ratio 0.6. For achieving higher penetration the transducer frequency can be reduced down to e.g. 2 MHz.

Comparing both cases of w/c ratio of 0.6 and 0.8, the velocity profile shapes were better for w/c 0.8 and a signal penetration up to the center of the pipe was achieved. In the case of w/c ratio 0.6, the signal penetration was nearly up to the center of the pipe. Other transducers should be tried for better penetration. Volumetric flow rates were not identical which implies that they cannot not be readily compared.

### 8.2.2 Rheology by Gradient method

Shear rate dependent viscosities and rheological model parameters can be obtained in two ways. Either from a non linear curve fit of the measured velocity profiles and pressure drop data to suitable rheological models, or directly from the velocity profile and pressure drop using the gradient method. The gradient method is unique for the UVP-PD method and has the advantage that it requires no 'a priori' knowledge of the flow behavior, however it requires high spatial resolution and high quality data (Wiklund et al, 2007). The principle of the gradient method is shown in Figure 8.12. The shear rate can be obtained by taking the gradient of the velocity profile for each radial position in the pipe. The shear stress is determined from the pressure difference, radial distance and distance between the pressure sensors which are all known. From the plug radius that is measured in the graph, the yield stress can be estimated.

Hence, with the gradient method the rheological properties can be directly estimated and the flow curve can be established without using any curve fitting to rheological models.



Figure 8.12 Principles of the gradient method Figur 8.12 Principer för gradient metoden

The gradient method is convenient if the type of fluid is unknown. The disadvantage of this method is that it is very sensitive to noise in the data and the gradient can be difficult to measure and determine. If the noise ratio is high then data will be distorted and hence measured properties will be erroneous

The data processing and fundamental difference between a curve fitting method and gradient method can be principally shown as in Figure 8.13 and 8.14.



Figure 8.13 Flow chart of the data processing of curve fitting procedure Figur 8.13 Flödesschema för kurvanpassnings proceduren



Figure 8.14 Flow chart of the data processing of gradient method Figur 8.14 Flödesschema för gradient metoden

In the curve fitting procedures, there can be errors in the flow data itself and simplification errors while fitting the data to a mathematical model, as the cement grout is neither a Bingham fluid nor a H-B fluid exactly. However in the gradient method the velocity profile is obtained from the beginning without having to use models and curve fittings. In most practical applications the velocity profile is needed and a great advantage of the UVP-PD method is that it measures the velocity profile directly.

### 8.3 Flow curves from in-line measurements

The flow curve (shear stress vs shear rate) for w/c ratio 0.6 and 0.8, with and without SetControl II, is shown in Figure 8.15. The shear rate range was not same for each sample because the measurements were taken at different pressures. The shear stress for w/c ratio 0.6 was higher than that of 0.8 at a specific shear rate, which was expected. However, it should be noted that the in-line measured data were not sampled at identical time and condition which means that they cannot be directly compared.



Figure 8.15 Flow curve for different w/c ratio with and without SetControl II using the H-B model Figur 8.15 Flödeskurvor för olika w/c tal, med och utan SetControl II, och H-B anpassning

The effect of SetControl II is visible here. SetControl II was primarily used to reduce the shear rate dependent viscosity and the yield stress of the cement grout. Here higher shear stress was observed due to the hydration of the cement but also due to the accelerating effect of SetControl II.

The rheological parameters obtained by the H-B model for different w/c ratios are shown in Table 8.3.

Sample Description	Time after mixing (min)	n	K	R <sub>0</sub> (mm)	Yield Stress (Pa)	R <sup>2</sup>
w/c=0.8, No SC	30	0.5	0.34	0.72	0.45	0.99
w/c=0.8, SC 2%	150	0.2	1.93	2.14	2.86	0.97
w/c=0.6, No SC	60	0.3	1.07	1.05	1.3	0.99
w/c=0.6, SC 2%	30	0.4	1.04	1.04	1.64	0.99

Table 8.3 Rheological parameters obtained by the H-B model for different w/c ratioTabell 8.3 Reologiska parametrar erhållna med H-B modellen för olika w/c tal

From the Table 8.3 it can be seen that the time of measurement after mixing were different for each sample. Since the cement grout changes its properties with time during hydration of the cement, it is difficult to make direct comparisons when the measurements are made at different times after mixing. The velocity of sound changes with temperature which means that also differences in temperature will have an effect on the properties. The maximum yield stress was observed in case of w/c ratio 0.8 with SetControl II and the measurement was taken 150 minutes after the mixing of the grouts. The yield stress increases with time so it can be said that here the higher yield stress was most probably due to hydration.

Figure 8.16 shows the flow curve obtained by gradient method and H-B model fitting for w/c ratio 0.6 without SetControl II. At high shear rate both of the procedures gave the same results but at low shear rate there were some deviations. Low shear rate region was at the center of the pipe where the velocity resolution was lowest for the UVP-PD technique and data was distorted due to the noise of multiple scattering near the center of the pipe during in-line measurement.



Figure 8.16 Flow curve obtained by gradient method and H-B fitting for w/c ratio 0.6 without SetControl II

Figur 8.16 Flödeskurva erhållen med gradient metoden och kurvanpassning till H-B modellen, för w/c tal 0.6, utan SetControl II

### 8.4 Shear rate dependent viscosity

Most of the tested grouts show a shear thinning behavior i.e. have a shear rate dependent viscosity. Figure 8.17 shows the distribution of shear rate dependent viscosity as a function of shear rate for different w/c ratio, with and without SetControl II. The sample collection time is shown in Table 8.2.


Figure 8.17 Shear rate dependent viscosity of different w/c ratio with and without SetControl II

Figur 8.17 Deformationshastighetsberoende viskositet för olika w/c tal, med och utan SetControl II

The viscosity was lowest for w/c ratio 0.8 without SetControl II as expected. Due to high fluctuations of pressure the velocity profile shapes were continuously changed. To compare the results one must have a stable flow rate which can be obtained by using e.g., other types of pumps. The in-line result for viscosity is very important for a field application, since it can be used to directly observe how the viscosity is changing with time and with additives.

#### 8.5 Comparison of volumetric flow rate

Volumetric flow rate was continuously measured directly in-line using the UVP-PD method as it is derived from the integral of the velocity profile. The LOGAC device was used as a reference to compare the volumetric flow rate with the in-line measurement. The volumetric flow rate measured in-line and by the LOGAC over the same time period is shown in Figure 8.18. By the UVP-PD method volumetric flow rate was measured in milli-seconds but as the LOGAC is a Coriolia mass flow meter, this was much slower than the UVP-PD method. However, as can be seen in the Figure 8.18, the two methods were showing results in the same order of magnitude. As mentioned earlier, one important findings of this feasibility study and research work was the fluctuation of the flow rate when a piston type pump was used. It was found that the



piston pump creates rapid changes in the flow rate due to the movement and position of the piston.

Figure 8.18 Comparison of volumetric flow rate measurement Figur 8.18 Jämförelse av olika födesmätningar

In Figure 8.18, it can be seen that from UVP-PD measurement the flow rate fluctuated from 15-25 l/min but the LOGAC showed fluctuations from 17-22 l/min. Figure 8.18 also shows that a stable flow was not achieved. In the UVP-PD method it is important to attain a stable flow rate in order to achieve the correct measurements of the velocity profile. This result shows that apart from measuring the rheological properties and flow rate, the UVP-PD method can also be a very good method to measure the efficiency and characteristics of the used pump.

# 8.6 Off-line measurements

Off-line measurements were performed using an ARES G2 rheometer, in order to calibrate the results obtained by the UVP-PD method. Experiments were made with w/c ratio 0.6 and 0.8, with and without SetControl II. The results are shown in Table 8.4.

W/C	Set	Time	Yield	Yield	Viscosity,	Power Law		$\mathbf{R}^2$	H-B		$\mathbf{R}^2$	$\mathbb{R}^2$
Ratio	Control	min	Stress,	Stress	Bingham,	Parameter		Power	Parameter		H-B	Bingh
	(%)		Bingham	H-B,	mPa.s			law				am
			Pa	Ра		n	k		n	k		
0.8	No	30	2.14	0.9	15.9	0.4	0.8	0.98	0.5	0.39	0.98	0.92
0.8	2	180	5.54	1.39	25.9	0.2	3.22	0.96	0.4	0.76	0.97	0.84
0.6	No	60	7.3	1.13	56.8	0.4	3.19	0.98	0.4	2.53	0.97	0.88
0.6	2	110	6.89	0.72	33.1	0.2	3.94	0.97	0.3	3.37	0.98	0.82

# Table 8.4 Off-line measurements with rheometerTabell 8.4 Off-line mätningar med Rheometer

The measurements were taken in shear rate range 0-600/s. From the curve fitting it can be seen that the cement grout of w/c ratio 0.6 and 0.8 follows the Herschel-Bulkley model rather than the Bingham model. The Bingham fit was poor which shows that cement grouts are not really described by this rheological models. The effect of SetControl II cannot be compared as the samples were not measured at identical time and condition. The H-B fitted flow curve obtained by rotational rheometer for w/c ratio 0.8 and 0.6 are shown in Figure 8.19 and 8.20 respectively. The off-line measurements performed with Brookfield viscometer are presented in Appendix A.



*Figure 8.19 Off-line measurement of w/c ratio 0.8 with and without SetControl II Figur 8.19 Off-line mätningar för w/c tal 0.8, med och utan SetControl II* 



*Figure 8.20 Off-line measurement of w/c ratio 0.6 with and without SetControl II Figur 8.20 Off-line mätningar för w/c 0.6, med och utan SetControl II* 

From Figure 8.19 and 8.20 it was found that the shear stress was lower for w/c ratio 0.8 which was expected. The effect of SetControl II was not comparable in case of w/c ratio 0.8 due to hydration effect.

## 9. CONCLUSION AND RECOMMENDATIONS

## 9.1 Conclusion

The results obtained with the UVP-PD method were found to be very promising for direct in- line velocity profile measurements of cement based grouts. The primary objective was to investigate the feasibility of this method for cement based grouts under field like conditions. The results show that it is possible to obtain the velocity profiles for w/c ratio 0.6 and 0.8. Based on this result, It was subsequently possible to determine the rheological properties, such as, viscosity and yield stress. In addition to the rheological models (e.g. Herschel-Bulkley), a non-model approach (Gradient method) was also applied. It was found that with the gradient method the true flow curve (shear stress vs shear rate) can be obtained. With this method, the volumetric flow rate can also be determined, i.e. integrated, from the velocity profiles. One important observation was that the piston pump creates fluctuating flow due to cyclic pressure and the effect of this on grouting efficiency needs to be investigated further.

A standard grouting equipment UNIGROUT E22H and flow/pressure meter LOGAC was used in the flow loop. This shows that the UVP-PD technology can also be used in the field after further development and optimization. The UVP-PD method can thus be a very effective tool for quality control in addition to the determination of the rheological properties of the used grouts..

From all the results of the present study, it can be concluded that it is feasible to measure the velocity profiles and determine the rheological properties of cement grouts directly in-line under field conditions. This is a totally new construction industry application for the UVP-PD method and further research should be carried out to establish this technology for field usage.

## 9.2 Recommendations

In order to apply the UVP-PD method for grouting and make it increasingly compatible with cement based grouts, several improvements should be made. Examples of such improvements are presented below.

- To obtain a higher penetration depth while using high concentration cement grouts, i.e., with low w/c ratio, ultrasound transducers of 2 MHz should be tried.
- The transducer design should be optimized. The beam diameter of the transducer used in the present study was too large for cement particles.
- The flow adapter can be redesigned with different types of transducers. In this way the same flow adapter can be used for different w/c ratios.
- The type of pump should be changed for UVP-PD measurements. The fluctuation of the flow rate is unacceptably high when using a piston pump and different type of pumps should be tried. Works should be performed to synchronize the UVP-PD measurements and the pressure of the pump so that a stable flow rate can be achieved.
- More sophisticated valves should be used to change the flow rate. Ball valves are too simple to maintain high precision of the work.
- Calibrations should be performed for the velocity of sound for different w/c ratio of cement grouts so that the concentration can be measured directly in-line.
- Off-line measurements should be performed at identical time and conditions, and subsequently compared with the in-line results of the UVP-PD for cement grouts.
- The UVP-PD setup should be tried in a laboratory environment for customization of the ultrasound transducers and pump types.
- The ultimate goal is that the method should be tested in the field in order to find out how it works as a measure for rheological properties and quality control. It should also be tried in the field together with the 'RTGC' method.

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## **APPENDIX** A

Flow curves (Shear stress vs Shear rate) obtained by off-line measurements are presented in Appendix A. Flow curves were obtained by ARES G2 rheometer and Brookfield DV-II<sup>+</sup> pro (LV) Viscometer. Water cement ratio 0.6 and 0.8 were used. Then the flow curves were fitted using Power law, Bingham and Herschel Bulkley model.

## A1. Flow curves measured by ARES G2 rheometer

Samples of w/c ratio 0.8 were collected for measurements at random time intervals of 30, 60, 180, 198 minutes. Samples of w/c ratio 0.6 were collected for measurements at random time intervals of 66, 71, 110 and 120 minutes.



Figure A.1 Flow curves for w/c ratio 0.8 with and without SetControl II



Figure A.2 Flow curve fitted with Bingham model for w/c ratio 0.8 with and without SetControl II



Figure A.3 Flow curve fitted with Power Law model for w/c ratio 0.8 with and without SetControl II



Figure A.4 Flow curve fitted with H-B model for w/c ratio 08 with and without SetControl II



Figure A.5 Flow curve for w/c ratio 0.6 with and without SetControl II



Figure A.6 Flow curve fitted by Bingham model for w/c ratio 0.6 with and without SetControl II



Figure A.7 Flow curve fitted by Power law model for w/c ratio 0.6 with and without SetControl II



Figure A.8 Flow curve fitted by H-B model for w/c ratio 0.6 with and without SetControl II



# A2. Flow curves measured by Brookfield DV-II<sup>+</sup> pro (LV) Viscometer

Figure A.9 Flow curve fitted by Power law, Bingham and H-B model for w/c ratio 0.8 without SetControl II just after mixing



Figure A.10 Flow curve fitted by Power law, Bingham and H-B model for w/c ratio 0.8 without SetControl II after agitating 2 hours



Figure A.11 Flow curve fitted By Power law, Bingham and H-B for w/c ratio 0.6 with SetControl II just after mixing



Figure A.12 Flow curve fitted by Power law, Bingham and H-B for w/c ratio 0.6 with SetControl II after agitating 2 hours

# **APPENDIX B**

The summary of the experimental works carried out at the Swedish Research Institute of Food and Biotechnology (SIK), Gothenburg are presented in Appendix B.

Summary of the experimental works at SIK, 17/06/2010 - 01/07/2010

Date	Time	Description of the work	W/C ratio	Cemen t	Water (L)	Set Contro	Remarks
				(KG)		1 (L)	
17/06	13.00 16.35 -	Arriving at SIK. Fine tuning of the Unigrout and Logac. Trial testing done					Equipments and machines assembled and fine tuned.
	17.26	with water					
18/06	14.30	Trial testing with water.	0.8	80	64	No	Trial testing done with water and grout.
	16.00	Trial testing with grout					Signals were not good enough.
							4 MHz transducers were used.
21/06	11.20	Trial testing with water.	0.6	100	60	1.4	Set Control was added with Batch 1.
	12.57 - 13.01	Mixing cement grout, Batch – 1					Sample was taken for off line measurements of
	14.00	Sample collected for off-line measurements					batch -1 without set control.
	14.16	Set Control added					No set control was added to batch 2.
	16.30	Cement grout – batch 2	0.6	100	60	No	Acoustic measurements were done with batch 2
22/06	10.40	Mixing cement grouts.					Objective was to obtain good UVP

			0.8	80	64	No	measurements.
	12.07 13.49	UVP measurements started UVP measurements started after lunch					Relatively stable flow and properties were observed after 1 hour
23/06	13.12 13.30 14.30	Mixing started with water for transducer distance calibration. Mixing started. Mixer clogged with the cement. Mixing of cement grouts.	0.8	3 bags 80	64	No	Batch cancelled. Objective was to take measurements at 15 minutes interval and observer the change of the properties over time.
24/06	09.50 10.55 11.58	Testing with water for transducer distance calibration. Testing with water with 2 MHz transducers. Mixing started with cement grouts.	0.8	80	64	No	<ul> <li>2 MHz transducers with old flow cell were used but good signal was not observed.</li> <li>Objective was to take readings at every 15 minutes and check the properties of cement grout at early stage and after agitating 2 hours</li> </ul>
28/06	09.50 10.56 - 11.01	Calibration done with water. Mixing of cement grout. Set control added.	0.8	80	64	1.2	Set control was used. First 30 minutes data were taken at every 5 minutes or less. Two samples were

	11.23 11.50 14.00	Measurements taken by UVP Sample taken for off line measurements. Sample taken for off line measurements.					<ul><li>taken for off line measurements to check the properties at early stage and after agitating longer period.</li><li>High fluctuation of flow rate observed.</li></ul>
30/06	10.32	Calibration started with water. Mixing of cement grouts. Set control added.	0.6	80	48	1.1	2 MHz transducer was used with new flow cell. 4 MHz and 2 MHz both type of transducers were tried to achieve good signals, velocity profiles and rheological properties.
01/07	13.30	Calibration started with water. Mixing of cement grouts. Set control added.	0.8	60	48	0.8	Objective was to measure the velocity of sound directly in line for both 2 MHz and 4 Mhz transducers. Temperature sensor was installed. A time lag was added to find the right peak for estimating sound velocity.





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