Md. Mashuqr Rahman

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Division of Soil and Rock Mechanics

Department of Civil and Architectural Engineering

Royal Institute of Technology (KTH)

SE-100 44 Stockholm

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Abstract

In-Line Rheological Measurements of Cement Based Grouts Using the UVP-PD Method

Md. Mashuqur Rahman

Graduate Student
Infrastructure Engineering
Division of Soil and Rock Mechanics
School of Architecture and the Built Environment
Royal Institute of Technology (KTH)
SE- 100 44 Stockholm

rahman9@kth.se

Abstract: In underground construction grouting is performed to seal tunnels and caverns against excessive water inflow or to reduce the lowering of the ground water table. The rheological properties, such as viscosity and yield stress, of the used grouts play a fundamental role in grouting. No method has been developed yet to measure these properties in-line in the field during grouting. Methods used today are rather primitive and not robust enough for field use and they are mainly performed in order to verify and fulfil stipulated quality criteria. Modern grouting rigs are today equipped with continuous measurement of flow and pressure but instruments for continuous monitoring of rheological properties and their changes with time in the field are still lacking. A relatively new method, known as ‘UVP-PD’, for continuous in-line measurements of the rheological properties of cement grouts, was tested in this work. Standard grouting equipment (UNIGROUT) and flow meter (LOGAC) was used to ensure field conditions. The objective of this work was to determine the feasibility of the ‘UVP-PD’ method for cement based grouts. After performing full scale experimental works, this method was found feasible for measuring the rheological properties of cement based grouts directly in-line.

KEY WORDS: Rheology, In-line measurements, Cement grouts, UVP-PD method
Preface

This master’s thesis was conducted at the Division of Soil and Rock Mechanics, Royal Institute of Technology (KTH), Stockholm. The work was carried out under the supervision of Adj. Professor Ulf Häkansson whom I would like to thank for his continuous guidance, support and encouragement all through the work which helped me for better understanding and writing. I would like to thank Dr. Johan Wiklund for giving me the opportunity to perform experimental works at The Swedish Institute for Food and Biotechnology (SIK) and co-supervising my work. I also want to thank Fernando Martins from Atlas Copco for providing the UNIGROUT E22H and LOGAC equipment and organizing training session which helped me to get familiar with the equipment. I would also like to thank Almir Draganovic for the laboratory work at KTH. I want to thank Professor Håkan Stille and Professor Stefan Larsson for examining the thesis.

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Md. Mashuqur Rahman
List of Symbols

List of symbols

$A$  cross sectional area

$A_r$  amplitude of the reflected wave

$A_i$  amplitude of the incident wave

$D$  Tunnel diameter

$F$  force

$f$  emitted frequency

$f_0$  basic ultrasound frequency

$f_r$  received signal frequency

$f_e$  emitted frequency

$f_d$  doppler shifted frequency

$f_{\text{max}}$  maximum measurable frequency

$F_{\text{PRF}}$  pulse repetition frequency

$H$  pressure of water head

$h_b$  height of the bob of concentric cylinder

$I$  maximum penetration depth of grout

$I_D$  relative grout penetration

$I_1$  intensity of incidence wave

$I'_1$  intensity of reflected wave

$K$  Groutability parameter

$k$  consistency index

$k_{\text{grout}}$  conductivity of the grouted zone

$L$  length

$m_s$  mass of grout
List of Symbols

N      near field distance
n      flow index
\(P_{max}\)  maximum penetration depth
Q      flow
r      radial position
R      real part of complex shear impedance
T      torque
t_{D}   relative grouting time
t_{0}   characteristics grouting time
\(n\)   velocity
\(v(g)\)  slip velocity at the wall
\(v^*(r)\)  parabolic velocity function
\(\mu_{g}\)  viscosity of grout
\(\mu_{B}\)  bingham viscosity
\(\tau\)  shear stress
\(\tau_{0}\)  yield stress
\(\tau_{w}\)  shear stress at pipe wall
\(\Delta P\)  pressure difference
\(\epsilon\)  skin factor
\(\dot{\gamma}\)  shear rate
\(\eta(\dot{\gamma})\)  shear rate dependent viscosity
\(\beta\)  term in the Rabinowitsch correction factor
\(\Omega\)  angular velocity
\(\omega\)  amplitude
\(\rho\)  density

v
List of Symbols

\( \rho_g \)  \hspace{1cm} \text{density of grout}

\( \kappa_{AD} \)  \hspace{1cm} \text{adiabatic linear bulk modulus of elasticity}

\( \kappa_1 \)  \hspace{1cm} \text{transmission coefficient}
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Chapter 1: Introduction

Introduction

Grouting is the process of injecting fluids into the joints or voids of a rock mass in order to control the water ingress into the underground facilities. It is performed in order to change or improve its physical properties such as permeability, deformability and strength of the material. Grouting technology was initially developed for dam foundation purposes and subsequently its applicability has been increased to other fields, i.e. water proofing in tunnels and toxic waste disposal or storage. Grouting creates a water proofing layer which prevents leakage and further adverse effects on the ground water table. The usages of underground facilities are constantly increasing and as an example one can mention repositories for spent nuclear fuel which demands strict inflow requirements into the facility. An underground facility with high inflow can cause vital environmental damage and therefore grouting is becoming important not only in a technical point of view but also when it is necessary to satisfy strict environmental regulations.

Difficulties in the measurement of rheological properties of suspensions, in general, and cement-based grouts, in particular, are well known (Håkansson, 1993). Cement based grouts are the most commonly used grouting materials and the suspensions are injected through the joints of the rock mass during grouting. Interparticle forces affect the fluid flow by introducing a yield stress and the rheological properties influence the relationship between pressure and fluid flow. Therefore it is very important to accurately know the rheological properties of grouts and their change with time, for design purposes. Unfortunately the methods used today to determine the properties in the field are rather primitive and conventional rotational rheometers are not robust enough to be used under harsh field conditions.

This thesis involves the feasibility study of new measuring technique for rheological properties of cement based grouts.

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. Håkansson (1993) concluded that future improvement for grouting also involves the continuous in-line monitoring of the rheological properties during a grouting operation. 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 it is independent of the joint aperture, according to following equation

\[ t_0 = \frac{6 \cdot \mu \cdot \Delta p}{\tau_0^2} \]
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.

In this thesis a promising technology for in-line measurements of the rheological properties of cement-based grout, the so-called “Ultrasound Velocity Profiling (UVP) – Pressure Difference (PD)” method, has been introduced and applied. This technique has been used previously in medical science for blood flow measurement and also used in the food and paper pulp industry to investigate the flow of suspensions. To the best of the author’s knowledge, this is the first time that this technology has been used, in the field of construction and cement based grouting. 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).
Chapter 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 rheological properties of commonly used cement based grouts. The UVP-PD method was used to determine how accurate and effective it is in measuring the rheological properties of grouts with different water-cement ratios directly in-line. A standard cement grout mixing equipment UNIGROUT E22H and LOGAC flow meter was used to keep the conditions the same as in the field. Secondary objectives of the investigation were as follows.

1. To determine the velocity of sound and velocity profiles of cement based grouts directly in-line using the ultrasound velocity profiler (UVP) and customized flow loop.

2. To determine the shear stress and the shear rate in order to achieve flow curves of cement based grouts directly in-line.

3. To determine the rheological properties e.g. viscosity and yield stress of cement based grouts by curve fitting mathematical models to the data achieved by directly in-line and comparing the results with off-line measurements.

4. To determine the shear rate, yield stress and viscosity of cement based grouts directly from the velocity profiles by the gradient method.

5. To determine the volumetric flow rate directly in-line by using the UVP-PD method and comparing the results with a conventional flow meter (LOGAC).

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 as follows.

In order to have accurate measurements of velocity profiles and subsequent determination of rheological properties, it is very important to have a stable flow inside the flow loop. The UNIGROUT E22H is equipped with a piston type of pump. As a consequence, the movement of the piston creates high fluctuations in the flow rate which was observed in all the measured velocity profiles. This made it difficult to determine which velocity profile to use as it was constantly changing with time.
Ball valves mounted on the UNIGROUT E22H were used to change the flow rate. The ball valves were found to be too crude to control the flow rate accurately enough for this type of measurements. It was almost impossible to change the flow rate by a very small amount.

In the measurements a 4 MHz transducer was used. With this transducer it was possible to obtain a signal penetration up to the center of the pipe for w/c ratio 0.8 and 0.6, respectively. However, for w/c ratio 0.6 the signal was not sufficiently penetrating in all experiments and the effect of attenuation were clearly visible. The beam diameter of the transducer was obviously too large for the used cements.

Cement grouts were tested both with and without SetControl (SC). Since the objective was to only measure the velocity profiles and rheological properties using the UVP-PD, no comparisons were within the scope of work. Also, as the sampling time and other conditions were not identical, the results with or without SetControl 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.
Chapter 3

Background

3.1 Rock Grouting

The general objective of rock or soil grouting is to improve the physical properties of the ground, i.e. to make the ground less permeable, stiffer or stronger than its natural state. This work concerns permeation grouting of jointed hard rock, whereby a grout material is injected into voids and discontinuities of a geological formation, with the aim to decrease its permeability.

To eliminate or reduce excessive inflow of ground water into e.g. a tunnel is important. The lowering of the ground water table can create adverse settlement to buildings in the vicinity of the tunnel or decrease the output from nearby wells. In general, the contractual requirements for grouting are becoming increasingly strict, mainly due to the fact that environmental considerations are taken more seriously in our society today. The requirements are often stipulated by environmental court directives, expressed as a limitation to the amount of water that is allowed to flow into e.g. a tunnel, during construction and during operations, respectively. The limitation is usually given as litres/minute and 100 m of tunnel. Strict requirements are in the range 0.5-5 litres/minute and 100 m, and moderately requirements are in the range 10-20 litres/minute and 100 m. Relaxed requirements are in the range 40-80 litres/minute and 100 m. Requirements of 1-10 litres/minute and 100 m are common today. Other requirements can be restrictions in the use of chemical grouts or chemical additives and admixtures in cement-based grouts.

Factors that influence a practical grouting operation are e.g. the rock mass and its discontinuities, the grout material and its properties, the grouting equipment and the grouting design. These factors of influence are described below.

3.1.1 Rock Mass

Rock mass is a discontinuous, inhomogeneous, anisotropic and non-linear elastic material. Its property varies in different places and usually it consists of rock faults and joints. From engineering point of view estimating the strength of rock mass is very difficult. Rock masses are made of interlocking matrix of separate blocks. These blocks may be weathered and altered to varying degrees. It is not practical to determine the strength of an in situ rock mass by laboratory testing. So the strength is measured from geological observation and by testing the sample rock pieces that have been removed from the rock mass. At the starting of a project very little information of rock mass, its stress and hydrological properties are available.

All rock masses consist of discontinuities such as bedding planes, joints, shear zones and faults. Faults are features in which identifiable shear displacements have taken place. Joints are breaks of geological origin along which there is no visible displacement. Joints and rock faults are the weaker zones of a rock mass. Faults and joints have high permeability so there might be water leakage in to the tunnel.
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Water leakage in tunnels must be avoided because it may lower the ground water table and also reduce the efficiency of the construction. So it is necessary to make the tunnel waterproof.

Grouting has always been considered as one of the most cost effective ways to reduce the water leakage into the tunnels of jointed hard rocks. In general grouting is done to control the underground water penetration into the tunnels. It is a process where cement mixtures or chemical materials are injected into the cracks and voids due to discontinuities of geological formation. The basic objective of grouting is to increase the strength of the ground including discontinuities and reduce the permeability.

Water infiltration into the tunnel depends on the hydraulic conductivity and the depth of the water pressure of the rock mass. The rule of thumb is that the effect of joint opening on hydraulic conductivity follows the cubic theory. So an opening of one unit wider aperture will cause 3 times higher water inflow. Although it depends on conditions that the opening remains constant. Joint roughness, contact area etc also affects the water flow. Porosity is also an important characteristic of rock mass which determines the amount of grout that can be injected. In general the porosity of the normal rock mass is .01-1 per mil. The requirement for water sealing is also fixed for the excavations. The requirement for underground construction is 0.5-5 l/min/100m is set for road and railway tunnels. Grouting can be performed in two ways as pre-grouting and post-grouting. Pre-grouting is done in most of the cases and post-grouting is done if necessary. Pre-grouting is very important for water sealing. It is done with higher grout pressure for wider grout spread and greater penetration.

3.1.2 Grouting Materials

The grouting material should be a good sealing agent. It will also depend on the environmental requirements and the geological, hydrological properties of the rock mass. It should have sufficient penetrability, durability and quick setting property without shrinkage (Dalmalm, 2001). For rock grouting, cement based and chemical, these two types of grouts are available. Chemical grouts are not environmentally friendly and sometimes expensive so cement based grouts are mostly used. Cement based grouts have lower sealing effect comparing with chemical grouts but their properties and environmental effects are well known. Ordinary Portland Cement (OPC) is mostly used for cement grouting. Slag cement and aluminum cement can also be used. Slag cement has a longer curing time and it is a mixture of slag and ordinary Portland cement. Cements used for grouting can be divided into several classes according to their grain size distribution (Tolppanen and Syrjänen, 2003)

- Ordinary Portland cement (OPC, \( d_{95} < 128 \mu m \))
- Rapidly setting cement (\( d_{95} < 64 \mu m \))
- Microcement, \( d_{95} < 20 \mu m \) (MFC, SFS-EN 12715)
- Ultrafine cement (UFC)

The grouting cement should be such that it can penetrate into the pores and joints of the rock fractures. A rule of thumb is used that joint openings that are less than 3-5 times the particles size are not suitable for penetration. For grouting joint openings, less than 0.1 mm micro cement or chemical grouts are used (Håkansson, 1991). In Sweden Injektering 30 is mostly used for grouting (Tolppanen and Syrjänen, 2003). Water cement ratio is also an important factor for cement grouting. With
changing of the concentration, the flow properties change. So it should be tested before using in the field.

To modify the flow properties and control the hydration of the cement paste, additives are used. By using the additives the hydration rate of the cement is sometimes accelerated and sometimes decreased. The accelerators are of two types (Tolppanen and Syrjänen, 2003)

- Binder accelerators for early hydration of the grout.
- Strength accelerator to increase the strength at initial stage.

The most common additive is calcium chloride. It is added in the solution in a dosage of 15%-20% of the water volume. It accelerates both the binding and early strength gaining. Superplasticizers are also used to modify the flow properties. Naphthalene and melamine based superplasticizers are mostly used. Pre testing should be done to select the proper dosage before using superplasticizers. Bentonite is used for thicker and stable grout. Since Bentonite is larger than the micro cements so its use is limited.

3.1.3 Grouting Equipment

The grouting work is started by drilling the holes. The grout is mixed first and then agitated before pumping. The pump is connected to a valve called packer by a hose. The grouting holes are made by a drilling jumbo. Drilling jumbos are equipped with a rod changer so it is possible to drill long grouting holes. The hole diameter depends on the grouting unit, normally 50-65 mm, 64 mm preferable.

All grouting equipments are kept on a platform. Before mining vessels were used as platforms but in modern system all the equipments are installed permanently on a truck. This gives more freedom to move inside the site or from site to site if necessary. To mix the particles of cements, cement and water is fed in to the mixer. For microcements, colloidal type mixers are used. The rpm of the colloidal mixers should be minimum 1500 and the mixing time should be in the range of 3 minutes. If the cement is mixed with water for long time, it will generate excessive heat which may affect the grout properties and if it is mixed for short time, the particles may not mix properly.

After mixing the cement grouts, it is transferred in to the agitator. Agitator actually plays the role of a holding tank where grouts are ready for grouting. The grout is always agitated here in order to keep a low viscosity and to prevent sedimentation.

For performing satisfactory grouting operation the pump flow and pressure must be sufficient. Two types of pump are popular for grouting operation. One is the progressive cavity pump (pump without valves) and another is valve type pump or piston pump.

For measuring the pressure and flow continuously, pressure and flow meters are used. But it is suggested to continuously measure other parameters such as density, viscosity, yield stress etc directly in-line. If the ground has to be grouted, tested or sealed, packers are used to close-off the full length or part of the bore hole. When the hole is closed, pressure is confined in the bore hole and the grout is forced in to the cracks. Two types of compression packers are available such as reusable packers and disposable packers.
3.1.4 Grouting Design

Grouting design is complicated. It can be based on the recent designs with some empirical adjustment or by investigating the rock mass. While investigating the rock mass several properties like joint length, joint filling, joint aperture, transmissivity are defined and in the laboratory a grout suitable for these properties is determined. The investigation process can be found in Houlsby (1990) and Kutzner (1996). According to Houlsby (1990) during investigating rock mass different properties as spacing of joints, joint widths and continuity, joint inclination, uniformity of the site, rock soundness, strength, stress in rock, piping etc should be defined.

Hydraulic conductivity of rock mass is an important property for grouting design. It shows the permeability of the rock mass. So it is necessary to determine the grout mix, grout order, grout take and connection between fractures (e.g. Dalmalm, 2001). Following field tests are done to measure the hydraulic conductivity:

- Pressure building test
- Packer tests (water loss measurement), with constant head
- Packer tests (water loss measurements), with different head
- Pulse tests

Grouting is done to seal the fractures and strengthen the joints of the grouted zone. The inflow of water depends on geometry, location, water pressure, rock mass conductivity etc. To find out the sealing efficiency we have to first know the water ingress in to the tunnel. The water ingress in to a deep tunnel can be determined by

\[
Q(l/s,m) = \frac{2\pi \cdot H \cdot k}{\ln \frac{4H}{D} + \varepsilon} \approx H \cdot k
\]

Where

- \( Q = \) flow of water
- \( k = \) conductivity of the rock mass
- \( H = \) Pressure of water head
- \( D = \) Tunnel diameter
- \( \varepsilon = \) Skin factor

The water ingress in a grouted tunnel can be determined by
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\[ Q(l/s, m) = \frac{2\pi \cdot H \cdot k \cdot \ln \left( \frac{4H}{D} + \left( \frac{k}{k_{\text{grout}}} - 1 \right) \cdot \ln \left( \frac{D + 2t}{D} \right) \right) + \varepsilon}{\ln \frac{4H}{D}} \approx 2\pi \cdot H \cdot k_{\text{grout}} \]  \hspace{1cm} \ldots..3.2

Where

\begin{align*}
Q & = \text{flow of water} \\
k & = \text{conductivity of the rock mass} \\
H & = \text{Pressure of water head} \\
D & = \text{Tunnel diameter} \\
\varepsilon & = \text{Skin factor} \\
k_{\text{grout}} & = \text{conductivity of the grouted zone} \\
t & = \text{thickness of the grouted zone}
\end{align*}

The typical sealing requirement as of today is 0.5-10 l/min and per 100 m of the tunnel. Depending on the hydraulic conductivity of rock mass, geometry and water pressure head, high grouting is required sometimes. The sealing efficiency required might be from 90%-100% as most of the water ingress has to be sealed. The sealing efficiency can be defined as (Dalmalm 2001)

Sealing efficiency [%] = \( 1 - \frac{\text{inflow after grouting}}{\text{inflow before grouting}} \)

From the above two equations of tunnel water ingress we can find out the conductivity of the grouted zone. The sealing efficiency and conductivity of the grouted zone leads us to the required grout type.

**Stop Criteria**

Stop criteria is one of the most important property of grouting design. It tells us when to stop grouting. Injecting large volume of grouts is uneconomical and since the grouting pressure is very high so it may cause rock mass lifting. So stop criteria must be set. Stop criteria is set mainly based on practical knowledge as following (Houlsby, 1990)

- Grouting is completed when the grout flow is less than a certain value at maximum pressure
- Grouting is completed when the grout take is above a certain value

Such a criteria which combines the grout pressure and volume is the GIN principle (Lombardi and Deere, 1993). This was developed to minimize the risk of hydraulic fracture and uniform grout spreading. The GIN value can be taken as the total volume of energy that is injected in to the rock mass (Dalmalm, 2001). GIN is expressed as

\[ GIN = p \cdot V = \text{Grouting energy} \]  \hspace{1cm} \ldots.............3.3
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Where,

\( p = \) grout pressure at zero flow

\( V = \) grouted volume per meter drill hole at stop pressure

Maximum values of pressure and volume is determined in this criteria so when the maximum value of the multiplication of pressure and grout volume is attained, grouting is stopped. The standard GIN curves for measuring the INJ value is shown in Figure 3.1.

![GIN curves](image)

*Figure 3.1 Some standard GIN curves (Lombardi and Deere, 1993)*

Based on the GIN principle, determination of the INJ value is developed. The INJ value depends on the grout penetration and time dependent grout properties. The INJ value can be expressed as

\[
INJ = \frac{GIN}{\tau_0} = \frac{p \cdot V}{\tau_0} = \left( \frac{V}{K} \right)^3
\]

Where

\( \tau_0 = \) Yield value of the grout fluid

\( I = \) Maximum penetration length

\( K = \) groutability parameter

The problem with the GIN principle is that it does not relate the grout penetration. So the grouting might be uneconomical.

Further research has been continuously carried out to establish the relationship between the grouting principle, grouting time and penetration. Recent research showed that the relative grout penetration,
which is the ratio between the actual penetration and the maximum penetration, does not depend on the aperture. This implies that the relative grout penetration is the same in all fractures (Gustafson and Claesson, 2005).

The relative penetration can be expressed as

\[
I_D = \frac{t_D^2}{4(1 + t_D)} + \frac{2 \cdot t_D}{1 + t_D} - \frac{t_D}{2(1 + t_D)} \quad \text{.................3.5}
\]

Where

\[
I_D = \frac{I}{I_{\text{max}}} = \text{relative grout penetration}
\]

\[
t_D = \frac{t}{t_0} = \text{relative grouting time}
\]

Based on this study, theoretically based stop criteria for rock grouting was examined and presented (Gustaffson and Stille, 2005). The stipulated characteristic grouted time can be expressed by

\[
t_0 = \frac{6 \cdot \mu_{g} \cdot \Delta p}{\tau_0^2} \quad \text{.................3.6}
\]

Where

\[
t_0 = \text{characteristic grouting time}
\]

\[
\mu_{g} = \text{viscosity of the grouting fluid}
\]

\[
\tau_0 = \text{yield stress of the grouting fluid}
\]

It can be seen that the characteristic grouting time is proportional to the viscosity and the pressure difference and inversely proportional to the yield stress of the grout to the power of two. The important finding is that the pressure and the grout properties alone determine the time scale which is independent of the aperture. Consequently, the designer and the grouting operator can decide the time scale at their own choice.

Assuming the Bingham model for the grout, Gustafson and Stille (2005) showed the influence on the flow rate and penetration by changing the pressure, viscosity and yield stress, respectively.

In recent research a new methodology called ‘Real Time Grouting Method’ shows that the design and control of a grouting operation depends on the knowledge of the rheological properties and their change with time (Kobayashi et al, 2008).
Needless to say, the rheological properties of the cement grout plays a crucial role in grouting design and it is therefore necessary to determine the properties of the grout during the grouting operation.

3.2 Rheology of cement based grouts

Rheology is the science of deformation and flow of matter. The term Rheology comes from the Greek word ‘rheos’ which means flowing or streaming. The science of Rheology relates the Newtonian fluid mechanics that is the direct relationship between the shear stress and shear rate of a fluid and the Hook’s elasticity. In general Rheology is the viscous characteristics of slurry, more precisely the relationship between the shear stress and the shear rate. In this work the rheology of cement based grouts of different characteristics will be discussed.

The rheological behavior of cement based grout is considered complex (Håkansson, 1993). The grout might be non-Newtonian, thixotropic and have a yield stress. The hydration of the cement also plays a key role as the rheological properties change with time.

3.2.1 Viscosity

The concept of viscosity was found in ‘Principia’ of Isaac Newton (1687). If one fluid tends to flow over another fluid some resistance arise. According to ‘Principia’ this resistance arises from the lack of slipperiness and this lack of slipperiness is known as viscosity. As shown in Figure 3.2, if we consider two very thin layer of fluid flow ‘dy’ distance apart and the fluid is subjected to a force $F$ then we can say that the shear stress is force per unit area. That is

$$\tau = \frac{F}{A}$$

According to Principia we can say that the shear stress required to produce the motion is proportional to the shear rate, $\gamma$. Shear rate is expressed as the velocity gradient in the direction perpendicular to that of the shear force. It is a function of the shear stress acting on the fluid. From the Figure 3.2 we can say that shear rate, $\gamma = \frac{dv_x}{dy}$, where $dv_x$ and $dy$ are the relative velocity and distance between the adjacent layers. So shear stress,
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\[ \tau = \mu \cdot \frac{dv_x}{dy} = \mu \cdot \gamma \] .......................... 3.8

The constant proportionality, \( \mu \) is called the Newtonian viscosity and is independent of shear rate \( (\gamma_{yx}) \) or shear stress \( (\tau_{yx}) \) and depends on the material, temperature and pressure.

### 3.2.2 Yield Stress

Yield stress is a common rheological property which many concentrated suspensions may have. The yield stress is the minimum stress that makes the fluid to flow like a viscous material. Inter particle forces between the solids in a suspension results in a yield stress that must be exceeded to start the flow. If the stress applied to the fluid is less than the yield stress then the fluid doesn’t flow but it deforms like a solid. The yield stress can be considered as material property and it is the transition state between a solid like and liquid like behavior (Nguyen and Boger, 1983).

Yield stress is used in non-Newtonian fluid models like Bingham model and Herschel Bulkley model. For a Bingham fluid, the yield stress is defined in British standards (BS 5168) as: “the stress below which the material is an elastic solid and above which it is a liquid with plastic viscosity”.

Yield stress is generally exhibited in flocculated suspensions where inter particle interactions creates attraction between individual particles and as a result they form flocs. Cement grouts, mixtures, concrete shows yield stress. A precise knowledge of yield stress is needed for handling, storage and transportation of these materials. Some suspensions with high solids content may have the static yield strength at zero shear rates and also the dynamic yield strength (Keating and Hannant, 1989). Due to thixotropic characteristic, the suspension will have dynamic shear strength less than the static yield strength at zero shear rate. At small stress, the system deforms elastically but if the stress exceeds the gel strength (yield stress at zero shear rate) the deformation increases continuously at a lower shear stress. This lower stress is known as dynamic yield strength. This is a dynamic property of the suspension. The characteristics is shown in the Figure 3.3.

![Figure 3.3](image)

**Figure 3.3** Relation between shear stress and time to define gel strength and dynamic yield. (Keating and Hannant, 1989)

There are several methods to measure the yield stress. Both direct and direct methods have been proposed and used (Nguyen and Boger, 1983). The most common methods are the vane method, stress
relaxation method, marsh cone etc. In vane method the conventional analysis assumes that the stress is uniformly distributed on a cylindrical sheared surface to calculate the yield stress for the maximum torque and vane dimension (Nguyen and Boger, 1985). With conventional rotational viscometers it is not possible to interpret shear stress-shear rate data and to obtain the shear stress in zero shear rate limits due to lack of data at low shear rate. So some constitutive equations and rheological models are used to extrapolate the resulting fitted curve to zero shear rates.

There are some doubts of the existence of the yield stress. In some literatures its said that yield stress is the stress below which it cannot be measured. Some materials take a long time to flow if the zero shear rate viscosity is too high. Using conventional rheometer it is impossible to obtain accurate results at shear rates below $10^{-3}$ s$^{-1}$. But using some constant stress instruments (e.g. Deer Rheometer) accurate stress measurements can be made at a shear rate of $10^{-6}$ s$^{-1}$. By measuring shear stress at that low shear rate it was said that there is no yield stress and it was not measured by anyone (Barnes and Walters, 1985).

High range water reducer (HRWR), viscosity enhancing admixtures (VEA), rheology modifying admixtures (RMA) etc are used to change the properties of the grout and make it more suitable for practical usage. These admixtures are normally known in the grouting industry as plasticizers or super-plasticizers (author’s comment). RMA is used with high performance grout to increase the resistance against wash out, bleeding, sedimentation. These admixtures also have effect on the yield stress. VEA ensures the shear thinning behavior of grouts. So the resistance against deformation can be increased and it will have an effect on the test procedure and maximum shear rate. Yield stress is normally determined by using various mathematical models such as Bingham, Power Law, Herschel Bulkley, Casson etc. It has been found that Bingham model gives the slightest acceptable fitting of the experimental parameters comparing to other analytical models (Yahia and Khayat, 2003). It is shown in Figure 3.4. It is found that Bingham model results poor experimental data at low shear stress. The shape of the Bingham model is linear and it doesn’t give accurate fitting corresponding to the area of low shear rates. At high shear rate Bingham model gives accurate shear stress. So the yield stress predicted by the Bingham model is always higher than the actual one.
For grouting operation the rock geometry is hardly known so there is not much importance of a sophisticated rheological model. Using rheological models consisting yield stress is convenient and using Bingham model is sufficient. The flow rate and the filling capacity of cement based mixture depends on yield stress. For cement mixtures placed without vibration, yield stress is very important. So yield stress can be regarded as a quality control index of self consolidating and self leveling systems (Yahia and Khayat, 2001).

### 3.3 Fluid Behavior

In this section two types of fluid behavior will be presented and discussed – Newtonian and Non-Newtonian fluids.

#### 3.3.1 Newtonian Fluid

Let’s consider a thin layer of fluid flowing between two parallel planes $d$, apart as shown in the Figure 3.5. Now, if under steady state conditions, the fluid is subjected to a shear by the application of a force $F$ as shown, this will be balanced by an equal and opposite internal frictional force in the fluid. For an incompressible Newtonian fluid in laminar flow, the resulting shear stress is equal to the product
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The first subscript on both $\tau_{yx}$ and $\gamma_{yx}$ indicates the direction normal to that of shearing force, while the second subscript refers to the direction of the force and the flow. The plot of shear stress ($\tau_{yx}$) against shear rate ($\gamma_{yx}$) for a Newtonian fluid, the so-called ‘flow curve’ or ‘rheogram’, is therefore a straight line of slope, $\mu$, and passing through the origin; the single constant, $\mu$, thus completely characterizes the flow behavior of a Newtonian fluid at a fixed temperature and pressure. The characteristics of Newtonian fluid can be found in *Barnes et al, 1989*.

### 3.3.2 Non-Newtonian Fluid

Fluids which do not follow equation 3.8 and the characteristics of Newtonian fluid are known as non-Newtonian fluid. Most of the fluids in reality including the cement based grouts are non Newtonian fluids. The main characteristic of non Newtonian fluid is that they do not have a constant viscosity rather their viscosities are shear rate dependent, more precisely a function of shear rate. The relationship between shear stress and shear rate can be expressed as

$$\tau = \eta(\gamma) \cdot \gamma$$

Where $\eta(\gamma)$ is the shear rate dependent viscosity. While measuring a fluid with shear rate dependent viscosity it is very important that the measurements are done in the same range of shear rates as the hand application. If $\eta$ decreases with the increasing shear rate then the fluid is known as “pseudoplastic” or “shear thinning”. Shear thinning behavior is common in most of the fluids. The behavior of different kinds of non Newtonian fluids is shown in Figure 3.6.
If \( \eta \) increases with increasing shear rate the fluid is known as “dilatant” or “shear thickening”. Shear thickening behavior is normally observed in highly concentrated suspensions. Viscosity is usually temperature dependent and decreases with increasing temperature but for gases it reacts vice versa.

### 3.4 Rheological Models of Fluid Behavior

Several mathematical models have been derived to introduce and calculate the rheological properties. Rheological models for Newtonian and Non Newtonian models will be discussed here.

#### 3.4.1 Newtonian model

Newtonian model is described by a constant viscosity, \( \mu \), which is independent of the shear rate and shear stress but depends on the material properties and temperature. The relation of shear rate and shear stress varies in this model by the following equation

\[
\tau = \mu \cdot \gamma 
\]

\[ \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots 3.10 \]

Newtonian behavior is observed in fluids where the dissipation of viscous energy is due to the collision of comparatively small molecular particles. All glasses and liquid and solutions of low molecular weight fall in this category (Wilkinson, 1960). The particles increase the viscosity comparing to the suspending fluid medium. Decreasing the particle size or increasing the degree of asymmetry of the particles lead to a lower limiting concentration (Håkansson, 1993). If the concentration increases the viscosity is also increased and after a highest limit of the solid concentration the behavior becomes non Newtonian.
3.4.2 Non Newtonian Models

Non Newtonian models are used to measure the rheological properties of non Newtonian fluids. Power Law, Bingham and Herschel Bulkley model will be discussed here.

Power Law Model

Power Law model is used for the curve fitting of the flow curve of pseudoplastic fluids. Pseudoplastic fluids do not have a yield stress and the typical flow curve for these materials indicates that the ratio of shear stress to the shear rate. Which is termed as apparent viscosity, falls progressively with shear rate and the flow curve becomes linear only at very high shear rate. The model is described by a power law relationship of the form

$$\tau = k \cdot \dot{\gamma}^n$$  \hspace{1cm} .................3.11

In this equation $k$ and $n$ are mathematical curve fitting parameters and they are known as ‘consistency index’ and ‘fluid behavior index’, respectively. Depending on the value of the fluid behavior index, $n$, the following rheological models can be described by the power-law model

- Pseudoplastic or Shear Thinning  \hspace{0.5cm} (If $n < 1$)
- Dilatant or Shear Thickening  \hspace{0.5cm} (If $n > 1$)
- Newtonian  \hspace{0.5cm} (If $n = 1$)

Pseudoplastic models are frequently observed in suspension rheology. For this kind of fluids the apparent viscosity decreases as the shear rate increases. This is a characteristic of suspension of asymmetric particles of high polymers. Which means with increasing shear rate the asymmetric particles are progressively aligned (Wilkinson, 1960). Suspension of isometric particles may exhibit pseudoplastic behavior if interactions between the particles lead to a mutual attraction. The particles are flocculated due to attraction and forms aggregate which can be broken down under shear causing reduced apparent viscosity (Håkansson, 1993).

Bingham Model

The Bingham model is characterized by a flow curve of straight line having a yield stress $\tau_0$. This is the simplest model describing the flow behavior of a non Newtonian fluid with yield stress. The rheological model for a Bingham plastic may be expressed as

$$\tau = \tau_0 + \mu_B \cdot \dot{\gamma}$$  \hspace{1cm} .................3.12

Here $\mu_B$, the plastic viscosity is the slope of the flow curve. The Bingham model predicts a linear relationship between shear stress and shear rate when the yield stress, $\tau_0$, is exceeded. The concept of idealized Bingham plastic is very convenient in practice because many real fluids closely approximate this type of behavior. The common examples of this kind of fluid are the slurries, drilling mud, oil
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paints, tooth paste etc. When the Bingham fluids are at rest they contains a three dimensional structure of sufficient rigidity to resist any stress less than the yield stress \( \tau_0 \). When the yield stress is exceeded the structure completely disintegrates and the fluid behaves as a Newtonian fluid (Wilkinson, 1960).

**Herschel Bulkley Model**

The Herschel-Bulkley model usually represent the non linear curve for the pseudoplastic fluid behavior. The model can be expressed by the following equation

\[
\tau = \tau_0 + k \cdot \dot{\gamma}^n
\]

……………………………………3.13

In fact this is a simple generalization of the power law model consisting a yield stress. For \( n < 1 \) the suspension is yield pseudoplastic, for \( n > 1 \) the suspension is yield dilitant and for \( n = 1 \) the model reduces to the Bingham model.

**Gradient Method**

The gradient method is a unique feature for the UVP-PD technique. In this method, the gradient \( -\frac{dv}{dr} = \dot{\gamma} \) is determined along different points in the measured radial velocity profile and thus, the shear rate can be obtained directly. This is an alternative method to the conventional curve fitting to mathematical models and was proposed by e.g. Muller et al, 1997. The shear stress, \( \tau \), at any individual point is obtained by using the pressure difference \( \Delta P \) with the formula \( \tau = \frac{r \Delta P}{2L} \). The apparent viscosity is subsequently achieved by the relationship, \( \eta = \frac{\tau}{\dot{\gamma}} = \frac{r \Delta P}{\dot{\gamma} 2L \dot{\gamma}} \).

This method was for example, applied by Birkhofer et al, 2007 for chocolate suspension and cocoa butter and the result was compared with conventional rheological modeling.

**3.5 Circular Pipe Flow**

In this section the fluid flow behavior in a circular pipe will be discussed. Flow in a long pipe is considered. Assumptions made for the calculation are the followings

1. Incompressible fluid
2. Laminar flow
3. Fully developed, steady flow
4. Flow only in z direction
5. No slip at the wall

The schematic diagram of a circular pipe flow is shown in Figure 3.7. If the fluid properties are independent of time the rheological equations shear rate and shear stress can be written as
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\[ \gamma = f(\tau) \] ..........................3.14

So the pipe flow becomes

\[ -\frac{du}{dr} = f(\tau) \] ..........................3.15

Here \( \tau \) is the shear stress at a radius \( r \). As there is no angular velocity, the force balance on a fluid element at distance \( r \) can be written as (Wilkinson, 1960)

\[ 2\pi L r \tau = \pi r^2 \Delta P \] ..........................3.16

or,

\[ \tau = \frac{r\Delta P}{2L} \] ..........................3.17

For shear stress at the wall, \( \tau_w \), we can say

\[ \tau_w = \frac{R\Delta P}{2L} \] ..........................3.18

Figure 3.8 represents the shear stress and velocity profile distribution in a fully developed laminar flow in a pipe.
This shows the laminar shear stress distribution across the pipe cross section and the shear stress is zero at the center of the axis and highest at the wall.

### 3.5.1 Newtonian Pipe Flow

For proper velocity distribution a rheological model must be applied. For a Newtonian fluid equation 3.17 can be written as

$$-\mu \frac{dv_z}{dr} = \frac{\Delta P}{L} \cdot \frac{r}{2}$$

..................3.19

Integrating with respect to \( r \) we can say that

$$-v_z = \frac{\Delta P}{L} \cdot \frac{r^2}{4\mu} + c_2$$

..................3.20

The boundary condition is, when

\[
\begin{align*}
  r &= R \\
  v_z &= 0
\end{align*}
\]

So we can find the velocity distribution as

$$v_z = \frac{1}{4\mu} \cdot \frac{\Delta P}{L} \cdot (R^2 - r^2)$$

..................3.21

The maximum velocity occurs at the center of the pipe, when \( r = 0 \), so

$$v_{z,\text{max}} = \frac{R^2}{4\mu} \cdot \frac{\Delta P}{L}$$

..................3.22
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The volumetric flow rate is obtained by integrating equation 3.21 with respect to r, over the cross sectional area, so

\[ Q = \int_0^R 2\pi r v_z(r) \, dr = \frac{\pi \cdot \Delta P \cdot R^4}{8\mu L} \] ..........3.23

The average velocity can be found by dividing the volume flow rate, Q with the cross sectional area, so

\[ \overline{v_z} = \frac{Q}{\pi \cdot R^2} = \frac{\Delta P \cdot R^2}{8\mu L} \] ..........3.24

The shear rate at the wall can be achieved by

\[ \dot{\gamma}_R = \left(-\frac{dv_z}{dr}\right)_R = \frac{\Delta P \cdot R}{2\mu L} \] ..........3.25

By comparing equation 3.24 & 3.25 we can say that

\[ \dot{\gamma}_R = \frac{4Q}{\pi R^3} = \frac{8\overline{v_z}}{D} \] ..........3.26

For a more general case where the rheological model of the fluid is unknown the relationship between the wall shear stress \( \tau_w \), the volumetric flow rate Q and the shear stress \( \tau_r \), are as follows:

\[ \frac{Q}{\pi R^3} = \frac{1}{\tau_R} \int_0^{\frac{\tau}{\tau_R}} \tau^2 \dot{\gamma} \, d\tau \] ..........3.27

By integrating with respect to \( \tau_R \) and rearranging the equation we can find the Rabinowitsch equation which can be written as (Bird et al, 1989)

\[ \gamma_R = \frac{1}{\pi R^3 \tau_R^2} \frac{d}{d\tau_R}\left(\frac{\tau_R^3}{3} \cdot Q\right) \] ..........3.28

Equation 3.28 is used to plot the flow curve; shear stress Vs shear rate for any fluid from the experimental data of the pressure drop (\( \Delta P \)) and volume flow rate (Q). The assumption needed here is, the fluid is homogeneous and there is no wall slip. The equation can be transformed to

\[ \gamma_R = \frac{4Q}{\pi R^3} \left(\frac{3+\beta}{4}\right) \] ..........3.29

Where,
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\[ \beta = \frac{d \log \left( \frac{4Q}{\pi R^4} \right)}{d \log \tau_R} \] \quad \text{………………………………}3.30

Equation 3.28 is used to convert an apparent shear rate at the pipe wall to a true shear rate. The factor \( \frac{3 + \beta}{4} \) is known as Rabinowitsch correction term. Equation 3.29 is a general equation and can be applied to all fluids without knowing their rheological model.

3.5.2 Non Newtonian Pipe Flow

In this section velocity profiles and flow curves of Power Law, Bingham Plastic and Herschel-Bulkley model will be discussed. Rheological properties depend on the shape of the velocity profile of the pipe flow. For calculating the rheological parameters several formulas have been developed depending on the fluid behavior.

**Power Law Fluid**

In power law model the variation of the shear stress with respect to the shear rate is presented by

\[ \tau = k \cdot \dot{\gamma}^n \]

The assumption is zero velocity at the pipe wall. By combining and integrating equation 3.11 &3.17 radial velocity profile, shear rate and viscosity are found as

\[ v = \left( \frac{nR}{n+1} \right) \left( \frac{R \cdot \Delta P}{2LK} \right) \left( 1 - \left( \frac{r}{R} \right)^{1+\frac{1}{n}} \right) \] \quad \text{………………..}3.31

\[ \dot{\gamma}_R = \left( \frac{r \cdot \Delta P}{2LK} \right)^{\frac{1}{n}} \] \quad \text{………………..}3.32

\[ \mu = \frac{\tau}{\dot{\gamma}} = K \left( \frac{r \cdot \Delta P}{2LK} \right)^{-\frac{1}{n}} \] \quad \text{………………..}3.33

The wall shear rate and the viscosity at wall can be found by

\[ \dot{\gamma}_{Rw} = \left( \frac{R \cdot \Delta P}{2LK} \right)^{\frac{1}{n}} \text{ and} \quad \mu_w = \frac{\tau_w}{\dot{\gamma}_w} = K \left( \frac{R \cdot \Delta P}{2LK} \right)^{-\frac{1}{n}} \] \quad \text{………………..}3.34

The power law predicts an unrealistic infinite shear viscosity at the center of the pipe \( r = 0 \) for pseudoplastic fluids \( n < 1 \) but a realistic finite viscosity at the pipe wall \( r = R \) (Wiklund et al, 2002). The flow rate is obtained integrating the velocity profile.
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\[
Q = 2\pi \int_{0}^{R} r \cdot v \cdot dr = \frac{\pi R n^3}{3n + 1} \left( \frac{R \cdot \Delta P}{2LK} \right)^{\frac{1}{n}}
\]  

[3.36]

For Newtonian fluid \( n = 1 \), \( k = \mu \) and the equation reduces to Poiseuille form (Wilkinson, 1960).

**Bingham Plastic Fluid**

A fluid with a yield stress will flow if the applied stress (proportional to the pressure gradient) is larger than the yield stress. There will be a solid plug like core in the middle of the pipe where shear stress is less than the yield stress. The plug has an important practical significance. When the shear stress at the wall decreases, the plug increases and finally reaches the wall leading to a zero velocity. It is shown schematically in the Figure 3.9.

\[
\tau_{rz} = \tau_{0B} + \mu_B \left( -\frac{dv_z}{dr} \right)
\]  

[3.38]

Now combining equation 3.17 and 3.38 and integrating we can find the velocity profile as
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\[ v_z(r) = \frac{\Delta P \cdot R^2}{4\mu_B L} \left( 1 - \frac{r^2}{R^2} \right) - \frac{\tau_0 R}{\mu_B} \left( 1 - \frac{r}{R} \right) \]  

...............3.39

Equation 3.38 can be applied when \( \tau_r > \tau_0^B \) and \( r \geq R_p \). The velocity at the plug region can be found by substituting \( r = R_p \). The constant plug velocity is

\[ v_{plug} = \frac{\Delta P \cdot R^2}{4\mu_B L} \left( 1 - \frac{R_p^2}{R^2} \right) \]  

...............3.40

The shear rate at the wall is

\[ \gamma_R = \frac{\Delta P \cdot R}{2\mu_B L} - \frac{\tau_0}{\mu_B} \]  

...............3.41

The expression for volumetric flow rate is obtained by evaluating the following integral

\[ Q = \int_0^R 2\pi r v_z \cdot dr = \int_0^{R_p} 2\pi r v_{plug} dr + \int_{R_p}^R 2\pi r v_z dr \]  

...............3.42

**Herschel-Bulkley Fluids**

For fluid with a yield stress and power law behavior above yield stress Herschel-Bulkley model is used. By combining equation 3.13 & 3.17 and integrating assuming zero velocity at the pipe wall we can find the radial velocity profile, shear rate and viscosity profile as follows

\[ v = \left( \frac{n}{1+n} \right) \left( \frac{\Delta P}{2LK} \right)^{\frac{1}{n}} \left( R - R_p \right)^{\frac{1}{n}} - \left( r - R_p \right)^{\frac{1}{n}} \]  

...............3.43

\[ \gamma = \left( \frac{\Delta P}{2LK} \right)^{\frac{1}{n}} \left( r - R_p \right)^{\frac{1}{n}} \]  

...............3.44

\[ \mu = \frac{\tau}{\gamma} = K \left( \frac{\Delta P}{2LK} \right)^{-\frac{1}{n}} \left( \frac{r}{\left( r - R_p \right)^{\frac{1}{n}}} \right) \]  

...............3.45

Shear rate and the viscosity at the pipe wall can be found as
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\[ \gamma_w = \left( \frac{\Delta P}{2LK} \right)^{\frac{1}{n}} \left( R - R_p \right)^{\frac{1}{n}} \]  
\[ \mu_w = \frac{\tau_w}{\gamma_w} = K \left( \frac{\Delta P}{2LK} \right)^{1-\frac{1}{n}} \left( \frac{R}{R - R_p} \right)^{\frac{1}{n}} \]  

\[ Q = \pi v_p R_p^2 + \frac{\pi n R^2 (R - R_p)^{\frac{1}{n}}}{n + 1} \left[ \Delta P \left( \frac{1}{2LK} \right)^{\frac{1}{n}} \right] \left[ 1 - \frac{2n}{3(n + 1)} \left( 1 - \frac{R_p}{R} \right)^2 \right] - \frac{2n R_p}{(2n + 1)R} \left( 1 - \frac{R_p}{R} \right) - \left( \frac{R_p}{R} \right)^2 \]

Volumetric flow rate can be shown as

Where plug velocity can be obtained by substituting \( r = R_p \) in equation 3.43.
4.1 Off-Line measurements of the rheological properties of cement based grouts

Off-line measurements are performed in the laboratory and in the field. Instruments that are used in the laboratory are sophisticated but not robust and instruments that are used in the field are primitive and the results might be unreliable (Håkansson and Rahman, 2009). Different methods to determine the rheological properties in the laboratory and in the field are discussed here.

4.1.1 Laboratory measurements

For laboratory measurements, rotational and capillary viscometers are commonly used and will be presented here.

Rotational Viscometer

Rheological properties can be measured by rotational viscometers using indirect and direct techniques. Indirect measurements are performed by following methods (Nguyen and Boger, 1983)

- Direct extrapolation of the rheological shear rate-shear stress data
- Extrapolation of the flow curves assuming the Bingham flow model
- Extrapolation of the flow curves assuming non linear plastic models, as Herschel-Bulkley and Casson model

Direct measurements are performed in the following methods

- Shear stress relaxation method
- Shear vane method.

The rotational viscometers usually consist of concentric cylinders where an inner cylinder is rotating while the outer cylinder is stationary. Cylinders are mostly common but other geometries such as cones, disks and vanes are also used. An advantage is that rotational viscometers only require a small sample and that the sample can be measured for a long period of time.

Concentric cylinders

There are two ways to apply the rotation and the couple measurement. One way is to drive one member and measure the couple on the same member and the second way is to drive one member and measure the couple for other member. Typically the outer cylinder rotates and the torque T on the
inner cylinder suspended from a wire is measured. Figure 4.1 shows a partial section of a concentric cylinder viscometer.

There are established formulas to determine the torque to measure the yield stress and the angular velocity to measure the shear rate (Barnes et al., 1989). If the gap between two cylinders is small and they are in relative rotation, the liquid inside the gap is almost in a constant shear rate. If the radius of outer and inner cylinder is \( r_0 \) and \( r_1 \) respectively and the angular velocity of the inner cylinder is \( \Omega_1 \), the outer cylinder is stationary and the shear rate is \( \dot{\gamma} \) than we find that

\[
\dot{\gamma} = \frac{r_0 \Omega_1}{r_0 - r_1}
\]

The working formulas in many cases do not consider the curvature of the surface of the measuring geometry. The determination of shear rate and shear stress is valid only for very small gap between the cylinders and the ratio between the inner to outer radius must be greater that 0.99 (Chhabra and Richardson, 1999).

If the ratio of the radius is >0.99 then the shear stress is given by

\[
\tau = \frac{T}{2\pi r_1^2 h}
\]

Where \( h \) is the sample height.

Some designs have been developed to overcome the end effects due to shear flow at the bottom of the concentric cylinder. One of them is the ‘Mooney-Ewart’ design with a conical bottom of a suitable cone angle. Here the shear rate at the bottom is the same as the narrow gap between the cylinders. It has also been covered by roughening the cylinders that is by attaching a layer of fine sand on the surface (Håkansson, 1993).
Concentric cylinder has some limitations also. Most of the errors are caused by the end effects, wall slip, inertia and secondary flows, viscous heating effects and eccentricities due to misalignment of the geometry (Macosco, 1994). The measuring techniques of the rheological properties of cement grouts are vastly discussed in other literatures (Hässler1991, Håkansson 1993).

The viscometer is connected to a computer. Rotational viscometers can be run for a long period of time so it is possible to observe the change of the properties over time, especially the grout’s hardening process. During the measurements the angular velocity is changed from a lower rate to higher or vice versa so different sequences can be observed. It is assumed that during a short period of time (2-5 minutes) the properties remain constant. All data’s of different sequences are stored in the computer and by suitable simulation continuous change of the shear stress and the viscosity over time can be determined (Macosco 1994, Hässler 1991, Håkansson 1993).

It is important that the cement suspensions will be stable while grouting with cement suspensions. Otherwise some particles will settle and reduce or may block the penetration. We can make stable cement grouts by using low water/cement ratio or by using admixtures, as bentonite. While using rotational viscometers sedimentation of cement can appear as a problem. The mixture will become thinner if lots of particles are settled. A mixer can be connected to the measuring cylinder to avoid this problem. After taking some measurements, the mixer is operated and it force the grouts inwards hence prevents settling (Håkansson1993).

**Shear Vane Method**

Wall slip effect is a major problem of rotating cylindrical viscometer. For measuring the yield stress and rheological properties of cements, rotational viscometers are commonly used. While running the rotational viscometer a cement concentrated layer develops due to the displacement of the scattered cement particles at the smooth walls of the cylinder. The development of such a low viscosity layer at the wall means that there will be lubricating effect and it will be easier to flow over this layer. This phenomenon is known as ‘slip’ (Barnes, 1995).

Wall slip is caused by both static and dynamic geometric depletion effect. When the cement particles come in contact with the smooth cylinder wall, physical depletion occurs and the microstructures are attracted by the wall. It also happens if there is no rotation so it’s known as static geometric depletion. For very small particles local isotropy of Brownian motion is destroyed near walls. Electrostatic and steric effects arise between the wall and particles and they may cause slip. The dynamic effect arises from hydrodynamic forces which move the particles away from the wall when a torque is applied. When the torque is increased, the slip is also increased due to the increment of shear force gradient.

For reducing the slip effect, vane method was introduced. It has been vastly used in soil mechanics for measuring the shear strength of cohesive soil. Shear vane method is a direct method for measuring the static yield stress. The advantages of the vane test are its geometry. In vane test the material is yielded in a static condition within the material itself. So the problems and errors caused by the slip flow on smooth surfaces can be neglected. Also the vane doesn’t cause any significant disturbance to the sample before the measurements which is important for thixotropic materials. A typical four bladed vane and the vane technique is shown in Figure 4.2.
Vane test can be done in rate control mode or stress control mode. There are some assumptions for the vane test. One is the material yields along a cylindrical surface of the diameter and height of the vane. The second is the shear stress distribution is uniform on the cylinder and equal to the yield stress when the torque is applied. In vane test, usually a four bladed vane is completely immersed in the sample suspension. For minimize any effect in the cylinder boundary, the depth of the suspension and diameter of the cylinder should be twice as large of the dimensions of the vane. Then the vane is started rotate slowly at a constant rate. The torque is continuously measured and the yield stress is calculated from the maximum torque measured by the following equations (Nguyen and Boger, 1983)

\[
\tau_0 = \frac{T}{k}
\]

\[\text{Where, } k = \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3}\right)\]

So

\[
\tau_0 = \frac{2T}{\pi D^3 \cdot \left(\frac{H}{D} + \frac{1}{3}\right)}
\]

\[\text{………………..4.3} \]

\[\text{………………..4.4} \]

\[\text{………………..4.5} \]

A typical torque-time curve is shown in Figure 4.3. Here we can see an initial linear region and then a non linear region. The vane starts rotating from rest so the suspensions close to the edges of the vane blades deform elastically. The non linear region shows the viscoelastic flow where the network bond is

Figure 4.2  Schematic diagram of a four bladed vane and the vane technique (Chhabra and Richardson, 1999)
stretched exceeding their elastic limit and breaking occurs. As more bonds are stretched and resistance to deformation increases so the torque required to keep the rotation constant also increases. At the maximum stress the majority of the bonds are deformed and the flow starts and the material is yielded. The first yield stress $\tau_{y(s)}$ is said to be the onset viscoelasticity of the material. As microscopic flow is not yet started so it is known as the static yield stress. The peak or the second yield stress, $\tau_{y(d)}$ is the true yield stress or the dynamic yield stress as after this viscous flow occurs (Liddell and Boger, 1996). The peak of the yield stress value is defined as the static yield stress by Håkansson et al, 1993.

Some corrections had been made of the assumptions of the vane test. It has been found that the diameter of the sheared surface may be 5% larger than the blade dimension. It was also assumed that the stress distribution is uniform over the cylindrical surface but actually it peaks at vane end point. Using finite element modeling it is also found that the stress is highest at the vane edges (Nguyen and Boger, 1985).

**Stress Relaxation Method**

Stress relaxation method is a direct method for measuring the dynamic yield stress. A concentric cylinder consisting cup and bob is used in this experiment. In this method the residual stress acted on the bob by the sample is regarded as the yield stress. For reducing the slip of the suspension at the cylinder surface, vertical grooves are made. The test can be described in following steps

- The cement is mixed with up to desirable w/c ratio and then placed in to the gap between the cylinders.
- The bob is rotated at a chosen speed and the torque exerted on the bob is measured as a function of time.
- When the measured torque becomes constant the motor is suddenly stopped or it can be gradually decreased to a rotation of zero.

If the motor is suddenly stopped the remaining stress in the bob in a relaxed state is taken as the dynamic yield stress. If the suspension possesses a true yield stress then the remaining stress will be preventing the bob to come to its zero shear position. The bob will take long time to come to a relax

*Figure 4.3 A typical torque time curved observed by the vane method (Saak et al, 2001)*
state and the residual stress will be finite (Nguyen and Boger, 1983, Hakansson 1993). The torque is continuously measured and the dynamic yield stress can be calculated by equation 4.6.

\[ M = \tau \cdot 2\pi r^2 h_b \] ..........4.6

Here M is the measured torque and r and h are the radius of the cylinder and height of the bob respectively. Yield stress determined by the stress relaxation method is shown in Figure 4.4.

\[ \text{shear stress} \]
\[ \text{dynamic yield stress} \]
\[ \text{time} \]

Figure 4.4 Typical output from stress relaxation method. The minimum value denotes the dynamic yield stress (Håkansson, 1993)

**Bucket Rheometer**

The Bucket rheometer concept is developed using the same principle of the vane method as discussed in previous section. But instead of a cylinder here the vane is immersed in an infinite medium of geometry. In concentric cylinder viscometers the gap is small between the cup and bob and it is assumed that the stress is constant across the gap. Also in concentric cylinder viscometers we have wall slip, end effects, particle size distribution related problems. The vane method eliminates most of the problems but it is limited inside the cup. So the bucket rheometry is developed for an infinite geometry where no cup is needed. This method is vastly discussed by Fisher et al, 2007.
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In Figure 4.5 we have a schematic diagram of a concentric cylinder. Here the radius of the bob is $R$, radius of the cup is $\varepsilon R$, radius of the sheared region is $R_y$, and the bob is rotated at an angular velocity $\Omega$. If the vane height is $H$, at angular velocity $\Omega$, the measured torque is $T$ then the shear stress at the vane edge is

$$\tau_i = \frac{T}{2\pi R^2 H} \quad \text{..........................4.7}$$

The shearing radius, $R_y$ can be obtained by substituting the vane radius, $R$ and substituting the yield stress ($\tau_y$) for shear stress ($\tau_i$). It can be showed as

$$\tau_i = \frac{R_y^2}{R^2} \cdot \tau_y = \varepsilon^2 \cdot \tau_y \quad \text{..........................4.8}$$

From equation 4.8 we can find the critical cup diameter. If the cup radius is smaller than the shearing radius $R_y$ than the whole suspension will be sheared and if the cup radius is larger than the shearing radius than the suspension beyond $R_y$ will be static or very have a small stress. From the stress distribution diagram (Figure 4.5) we can say that when $\tau_1 > \tau_2 > \tau_y$, all the suspension inside the gap are completely sheared and when $\tau_1 > \tau_y > \tau_2$, partial shearing is occurred and the shear stress is reduced to zero at or before the wall. As the wall has no effect in measurements so this condition is considered as infinite. For this infinite region the relationship between angular velocity and shear rate is derived as

$$\Omega = \int_{R_y}^{R} \frac{\gamma}{r} \, dr = \frac{1}{2} \int_{\tau_1}^{\tau_y} \frac{f(\tau)}{\tau} \, d\tau \quad \text{..........................4.9}$$
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The expression of shear rate is derived as

\[ \dot{\gamma} = 2\tau_1 \frac{d\Omega}{dt} = \frac{2\Omega}{n} \]

Here \( n \) is the local gradient of a log-log plot of torque versus angular velocity. Equation 4.10 is valid for both cases, with or without yield stress. In shear vane method we measure the shear stress from equation 4.4. Equation 4.7 underestimate the actual shear stress and equation 4.3 over estimates the shear stress. End effect is a problem while using concentric cylinder viscometer or vane method. It has been showed that if height to radius ratio of the vane increases the significance of the end effect become less. If we use a height to radius ratio 12 and measure the stress using equation 4.7, the end effect can be eliminated and the error will be less than 5%. (Fisher et al, 2007).

The problem associated with concentric cylinder is that the determination of the shear rate and shear stress could be erroneous. Shear rate and shear stress are not uniformly distributed over the gap of the cylinder and there is no accurate method of determining the shear rate if the fluid model is unknown. The problems associated with determining the shear rate for partially sheared concentric cylinder consisting yield stress material is examined in detail by Nguyen and Boger (1987). However the yield stress has to be known for this measurement.

**Cone and Plate Viscometer**

In cone and plate geometry the sample is spread between a rotating cone and a fixed plate. The fluid is spread such a way so that it fills the gap between the cone and plate. In Figure 4.6 a cone and plate geometry is shown with 40 mm diameter and 1°59' angle. The gap between the cone and plate is

![Cone and Plate geometry](image)

The problems associated with determining the shear rate for partially sheared concentric cylinder consisting yield stress material is examined in detail by Nguyen and Boger (1987). However the yield stress has to be known for this measurement.

**Figure 4.6  Cone and Plate geometry (Chhabra and Richardson, 1999)**

51\(\mu\)m. The angle is kept less than 4° so the shear rate is less constant in the shearing gap. The small angle also causes some errors due to eccentricities and misalignment. All cone and plate viscometers allow the cone to be moved away from the plate to facilitate while sample changing. The cone and plate must be reset so that the tip of the cone is in the surface of the plate. For 1° gap angle and a cone radius of 50mm, every 10\(\mu\)m error in the axial separation produces an extra 1% error in shear rate. To avoid this error the cone can be truncated by a small gap (Barnes et al, 1989). The gap between the cone and plate is dependent on the particle size distribution. Usually a gap to maximum particle size ratio of 100 or more is used for the adequate measurement of bulk material properties. The relationship
between the shear rate and shear stress is determined from the torque required to rotate the cone and the angular velocity of the rotating element. Thus the flow curve is obtained. The shear rate and shear stress of the cone and plate geometry can be obtained by the following relations

\[ \tau = \frac{3T}{2\pi R^3} \] ........................4.11

\[ \gamma = \frac{\frac{\Omega}{\tan \alpha}}{R} \] ........................4.12

Here R is the radius of the cone, T is the torque, \( \Omega \) is the angular velocity and \( \alpha \) is the cone angle.

**Parallel Plate Viscometer**

In parallel plate viscometric geometry the sample is kept between an upper rotating and a lower stationary plate. The large gap between the plates overcomes the problems of misalignment and eccentricities comparing with cone and plate geometry. Due to the large gap, shear rate varies along the radius of the plates and it reduces the occurrence of plug flow. Figure 4.7 shows the schematic diagram of parallel plates. The shear rate and shear stress of parallel plate geometry can be obtained by following relationships

\[ \tau = \frac{2T}{\pi R^3} \] ........................4.13

\[ \gamma = \frac{\omega R}{h} \] ........................4.14

Here h is the plate distance, R is the plate radius, \( \omega \) is the angular velocity and T is the applied torque. Kuder et al, 2007 developed a customized parallel plate rheometer to measure the rheological properties of fiber reinforced cement paste with a /c ratio of 0.3, 0.35. They used square grooved plate to reduce the slip effect. The bottom stationary plate can be surrounded by a glass wall. This will introduce some frictional error but using a large diameter to gap ration (usually greater than 10) it can be minimized.
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Capillary Viscometer

Capillary viscometer is developed using the theory of circular pipe flow. If fluid is flowing through pipe where the radius ($r$), pressure difference ($\Delta P$) and volumetric flow rate ($Q$) is known over a definite distance ($L$) then the wall shear stress and the shear rate can be measured by equation 3.26 and 3.18 respectively for a Newtonian fluid. In case of Newtonian fluid the shear rate is zero at the center line. If the viscosity is changing over time then we have to make some corrections. In case of non-Newtonian fluids we have to change the apparent shear rate at the pipe wall to true shear rate. The apparent shear rate is given by equation 3.26 and the true shear rate is given by equation 3.29. The correction factor $\beta$ used here is known as Robinowitsch correction factor. The rheological property that means the flow curve and yield stress can be obtained by plotting the shear stress versus shear rate curve. For measuring $\beta$ we have to plot the log-log graph of $\frac{4Q}{\pi R^4}$ versus shear stress ($\tau_R$) measure the gradient. If we know the rheological model of the fluid then we can use it to find the rheological parameters.

While measuring the pressure drop per unit length extra care has to be taken. If the ratio of the tube length to the radius is very large (>100), entrance and exit effect has to be considered (Barnes et al, 1989). Wall slip error is another major problem in capillary viscometer. It occurs in case of concentrated dispersion where the particle layer is comparatively dilute near the wall. The thin, dilute layer near the wall has a lesser viscosity, resulting in apparent slippage of the bulk fluid along the wall.

The wall slip in capillary Rheometer was measured by Glieble and Windhab, 1985. They showed that the velocity distribution in circular pipe is the summation of the parabolic velocity and the wall slip velocity and can be shown as

$$v(r) = v_G + v^*(r)$$

Where $v_G$ is the slip velocity at the wall, $v^*(r)$ is the parabolic velocity function and $v(r)$ is the combined velocity profile. Two capillaries were used with the same pressure difference and the same $R/L$ ratio. The wall slip velocity can be measured as the gradient of the $V/\pi R^4$ versus $1/R$ curve (from transformed Mooney equation) when the shear stress is constant. In case of Kaolin-water it was found that the wall slip velocity increases rapidly with increasing shear stress.

Tube viscometer consisting of three pipes with different diameter is shown by Kotze, 2007. For laminar flow, the flow curves for different diameter pipes will coincide for the same set of data. The laminar flow log-log data are fitted with a mathematical function to obtain the first derivative which is the correction factor $\beta$. True shear rate is obtained and the shear stress versus true shear rate is plotted on a logarithmic scale. The data are fitted with appropriate mathematical model and hence rheological parameters are achieved.
4.1.2 Field Measurements of Rheological Properties

All these methods described above are not robust and rather sophisticated. They can be implemented only in the laboratory. So different procedures are followed in the field. In this section different methods that are used in the field for measuring rheological properties are discussed.

Marsh Cone

The Marsh cone is a simple tool, originating from the oil industry, that is used to measure the fluidity of cement grouts in field. Actually it can be said as a workability test to check the specification and quality control of cement pastes and grouts. It consists of a truncated cone at the lower end and a pipe shaped nozzle. The time needed for a certain amount of grout to flow out of the cone is measured. The longer the time for the grout to flow out of the cone, the lower the fluidity. The geometry of a marsh cone is shown in Figure 4.8

![Geometry of a Marsh cone](image)

**Figure 4.8 Geometry of a Marsh cone, (Roy and Roussel, 2005).**

A theoretical model was developed by Håkansson, 1993 to measure the flow time required for the grouts assuming it as a Bingham fluid. By using the model the concern was to correlate the fluidity with rheological properties such as viscosity. By using known yield stress and known density of the grout as input it was possible to determine the viscosity, by using specific nomogram. He also indicated that it is very important to know the shear rate range while measuring viscosity and yield stress using different methods. A comparison of determination of measuring viscosity using the rotational viscometer and marsh cone is shown in Figure 4.9. It was found that due to different range of shear rate, the linear regression of Bingham model was made at low shear rate range in case of rotational viscometer and the measured viscosity was higher than that measured by marsh cone.
Roussel & Roy, 2005 also established a relationship between the fluidity and the rheological properties e.g. viscosity, yield stress by using analytical models to predict the flow time. Two cones, slightly differed in their nozzle configurations are used so that different flow time values can be obtained. Using these two flow time values other parameters are calculated and the method is known as ‘two cone test’.

Nguyen et al, 2006 established a semi analytical and numerical model to measure the flow time of cement grouts. The values obtained by their model shows a deviation of less than 15% with the experimental values. However, the authors concluded that further work have to be done in order to measure the rheological properties by this model.

**Plate Cohesion Meter**

With the Marsh cone one can only measure the fluidity of the sample, i.e. the time taken for a certain volume to flow out of the cone. The flow time defines only a series of values of the two properties, viscosity and yield stress. To find out the viscosity the yield stress (cohesion) has to be known. A simple method known as ‘plate cohesion meter’ was designed by Lombardi, 1985. In this method a thin steel plate with rough surface where grout can stick. If the specific weight and the amount of the grout sticking on the plate are known then the cohesion per unit weight $\frac{C}{\delta}$ can be determined. It is the thickness of the grout layer sticking on both side of the plate. A graphical relationship of flow time versus cohesion and viscosity has been developed as shown in Figure 4.10. By using the cohesion per unit weight obtained from plate cohesion meter, the viscosity can be determined.
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**Raise Pipe**

The Raise pipe is a simple device to measure the yield stress of cement grouts developed by Håkansson, 1993. Here the grout is intruded into a vertical raise pipe and the principle is that the flow stops when the maximum shear stress at the pipe wall is below the yield stress. The density of the fluid must be known for this test.

Here one large radius pipe (R=30 mm) and two small radius pipes (R=2-4 mm) are connected and placed on a bottom plate. The grout flows from the the large pipe and into the smaller pipes. When the grout reaches the bottom plate it will start rising in the smaller pipes. If the grout has a yield stress the height, at steady state, of the fluid in the large pipe and the small pipes will be different. By measuring the difference in height in between the pipes, the yield stress can be estimated. When the grout stops rising in the smaller pipes, the plug has reached the pipe wall and hence the shear stress at the wall is equal to the yield stress. By using the formula of vertical force balance it can be showed that

\[
\tau_0 = \gamma R \left( \frac{\Delta h}{h_1 - \Delta h} \right)
\]  

\[\text{.................4.15}\]

Where \( \gamma \) is the specific weight of the fluid, \( h_1 \) is the height of the fluid inside the large pipe and \( \Delta h \) is the height difference of the fluid between the two pipes. A schematic diagram of the raise pipe is shown in Figure 4.11
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**Stick Method**

*Axelsson and Gustafson, 2006* proposed a simple and robust method to measure the yield stress of cement based grouts. They developed it for field use as yield stress is an important parameter to determine the grout penetration and also the existing methods that are used in the field are primitive. A simple stick with a weight attached at the bottom is used here. The stick is allowed to sink in the grout and the sink depth is measured. A schematic diagram of the test is shown in Figure 4.12.

![Figure 4.12 Schematic diagram of the stick method. \( l \) is the length of the stick penetrated. (*Axelsson and Gustafson, 2006*)](image)
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The yield stress is measured by the following equation

\[ \tau_0 = \frac{m_s \cdot g - l \cdot \pi r^2 \cdot g \cdot \rho_g}{2\pi \cdot l \cdot r + 9 \cdot \pi r^2} \]

………………..4.16

Here \( l \) is the length of the stick in the grout, \( r \) is the radius of the stick, \( \rho_g \) is the density of the grout, \( m_s \) is the mass of the stick, \( g \) is the acceleration due to gravity and \( \tau_0 \) is the yield stress of the grout. So the yield stress can be measured once the stick penetration is known.

The values obtained from the stick method was compared with the values obtained by rotational viscometer and the maximum deviation was found approximately 20% in the regression analysis. The shear rate was found in between 0.2 and 2s\(^{-1}\) which is mentioned as in the same shear rate range with the rotational viscometer. Moreover it follows same pattern while grout penetrates into the fractures.
Chapter 5

Ultrasound Physics

5.1 Introduction

Sound wave propagates through air or water by mechanical vibration. Sound is propagated 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 a form of the sound wave. Ultrasound is just like the sound waves. Sound waves can be propagated transversely or longitudinally. It is incapable of travelling through vacuum because energy or momentum is transferred in wave motion through the molecules. Transverse waves are also called shear wave 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.

Some technical terms are discussed also in this section to give a clear view on ultrasound. The amplitude of a sound wave comes from the change of the 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 measured from the product of the frequency and wavelength. It can be showed by the following equation

\[ c = f \times \lambda \], where
\[ c = \text{Acoustic velocity in the medium (m/s)} \]
\[ \lambda = \text{Acoustic wavelength (m)} \]
\[ f = \text{Frequency (Hz)} \]

The audible sound frequencies are below 15 kHz-20 kHz. Any sound waves consisting frequencies higher than the human hearing threshold, i.e. 20 kHz-105 MHz is known as ultrasonic waves.

5.2 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 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
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\[ \kappa_{AD} = \rho_0 \left( \frac{dP}{d\rho} \right) \], where \[ \kappa_{AD} = \text{adiabatic linear bulk modulus of elasticity (N/m}^2) \]
\[ \rho_0 = \text{equilibrium density} \]

The adiabatic linear bulk modulus of elasticity indicates the stiffness of the resistant 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 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

\[ \chi_{AD} = \kappa_{AD}^{-1} = \rho_0^{-1} \left( \frac{d\rho}{dP} \right)_{p \rightarrow 0} \]  \[ \kappa_{AD} \rightarrow \text{adiabatic linear bulk modulus of elasticity (N/m}^2) \]
\[ \rho_0 \rightarrow \text{equilibrium density} \]

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

\[ c = \sqrt{\frac{\kappa_{AD}}{\rho_0}} = \sqrt{\frac{1}{\chi_{AD} \cdot \rho_0}} \], where \[ c = \text{Sound velocity in liquid (m/s)} \]

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

\[ \alpha_0 = \left( \frac{-1}{A_{Max}} \right) \left( \frac{dA_{Max}}{dx} \right) \], where \[ A_{Max} = \text{wave amplitude of the transmitted ultrasound wave} \]

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

\[ A_{Max} = A_{Max0} \cdot e^{-\alpha_0 \cdot c} \]  \[ \alpha_0 \rightarrow \text{spatial attenuation coefficient} \]
\[ c \rightarrow \text{wave amplitude of the transmitted ultrasound wave} \]

It is shown in Figure 5.1.
The attenuation coefficient depends on the material. So while choosing the pipe material, materials with a high attenuation coefficient should be avoided.

**Acoustic Impedance**

Acoustic impedance is a physical property of the material. It measures the refraction and transmission of energy at the boundary between two successive medium. While choosing the medium, acoustic impedance is an important factor for optimizing the energy transfer from one medium to another. The acoustic impedance, $Z$ can be expressed as

$$Z = \rho \times c,$$

where

$Z = \text{Acoustic impedance (kg/m}^3\text{s = Ray)}$

$C = \text{sound velocity (m/s)}$

$\rho = \text{density of transmitting medium (kg/m}^3\text{)}$

It is possible to obtain the non invasive measurements through pipe wall if the acoustic impedance of the wall material is greater than the acoustic impedance of the liquids used. But the acoustic impedance of the wall material should not be larger than twice or thrice of the acoustic impedance of the liquid.

### 5.2.1 Propagation of Ultrasound through the Interface between Two Layers

Reflection and refraction occurs when ultrasound waves pass through the boundary of two layers. While passing through every interface between two layers ultrasound waves will reflect and refract at different angles. The change of angles depends on the acoustic properties of the two materials. A correlation between the incident angle and refractive angle, sound velocities of different layers is established by snell’s equation. It was established for optics but found valid for all wave propagation and it can be expressed as
\[ \frac{\sin \theta_1}{\sin \theta_2} = \frac{c_1}{c_2}, \text{ where} \]

\[ \theta_1 = \text{angle of incidence} \]
\[ C_1 = \text{sound velocity in medium 1} \]
\[ \theta_2 = \text{angle of refraction} \]
\[ C_2 = \text{sound velocity in medium 2} \]

It is explained by Figure 5.2.

\[ I_1 = \text{incident intensity} \]
\[ I_2 = \text{transmitted intensity at layer 2} \]
\[ I_1' = \text{reflective intensity at layer 1} \]

The distance between the boundary layers is \( d \) along the \( x \) axis. Here 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 \( \theta_2 \). Depending on the acoustic properties of the materials in the same way it propagates from layer 2 to layer 3. From Snell’s law we can say that the higher the sound velocity in layer 2 the larger the refraction angle \( \theta \) will be.

While propagating through the boundary layers the ultrasound wave will loss some energy due to the acoustic impedance of the materials. In Figure 5.2 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 \( I_1' \). So the initial intensity of the ultrasound wave can be expressed as

\[ I_1 = I_2 + I_1' \]

The reflection coefficient can be expressed as

\[ \text{Figure 5.2 Ultrasound wave propagating through two materials (Ouriev, 2000)} \]
The transmission coefficient can be written as
\[ \kappa_i = \frac{I_2}{I_1} \] ..........................5.11

The total acoustic energy at the boundary of two layers consists of the incidence intensity, transmitted intensity, reflective intensity and can be expressed as
\[ r_i + \kappa_i = 1 \] ..........................5.12

The reflection coefficient can be showed as the function of the material acoustic impedance and the corresponding angles and can be expressed as
\[ r_i = \left( \frac{z_1 - z_2}{z_1 + z_2} \right)^2 \] ..........................5.13

From equation 5.12 it can be showed as
\[ r_i = 1 - \kappa_i = \frac{4z_1z_2}{(z_1 + z_2)^2} \] ..........................5.14

The incident wave will be fully reflected when \( \theta = 90^\circ \). The corresponding \( \theta_i \) 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
\[ \theta_i = \arcsin \left( \frac{c_1}{c_2} \right) \] ..........................5.15

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.

5.2.2 Ultrasound Transducer

For ultrasonic measurements, transducers developed by Met Flow, SA has been used by most of the authors (Wiklund 2007, Birkhofer, 2007, Kotze 2007). Transducer frequencies are available of 0.5, 1, 2, 4, 8 Hz and the suitable frequencies had been used. The sound field generated by the transducer is divided in to two fields, near field and far field. In near field, measurements are avoided because
ultrasonic 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

\[ N = \frac{D^2 f_0}{4c} , \text{ where} \]

\[ \frac{N}{D} = \frac{f_0}{4c} \]

\( N \) = near field distance,
\( D \) = active element diameter
\( f_0 \) = basic ultrasound frequency
\( c \) = sound velocity

The acoustic wave propagation from an ultrasound transducer is shown in Figure 5.3

![Figure 5.3 Schematic diagram of the sound wave generation from an ultrasound transducer (Met-Flow, 2000)](image)

The near field distance, \( N \) is the natural focal point of the transducer.

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

Several ultrasound transducers, 1-3 can be used and the data’s are combined. For non invasive 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 flush with the pipe through small cavities in front of the transducers as shown in Figure 5.4. They are installed in such a manner that the near field region is equal to the distance of the transducer from the pipe wall. This is done to avoid the near field problem and measurements in near field zone where the sound field is irregular and not fully developed. If the transducer is mounted in wall then there will be loss of ultrasound energy due to the pipe wall. The doppler angle will be incorrect due to the refraction of ultrasonic wave in pipe wall. It was found that
it is possible to achieve good results when the transducer is in direct contact with the fluid. It is possible to use two transducers in the opposite direction and it is used while the acoustic measurements.

It is also possible to use a thin wall membrane, e.g. polymethyl methacrylate (PMMA) in the near field region to separate the transducers from the flow channel.

Figure 5.4  Schematic diagram of flow adapter cell with two transducers
Chapter 6

Principles of Ultrasound Velocity Profiling (UVP)

6.1 Ultrasound Doppler Theory

Using sound waves for measuring the distance between two points was invented by mankind due to their own need of measuring distances and this method has been continuously developed since last century. Bats use sound waves for their navigation and to measure the distances with 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 arises 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 introduced 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’ leaded the research of applying ultrasound in non destructive material testing and now a days it is one of the most frequently used application (Wiklund, 2003).

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

6.1.1 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, then the frequency of the wave to the observer is different than the source. This phenomenon is said to be Doppler shifted and known as Doppler effect. The Doppler 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 than a
different frequency will be observed and the sound intensity will be changed. The basic frequency of an ultrasonic wave can be expressed by the following equation

\[ f = \frac{c}{\lambda}, \text{ where} \]

\[ f = \text{emitted frequency} \]
\[ c = \text{velocity of sound wave in the medium} \]
\[ \lambda = \text{wavelength of the emitted ultrasound wave} \]

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

\[ f_{\text{observer}} = f_{\text{source}} \left[ \frac{c - v_{\text{observer}}}{c - v_{\text{source}}} \right], \text{ where} \]

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

6.2 Principles of Ultrasound Doppler Velocimetry (UDV)

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 measured. This method was subsequently extended by Takeda to apply it in other fields of engineering. The principles of ultrasound Doppler velocimetry are vastly discussed in his literatures (Takeda 1985, 1990, 1995).

The principles of ultrasound doppler velocimetry are shown in Figure 6.1. 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.
Chapter 6: Principles of Ultrasound Velocity Profiling (UVP)

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

\[ \text{Position: } x = \frac{c \tau}{2} \quad \text{Velocity: } v_x = f_D c / f_0 \]

Figure 6.1  Principles of Ultrasound doppler velocimetry (a) transducer emitting ultrasound wave (b) received ultrasound signal (c) velocity profile (Takeda, 1995) (Bottom) Transmission and reflection from a moving particle inside the acoustic beam (Ouriev’2000)

The transducer is static with respect to the reflector particle and if the particle is moving with non zero velocity inside the acoustic wave then doppler shift occurs. The received signal frequency is doppler shifted and can be expressed as
Chapter 6: Principles of Ultrasound Velocity Profiling (UVP)

\[ f_r = f_e \left[ \frac{c - v_{Tr}}{c + v_{Tr}} \right] \]

where \[f_r\] is received frequency, \[f_e=f_0\] is the emitted frequency and \[v_{Tr}\] is the velocity of the transducer which is static. In Figure 6.1 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

\[ v = \frac{c \cdot f_d}{2f_e \cos \theta} \]

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 than the distance of the moving particle from the transducer, \(x\) along the measuring axis can be showed as

\[ x = \frac{c \cdot t}{2} \]

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 time period is very small and it is known as gate. Each gate gives a single value of velocity. This is measured from the mean value of total doppler energy of that certain time. It should be set before the experiment when the gate will be open after the pulse transmission and the desired sampling time should be set. The gate is closed after each transmission and the next pulse is transmitted after the previous one has reached to its maximum depth. It is known as pulse repetition frequency. This process is repeated until enough gates are available for a complete velocity profile. Windowing function is used to fix the measurement volume and their distance from each other. To obtain a good resolution a large number of gates are desirable. In Figure 6.2 schematic diagram of a measuring window is shown. In UVP-DUO-MX from Met-Flow SA, Switzerland it is possible to choose 10-2048 gates as required.
Chapter 6: Principles of Ultrasound Velocity Profiling (UVP)

6.6

6.7

The sampling frequency and the aliasing

Analog data’s are received in pulsed doppler ultrasound instruments and the analog signal is converted to digital signal by sampling the signal in certain points. Sampling frequency is determined from sampling interval which is the time between two successive pulse emissions. The sampling frequency is the sampling rate in unit time. It is known as 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. So it can be expressed as

$$ f_{\text{max}} \leq \frac{F_{prf}}{2} $$

Here $f_{\text{max}}$ is the Nyquist frequency. If the measured frequency is higher than the Nyquist frequency than 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 ultrasonic 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

$$ P_{\text{max}} \leq \frac{c}{2F_{prf}} $$

Figure 6.2 Diagram of measuring window in ultrasound beam (Ouriev, 2000)
Here $P_{\text{max}}$ is the maximum measurable depth, $c$ is the velocity of transmitted pulse in the fluid medium and $F_{\text{prf}}$ is the pulse repeated frequency. So we can see that for a larger depth lower frequencies should be used.

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

$$V_{\text{max}} = \frac{cF_{\text{prf}}}{2F_0}$$

So the limiting condition can be obtained from equation 6.7 and 6.8 and can be expressed as

$$P_{\text{max}} \cdot V_{\text{max}} = \frac{c^2}{8F_0}$$

We can see that in constant pulse velocity and emitted frequencies maximum depth and velocity is dependent on each other hence a consideration has to be made (Takeda, 1991). In higher velocities the penetration depth will reduce and in higher velocities vice versa. Higher velocities can be achieved at the same penetration depth by reducing the transducer frequency.

*Doppler shifted frequency or time-phase lag*

In UVP we do not use the doppler shifted frequency ($f_d$). We measure the time-phase lag for the reflection of emitted US wave for each particle. The pulse repetition frequency ($F_{\text{prf}}$) is used to determine the velocity of the particles. We use 4MHz or 2 MHz ultrasound transducer for ultrasound wave emission and the occurred doppler shift is very small around 4 Hz. So it is impossible to detect such small doppler shift by these transducers. There is an ongoing debate that if it should be called a doppler shifted method.

*Doppler angle*

Doppler angle is not the inclined angle between the transducer and the adapter wall. It is the angle between the ultrasound beam and the direction of the particles along the velocity stream. The ultrasound doppler instruments measure velocities along the ultrasound beam axis, so it should be multiplied by a factor $1/\cos\theta$ to achieve the velocity in the direction of the main flow. An illustration of doppler angle is showed in Figure 6.3. Here both the particles in Figure 6.3 are moving with the same velocity so travelled distance $\Delta s$ is same for the both case. $\Delta d_1$ and $\Delta d_2$ are the
distance travelled relative to the beam direction. If the doppler angle is lower, as we can see in the Figure 6.3, \( \theta_1 < \theta_2 \), the perpendicular distance \( h_1 \) is larger than \( h_2 \). A higher perpendicular distance will cause a velocity spread for all the particles inside the beam and generate a spectrum of doppler shift frequencies. The accuracy of the measured velocity is dependent on the pulse doppler spectrum so a narrow spectrum is desirable. Based on literatures a doppler angle of 60°-80° is optimum for the accuracy of velocity estimation (Birkhofer, 2007).

### 6.2.1 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 so the time resolution is limited to 10 ms. Time domain algorithms are superior to FFT but does not provide the whole spectra. Not so much information about the quality of the measurements are available in time domain. So time domain method is found disadvantageous for real time and high time resolution equipments. From FFT we can have the spectral distribution of the velocity profile. So we know actually in what volume we are measuring the velocity profile and the quality of the measurement is also known.

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 6.4.
6.2.2 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 UVP monitor delivers the outcome. It is based on the ultrasound pulse doppler velocimetry principle. Wiklund 2007, Birkhofer 2007, Kotze 2007 used UVP-Duo instrument of Met-Flow, SA. UVP Duo can emit pulses of 1 cycle to 32 cycles with a base frequency of 0.5 Hz, 1 Hz, 2 Hz, 4 Hz and 8 Hz. The emitted voltage can be 30, 60, 90 and 150 V peak-peak. The maximum pulse repetition frequency is 244 Hz. It is possible to use up to 2048 gates/channels to obtain a good resolution of the velocity profile. The monitor has an integrated multiplexer with 20 BNC connections. So it is possible to use multiple transducers and flow mapping. The UVP monitor adjusts the attenuation of the received echo signal as it increases with increased depth. It is done by an amplification procedure which follows the exponential law. A detail description can be found in Birkhofer 2007, Met-Flow, 2002.
Chapter 7

UVP-PD Method

7.1 Introduction

The term UVP – PD represents ultrasound velocity profiling using pressure difference. It consists of the continuous measurement of various rheological parameters using ultrasound pulsed doppler velocity profiling and the pressure difference between two certain points. The concept of combining UVP with pressure difference was developed before 1993 but it was implemented after 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 done by Ouriev (2000). Recent developments were done by Wiklund (2007), Birkhofer (2007).

7.2 Principles of UVP-PD

The basic of the UVP-PD comes from the pipe viscometry concept. The working principle of pipe and capillary viscometer can be derived from a force balance over a cylindrical fluid element in fully developed, steady state, laminar flow in a pipe. Single point rheological parameters are obtained from pressure drop and volumetric flow rate. The derivations for the shear stress, shear rate, viscosity, yield stress is shown at chapter 3. UVP-PD instrument is basically a tube viscometer with multi point measurement. The basic principle of the UVP-PD and the link between the velocity distribution, shear stress, shear rate, viscosity can be shown by Figure 7.1

![Figure 7.1](image)

*Figure 7.1 Link between velocity distribution, shear stress, shear rate and viscosity for a Newtonian fluid (n=1) and shear thinning fluid (n<1)*
Examples of Newtonian and shear thinning fluid are shown in the Figure 7.1 for five moving reflectors inside a pipe. In the first segment we can see the velocity distribution over the diameter of the pipe. In the second segment distribution of the shear stress which is independent of the velocity profiling is showed. The third segment shows the distribution of shear rate over the pipe diameter which is dependent on the velocity profiling. In the fourth segment the viscosity and yield stress is shown which is derived from the shear rate and shear stress.

7.3 Data Acquisition and Software

The UVP-PD method uses the principle of ultrasound doppler velocimetry (chapter 6) in combination of the pressure difference measurements. The velocity in different point is obtained from the velocity profile and pressure difference is calculated from the pressure sensors. By using the rheological models flow curve and other parameters are determined. The UVP-PD setup consists of 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 centrifugal pump. A personal computer with data acquisition software is also connected. The data acquisition and processing steps are shown in Figure 7.2. The data acquisition steps and the UVP-PD set up varies on the type of the experiment. A MATLAB (Math Works, Natic, MA,USA) based application with Graphical User Interface (GUI) has been developed (Wiklund et al 2007) for data acquisition and processing. The following tasks can be done by this software as shown in Figure 7.2

- Data acquisition and processing from UVP monitor, pressure sensors and temperature sensor.
- Measurement of the velocity profile
- Measurement of rheological properties from the velocity profile and pressure drop
- Post data processing
- Flow curve visualization
The data is acquired and stored in the memory as Matlab file. It is possible to read a saved MatLab file associated with UVP monitor application or raw binary data. So it can be stored in-line also used for post processing. The velocity profile is estimated from the power spectra of the baseband signal. It is possible to fit the flow curve using rheological models such as power law, Herschel Bulkley etc or by using the gradient method. The following data visualizations can be possible after data processing.

Figure 7.2 Data acquisition and processing flowchart of the UVP-PD method (Wiklund, 2007)
Chapter 7: UVP-PD Method

- Power spectra of a single channel of a single profile or the average of several profiles including the velocity estimation
- Velocity profile (from time domain), RMS and peak-peak amplitude of the base band signal, amplitude of the RF signal, SNR from the power spectra, first and second derivative of the velocity profile
- Rheological properties such as viscosity, yield stress and shear stress, shear rate, plug radius, the resulting curve from fitted model.
- Consistency index such as n, k, pressure drop, velocity of sound, temperature and maximum flow velocity.
- Animations of the velocity profile over time.

These features are under continuous development and depend on the individual requirements of the various research groups.

7.4 Acoustic Characterization

Sound velocity is an important parameter in UVP-PD method. If the measured sound velocity is incorrect the velocity of the fluid particles will be incorrect also. Individual method and flow adapter has been developed to measure the sound velocity both off-line and in-line. In Figure 7.3 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 other works as a receiver.

![Schematic diagram of the transducer setup for acoustic measurements off-line](Wiklund 2007)

This technique is known as ‘pulse echo time of flight’ or ‘acoustic time of flight measurement’. The ultrasonic pulse emitting transducer is connected to a UVP monitor and the receiver transducer is connected to an oscilloscope. The oscilloscope is connected to 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. Attenuation decreases with an
increasing concentration of solids in the fluid. Distilled water is used as reference sample to measure the variation of attenuation and peak-peak voltage. The velocity of sound measurement is temperature dependent. The sound velocity increases approximately 50 m/s over the temperature range of 25-50°C. The acoustic measurements for UVP-PD setup is vastly described by Wiklund, 2007.

7.5 Previous Studies Based on UVP-PD Method

This section presents a brief description of various published research studies based on ultrasound doppler velocimetry in combination with pressure difference technique. Lots of verities of fluids have been used so far so it’s rather difficult to put them together in different groups and certainly there are other groups who are also using this method.

P. Brunn, University Erlangen-Nurnberg, Germany

The idea of combining pressure difference with the ultrasound doppler velocimetry was first presented by Brunn et al, 1993. They introduced three rheometers, two optical instruments and one acoustic rheometer based on the UVP-PD combination. Flow curve of aqueous hydroxypropyl solution and aqueous polyacrylamide solution were achieved for different concentration. These results were compared with a rotational viscometer and showed good agreement. Muller et al, 1997 used the same flow loop to investigate the feasibility of UVP-PD method. 4000 ppm aqueous polyacrylamide solution with very high non-Newtonian behavior was analyzed and the achieved shear viscosities were also compared with commercial rotational viscometer and good agreement was found. Wunderlich and Brunn, 1999 used ultrasound pulse doppler method combining pressure difference to measure the flow process of polyacrylamide solution. The velocity profile was fitted using power law model. It was an experiment to find the applicability of UVP-PD as a quality control. Brunn et al. 2004 used the UVP-PD method to measure the rheological properties of body lotion. They measured the velocity over radius, change of shear stress with shear rate and the slip velocity over wall shear stress.

E. Windhab, ETH Zurich

The feasibility studies of UVP-PD method for characterization of rheological flow behavior was first studied in ETH in a semester work (Cantz, 1994) and in a diploma work (Drost et al, 1994). Later on developments of the method were done by his PhD student B. Ouriev at the lab of ETH. Ouriev published lots of research papers from his PhD work (Ouriev, 2000). Power law model was applied by Ouriev and Windhab for measuring the flow process of highly concentrated non-transparent food suspension. At one application they made direct in-line measurements in transporting pipe of chocolate crystallization process (Ouriev and Windhab, 1999) and in other it was done in the transporting pipe of the fat crystallization (Ouriev et al, 1999). It was found that the experimental process strongly affects the rheological flow properties. Wall slip measurements of shear thickening fluids were done by Ouriev (2002) and discussed in detail. Corn starch in glucose syrup diluted by water was used as shear thickening material and the sound velocity was measured 1830 m/s. Time averaged flow mapping was done by Ouriev and Windhab (2003) for shear thinning and shear thickening suspensions. Ouriev and Windhab (2002,2003) discussed the transient flow and the pressure driven shear flow of highly concentrated solutions. Power law fit and Herschel Bulkley fit both was used in this case to measure the yield stress and plug radius. Measurements of pulsating pipe flow of chocolate suspension in precrystallization process were discussed by Ouriev et al’2004. The
material was chocolate suspension including cocoa butter with 60% solids. It was an industrial application. During the PhD studies Beat Birkhofer, fat crystallization process of cocoa butter was monitored using the UVP-PD method (Birkhofer et al, 2007).

J. Wiklund and M. Stading, SIK, Sweden and R. Kotze, CPUT, South Africa

Johansson and Wiklund (2001) made the measurements of shear thinning surfactant solutions and cellulose suspensions using the UVP-PD method with power law fit and Herschel Bulkley Fit. It was a diploma work and was done at the laboratory of the ETH. These results were also compared with the conventional off-line methods. The findings were presented by Wiklund et al 2001. Later on he continued his PhD studies at SIK. He made a literature review of the ultrasound doppler based applications combining pressure difference technique used in the food industry (Wiklund, 2003). A comparative study of UVP and LDA techniques for pulp suspensions was done by Wiklund et al, 2004 and Wiklund et al, 2006. It showed that the UVP technique can be used to determine the yield stress directly in-line. It was also found that by using UVP a velocity gradient close to the pipe wall can be achieved. Wiklund and Stading (2008) and Wiklund et al (2010) discussed the UVP-PD measurements of various food and industrial suspensions such as cheese sauce, fruit yoghurt, vegetable sauce, glycerol, slurries, tomato sauce, sunflower oil, aqueous syrup etc. These were done in order to find the applicability of UVP-PD method to highly concentrated suspensions containing anisotropic particles of various sizes and shapes. Wiklund et al (2007) discussed the methodology of the UVP-PD technique including the other details of signal processing, statistical methods etc. All these literatures are combined in the PhD thesis, Wiklund (2007). Kotze (2008) measured the rheological properties of highly concentrated mineral suspensions using UVP-PD method. He also compared the results with capillary viscometer and other off-line rheometers and found a good agreement. It was a masters thesis and was done at SIK. Later on he continued his PhD studies. Measuring flow behavior of concentrated suspensions in complex geometries using UVP-PD was presented by Kotze et al (2010). They showed that UVP-PD is a suitable method for measuring the flow behaviors in complex geometry.

R. Powell, K. McCarthy and M. McCarthy, UC Davis, USA

Lots of works with the UVP-PD method were done at the laboratory of M. J. McCarthy, UC Davis, USA. A study on complex fluid was done by Powell et al, 2006. As complex fluids they studied Microcrystallinecellulose gel, Xantham gum solution, modified and unmodified starch gels, polymer melts etc. The obtained data were compared with conventional rheometers results and it showed a good agreement. As this method can be used to measure shear viscosity data over a wide range of shear rate with a single measurement so the shear rate limitations were investigated and a design curve was generated to predict the deviation of shear viscosity from the rotational rheometers at low shear rate. Dogan et al, 2002 used the UVP-PD method to measure the rheological properties of diced tomatoes suspended in tomato slurries. The diced tomato suspension comprised of 1-3 mm particles suspended in tomato slurrries. They obtained velocity profiles in four different flow rates. Yield stress and apparent viscosity were measured and compared with conventional rotational viscometer results. Dogan et al, 2003 measured the flow of tomato concentrated with three different amount of solid content. They used power law and casson model to fit the data. A shear thinning behavior and yield stress was achieved. These data were compared with capillary viscometer. Dogan et al, 2005a investigated the rheological properties of 6% (w/w) acid thinned and native corn starch including gel. They observed an increment of consistency index and a decrease of flow behavior index with cooling and a thermo reversible change in the flow behavior index was observed in the native corn starch after
storage. Dogan et al, 2005b investigated the viscosity, shear stress at different shear rate range, velocity profile of the polymer melt suspensions and compared it with the conventional viscometers results. Choi et al, 2002 measured the flow properties, i.e., viscosity, yield stress of 65.7° Brix corn syrup and 4.3° Brix tomato juice using UDV and compared it with the results obtained by MRI. Choi et al, 2005 developed a Matlab based graphical user interface program and implemented for MRI and UDV based viscometry. The main data processing consists of velocity image reconstruction, velocity profile generation, velocity profile curve fitting, rheograms construction and constitutive model fitting.
Due to ease of preparation and use, wide availability and relative low cost, cement-based 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 achieve as fine cement as possible. However, it has been found that very fine cement are difficult to handle due to the increased interaction between the particles that comes with increasing specific surface.

In this work, a relatively fine cement has been used, Cementa IC30. It has been found that this has superior characteristics with respect to penetrability compared to more fine cements (Draganovic, 2010).

8.1 Micro Cement

Micro cement Injektering 30 from Cementa was used to prepare the mixture of the cement grouts. Injektering 30 is a sulphate resistant, chromate reduced and low alkaline injection cement.

Particle size distribution

Injektering 30 has a particle size distribution where 95 percent of the materials are less than 30 μm in particle size. The particle size distribution is shown in Figure 8.1.
Properties of micro cement Injektering 30 are shown in Table 8.1

Table 8.1 Properties of Injektering 30 from Cementa

<table>
<thead>
<tr>
<th>Properties</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Physical properties</td>
<td>Compact density approximately 3100-3200 kg/m³. Bulk density 800-1500 kg/m³.</td>
</tr>
<tr>
<td>Chemical properties</td>
<td>MgO maximum 5% by weight. SO₃ maximum 3.5% by weight and chloride maximum 0.1% by weight.</td>
</tr>
<tr>
<td>Setting time</td>
<td>Setting time for Injektering 30 is 100 minutes.</td>
</tr>
</tbody>
</table>

8.2 Additive

Cementa SetControl II was used as additive. Cementa SetControl II is a high performance binding time regulator and dispersing additive that is especially suitable for grouts based on Injektering 30, Ultrafin 16 and Ultrafin 12. It is based on sulphonated naphthalene polymers and nitrate. The color of the SetControl 2 is yellowish brown. Its density is 1476 kg/m³, pH value approximately 6 and dry content 45%.
8.3 Sample preparation

Total 8 batches of experiments were made using w/c ratio 0.8 and 0.6 with and without set control. Sample preparations for different batches are shown in Table 8.2. The mixing time was 4 minutes.

Table 8.2 Sample preparation for different w/c ratio

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>W/c ratio</th>
<th>Set Control (w/w)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch 1</td>
<td>0.8</td>
<td>-</td>
</tr>
<tr>
<td>Batch 2</td>
<td>0.6</td>
<td>2</td>
</tr>
<tr>
<td>Batch 3</td>
<td>0.8</td>
<td>-</td>
</tr>
<tr>
<td>Batch 4</td>
<td>0.8</td>
<td>-</td>
</tr>
<tr>
<td>Batch 5</td>
<td>0.8</td>
<td>-</td>
</tr>
<tr>
<td>Batch 6</td>
<td>0.8</td>
<td>2</td>
</tr>
<tr>
<td>Batch 7</td>
<td>0.6</td>
<td>2</td>
</tr>
<tr>
<td>Batch 8</td>
<td>0.8</td>
<td>2</td>
</tr>
</tbody>
</table>
To test the feasibility of the UVP-PD equipment under practical grouting conditions it has been important to use as much standard equipment as possible. In order to achieve field like conditions, an ordinary grouting rig has been used, including normal pressure hoses. The grouting rig is placed outside on a parking lot and connected to the UVP-PD equipment, placed under a garage roof, and combined into an experimental flow loop.

9.1 Flow Loop Characteristics

The experimental flow loop consists of the following instruments

- UNIGROUT E22H
- LOGAC
- UVP flow adapter
- Pressure sensor
- Temperature sensor
- Grouting hose
- Stainless steel pipe

A schematic diagram of the flow loop is shown in Figure 9.1. Two pressure sensors in the flow adapter were separated by 1.3 m for 4 MHz transducers. 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 degree Celsius. One ball valve was added with the agitator to control the flow of the grout mixture when it was returning back to the agitator tank. A gate, or lever, was integrated with the mixer to control the flow from the mixer to the agitator. Another ball valve was integrated with the pump to control the flow from the agitator to the UVP-PD test section. This valve was also used to set the desired pressure before the circulation of the flow, see Figure 9.1.
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9.1.1 UNIGROUT E22H

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

*Grout mixer, Cemix 203H*

This 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 was 200 L and mixing capacity 0-3 m$^3$/h.

*Figure 9.1 Schematic diagram of the experimental setup*
Chapter 9: Experimental Set-Up

**Grout agitator, Cemag 402H**

This is the agitator containing cylindrical container with angular base, slow running and inclined mixer shaft fitted with two pair of blades. The volume of the agitator is 400L and rotation speed of the agitator shaft is 60-70 rpm.

**Grout pump, Pumpac**

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

The UNIGROUT E22H used for the experiment is shown in Figure 9.2

![UNIGROUT E22H used for the experimental work](image)

**Figure 9.2 UNIGROUT E22H used for the experimental work**

### 9.1.2 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. The data was recorded on the card at every 10th 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
Chapter 9: Experimental Set-Up

In the presented case the flow rate was always maintained within 15-30 L/min. The LOGAC used for the experiment is shown in Figure 9.3.

![LOGAC for experimental work](image)

Figure 9.3  LOGAC used for the experimental work

9.1.3. UVP-PD Instruments

**Flow adapter**

A flow adapter cell was developed and fitted with one pair of 4MHz transducers. The inner diameter of the flow adapter was 22.5 mm. Flow adapter material was stainless steel. Transducers were mounted in flush with pipe through the cavities with diameter equal to the housing diameter of the transducers. Transducers were installed at a distance equal to the near field distance to avoid the near field region where the ultrasound field is highly irregular. The flow adapter and transducer installation used for this experiment is shown in Figure 9.4.

**Ultrasound transducer**

In this experiment two 4MHz ultrasound transducers (TR0405LH-X; Signal-Processing SA, Savigny, Switzerland), high temperature, were fitted with the flow adapter. The flow adapter consisting 4 MHz ultrasound transducers used for the experiment is shown in Figure 9.4. These high temperature transducers allow measurements directly from the transducer front, so more or less zero velocity at the wall can be recorded. The active and outer diameters of the transducers are 5mm and 8mm respectively. The transducers were fixed inside the flow adapter in a horizontal plane opposite to each
other with a doppler angle (between the flow direction and the transducer axis) of $70^\circ$ and $110^\circ$ respectively to minimize the sedimentation effect. The near field distance for these transducers are 7 mm. The material was stainless steel and the pressure limit was 20 bars.

**UVP DUO**

The velocity profile measurements were done 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 for 4 MHz transducers.
Chapter 9: Experimental Set-Up

**UVP-PD experimental parameters**

The UVP-PD experimental parameters used for this experiment are shown in Table 9.1.

*Table 9.1 Experimental parameters for UVP-PD*

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasound frequency (MHz)</td>
<td>4</td>
</tr>
<tr>
<td>Cycles per pulse</td>
<td>2-4</td>
</tr>
<tr>
<td>Voltage (V)</td>
<td>50-150</td>
</tr>
<tr>
<td>Transducer active element diameter (mm)</td>
<td>5</td>
</tr>
<tr>
<td>Spatial resolution (mm)</td>
<td>0.37-0.74</td>
</tr>
<tr>
<td>Repetitions per pulse</td>
<td>128-512</td>
</tr>
<tr>
<td>Sampling time per profile (ms)</td>
<td>104-115</td>
</tr>
<tr>
<td>Doppler angle (°)</td>
<td>70-110</td>
</tr>
<tr>
<td>Number of Time domain profiles (steady state flow)</td>
<td>500</td>
</tr>
<tr>
<td>Number of FFT profiles (steady state flow)</td>
<td>30</td>
</tr>
<tr>
<td>Sampling time for pressure difference (Hz)</td>
<td>100</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>16-20</td>
</tr>
<tr>
<td>Length of pipe between pressure sensors (m)</td>
<td>1.3</td>
</tr>
<tr>
<td>Volumetric flow rate range (L/min)</td>
<td>15-30</td>
</tr>
</tbody>
</table>

**9.2 Experimental Procedure**

The experiments were performed in the following steps:

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

2. The mixer is filled with the required amount of water for a certain water cement ratio. Cement is added after the high speed mixer is switched on.
Chapter 9: Experimental Set-Up

3. The mixing is performed for 4 minutes. SetControl is added after mixing of 2 minutes.

4. The mixture is shifted to the agitator.

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

6. The mixture is 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 10th seconds’.

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

9.3 Off-line Measurement Instrument

ARES-G2 Rheometer

ARES stands for Advanced Rheometric Expansion System. 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 9.2

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geometry</td>
<td>27mm DIN, Concentric cylinder</td>
</tr>
<tr>
<td>Temperature(°c)</td>
<td>20</td>
</tr>
<tr>
<td>Soak time (s)</td>
<td>10</td>
</tr>
<tr>
<td>No Flow sweeps</td>
<td>2 (low to high shear rate and vice versa)</td>
</tr>
<tr>
<td>Shear rate(1/s)</td>
<td>0.1-1000</td>
</tr>
<tr>
<td>Points per decade</td>
<td>10</td>
</tr>
<tr>
<td>Equilibration time(s)</td>
<td>5</td>
</tr>
<tr>
<td>Averaging time (s)</td>
<td>5</td>
</tr>
</tbody>
</table>
Chapter 9: Experimental Set-Up

Brookfield DV-II’ pro (LV) Viscometer

Brookfield DV-II’ pro (LV) viscometer from Brookfield Engineering was used for off-line measurements. This experiment was performed at the laboratory of KTH. The experimental parameters are shown in Table 9.3. 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.

Table 9.3 Experimental parameters for Brookfield viscometer

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geometry</td>
<td>Concentric cylinder</td>
</tr>
<tr>
<td>Spindle</td>
<td>SC4-31</td>
</tr>
<tr>
<td>Temperature(°C)</td>
<td>25-27</td>
</tr>
<tr>
<td>Shear rate (1/s)</td>
<td>0.1-200</td>
</tr>
<tr>
<td>Speed (rpm)</td>
<td>5-200</td>
</tr>
</tbody>
</table>
Chapter 10

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 UVP-PD method works for cement grouts in field conditions. The primary objective was to measure the velocity profiles of cement grouts directly in-line using UVP-PD method. Other objectives were to determine the rheological parameters using mathematical models and 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 the field a standard grouting mixture machine UNIGROUT E22H and LOGAC flow meter was used. 4 MHz ultrasound transducers were used as a trial for this setup as it was 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 a very small scale. Acoustic off-line characterization measurements were done to study the pre-feasibility of UVP-PD method for cement based grouts 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 at 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.

10.1 Acoustic measurements

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 6) are based on the velocity of sound in the sample fluid medium, 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. The results are shown in Figure 10.1 and 10.2 and summarized in Table 10.1. 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 10.1 and 10.2 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 10.1 Received waveform pulse for w/c ratio 0.8 (Wiklund, 2009)

Figure 10.2 Received waveform pulse for w/c ratio 0.6 (Wiklund, 2009)
Chapter 10: Results

Table 10.1 Acoustic parameters for off-line and in-line measurements

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cement grout (off-line)</th>
<th>Cement grout (off-line)</th>
<th>Distilled water (off-line)</th>
<th>Cement grout (in-line)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W/C ratio (by cement weight)</td>
<td>0.8</td>
<td>0.6</td>
<td>N/A</td>
<td>0.8</td>
</tr>
<tr>
<td>Frequency (MHz)</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Voltage (V)</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>Gain (-)</td>
<td>5-7</td>
<td>5-7</td>
<td>5-7</td>
<td>5-7</td>
</tr>
<tr>
<td>Cycles (#)</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>3</td>
</tr>
<tr>
<td>Sound Velocity (m/s)</td>
<td>1525.6</td>
<td>1540.1</td>
<td>1485.4</td>
<td>1487.30</td>
</tr>
<tr>
<td>Time-of-flight (ms)</td>
<td>0.093</td>
<td>0.092</td>
<td>0.095</td>
<td>0.018</td>
</tr>
<tr>
<td>Transducer Distance (mm)</td>
<td>14.19</td>
<td>14.19</td>
<td>14.19</td>
<td>27.77</td>
</tr>
<tr>
<td>Peak-Peak voltage (V)</td>
<td>0.102</td>
<td>0.042</td>
<td>35.31</td>
<td>0.053</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>24.8</td>
<td>25.5</td>
<td>20.3</td>
<td>21.1</td>
</tr>
</tbody>
</table>

The sound velocity of distilled water was also measured in the flow adapter cell to compare with the results obtained from 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 cement grout is much more attenuating than distilled water. High attenuation in low w/c ratio was caused due to increased multiple scattering of ultrasound with increasing concentration and wide particle distribution of solids. The velocity of sound was lower for in-line measurement than the off-line measurement. During off-line measurements, sedimentation occurs into the flow cell so the velocity of sound is higher than the in-line measurements.

Within the scope of this master thesis, acoustic measurements were also performed directly in-line 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. The transducer distance was not correct all the time and it caused lower velocity of sound during the in-line measurements. Figure 10.3 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 different acoustic measurement parameters and the results are summarized in Table
Chapter 10: Results

10.1. The performed acoustic measurements show that it is possible to measure the velocity of sound in cement grouts.

The achieved waveform pulses for two in-line measurements are shown in Figure 10.3. The complete waveform was achieved over a distance of 27.77 mm. The diameter of the flow cell was to be 22.5 mm and the distance between the transducer was found 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 10.3 Received waveform pulse for W/C ratio 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 rheological parameters. It was thus found that 3 cycles/pulse were found optimum for 4MHz transducers in case of in-line measurements. The performed in-line acoustic measurements show that it is also possible to measure the velocity of sound in cement grouts under field conditions.
10.2 Velocity profiles measured by UVP

In this section, velocity profiles measured by the ultrasound velocity profiling (UVP) technique 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 (SC) as additive. SetControl was used as a high performance binding time regulator and dispersing additive which is suitable for grouts based on Injektering 30. SetControl 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 to present the velocity profiles in the same time when samples for off-line measurements were taken. The timings for the off-line measurements are shown in Table 10.4. 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. SetControl, UNIGROUT E22H and LOGAC were used to keep the condition the same as in the field. The samples and time after mixing used for velocity profile measurements and corresponding determination of rheological parameters are shown in Table 10.2. Firstly, all the velocity profiles obtained over a certain period of time are shown in Figure 10.4 and Figure 10.6 and subsequently, the average of the profiles for that certain time period is determined and presented in Figure 10.5 and Figure 10.7.

Table 10.2 Samples used for measuring velocity profile and rheological parameters

<table>
<thead>
<tr>
<th>w/c ratio of cement grout</th>
<th>Set Control used by cement weight (%)</th>
<th>Time after cement mixing (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8</td>
<td>-</td>
<td>30</td>
</tr>
<tr>
<td>0.8</td>
<td>2</td>
<td>150</td>
</tr>
<tr>
<td>0.6</td>
<td>-</td>
<td>60</td>
</tr>
<tr>
<td>0.6</td>
<td>2</td>
<td>30</td>
</tr>
</tbody>
</table>

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 6.2. 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 10.4 and 10.6 for water cement ratio 0.8 and 0.6, respectively. In Figure 10.4, 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. The ultrasound voltage was 150 v. The profile near the far wall is distorted due to the loss of signal but up to half of the profile a very good signal is achieved and due to pipe geometry, 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 10.4 Captured velocity profiles for w/c ratio 0.8, 90 minutes after mixing](image)

**Figure 10.4 Captured velocity profiles for w/c ratio 0.8, 90 minutes after mixing**

As shown in Figure 10.5, 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 3rd region of ‘above 0.5 m/s’.
In Figure 10.6, 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 10.5 Average velocity profiles for w/c ratio 0.8, 90 minutes after mixing

Figure 10.6 Captured velocity profiles for w/c ratio 0.6, 60 minutes after mixing
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As shown in Figure 10.7, 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 3rd region of ‘above 1.2 m/s’.

![Average velocity profiles for w/c ratio 0.6, 60 minutes after mixing](image)

**Figure 10.7  Average velocity profiles for w/c ratio 0.6, 60 minutes after mixing**

From Figure 10.6 it can be seen that a good signal is 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 10.7.

From the Figure 10.4 and 10.6 it can be seen that a high volumetric flow rate was achieved with a thicker grout. The reason for this situation 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, this was due to the dual-piston pump used in the Unigrout equipment ‘pumpac’. 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.

### 10.2.1 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. It was possible to obtain a very good fit. In all cases the velocity profile and pressure drop is measured, averaged over a specific time interval and then fitted to the H-B model. 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 R0 and the coefficient of determination R² is also shown with the fitted profiles. Figure 10.8 shows the measured velocity profile and the fitted H-B velocity profile for w/c ratio 0.8 without SetControl. Here the fitted in-line profiles are presented according to the time after mixing shown in Table 10.2, i.e., the measurement was taken 30 minutes after mixing the cement grout.

Figure 10.8  Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.8 without SC, 30 minutes after mixing the cement grout

Figure 10.9 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 10.8 and 10.9, it can be seen that the flow index, n for w/c ratio 0.8 with SetControl 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 strength probably had increased due to hydration of the cement. The profile shape was 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.
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Figure 10.9  Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.8 with SC, 150 minutes after mixing the cement grout

Figure 10.10 and 10.11 show velocity profiles for w/c ratio 0.6 with and without SetControl, respectively. In Figure 10.10, for w/c ratio 0.6 without SetControl 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 cement grout.

Figure 10.10  Measured velocity profile and H-B fitted velocity profile for w/c ratio 0.6 without SC, 60 minutes after mixing the cement grout

Figure 10.11 shows the H-B fitted velocity profile for w/c ratio 0.6 with SetControl. The measurement was taken 30 minutes after mixing of the cement grout. The little distortion in some regions of the
measured profile of Figure 10.10 and 10.11 were because of the high fluctuations of the velocity profiles.

![Graph of velocity profile](image)

*Figure 10.11 Measured velocity profile and H-B fitted profile for w/c ratio 0.6 with SC, 30 minutes after mixing the cement grout.*

The values of coefficient of determination ($R^2$) for w/c ratio 0.6 were lower than those obtained for w/c ratio 0.8 was due to the unstable flow rate due to the piston pump and it was also 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.

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, 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.

### 10.2.2 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 but it requires high spatial resolution and high quality data (Wiklund et al, 2007). The principle of the gradient method is shown in Figure 10.12. 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.
Chapter 10: Results

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.

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.
10.2.3 Comparison of velocity profiles obtained by different methods

Figure 10.13 shows a comparison of a velocity profile measured by direct in-line readings, H-B curve fitting and gradient method for w/c ratio 0.6 without set control. Here the sample was the same as one used in figure 10.10. It can be seen that for the same measured profile the gradient method gives the best fit. Near the center of the pipe the data is little distorted due to noise in the data.

![Comparison of velocity profiles](image)

**Figure 10.13** Comparison between the velocity profile obtained by direct in-line reading, mathematical model (H-B) and gradient method

The data processing and fundamental difference between a curve fitting method and gradient method can be principally shown as in Figure 10.14 and 10.15.

![Flow chart of data processing](image)

**Figure 10.14** Flow chart of the data processing of curve fitting procedure

![Flow chart of data processing](image)

**Figure 10.15** Flow chart of the data processing of gradient method
Chapter 10: Results

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.

10.3 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, is shown in Figure 10.16. 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.

![Flow curve for different w/c ratio with and without SetControl using the H-B model](image)

Figure 10.16 Flow curve for different w/c ratio with and without SetControl using the H-B model

The effect of SetControl is visible here. Set Control was used to reduce the yield strength and the shear stress of the cement grout. Here higher shear stress with Set Control was observed due to the hydration of cement.

The rheological parameters obtained by the H-B model for different w/c ratios are shown in Table 10.3.
Table 10.3 Rheological parameters obtained by the H-B model for different w/c ratio

<table>
<thead>
<tr>
<th>Sample Description</th>
<th>Time after mixing (min)</th>
<th>n</th>
<th>R₀ (mm)</th>
<th>Yield Stress (N/m²)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>w/c=0.8, No SC</td>
<td>30</td>
<td>0.5</td>
<td>0.34</td>
<td>0.72</td>
<td>0.45</td>
</tr>
<tr>
<td>w/c=0.8, SC 2%</td>
<td>150</td>
<td>0.2</td>
<td>1.93</td>
<td>2.14</td>
<td>2.86</td>
</tr>
<tr>
<td>w/c=0.6, No SC</td>
<td>60</td>
<td>0.3</td>
<td>1.07</td>
<td>1.05</td>
<td>1.3</td>
</tr>
<tr>
<td>w/c=0.6, SC 2%</td>
<td>30</td>
<td>0.4</td>
<td>1.04</td>
<td>1.04</td>
<td>1.64</td>
</tr>
</tbody>
</table>

From the Table 10.3 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 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 10.17 shows the flow curve obtained by gradient method and H-B model fitting for w/c ratio 0.6 without set control. 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 as shown in Figure 10.13.
Chapter 10: Results

Figure 10.17  Flow curve obtained by gradient method and H-B fitting for w/c ratio 0.6 without SetControl

Figure 10.18 shows the distribution of shear rate dependent viscosity as a function of shear rate for different w/c ratio, with and without set control. The sample collection time is shown in Table 10.2.

Figure 10.18  Shear rate dependent viscosity of different w/c ratio with and without SetControl
Chapter 10: Results

The viscosity was lowest for w/c ratio 0.8 without SetControl as expected and this was also shown in Figure 10.18.

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.

10.4 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 10.19. By the UVP-PD method, volumetric flow rate was measured in milli-seconds but as the LOGAC is a Coriolis mass flow meter, this was much slower than the UVP-PD method. However, as can be seen in the Figure 10.19, the two methods were showing results in the same order of magnitude. 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.

In Figure 10.19, 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 10.19 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.
Chapter 10: Results

10.5 Off-line Measurements

Off-line measurements were performed using 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. The results are shown in Table 10.4.

Table 10.4 Off-line measurements with rheometer

<table>
<thead>
<tr>
<th>W/C Ratio</th>
<th>Set Control (%)</th>
<th>Time (min)</th>
<th>Yield Stress, Bingham Pa</th>
<th>Yield Stress H-B, Pa</th>
<th>Viscosity, Bingham, mPa.s</th>
<th>Power Law Parameter</th>
<th>R² Power law</th>
<th>H-B Parameter</th>
<th>R² H-B</th>
<th>R² Bingham</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8</td>
<td>No</td>
<td>30</td>
<td>2.14</td>
<td>0.9</td>
<td>15.9</td>
<td>0.4 0.8</td>
<td>0.98</td>
<td>0.5 0.39</td>
<td>0.98</td>
<td>0.92</td>
</tr>
<tr>
<td>0.8</td>
<td>2</td>
<td>180</td>
<td>5.54</td>
<td>1.39</td>
<td>25.9</td>
<td>0.2 3.22</td>
<td>0.96</td>
<td>0.4 0.76</td>
<td>0.97</td>
<td>0.84</td>
</tr>
<tr>
<td>0.6</td>
<td>No</td>
<td>60</td>
<td>7.3</td>
<td>1.13</td>
<td>56.8</td>
<td>0.4 3.19</td>
<td>0.98</td>
<td>0.4 2.53</td>
<td>0.97</td>
<td>0.88</td>
</tr>
<tr>
<td>0.6</td>
<td>2</td>
<td>110</td>
<td>6.89</td>
<td>0.72</td>
<td>33.1</td>
<td>0.2 3.94</td>
<td>0.97</td>
<td>0.3 3.37</td>
<td>0.98</td>
<td>0.82</td>
</tr>
</tbody>
</table>

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 Bingham fluids. The effect of SetControl cannot be compared as the samples were not measured in identical time and condition. For w/c ratio 0.6 the viscosity was reduced after adding SetControl but for w/c ratio 0.8 it was increased. The H-B fitted flow curve obtained by rotational rheometer for w/c ratio 0.8 and 0.6 are shown in Figure 10.20 and 10.21 respectively. The off-line measurements performed with Brookfield viscometer are presented in Appendix A.
From Figure 10.20 and 10.21 it was found that the shear stress was lower for w/c ratio 0.8 which was expected. The effect of SetControl was not comparable in case of w/c ratio 0.8 due to hydration effect.
Chapter 11

Conclusion and Recommendations for Future Work

11.1 Conclusion

The in-line results obtained with the UVP-PD method were found to be very promising for direct in-line rheological measurements of cement based grouts. The objective was to find the feasibility of this method for cement based grouts. It was possible to obtain the velocity profiles for w/c ratio 0.6 and 0.8. The velocity profiles at different times with and without SetControl were measured. It was possible to measure the rheological properties, such as, viscosity and yield stress of cement grout. Beside mathematical model (Herschel-Bulkley), a non-model approach (Gradient method) was also applied. It was found that with gradient method the true flow curve (shear stress Vs shear rate) can be obtained. The volumetric flow rate was measured directly in-line. The effect of SetControl is not compared in this work. It was out of the scope of the present study to compare the results with off-line measurements and SetControl.

One important conclusion is that the current flow meter may not be useful for grouting. Another important observation was that the piston pump creates unstable flow while grouting which has to be investigated if it is ideal for grouting.

We used standard grouting equipment UNIGROUT E22H and flow meter LOGAC in our flow loop. It resembles that this UVP-PD technology can be used in field after further development and optimization and can be a very effective tool for quality control.

From all the results of the present study we can conclude that it is feasible to measure the rheological properties of cement grots directly in-line under field condition using the UVP-PD method. This is a totally new application of the UVP-PD method in the construction industry and further research should be carried out to establish this technology in the working field.
Chapter 11: Conclusion and Recommendation for Future Work

11.2 Recommendations for Future Work

In order to apply the UVP-PD method in the grouting sector and make it 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 2MHz 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. A customization of the ultrasound transducers will yield improved results.

- 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. 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.
Bibliography


Appendix A


Appendix A


Appendix A


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-III 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 set control
Figure A.2  Flow curve fitted with Bingham model for w/c ratio 0.8 with and without set control

Figure A.3  Flow curve fitted with Power Law model for w/c ratio 0.8 with and without set control
Figure A.4  Flow curve fitted with H-B model for w/c ratio 0.8 with and without set control

Figure A.5  Flow curve for w/c ratio 0.6 with and without set control
Figure A.6  Flow curve fitted by Bingham model for w/c ratio 0.6 with and without set control

Figure A.7  Flow curve fitted by Power law model for w/c ratio 0.6 with and without set control
Figure A.8  Flow curve fitted by H-B model for w/c ratio 0.6 with and without set control
Appendix B

A2. Flow curves measured by Brookfield DV-II+ pro (LV) Viscometer

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

Figure A.10  Flow curve fitted by Power law, Bingham and H-B for w/c ratio 0.8 without S.C after agitating 2 hours
Figure A.11 Flow curve fitted by Power law, Bingham and H-B for w/c ratio 0.6 with S.C just after mixing

Figure A.12 Flow curve fitted by Power law, Bingham and H-B for w/c ratio 0.6 with S.C 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**

<table>
<thead>
<tr>
<th>Date</th>
<th>Time</th>
<th>Description of the work</th>
<th>W/C ratio</th>
<th>Cement (KG)</th>
<th>Water (L)</th>
<th>Set Control (L)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>17/06</td>
<td>13.00</td>
<td>Arriving at SIK. Fine tuning of the Unigrout and Logac.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Equipments and machines assembled and fine tuned.</td>
</tr>
<tr>
<td></td>
<td>16.35-17.26</td>
<td>Trial testing done with water</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>18/06</td>
<td>14.30</td>
<td>Trial testing with water.</td>
<td>0.8</td>
<td>80</td>
<td>64</td>
<td>No</td>
<td>Trial testing done with water and grout.</td>
</tr>
<tr>
<td></td>
<td>16.00</td>
<td>Trial testing with grout</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Signals were not good enough.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4 MHz transducers were used.</td>
</tr>
<tr>
<td>21/06</td>
<td>11.20</td>
<td>Trial testing with water.</td>
<td>0.6</td>
<td>100</td>
<td>60</td>
<td>1.4</td>
<td>Set Control was added with Batch 1.</td>
</tr>
<tr>
<td></td>
<td>12.57-13.01</td>
<td>Mixing cement grout, Batch – 1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Sample was taken for off line measurements of batch -1 without set control.</td>
</tr>
<tr>
<td></td>
<td>14.00</td>
<td>Sample collected for off-line measurements</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>No set control was added to batch 2.</td>
</tr>
<tr>
<td></td>
<td>14.16</td>
<td>Set Control added</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Acoustic measurements were done with batch 2.</td>
</tr>
<tr>
<td></td>
<td>16.30</td>
<td>Cement grout – batch 2</td>
<td>0.6</td>
<td>100</td>
<td>60</td>
<td>No</td>
<td></td>
</tr>
<tr>
<td>22/06</td>
<td>10.40</td>
<td>Mixing cement grouts.</td>
<td>0.8</td>
<td>80</td>
<td>64</td>
<td>No</td>
<td>Objective was obtain good UVP measurements.</td>
</tr>
<tr>
<td></td>
<td>12.07</td>
<td>UVP measurements started</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Relatively stable flow and properties were</td>
</tr>
<tr>
<td>Date</td>
<td>Time</td>
<td>Activity</td>
<td>Description</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------</td>
<td>-------</td>
<td>---------------------------------------------------------------------------</td>
<td>-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>23/06</td>
<td>13.49</td>
<td>UVP measurements started after lunch</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.12</td>
<td>Mixing started with water for transducer distance calibration.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.30</td>
<td>Mixing started. Mixer clogged with the cement.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>14.30</td>
<td>Mixing of cement grouts.</td>
<td>0.8 80 64 No Batch cancelled.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>24/06</td>
<td>09.50</td>
<td>Testing with water for transducer distance calibration.</td>
<td>2 MHz transducers with old flow cell were used but good signal was not observed.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.55</td>
<td>Testing with water with 2 MHz transducers.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.58</td>
<td>Mixing started with cement grouts.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/06</td>
<td>09.50</td>
<td>Calibration done with water.</td>
<td>Set control was used. First 30 minutes data were taken at every 5 minutes or less.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.56</td>
<td>Mixing of cement grout. Set control added.</td>
<td>Two samples were taken for off line measurements to check the properties at early stage and after agitating longer period.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.01</td>
<td></td>
<td>High fluctuation of flow rate observed.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.23</td>
<td>Measurements taken by UVP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>11.50</td>
<td>Sample taken for off line measurements.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>14.00</td>
<td>Sample taken for off line measurements.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30/06</td>
<td>10.32</td>
<td>Calibration started</td>
<td>2 MHz transducer was observed after 1 hour</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Date</td>
<td>Time</td>
<td>Description</td>
<td>Speed</td>
<td>Temperature</td>
<td>Rheology</td>
<td>Notes</td>
<td></td>
</tr>
<tr>
<td>-------</td>
<td>-------</td>
<td>------------------------------------------------------------------------------</td>
<td>-------</td>
<td>-------------</td>
<td>----------</td>
<td>----------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td>01/07</td>
<td>12.27</td>
<td>Mixing of cement grouts. Set control added.</td>
<td>0.6</td>
<td>80</td>
<td>48</td>
<td>1.1</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>used with new flow cell. 4 MHz and 2 MHz both type of transducers were tried</td>
<td></td>
<td></td>
<td></td>
<td>to achieve good signals, velocity profiles and rheological properties.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>01/07</td>
<td>Calibration started with water.</td>
<td>0.8</td>
<td>60</td>
<td>48</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.42</td>
<td>Mixing of cement grouts. Set control added.</td>
<td></td>
<td></td>
<td></td>
<td>Objective was to measure the velocity of sound directly in line for both 2 MHz and 4 Mhz transducers.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Temperature sensor was installed.</td>
<td></td>
<td></td>
<td></td>
<td>A time lag was added to find the right peak for estimating sound velocity.</td>
<td></td>
</tr>
</tbody>
</table>