. \( P \) \quad \text{Total pressure drop}

\( P_s \) \quad \text{Pressure to start flow}

\( R_b \) \quad \text{Radius of bob (inner cylinder)}

\( R_p \) \quad \text{Radius of pipe}

\( T \) \quad \text{Torque}

\( T(t) \) \quad \text{Torque at time } t

\( t \) \quad \text{Time}

\( t_1 \) \quad \text{Minimum time of shearing}

\( t_2 \) \quad \text{Maximum time of shearing}

\( V_m \) \quad \text{Mean fluid velocity}

\( V_p \) \quad \text{Linear velocity of plug}

\( x \) \quad \text{Thickness of sheared layer}

\( \Delta \ell \) \quad \text{Length of element}

\( \Delta P \) \quad \text{Pressure difference}

\( \Delta t \) \quad \text{Time taken to travel distance } \Delta \ell

\( \Delta \tau \) \quad \text{Shear stress increment at the wall of pipe}

\( \tau \) \quad \text{Shear stress at the wall of pipe}

\( \tau_a \) \quad \text{Average shear stress}

\( \tau_b \) \quad \text{Shear stress at the surface of bob}
RHEOLOGY OF A SPECIFIC
OILWELL CEMENT

by

A. M. HAIMONI, B.Eng.

A thesis submitted for the degree of Doctor of Philosophy

Department of Civil Engineering

University of Surrey

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SYNOPSIS

A brief review is made of the rheology of suspensions in general and cement slurries in particular. The factors influencing the flow behaviour of these systems are discussed. It is pointed out that the rheological properties of cement slurries cannot be determined by theory alone from the original constituents, and experimental tests combined with empirical formulae are needed.

The co-axial cylinder viscometer was used to characterise the rheological behaviour of the cement slurry used in this investigation, the slurry being made highly thixotropic by the use of additives. It is shown that no single flow model, however complex, can correctly fully describe the flow behaviour of such materials.

The cement slurry used in this research programme had a structure which changed continuously with time and, when pumped through a pipe, formed a high water content slip layer on the pipe surface. An accurate theoretical solution for this type of pipe flow is lacking in the literature. A new method to predict the pressure gradient of such suspensions flowing in pipes is proposed and the technique was tested using a small pumping line and also with data extracted from the literature.

The shear vane test, commonly used to measure shear strength of soils, was developed to measure gel strength of the cement slurry used in this investigation. It is shown that the gel strength measured with this technique is of considerable benefit when assessing the real material behaviour.
TO MY PARENTS
ACKNOWLEDGEMENTS

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GLOSSARY OF RHEOLOGICAL TERMS

The terminology followed for rheological terms will be, where possible, that of BS5168:1975, from which most of the following definitions are borrowed.

<table>
<thead>
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<th>Term</th>
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<tr>
<td>Aggregate</td>
<td>A group of particles held together in clumps.</td>
</tr>
<tr>
<td>Anisotropic</td>
<td>Not having the same properties in all directions.</td>
</tr>
<tr>
<td>Anti-thixotropic</td>
<td>An increase of the apparent viscosity under shear stress followed by a gradual recovery when the stress is removed. The effect is time dependent.</td>
</tr>
<tr>
<td>Apparent viscosity</td>
<td>The quotient of shear stress divided by rate of shear when this quotient is dependent on the rate of shear.</td>
</tr>
<tr>
<td>Consistency</td>
<td>A general term for the property of a material by which it resists permanent change of shape.</td>
</tr>
<tr>
<td>Creep</td>
<td>The slow deformation of a material; usually measured under constant stress.</td>
</tr>
<tr>
<td>Deformation</td>
<td>A change of shape or volume or both.</td>
</tr>
<tr>
<td>Flow</td>
<td>A deformation, of which at least part is non-recoverable.</td>
</tr>
<tr>
<td>Flow curve</td>
<td>A curve relating stress to rate of shear.</td>
</tr>
<tr>
<td>Gel strength</td>
<td>Static rather than dynamic yield stress.</td>
</tr>
<tr>
<td>Isotropic</td>
<td>Having the same properties in all directions.</td>
</tr>
<tr>
<td>Negative thixotropy</td>
<td>See anti-thixotropy.</td>
</tr>
<tr>
<td>Rate of shear</td>
<td>The change of shear strain per unit time.</td>
</tr>
<tr>
<td>Relative viscosity</td>
<td>The quotient of the viscosity of suspension divided by the viscosity of fluid medium.</td>
</tr>
<tr>
<td>Rheology</td>
<td>The science of the deformation and flow of matter.</td>
</tr>
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<td>----------------------</td>
<td>-----------------------------------------------------------------------------</td>
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<tr>
<td>Rheomolaxis</td>
<td>An irreversible loss of consistency, on shearing of a material, induced by deformation.</td>
</tr>
<tr>
<td>Rheometer</td>
<td>An instrument for measuring rheological properties.</td>
</tr>
<tr>
<td>Rheopexy</td>
<td>See anti-thixotropy.</td>
</tr>
<tr>
<td>Shear thickening</td>
<td>An increase of viscosity with increasing rate of shear in steady flow.</td>
</tr>
<tr>
<td>Shear thinning</td>
<td>A reduction of viscosity with increasing rate of shear in steady flow.</td>
</tr>
<tr>
<td>Stress relaxation</td>
<td>The decrease of stress with time in a strained material.</td>
</tr>
<tr>
<td>Thixotropy</td>
<td>A decrease of apparent viscosity under shear stress, followed by a gradual recovery when the stress is removed. The effect is time-dependent.</td>
</tr>
<tr>
<td>Viscometer</td>
<td>An instrument for the measurement of viscosity.</td>
</tr>
</tbody>
</table>
| Viscosity            | i) Qualitatively, the property of a material to resist deformation increasingly with increasing rate of deformation.  
                        | ii) Quantitatively, a measure of this property defined as the quotient of shear stress divided by rate of shear in steady flow. |
| Yield point          | The point on the stress/strain or stress/rate of strain curve corresponding to the transition from elastic to plastic deformation. |
| Yield stress         | The stress corresponding to a yield point.                                    |
LIST OF SYMBOLS

CHAPTER 1

\[ \dot{\gamma} \quad \text{Shear rate} \]
\[ \tau \quad \text{Shear stress} \]

CHAPTER 2

\[ n_r \quad \text{Relative viscosity} \]
\[ V_A \quad \text{Attractive energy} \]
\[ V_R \quad \text{Repulsive energy} \]
\[ v \quad \text{Volume fraction of solid phase} \]
\[ v_m \quad \text{Maximum volume fraction of solid phase} \]

CHAPTER 5

\[ h \quad \text{Height of bob} \]
\[ R_b \quad \text{Radius of cup (outer cylinder)} \]
\[ r \quad \text{radius} \]
\[ T \quad \text{Torque} \]
\[ V_s \quad \text{Slip velocity} \]
\[ V_{sb} \quad \text{Slip velocity at the surface of bob} \]
\[ \dot{\gamma} \quad \text{Shear rate} \]
\[ \mu \quad \text{viscosity} \]
\[ \mu_p \] Plastic viscosity

\[ \tau \] Shear stress

\[ \tau_b \] Shear stress at the surface of bob

\[ \tau_p \] Shear stress at the surface of the bob when plug flow is eliminated in the gap

\[ \tau_0 \] Yield stress

\[ \Omega \] Angular velocity of outer cylinder

\[ \omega \] Angular velocity

CHAPTER 6

\[ D \] Diameter of tube

\[ d \] Diameter of particle

\[ g \] Acceleration due to gravity

\[ K \] Power law consistency parameter

\[ K' \] Metzner-Reed power law consistency parameter

\[ K'_{D} \] Metzner-Reed power law consistency parameter at constant pipe diameter

\[ \ell \] Tube length

\[ n \] Power law exponent

\[ n' \] Metzner-Reed power law exponent

\[ Q \] Volume flow rate

\[ Re \] Reynolds number
r  Radius
S  Coefficient of slip
Sc  Corrected coefficient of slip
Vm  Mean fluid velocity
Vs  Slip velocity
Vst  Settlement velocity
v  Velocity
Δp  Pressure drop
μ  Viscosity
μp  Plastic viscosity
ρ  Density
ρp  Density of particle
ρs  Density of fluid medium
τ  Shear stress
τw  Wall shear stress
τo  Yield stress

CHAPTER 7

A  Area under shear stress-time curve
H  Height of bob
l  Length of pipe
\( \tau_b(t) \)  
Shear stress at the surface of bob at time \( t \)

\( \tau_0 \)  
Gel strength

\( \omega \)  
Rotation speed of outer cylinder

CHAPTER 8

\( D \)  
Diameter of vane

\( dr, ds \ & dz \)  
Element dimensions as defined in Figure 8.8.

\( d\sigma_r \ & d\sigma_z \)  
Stress increment

\( H \)  
Height of vane

\( P \)  
Normal die pressure

\( P_e \)  
Extrusion pressure

\( R_1 \ & R_2 \)  
Maximum and minimum die radii respectively

\( r \)  
Radius

\( T \)  
Torque

\( \alpha \)  
Die angle (Figure 8.8)

\( \dot{\gamma} \)  
Shear rate

\( \sigma_r \ & \sigma_z \)  
Stress components

\( \tau \)  
Shear stress

\( \tau_y \)  
Yield stress
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INTRODUCTION

Concentrated suspensions in general and cement slurries in particular exhibit a very complex rheological behaviour which has yet to be fully understood. Extensive research has been carried out in this field in the past few decades which has improved our knowledge of the rheology of suspensions and the factors influencing it. Nevertheless, the picture is still a confused one and many years will elapse before a full understanding of concentrated suspensions can be achieved.

The following quotation from Barnes (1980) gives a good picture of the real problems and current position in the art/science of concentrated suspension.

"If all the techniques mentioned here and elsewhere were used to fully characterise the physical properties of a dispersion in terms of particle size and size distribution, shape and shape distribution, surface charge etc., etc., then is it possible using the current knowledge to predict the rheological properties? Unfortunately apart from a few simple systems the answer is no.

If it were possible to measure the rheological properties of these systems in a simple flow (i.e. a rheometer) is it then possible to predict the flow behaviour in a complicated flow field? The answer again, apart from a few examples, is in general negative."

In the oil drilling industry, cement slurries are mainly used to seal the annular space between the wall of the borehole and the steel casing, which is inserted to facilitate further drilling and eventually to carry any oil or gas to the surface. The conditions down the hole are complex and often unpredictable. The basic cement and water mixture is usually inadequate and may cause serious problems unless additives are included. Consequently, a large number of additives and admixtures have been developed to modify the behaviour of the basic system to suit variety of conditions and to fulfil many objectives. A viscometer or rheometer is usually used to assess the influence of these additives and admixtures on the rheology of cement
slurries, and to characterise the flow behaviour in order to choose the correct slurry formulation for a successful job completion.

The cement slurry used in this investigation is a slurry with special properties, and usually referred to in the industry as a thixotropic cement. It is claimed that such cements have the capability of developing a high gel structure as the shear rate approaches zero, whilst remaining thin and pumpable at relatively high shear rates. This property of the material has been used in the most difficult problems of fallback of the cement column, squeeze and lost circulation. Nonetheless, engineers often turn away from these cements and resort to more conventional slurries. Part of this preference for the more conventional slurries stems from the unique properties of the so called thixotropic cements which have never been fully understood. This lack of understanding is caused by the complex nature of cement slurries and the failure of rheologists to produce a suitable and trouble free instrument to measure and quantify the rheological properties of concentrated suspensions and thick pastes. Furthermore, the existing theoretical predictions for the flow of such materials in pipes and other geometries is not satisfactory.

This unfortunate state has led engineers to the use of "rule of thumb" or field experience in providing the desired objectives. In view of this lack of knowledge, many cementing operations are very uneconomical, and if an operation goes wrong, then the cement may harden and block the casing or, it may not effectively seal the annular gap, thus requiring very expensive remedial work. This state is not any longer considered to be acceptable by the drilling companies such as British Petroleum.

The main objectives of this investigation therefore, were to attempt to provide some basic information to assist in reducing the practical problems. The intention was to characterise the rheological behaviour of a specific thixotropic cement slurry from the physical and chemical variables and/or from viscometric data. Using this data, the accuracy of theoretical prediction of the flow behaviour of this slurry in tubes and other geometries was to be investigated. The theoretical study included prediction of the pressure to start and maintain flow in tubes, annuli and in other complex geometries.
CHAPTER 1

INTRODUCTION TO RHEOLOGY

1.1: Introduction

Rheology is the science of deformation and flow of matter. The word Rheology was first used by Bingham (Barnes (1980)) to describe that branch of physics concerned with the mechanics of deformable bodies. The idea was to establish a branch of science which is of interest to chemists, physicists and engineers.

A body is said to be deformed when the application of a force system alters the shape or the size of the body. A body is said to flow if its degree of deformation changes continually with time. The objective of rheology is to establish mathematical equations which relate the flow or deformation exhibited by the material to the forces that act on it. If the body under consideration is a fluid, application of any non-hydrostatic force, however small, will result in flow, and upon release of the force the body will not result in a return to the original undeformed state. However, if the body is solid, application of any force will result in a deformation but not flow. If the body returns to its undeformed state upon the release of the force, then the body is described as an elastic solid, but if the body does not return to its original undeformed state upon the release of the force, the body is described as an inelastic or a plastic solid.

The concepts of fluidity, solidity, elasticity or plasticity are idealisations which describe the behaviour of real materials in certain limiting cases. In practice solids may show elasticity, plasticity and fluidity by showing some relaxation of stresses and consequently creep. Hence, the Greek philosopher Heraclitus (Barnes (1980)) was led to say "Everything flows". On the other hand some fluids may have some characteristics of solids by showing some elasticity and supporting stresses without showing flow. The distinction between solids and fluids is of little rheological value, and it is more important to establish how the material is going to behave under the action of the stress present in the practical situation.
1.2: Fundamental Concepts Of Rheology

1.2.1: Stress

The deformation or flow produced by a force acting on, or within, a body depends on the force per unit area rather than the force itself. This value is usually called stress.

Consider a body in equilibrium subjected to the system of forces shown in Figure (1.1a). An element of area $\Delta A$, drawn in the body is acted on by a force $\Delta F$. This force can be resolved in three components along three mutually perpendicular axes, one normal to the surface $\Delta A$ and two in the plane of the surface. Let $\Delta F_{xx}$, $\Delta F_{xy}$, and $\Delta F_{xz}$ be the components of the force $\Delta F$ in the direction of the $x$, $y$ and $z$ system of axes respectively, Figure (1.1b). These components lead to the following definitions of the normal stress ($\tau_{xx}$) and shear stresses ($\tau_{xy}$ and $\tau_{xz}$).

\[
\tau_{xx} = \lim_{\Delta A \to 0} \frac{\Delta F_{xx}}{\Delta A} \quad \ldots \ldots (1.1)
\]

\[
\tau_{xy} = \lim_{\Delta A \to 0} \frac{\Delta F_{xy}}{\Delta A} \quad \ldots \ldots (1.2)
\]

\[
\tau_{xz} = \lim_{\Delta A \to 0} \frac{\Delta F_{xz}}{\Delta A} \quad \ldots \ldots (1.3)
\]

Consider the three dimensional element as shown in Figure (1.2). For infinitesimal elements (ignoring body forces) it can be shown by resolving the forces in the $x$, $y$ and $z$ axes that the normal stresses on opposite faces are equal in magnitude but opposite in direction. By considering the moment of the shear stresses about axes through the centre of the element, it can be shown that:-

\[
\tau_{xy} = \tau_{yx} \quad \ldots \ldots (1.4)
\]

\[
\tau_{yz} = \tau_{zy} \quad \ldots \ldots (1.5)
\]

\[
\tau_{zx} = \tau_{xz} \quad \ldots \ldots (1.6)
\]

Therefore the state of stress at a given point in a continuous medium can be characterised by six independent components of stress.
FIGURE 1.1: SYSTEM OF FORCES ACTING ON A BODY

FIGURE 1.2: THREE DIMENSIONAL ELEMENT
1.2.2: Strain and Strain Rate

A body is said to have experienced straining if the relative positions of two points in the body are altered. Consider the axially loaded member of Figure (1.3). Before the application of the Load, let the distance between points A and B be \( \Delta x \), and after application of the Load be \( \Delta x + \Delta u \). Then, if the deformation of the member is uniform, the normal strain is defined as:

\[
\varepsilon = \lim_{\Delta x \to 0} \frac{\Delta u}{\Delta x} = \frac{du}{dx}
\]  

....(1.7)

Now consider the two dimensional case in which all points in the body, before and after application of the Load remain in the same plane. The body has dimensions \( dx \) and \( dy \) and unit thickness, Figure (1.4a). The body can deform by a change of length of the side (normal strain), change in shape (shear strain), or combination of both. For simplicity these two cases of strain will be dealt with separately.

Referring to Figure (1.4b), if the extensions of the sides of the body in the \( x \) and \( y \) directions are \( \Delta u \) and \( \Delta v \) respectively, then the two normal strains can be defined on the bases of equation (1.7):-

\[
\varepsilon_x = \frac{du}{dx}, \quad \text{and} \quad \varepsilon_y = \frac{dv}{dy} \quad \text{....(1.8)}
\]

Referring to Figure (1.4c), the angular change between lines in the \( x \) and \( y \) directions is defined as the shearing strain and if \( dx \) and \( dy \) are small then:

\[
\alpha_x = \frac{dv}{dx}, \quad \text{and} \quad -\alpha_y = \frac{du}{dy} \quad \text{....(1.9)}
\]

But the shear strain is equal to \( \alpha_x - \alpha_y \) then the shear strain:

\[
\gamma_{xy} = \frac{du}{dy} + \frac{dv}{dx}
\]

In the case of a three dimensional element, a rectangular prism with sides \( dx \), \( dy \), and \( dz \), similar analysis leads to the following normal and shear strain then:-
Before loading

\[ A \quad B \]

\[ \Delta x \]

After loading

\[ A \quad B \quad F \]

\[ \Delta x + \Delta u \]

FIGURE 1.3: DEFORMATION OF ONE DIMENSIONAL MEMBER

(a)

\[ dx \]

\[ dy \]

(b)

\[ dx \]

\[ dy \]

\[ \Delta u \]

(c)

\[ \Delta u \]

\[ \sigma_x \]

\[ \sigma_y \]

\[ dx \]

\[ dy \]

FIGURE 1.4: DEFORMATION OF TWO DIMENSIONAL MEMBER
normal strains
\[ \xi_x = \frac{\partial y}{\partial x}, \xi_y = \frac{\partial v}{\partial y}, \xi_z = \frac{\partial w}{\partial z} \]  
\[ ... (1.10) \]
shear strains
\[ \gamma_{xy} = \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} \]  
\[ ... (1.11) \]
\[ \gamma_{yz} = \frac{\partial v}{\partial z} + \frac{\partial w}{\partial y} \]  
\[ ... (1.12) \]
\[ \gamma_{zx} = \frac{\partial w}{\partial x} + \frac{\partial u}{\partial z} \]  
\[ ... (1.13) \]
Consequently, similar to stresses, the state of strain at a given point in a continuous medium can be characterised by six independent components of strain.

In addition to fixed strains, strain rates are often used to describe the rate of stress in a medium. Corresponding to fixed strains there exist strain rates:
\[ \dot{\xi}_x, \dot{\xi}_y, \dot{\xi}_z, \dot{\gamma}_{zx}, \text{etc.}, \ldots \]

1.3: Rheological Classification Of Fluids

Many materials obey simple relationships between stress and strain, or strain rate. These materials are usually termed elastic solids, and Newtonian fluids. However there exist many materials of significant practical value which cannot be called Newtonian fluids or elastic solids. Fluids of this kind are usually known as non-Newtonian fluids. Many Mathematical models are used to describe the rheological behaviour of these materials, based on empirical or semi-empirical considerations. Some of these fluids are discussed in section 1.3.2.

1.3.1: Newtonian Fluids

For Newtonian fluids in laminar flow the shear stress is proportional to shear rate or velocity gradient, i.e.
\[ \tau = \dot{\gamma} \]
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This equation may be written as

$$\tau = \mu \dot{\gamma}$$

...(1.14)

Where $\mu$ is constant and usually called Newtonian viscosity, coefficient of viscosity, or simply viscosity of the fluid. For Newtonian fluid $\mu$ is independent of the shear rate. Thus if the shear stress is plotted against the shear rate, Equation 1.14 yields a straight line which passes through the origin (Figure 1.5). For most Newtonian fluids $\mu$ may be affected by the pressure and the temperature of the fluid.

The Newtonian fluid model, Equation 1.14 is an excellent one for fluids whose microscopic structure is not too complicated. All gases, liquids and solutions of low molecular weight come into this category.

1.3.2: Non-Newtonian Fluids

Complex systems, suspensions, colloids, paste, slurries and polymeric systems may show marked deviation from Newtonian behaviour. These fluids are usually classified into three broad types:-

1.3.2.1: Time Independent Fluids

Fluids of this type are described rheologically by an equation of the form

$$\tau = f(\dot{\gamma})$$

...(1.15)

According to the nature of Equation 1.15 rheologists usually subdivide time independent fluids into three different types as shown in Figure 1.5.

i) Pseudoplastic (or shear thinning) fluids - the viscosity decreases with the increase in shear rate.

ii) Dilatant (or shear thickening) fluids - the viscosity increases with the increasing shear rate.

iii) Viscoplastic fluids - for which the material will not flow
unless minimum value of shear stress (usually known as yield stress) is applied on the material.

1.3.2.2: Time Dependent Fluids

i) Thixotropic materials - Thixotropy is defined (BS5168:1975) as a decrease of the apparent viscosity under shear followed by a gradual recovery when the stress is removed. The effect is time dependent. Hence thixotropy is rather like pseudoplasticity in which the time required for the reduction in viscosity is not negligible.

If, after a period of rest, a thixotropic material is sheared at a constant rate the apparent viscosity ($\nu_{a}$) will decrease with time as shown in Figure 1.6. Many reasons have been proposed to explain thixotropy, for example, breakdown of linkages or structures in the material, flocculation or deflocculation, and alignment of irregular shaped particles or long chain molecules. Whatever the reason, the rate of viscosity decrease, during shearing at a given rate, will depend on the number of linkages available for breaking, or the number of particles for alignment, and must therefore decrease with time.

Thixotropy leads to a kind of hysteresis loop on the shear stress - shear rate graph as shown in Figure 1.7. On this figure, the curve is plotted for increasing shear rate (up curve), and then followed by decreasing shear rate (down curve). The up curve will be closer to the shear stress axis. There are some materials which show permanent or irreversible loss of viscosity, these materials are not thixotropic and this phenomenon is known as rheomalaxis or rheodestruction.

ii) Anti-thixotropic material - There are some materials which show the opposite effects to thixotropic materials, this behaviour is usually termed as anti- or negative - thixotropy. Also sometimes it is termed as rheopexy. For these materials a hysteresis loop is measured as for the thixotropic material but with a major difference, that is the down curve will be closer to shear stress axis.
FIGURE 1.5: FLOW CURVES FOR VARIOUS IDEAL RHEOLOGICAL BODIES

FIGURE 1.6: VARIATION OF APPARENT VISCOSITY WITH TIME FOR THIXOTROPIC FLUIDS WHEN SUBJECTED TO CONSTANT SHEAR RATE AFTER RESTING

FIGURE 1.7: Hysteresis Loop for Thixotropic Fluids
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1.3.2.3: Viscoelastic Fluids

These fluids show the characteristics of both solids and fluids. Like elastic solids they have the capacity to store mechanical energy, while at the same time they are able to dissipate energy as viscous fluids do.

Elastic solids respond to a suddenly applied and maintained shear stress by exhibiting a constant shear strain while viscous fluids respond by a continual increase in shear strain. If the fluid is time independent, the shear strain will increase at a constant rate as shown in Figure 1.8. Viscoelastic material, however, responds to a suddenly applied and maintained shear stress by a sudden jump followed by a gradual increase in shear strain, (Figure 1.9). If the material appears to approach a limiting value of strain, then the material is termed as a viscoelastic solid. But, if, after some time, the strain starts to increase at a constant rate, then the material is called a viscoelastic liquid (Figure 1.9).

1.3.2.4: Reacting Systems

There is an additional complication in the material which is the subject of this thesis and which is dealt with in more detail later. Fresh cement paste is a material which shows the characteristic of some of the more complex of the above systems but additionally is essentially a different material on every occasion when measurements are taken. This is because it changes from a liquid to a solid over a period of about a few hours by virtue of the complex chemical reaction of cement powder with water. Thus it is not to be expected that any single one of the above ideal systems will accurately represent the behaviour of cement paste during it's relatively short life span as a fluid.
FIGURE 1.8: CREEP TEST RESPONSE. (a) SHEAR STRESS APPLIED. (b) ELASTIC SHEAR STRAIN RESPONSE. (c) VISCOUS SHEAR STRAIN RESPONSE

FIGURE 1.9: CREEP CURVES. (a) SOLID-LIKE SHEAR STRAIN RESPONSE TO SUDDENLY APPLIED SHEAR STRESS. (b) FLUID-LIKE RESPONSE
1.4: Flow Models for Non-Newtonian Fluids

Flow models are mathematical equations which relate the stresses applied on the material to the strains or strain rates exhibited by it. Some of these equations were derived by theoretical considerations, from first principles, while others were empirically established. A large number of equations have been proposed. Cheng (1980) mentioned that he came across more than fifty equations which described the behaviour of dilatant fluids, forty three equations for pseudoplastic fluids and ten for viscoplastic fluids. Other models have been introduced to describe the behaviour of the more complex fluids such as viscoelastic or thixotropic fluids. Some of the more common flow models are described below.

1.4.1: Time Independent Fluids

i) Bingham Fluid Model

The flow model curve of a Bingham fluid, like that of a Newtonian fluid, is a straight line. The difference between the Newtonian fluid model and the Bingham one is that the Bingham fluid flow curve is a straight line with an intercept on the shear stress axis, which is usually termed as the Bingham yield stress or just a yield stress of the material in question, a general definition of the yield stress is given in Chapter 8, section 8.2.1: Above the yield stress the equation for the Bingham fluid can be written as:

\[ \tau = \tau_y + \mu_p \dot{\gamma} \]  

... (1.16)

Where \( \tau_y \) and \( \mu_p \) are constants and usually called Bingham yield stress and plastic viscosity respectively. The behaviour of many concentrated suspensions and emulsions, such as printing inks and clay slurries may sometimes be adequately represented over a limited range of strain rate by the Bingham model.
ii) Power Law Model

Generally known as the power law model but sometimes it is referred to by Ostwalde-de Waede model. This model represents fluids having curved flow curve with no yield stress. The equation may be written as:

$$\tau = k\dot{\gamma}^n$$

...(1.17)

where $k$ and $n$ are constants. From Equation 1.14 it can be seen that the Newtonian fluid is a special case of the power law model when $n=1$. In this case $k$ will be equal to $\nu$ the Newtonian viscosity.

The ratio of shear stress to shear rate at any point is function of shear rate and usually termed as apparent viscosity $\mu_a$, where

$$\mu_a = \frac{\tau}{\dot{\gamma}} = k\dot{\gamma}^{n-1}$$

If the apparent viscosity reduces with the increase in $\dot{\gamma}$, i.e. $n < 1$, the fluid is termed as pseudoplastic or shear thinning, and if the apparent viscosity increases with the increase in $\dot{\gamma}$, then the fluid is said to be dilatant or shear thickening fluid. For pseudoplastic and dilatant fluids the change in viscosity is time independent, so they should not be confused with thixotropic or rheopectic fluids for which the change in viscosity is shear rate and time dependent.

iii) Herschel Bulkley Model

Sometimes referred to as the generalised Bingham model, this model is in someway similar to Bingham model in the sense that the material has a yield stress, but above the yield stress the flow curve is no longer straight. The model can be written as:

$$\tau = \tau_y + k\dot{\gamma}^n$$

...(1.18)

On inspection of Equation (1.18) it can be seen that the Newtonian model, the Bingham model, and the power law model are all special cases of Herschel Bulkley model, so the model represents the behaviour of a large number of materials without being very complex to handle mathematically.
iv) Other Models

Other empirical or semi-empirical equations which describe the behaviour of inelastic time independent fluids are:

- Prandtl
  \[ \tau = A \sin^{-1} \left( \frac{\gamma}{c} \right) \]  
  \( \ldots (1.19) \)

- Eyring
  \[ \tau = \dot{\gamma} B + C \sin \left( \frac{\tau}{A} \right) \]  
  \( \ldots (1.20) \)

- Casson
  \[ \tau^{0.5} = \tau_y^{0.5} + k \dot{\gamma}^{0.5} \]  
  \( \ldots (1.21) \)

- Vocablo
  \[ \tau = (\tau_y^{1/n} + k \dot{\gamma})^{1/n} \]  
  \( \ldots (1.22) \)

where \( A, B, C, \tau_y, k \) and \( n \) are constants.

Power series model

\[ \tau = k_1 \dot{\gamma} + k_2 \dot{\gamma}^3 + k_3 \dot{\gamma}^5 \]  
\( \ldots (1.23) \)

\( k_1, k_2, \ldots \) are constants.

1.4.2: Time dependent Fluids

i) Slibar and Paslay

Based on the Bingham model, Slibar and Paslay (1964) proposed mathematical expression to account for the thixotropy, the viscosity function in the expression contained an exponential time effect. The full expression is:

\[ \dot{\gamma}_{ij} = \begin{cases} 0 & \text{for } \sqrt{\dot{\gamma}_2} < \tau_{\text{crit.}} \\ \frac{(\sqrt{\dot{\gamma}_2} - \tau_{\text{crit.}}) \cdot S_{ij}}{\sqrt{\dot{\gamma}_2}} & \text{for } \sqrt{\dot{\gamma}_2} > \tau_{\text{crit.}} \end{cases} \]  
\( \ldots (1.24) \)

\[ 2 \mu \dot{\gamma}_{ij} = (\sqrt{\dot{\gamma}_2} - \tau_{\text{crit.}}) \cdot S_{ij}/\sqrt{\dot{\gamma}_2} \text{ for } \sqrt{\dot{\gamma}_2} \geq \tau_{\text{crit.}} \]  
\( \ldots (1.25) \)

with

\[ \tau_{\text{crit.}}(t) = \tau_1 - \frac{t}{\int \sqrt{D_2} \cdot e^{-\alpha(t-t')} dt'} \]  
\( \ldots (1.26) \)

where

...
D₂ is the quadratic invariant of strain.
J₂ is the quadratic invariant of the reduced stress tensor, in the cartesian co-ordinates.
S_ij is the component of reduced stress tensor in the cartesian co-ordinates.
\dot{\gamma}_{ij} is the component of the deformation rate tensor in the cartesian co-ordinates.
t is the current time.
t' is a dummy variable in time and

\tau_0, \tau_1, \nu, \alpha and \beta are material parameters.

**ii) Harris**
A mathematical expression was proposed by Harris which has some similarities with that of Slibar and Paslay. He used unspecified memory function for the viscosity equation as shown in equation 1.24.

\[
\nu(t) = \nu_1 \int_{-\infty}^{t} f(D_2', D_3') \nu(t-t') \, dt' \quad \ldots (1.27)
\]

where D₂' and D₃' are the quadratic and cubic strain rate invariants at non current time t'.

\nu_1 is the undisturbed viscosity and \nu(t) is the viscosity at the current time t.

For materials with yield stress Harris used similar expression for the critical strain energy. He also introduced a time-dependent yield criterion.

**iii) Cheng**
Cheng and Evans (1965) followed a kinetic approach to thixotropy where they described the rheological behaviour by a set of two equations. The first is an equation of state which is a constitutive equation without functional, but with a time function \lambda which expresses the instantaneous structure of the material.
CHAPTER 1

\[ F = n(\lambda, D) D \quad \cdots (1.28) \]

The second is a rate equation which gives the instantaneous rate of change of the structure parameter \( \lambda \)

\[ \frac{d\lambda}{dt} = g(\lambda, D) \quad \cdots (1.29) \]

where \( D \) is the shear rate
and \( F \) is the shear stress.

Cheng and Evans generalised Equation 1.28 and 1.29 to Equation 1.30 and 1.31 below, based on work by Bird et al. (1960) on time independent non-Newtonian fluids. However they pointed out that the problem of the validity of this procedure has received little attention in the literature even for time independent fluids.

\[ \tau_{ij} = G_1(\lambda, D_2) \quad \cdots (1.30) \]

and
\[ \frac{d\lambda}{dt} = g'(\lambda, D_2) \quad \cdots (1.31) \]

where \( \tau_{ij} \) is element in the stress tensor and \( D_2 \) is the quadratic strain rate invariant.

The above models are by no means exhaustive but are a fraction of the large number of models proposed in literature. The choice of an equation is in many cases a matter of taste. Nevertheless, the choice can be influenced a great deal by the reasons why we wish to fit a mathematical equation to experimental data. Cheng (1980) put three reasons for data fitting.

i) To summarise the experimental data into a small number of material parameters. Any simple equation which fits the data within the experimental scatter will be adequate.
ii) To extrapolate outside the experimental range of shear rates for predictive purposes or for engineering calculations. Unless there is previous knowledge of the material behaviour, there is no way of estimating the error involved. It is recommended that the experimental data should cover the condition in question, but if this is not possible, Cheng suggested that it would be preferable to use several equations, together with engineering judgement to decide which prediction to accept.

iii) To investigate into the material science. In this case, the curve fitting process has a deeper significance. The equation used for curve fitting has to be derived from theory. A close matching between equation and data is taken to mean the vindication of the theory and the assumptions involved. In the ideal situation, the derived equation should have no adjustable parameters, but, in practice, this is not the case and the data is used to obtain these parameters. By doing so may limit the process to a curve fitting exercise. Nevertheless it could help us to understand the molecular and structural properties of suspensions.
CHAPTER 2

RHEOLOGY OF SUSPENSION

2.1: Introduction

This chapter is not intended to be a complete survey of literature, but a review of the main factors affecting the rheology of concentrated suspensions. An attempt will be made to present the problems and difficulties encountered by both practitioners and theoreticians when attempting to predict and model the rheological behaviour of concentrated suspensions. Suspensions have been given much attention in recent years, and the literature contains numerous papers on the subject, only the most relevant of which will be presented here. For further information the reader is recommended to excellent reviews by Heywood (1984), Cheng (1980), Mewis (1980), Goodwin (1975), Barnes (1980), Mackay (1984), Russel (1980) and many more, see Heywood (1984).

The rheology of suspensions is influenced by a variety of interactions which take place between the particles themselves and between the particles and the suspending medium. Heywood (1984) summarised the many types of interactions into four main categories:

i) Hydrodynamic interaction between the liquid and particles;

ii) Long-range particle-particle attraction which promotes the formation of flocs or structure in the suspension, which modify the hydrodynamic interaction;

iii) Short-range particle-particle repulsion, preventing or inhibiting floc formation.

iv) Particle-particle contact, which brings into play steric effects and frictional or granular behaviour.

The details of each interaction are governed by the other interactions and by a variety of factors, which are themselves influenced by the interactions. Below the most important factors are discussed.
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2.2: Factors Affecting Suspension Rheology

2.2.1: Particle Concentration

The change of viscosity of a continuous medium due to the addition of solid particles was first investigated by Einstein (1906). Einstein considered the hydrodynamic forces acting in a very dilute suspension of non-deformable, uncharged, and spherical particles. He neglected the effect of inertia and assumed no slip on the surface of the particle. The Einstein expression for the relative viscosity \( n_r \) was

\[
\frac{n_r}{\eta} = \frac{1 + v/2}{(1 - v)^2}
\]

(2.1)

where \( v \) is the volume fraction of solids.

On expansion Equation 2.1 yields

\[
n_r = 1 + 2.5v + 4v^2 + 5.5v^3 + \ldots
\]

(2.2)

for small \( v \)

\[
n_r = 1 + 2.5v
\]

(2.3)

The above expression for the relative viscosity was derived on the basis that particles do not interact hydrodynamically, i.e. it is only valid when the volume fraction approaches zero. For higher concentration the viscosity will increase owing to the hydrodynamic interaction of particles and the effective volume fraction of solids due to the immobilisation of liquid within doubles, triplets, and higher order multiplets. There are a very large number of equations, both theoretical and empirical, which relate the relative viscosity of a suspension to the volume fraction of solids. Rutgers (1962, 1963) who cited two hundred and eighty references, has reviewed these equations and divided them into ten categories. Many of these equations can be expressed as a power series.
The value of $k_1$ is usually taken to be equal to 2.5 as in Einstein's equation (Equation 2.2). Values for $k_2$ have ranged from 4 to 14.1 (Goodwin (1975)). The values of the coefficients of the higher order terms are much harder to obtain theoretically owing to the difficulty inherent in handling three and higher body collisions. Experimental estimates of the value of $k_3$ varied between 15 and 50 (Goodwin (1975)). The main drawback of the above equation is that termination of the series after the $v^2$ term will produce an unknown error, which will become very large at high particle concentration. Several studies were concentrated on predicting the viscosity at very high concentration when the particles are in closely packed form. Mooney (1951) arrived at a mathematical expression for the relative viscosity by considering the successive additions of uniform spheres to pure fluid. Mooney's expression is:–

$$n_r = v \exp \left[ \frac{2.5v}{(1-bv)} \right] \quad \ldots \ldots (2.5)$$

where $b$ is a crowding factor. The value of $b$ is chosen such that the viscosity will become infinite when $v = v_m$

where $v_m$ is the maximum packing volume fraction.

Frankel and Acrivos (1967) used an asymptotic technique to derive a limiting expression for the relative viscosity of a concentrated suspension as $v \rightarrow v_m$. Their solution is valid for monodispersed solid spheres.

$$n_r = \frac{9}{8} \left[ \frac{(v/v_m)^{1/3}}{(1-v/v_m)^{1/3}} \right] \quad \ldots \ldots (2.6)$$

Goddard (1977) arrived at an expression similar to that of Frankel and Acrivos (1967) by considering a thin film of Newtonian liquid confined between deformable solid surfaces. When $v \rightarrow v_m$ his expression reduces to:–
\[ n_r = \frac{Nv_m}{8} \left[ \frac{(v/v_m)^{1/3}}{(1-v/v_m)^{1/3}} \right] \] 

... (2.7)

where \( N \) denotes the number of nearest neighbours of representative particles, and depend on the type of packing of the particles.

An empirical expression was proposed by Casson (1979) for the relative viscosity.

\[ n_r = \frac{(1-av)}{(1-1.75 av)} \] 

... (2.8)

where \( a \) is a material parameter.

There are many more expressions in the literature some of which are valid for special cases such as dilute suspensions of spherical uncharged particles, and others are more general (See Rutgers (1962, 1963) and Heywood (1984)). On the other hand, new expressions continue to be proposed, but while a reasonable agreement exists between theory and experiment for dilute suspensions, the agreement is not so good for medium to high concentrations where the viscosity of the suspension depends on many factors other than the particle volume fraction. Also in this range, suspensions of the type covered in this thesis are hardly Newtonian and they exhibit almost every form of non-Newtonian behaviour, shear thinning, shear thickening, thixotropy etc.

2.2.2: Effect of Particle Size

A large amount of research work, both theoretical and experimental, has been carried out on the effect of particle size on the relative viscosity of suspensions and some of these papers have been reviewed by Heywood (1984).

In the majority of viscosity-concentration equations proposed (see section 2.1) the particle size was not included in the mathematical expressions. Einstein (1906), for example, who considered the hydrodynamic forces only, showed that particle size has no influence on the relative viscosity of dilute suspensions of uncharged spherical...
particles if the dimensions of the particles are small compared with those of the measuring apparatus. However, hydrodynamic forces are not the only forces acting on the particle, and other forces like colloidal, inertia and friction forces have, in some cases, a large effect on the rheology of suspensions. For small particles the colloidal forces dominate the hydrodynamic ones, while inertia to some extent only affects the large particles.

For this reason, contradictory results have been reported in the literature. A number of studies (for example Jinescu (1974) and Clarke (1967) have shown that a plot of the relative viscosity ($n_r$) versus particle size has a minimum at between 5-20 microns, while Schack et al. (1957) have observed a maximum viscosity at about 20 microns. Chong et al. (1971) found the viscosity to be independent of particle size over the volume-surface mean diameter range of 53.8 to 236 microns, Sweeny and Geckler (1954) found that the viscosity of glass beads increased with increasing particle size if the suspending medium was aqueous, but they found that the viscosity was independent of particle size if a non-aqueous medium was used. The increase in viscosity with the aqueous medium was attributed to the adsorbed layer effect which is caused by strongly bound water layer which effectively increases the volume of solids. Heywood (1984) who surveyed a large number of papers on the effect of particle size on the viscosity of dispersions, for both spherical and non-spherical particles, concluded that the absolute size of the particle has no influence on the dispersion rheology provided that the particles are well-dispersed and influenced by hydrodynamic forces only. However, Heywood added that the effect of particle size can be seen in rheological data for a variety of reasons as follows:

1) Adsorbed Layer Effect

The stability of many colloidal systems is dependent on the presence of an adsorbed layer of a surface active agent which either provides a charge barrier or steric barrier to coagulation. The effect of this layer, unless the layer is free draining, is to increase the effective volume fraction of solids to a value greater than that calculated from a knowledge of weight and density of the particles. If the thickness of the adsorbed layer is constant and independent of the particle size
the effective volume fraction will increase with the reduction in particle size, hence increasing the viscosity.

2) Brownian motion

As the particle size is decreased in the colloidal range, i.e. 0 → 1 micron, Brownian motion effects have to be added to the hydrodynamic ones. Brownian motion causes translational and rotational diffusion processes. The action of shearing stress tends to arrange the particles in some sort of organised form. Brownian motion, however, tends to randomise the state of particles in the dispersion, and consequently affects the mechanical response of the dispersion.

At low shear rates, where particle diffusion (Brownian motion) dominates particle convection (shear motion), the viscosity of the dispersion is expected to be higher than that at high shear rate where Brownian motion is small compared with shear motion. This in effect will produce a shear thinning behaviour. See Wagstaff and Chaffey (1977) and Chaffey and Wagstaff (1977).

3) Coulombic forces (Electrical forces)

When the particle size is reduced to below 1 micron the effect of Coulombic forces will become increasingly important owing to the huge surface/volume ratio of the small particles. This applies no matter how successful one is at suppression of the double layer of charge on the particle surfaces using appropriate types and concentration levels of electrolytes.

4) Large particles

For large particles, say >100 microns particle size may affect the viscosity in many ways:

i) Inertia forces will increase as the particle size is increased;

ii) If the density of the solid particles is larger than that of the suspending fluid, the suspension is usually unstable, and settlement may occur.
iii) Wall effect (comprises several factors such as wall slip and particle sliding on the surface). If the particles size is comparable with that of the measuring system, then wall effect will be present during testing, which may lead to large experimental errors.

iv) Large particles can rarely be spherical, unlike the very small particles, so that the particle shape must be accounted for.

### 2.2.3: Effect of Polydispersity

Polydispersity of particle size affects the rheology of a suspension. This has been verified experimentally by Parkinson et al. (1970), Sweeny and Geckler (1954) and others. Theoretical and experimental works on the effect of polydispersity were reviewed by Goodwin (1975) and Heywood (1984). Orr and Blocker (1955) fitted experimental data to equation of the form:

\[
\frac{\eta - \eta_0}{\eta} = av^k
\]  

...(2.9)

where \( \eta \) and \( \eta_0 \) are the viscosity of the suspension and the suspending medium respectively.

They found that \( 1/a \) related to the measured maximum value fraction \( v_m \) and \( k \) was related to the geometric standard deviation of the size data of the particles.

Theoretical treatment of polydispersed suspensions is limited. Most of the viscosity-concentrated equations derived in literature do not include the particle size or particle size distribution in the calculation. However, there is an inherent assumption of monodispersity in the equations which include the crowding factor or the maximum volume fraction of solids as a parameter, such as that of Mooney (Equation 2.5). The reason for this assumption is that the maximum volume fraction of polydispersed particles is different from that of monodispersed ones.

The best theoretical treatment of polydispersity was given by Farris (1968). This theory was developed for a discrete size distribution of
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particles, where the ratio of large particles to small ones is $> 10$, so that the suspension of the small particles can act as a homogeneous medium to the large ones. However, although the Farris treatment provides a good starting point, for the theory to be useful, it has to be extended to include particles of continuous size distribution and to account for the effects of other factors which determine suspension rheology.

2.2.4: Effect of Particle Shape

The rheological behaviour of suspensions can be affected to a great extent by the particle shape. Clarke (1967), who investigated the effects of a number of factors on suspension rheology, found that, for a given concentration, particle shape is more influential than particle size or particle size distribution.

A large amount of work, both theoretical and experimental, was carried out on the effect of particle shape on the viscosity of suspensions. Most of the work was performed on particles of regular shape such as prolate or oblate spheroids, ellipsoids and fibres, Heywood (1984). No work of significance has been made theoretically to study the effects of irregularly shaped particles. In the case of cement suspensions, the effect of individual particle shape may not always be so important because cement particles in an aqueous medium tend to aggregate into flocs resulting in clusters of irregular shape.

It is well established in the literature describing experimental results that for a given concentration, particles of irregular shape give higher viscosity than spherical particles. A comprehensive review of the work on the effect of particle shape is beyond the scope of this section. The experimental work by Clarke (1967) gives a clear picture of the effect of particle shape, as can be seen from Figure 2.1 and Figure 2.2.
FIGURE 2.1: EFFECT OF SHEAR RATE ON THE VISCOSITY OF SUSPENSIONS CONTAINING DIFFERENTLY-SHAPED PARTICLES IN WATER. \( n = 0.2 \) (CLARKE [1967])

![Graph showing effect of shear rate on viscosity](image)

- **Material**
  - glass rods
  - glass plates
  - quartz grains
  - glass spheres
  - polymethylmethacrylate spheres
- **Dimensions** (Micron)
  - glass rods: 30 \( \times \) 700
  - glass plates: 100 \( \times \) 400
  - quartz grains: 53 \( \times \) 76
  - glass spheres: 53 \( \times \) 76
  - polymethylmethacrylate spheres: 53 \( \times \) 76

FIGURE 2.2: EFFECT OF SOLIDS CONCENTRATION ON THE VISCOSITY OF SUSPENSIONS CONTAINING DIFFERENTLY-SHAPED PARTICLES IN WATER AT A SHEAR RATE OF 328 1/s. (CLARKE [1967])

![Graph showing effect of solids concentration on viscosity](image)

- **Material**
  - glass rods
  - glass plates
  - quartz grains
  - glass spheres
  - Einstein's equation
- **Density** (g/cm\(^2\))
  - glass rods: 2.01
  - glass plates: 2.52
  - quartz grains: 2.58
  - glass spheres: 2.36
- **Size** (Micron)
  - glass rods: 30 \( \times \) 700
  - glass plates: 100 \( \times \) 400
  - quartz grains: 53 \( \times \) 76
  - glass spheres: 53 \( \times \) 76
  - Einstein's equation: 53 \( \times \) 76
2.2.5: Physicochemical Effects

The physical factors discussed in the previous sections are not the only factors affecting the rheology of suspension. Van der Waals attraction forces, electrostatic repulsion forces, and steric hinderance may have a large contribution to the total forces acting in the system. Steric hinderance can be achieved by the presence of an adsorbed layer of polymers on the surfaces of the particles. Polymers were not constituents of the mix used in this work so that steric hinderance will not be discussed any further.

There are two physicochemical phenomena in suspensions which are worth mentioning. Firstly, with many solutions (for example, clays and cement slurries), the application of an electric field to the solution causes the particles, in the solution, to move towards one of the electrodes, and a reversal of the polarity of the field causes a reversal of the direction of the particles' motion. Secondly, the addition of small amounts of salt or electrolytes to a stable solution (i.e. the particles are well dispersed) causes the particles to aggregate or flocculate to form clusters of varying size or even, in some cases, to form a global three dimensional network which spans throughout the suspension. To help to explain and understand these phenomena some basic theories of colloidal chemistry will be introduced.

2.2.5.1: Van der Waals Attraction Forces.

For the particles in suspension, to flocculate or aggregate net attraction forces must exist between them. The main attraction forces between two particles in solutions are usually attributed to the attraction forces produced by the two mutually induced instantaneous dipoles in interacting atoms. These forces are usually referred to as Van der Waals forces. For a pair of atoms the Van der Waals forces are small and decay rapidly with distance, but, between a pair of atoms the Van der Waals forces are independent of the interaction with the other atoms. Therefore, the total interaction forces between a pair of particles containing a large number of atoms is equal to the sum of all the attractive forces between every atom of one particle and every atom of the other particle, leading to a relatively large force which decays less rapidly, with distance, than for a pair of
The magnitude of the Van der Waals force depends on the size and the shape of the particles, and to some extent on the character of the dispersion medium. The expression for the attractive energy between a pair of particles of radius, $a$, and separation, $R$, between the centres of the particles is (Mackay 1984):

$$V_A = -\frac{A_m}{6} \left( \frac{2a^2}{R^2} + \frac{2a^2}{R^2} + \frac{\ln (R^2 - 4a^2)}{R^2} \right) \quad \ldots \quad (2.10)$$

where $A_m$ is constant.

### 2.2.5.2: Electrical Double Layer

As mentioned above, particles of clay or cement in solution move towards one of the electrodes if the system is subjected to an electrical field, indicating that the particles are electrically charged. The solutions or suspensions do not have a net electrical charge and therefore the charge on the particles must be internally compensated to preserve the electrical neutrality of the system. The internal balance of charges in the solutions or suspensions is explained by the theory of electrical double layer. The theory was first proposed by Gouy (1917) and further refined by Stern (1924) and others, Olphen (1976). The inner layer of the electrical double layer (Figure 2.3) is presented by the charged particle surface. The counter ions are electrostatically attracted to the surface to form the outer double layer. These counter ions have a tendency to diffuse away from the surface towards the bulk solution where their concentration is lower to produce a near exponential decay of counter ion concentration, as shown in Figure 2.3. This layer is usually referred to as the diffuse or Gouy layer. Simultaneously, ions of the same sign as those on the particle surfaces will be repelled away from the particle, creating a deficiency of ions of the same sign in the diffuse layer (Figure 2.3). The mechanisms for the acquisition of the surface charge of a solid particle when immersed in a polar liquid are, [Olphen (1976)]:

1) Imperfection of the crystal structure of the particle
ii) Preferential adsorption of certain specific ions on the surface of the solid particles.

In the case of cement grains coated in a C-S-H (calcium silicate hydrate) gel membrane, the first mechanism is highly probable because the conditions of high supersaturation under which the membrane is precipitated produce a highly disordered amorphous structure. However, the second mechanism is equally possible, Tattersall and Banfill (1983).

The electrical double layer provides the electrostatic repulsion forces required to compete with the Van der Waal attraction forces. Depending on the relative magnitude of these forces, suspensions are usually classified as stable (i.e. the particles are well dispersed) or unstable or flocculated (i.e. the particles tend to flocculate or coagulate to form aggregates of varying sizes).

Cement pastes may be in either stable or flocculated state depending on the relative magnitude of the electrical and other forces present.

2.2.5.3: Total Energy Potential and Effects of Salt Concentration.

For stable suspensions, the Van der Waals attractions are successfully opposed by the electrostatic repulsion generated when the electrical double layers of two approaching particles overlap. Calculation, (Olphen, 1976) shows that the repulsive potential $v_R$ between two particles is dependent on the salt or electrolyte concentration, while, for hydrous suspensions, the Van der Waals attraction potential is little dependent on salt content of the system. Figure 2.4 shows the repulsive potential plotted as a function of distance for three electrolyte concentrations, together with the attraction potential of Van der Waals attraction forces.

In Figure 2.4, the repulsive potential, $v_R$ shows an exponential decay with distance, and the Van der Waals attraction potential, $v_A$ is dependent on some inverse power of the interparticle distance. A resultant curve for the interparticle interaction can be obtained if the repulsive and attractive potentials were added. Figure 2.5 shows
FIGURE 2.3: THE DIFFUSE ELECTRIC DOUBLE LAYER MODEL AND THE DISTRIBUTION OF IONS WITHIN THE DOUBLE LAYER. (TATTERSALL AND BANFILL (1983))

FIGURE 2.4: REPULSIVE AND ATTRACTIVE ENERGY AS A FUNCTION OF INTERPARTICLE DISTANCE. (TATTERSALL AND BANFILL (1983))
FIGURE 2.5: RESULTANT OF REPULSIVE AND ATTRACTIVE ENERGY CURVES AT THREE ELECTROLYTE CONCENTRATIONS. (TATTERSALL AND BANFILL (1983))

FIGURE 2.6: RESULTANT OF REPULSIVE AND ATTRACTIVE POTENTIAL ENERGY CURVE. (MACKAY (1984))
the net potential curve of three different electrolyte concentrations. This figure explains the flocculation or coagulation tendency of some suspensions when salt is added to the system. For no or low salt concentration the particles must have sufficient energy to overcome the repulsive energy barrier, presented by the maximum on the resultant potential curve, before reaching the primary minimum interaction well, Brownian motion will provide the particles with some energy to surmount this energy barrier but in practice, few particles may possess sufficient energy to do this, and the suspension will be virtually stable. As the concentration of salt is increased the energy barrier will get smaller and smaller, and hence more and more particles will have the energy required to overcome the energy barrier and fall into the deep energy minimum and stick together. At high concentration, no energy barrier exists, and the particles will fall into the deep energy minimum well and stick producing a rapid coagulation.

Figure 2.5 presents a general picture of the net interparticle interaction curve. The detailed characteristics of this curve are governed by the relative magnitude of the $v_A$ and $v_R$ over the distance of separation. A secondary energy minimum may exist at a relatively long distance, Mackay (1984), from the particle (see Figure 2.6). Consequently if the secondary energy minimum is greater than the diffusion energy of the Brownian motion, the particles will be held together at long separation (in the secondary energy minimum well) with a liquid layer between them. Suspensions in which the particles aggregate in this way are usually termed as flocculated. On the other hand, suspensions in which aggregation in the primary energy minimum well occurs are said to be coagulated. Aggregation in the secondary minimum is thought to be the cause of thixotropy (Mackay 1984), because the particles can easily be redispersed by a moderate shear force owing to the shallow energy well of the secondary minimum. This type of behaviour could explain some of the phenomena presented on cement paste in this thesis.

2.2.5.4: Effect of Physicochemical Interaction on Rheology of Suspension

The rheological response of suspensions depends to a great extent on the resultant interparticle interaction curve which determines the
state of the suspension. For negative interaction (i.e. dispersed systems), where the resultant electrostatic repulsion forces dominate the attractive forces, the electrostatic repulsion alters the relative viscosity of the suspension in three ways. Firstly, an increase in the relative viscosity by the increase in viscous drag forces on the particles as their counter ion clouds are distorted from spherical symmetry by the shear field. Secondly, and more important, is the increase in the relative viscosity by the repulsive forces of the electrical double layer which prevent the particles from close approach, and hence increase the effective particle diameter, or in other words, the effective volume fraction of solids. Thirdly, the relative viscosity may be altered by changes in the size and shape of macromolecular ions. These changes are caused by changes in the electrical properties of the system i.e. changing ionic strength and/or pH of the system. These mechanisms are usually referred to as the primary, secondary and tertiary electroviscous effects respectively.

In addition to altering the relative viscosity in the well dispersed suspension, the electroviscous forces may change the rheological characteristics of the suspensions. They may produce shear dependent effects, yield stress, and shear modulus (Mewis (1980)).

Experimental evidence of the electroviscous effects are well defined in literature. Some theoretical analyses have been successful for dilute dispersion of spherical particles, but for concentrated and non spherical particles the picture is not so clear (Mewis (1980)), (Mackay (1984)), (Russel (1980)).

When the attraction forces are dominant over the repulsion forces, the particle will stick on collision and clusters of light aggregates or loose flocs will form. Depending on the properties of the particles and the interaction between them, a variety of cluster shapes may result. Under certain conditions, the clusters may extend right through the suspension forming a three dimensional structure. The formation of the clusters influences the rheological behaviour of suspensions in a variety of ways. The immobilised liquid in the clusters will increase the relative viscosity of the system by increasing its effective volume fraction. The shape and size of the
clusters and the interaction between them will affect the hydrodynamic interaction. If the formation or breakdown of the clusters is enhanced under the action of shear forces, then a fluid of a shear thinning, shear thickening, thixotropic, or rheopectic nature may be the result. Deformation of flocs or aggregate under the action of shear force may be the cause of viscoelastic behaviour. If global three dimensional structure is formed, the suspension may behave like a solid with a rigidity modulus, and sustain limited shear stress, the yield stress, before flowing. Such materials are usually known as viscoplastic material. Before yielding the cement paste described later follows viscoplastic behaviour.

The factors discussed above are not the only factors affecting suspension rheology. Nevertheless, by considering these factors, it can be realised that the rheology of suspensions still presents a challenging problem to theoreticians and practitioners, especially in the complex regions of high solid volume fraction and high interparticle interaction. In these regions qualitative and quantitative analyses of the system are lacking. An attempt to present a qualitative analysis of suspensions was given by Cheng (1980). In Figure 2.7 he produced a plot which gives the rheological characteristics of a suspension in terms of its particle concentration and interparticle attraction. However, in a recent survey Cheng (1984) was led to the conclusion that the shear properties of dense suspensions cannot be characterised by a unique viscosity or flow curve, but rather it is described by a wide viscosity distribution or shear stress-shear rate flow band.

For aqueous cement suspensions the picture is even more complex. Cement in water systems are usually concentrated suspensions with strong interparticle interactions. In addition, the physical and chemical properties of the systems are continually being changed by the chemical reactions of the hydrating cement particles. These changes will continually alter the physical and chemical interactions of the suspension by influencing the factors which determine these interactions, and consequently making the cement-water suspensions one of the most complex systems in suspension technology.
FIGURE 2.7: RELATION BETWEEN DIFFERENT TYPES OF RHEOLOGICAL BEHAVIOUR AND THE THREE CATEGORIES OF INTERACTIONS.

(Cheng (1980))
2.3: Conclusion

This conclusion can only echo the conclusions of the many reviews on the subject (Cheng (1980), Mackay (1984), Mewis (1980), Russel (1980)). Theory may successfully analyse the flow behaviour of relatively simple systems of low particle concentration and low interparticle interactions. Increasing either particle concentration or interaction will increase the complexities of the rheological behaviour and hence the theoretical analysis of the system. Research work of recent years has increased our knowledge of the behaviour of suspensions. Nevertheless no existing theory can give a general treatment to suspension rheology and cope with the hydrodynamic interaction, electroviscous forces, Brownian motion, etc. Consequently, for many years to come, we may continue to rely on experiments to provide us with the data required to analyse suspensions (cement suspensions in particular) rheological behaviour and make engineering predictions.
RHEOLOGY OF CEMENT SLURRIES

3.1 Introduction

In Chapter 2 it was argued that concentrated suspensions still present a problem for rheologists and prediction of their flow behaviour from the original constituents is still lacking in literature. This is particularly so when other than hydrodynamic forces are involved. It has also been concluded in Chapter 2 that one should rely on experiments to measure and quantify the rheological behaviour of cement slurries. Despite the increasing amount of research work performed on fresh cement slurries, the picture is still a confused one and, as yet, there is no generally accepted method of obtaining the rheological parameters of cement slurries nor is there a generally accepted model to describe their rheological behaviour. This confused state stems mainly from the complex nature of cement slurries and the failure of rheologists to produce an instrument which can measure fundamental parameters for concentrated suspensions and thick pastes. Also many early workers failed to understand the shortcomings and limitations of their instruments, and did not recognise the importance of the experimental conditions or even report them.

Although a comprehensive review of the rheology of cement slurries is beyond the scope of this chapter, it is hoped that the reader will be introduced to the rheology of fresh cement slurries and the problems associated with them. Detailed reviews of the subject can be found in Tattersall and Banfill (1983), Dimond (1975), Lapasin (1982), Massazza and Costa (1982) and Helmuth (1980).

3.2 Viscometry on Cement Slurries

As noted above there exists no single instrument which has gained a general acceptance or a widespread use to measure the rheological properties of cement slurries. However, rotational viscometers in general and the co-axial cylinder viscometer in particular (described in chapter 5) has become increasingly popular in the past few decades. Another viscometer which has been widely used in fluid mechanics is the tube or capillary viscometer, but this has been less popular for
cement slurries than the co-axial cylinder viscometer. The theory and analysis of the results of the capillary viscometer is similar to that for pipe flow discussed in Chapters 6 and 7. A good description of and analysis of the many other instruments used to measure the rheological properties of fluids can be found in Wilkinson (1960), Van Wazer et al (1963) and Whorlow (1980).

The cone and plate, cone and cone, and plate and plate are other forms of rotational viscometers which have been used with fluids but they have some limitations. The plate and plate viscometer can be used without problems with Newtonian fluids but with non-Newtonian fluids a major problem arises owing to the large variation in shear rate throughout the fluid in the gap. The cone and plate geometry has the advantage of producing a constant shear rate in the test sample. Nevertheless, this geometry presents a problem with suspensions, since the cone must theoretically be touching the plate which results in the particles in suspension being trapped between the cone and the plate. This problem was limited by Jones and Taylor (1977) who used a truncated cone with a separation between the cone and plate of 98 microns. These authors reported that consistent results were obtained if cement particles passing through a 70 microns sieve were used. Another limitation in the use of the cone and plate geometry is the centrifugal force encouraging the fluid to flow out of the gap at high rotation speeds.

A variety of other instruments, where the flow field is not as well defined as with the aforementioned rheometers, has been used to study the rheology of cement slurries. Examples of such instruments are the helical impeller used by Bhattan and Banfill (1982), the turning tube viscometer designed by Hopkins and Cabrera (1985) and the rotating spindle used by Cao and Diamond (1982). Notwithstanding the fact that such instruments can be used to produce comparative results, they can not be used for the determination of the fundamental properties of the material. It is important to recognise that the equivalent or apparent shear rate usually associated with such instruments based on Newtonian fluids can not be assumed to be correct with non-Newtonian fluids, and two completely different materials may easily produce similar results. Nevertheless, these instruments are most useful when the other instruments with a well defined flow field fail or present a
serious problem. Bhatty and Banfill (1982), for example, used the helical impeller with slurries which tend to settle in the co-axial cylinder viscometer. The turning tube viscometer was intended for construction sites where the environment is not ideal for more sophisticated viscometers, and can give an idea of the material flow curve by using different balls. However, if the instrument is required only to generate comparative results, the Marsh cone (see Budge (1981) for description), well used on construction sites, can produce useful results for cement grouts with the advantage of being easier to handle.

In the oil drilling industry, the thickening time test is used as specified in the API Spec. 10 (1986). This test is carried out with either an atmospheric or pressurised consistometer and is basically a rotating blade (see Chapter 4 for description of the atmospheric consistometer) similar in its behaviour to the helical impeller used by Bhatty and Banfill (1982). The criticisms noted above are still applicable in this case. However, the pressurised consistometer has the capability of testing the material at temperatures up to 200°C and pressures up to 140 MPa, hence simulating down-hole conditions. It is usually assumed that the blade keeps the test specimen continuously sheared. If this is true then, the action of the blade may simulate the actual job conditions when the material is continuously sheared in the case of turbulent flow or if the material is flowing in eccentric annuli. For laminar plug flow, however, the situation is different and this test can not provide a simulative test.

In addition to the instruments and techniques mentioned above there exist specialised techniques which are designed to measure one particular property of cement slurries. For instance, the shear vane test and many other techniques (see Chapter 8) are used to measure the yield stress. A non-destructive measurement of the shear modulus was obtained by Hannant and Keating (1985) by specially designed apparatus. Furthermore, there exist a number of non-destructive techniques where the material rheological behaviour is assessed by indirect means such as zeta potential, ultrasonic pulse velocity and induction calorimetry.
3.3: Flow Curves and Flow Models

A viscometer is usually used to obtain the relationship between shear stress and shear rate for fluids. This relationship then can be used for flow predictions or to gain fundamental understanding of the material behaviour and the factors affecting it. Unfortunately most practical cement slurries possess a complex rheological behaviour which is influenced by a variety of factors, hence making the task of establishing a general flow equation difficult or even impossible.

One of the most common procedures for the characterisation of the flow behaviour of cement slurries is the hysteresis cycle, where the material is subject to increasing (up curve) and then reducing (down curve) rates of shear. As described in Chapter 1 for a time dependent material, a plot of the shear stress against shear rate will give a hysteresis loop. For a given slurry the shape of the curve depends on a number of factors such as the shear history, duration of hysteresis cycle, the maximum value of shear rate in the hysteresis cycle, and viscometer type and geometries. Hence it is important that the experimental procedure and apparatus are well defined and related to the practical situation. It is almost valueless to report hysteresis loops without a full description of how they were obtained as noted by Tattersall and Banfill (1983). Also Warrall and Tuliani (1964) have shown that two completely different materials can produce similar results. Cheng and Evans (1965), on the other hand, have noted that, if the tests are performed under varying shear stress and shear rate, such as in the determination of hysteresis loops, the flow equation can only be obtained by comparing the test results with theoretically assumed flow equations. These facts have not deterred many authors and hysteresis loops are continually reported in literature. Moreover different authors have used different methods and/or apparatus which have led to inconsistency and contradiction in the reported results. Hysteresis loops of varying shapes and characteristics were obtained by Banfill and Saunders (1981) who concluded that the experimental procedure and the viscometer geometry are the main reason behind these differences.

Another commonly used method for the measurement of the flow curves of cement slurries is adopted by the API Spec 10 (1986) and used by a number of authors such as Pierzchala (1970) and Jones and Taylor
In this method, if the duration of the test is short so that the effect of cement hydration during the test is negligible, the time factor is reduced or eliminated by starting with a material which has been sheared at higher shear rate than the maximum shear rate in the experiment. This procedure ensures that the material will not undergo a structural breakdown during the test. This method has the supposed advantage of eliminating some of the shear history dependence undergone by the material. However, with material such as cement slurries where the shear history may influence the hydration process a complete elimination of the effects of the shear history may not be possible. Moreover this procedure ignores an important factor in the material rheological behaviour, namely, time dependency.

The constant shear rate experiment may furnish a preferable method to examine the material flow behaviour. This method was first used by Tattersall (1955) followed by Nessim and Wajda (1965), Dimond and Tattersall (1976) and vom Berg (1980) to study the process of structural breakdown or build up of cement slurries. It must be realised that, again, the exact material behaviour measured by this method will be largely dependent on the experimental conditions but a well defined procedure can produce results relevant to practice.

Apart from the large number of papers which give hysteresis loops; see for example Ish-Shalom and Greenberg (1960), Roy and Asaga (1979), Asaga and Roy (1980), Banfill and Saunders (1981), Vom Berg (1979), and Massidda and Sann (1982), a number of papers reported a single flow curve to describe the flow behaviour of cement slurries. Pierzchala (1970) obtained the down curve using co-axial cylinder viscometer. His flow curves were linear between rotation speeds of 600 and 300 r.p.m. and convex towards the torque axis for the lower speeds. Pierzchala proposed a power law relationship to fit part of the curve, but was unable to find equations to fit the whole curve. Similar procedure are described for the measurement of the flow curve of cement slurries in the API Spec 10 (1986). Jones & Taylor (1977) used the cone and plate arrangement on the Weissenberg Rheogoniometer to obtain flow curves for cement slurries of W/C ratio between 0.3 and 0.5. They used a vibration method of mixing in order to obtain reproducible flow curves. Their results indicate that at 0.4 W/C ratio the material changes from shear thickening to shear thinning as
the W/C ratio of the paste is increased. The Herschel and Bulkley model and the Robertson and Stiff model (1976) were used to fit the resulting flow curves. Jones and Taylor found that although the Herschel and Bulkley model gives a good fit for cement slurry of 0.3 W/C ratio, the Robertson and Stiff model was superior for the whole range of W/C ratios investigated. Jones and Taylor's results are very interesting in the sense that they show shear thickening behaviour at low W/C ratios which is in contradiction to other works, as they noted. They suggested that this behaviour may be attributed to the different viscometers used, a suggestion supported by the observations made in the course of this work (see Chapter 5). In a recent paper Atzeni et al (1985) obtained rheological measurements on two Portland cements and a ferric type cement with varying W/C ratios. Flow curves and hysteresis loops were obtained in a ribbed co-axial cylinder viscometer. These curves were fitted to various flow models using the least square method. They found that the best fit was furnished with the Eyring's flow model and a parabolic equation. Another model derived from the Eyring's model but explicitly containing the term $\tau_0$ (yield stress) also produced a good correlation.

Ivanov (1980) who gave no details of his experimental procedure used a co-axial cylinder viscometer to obtain flow curve for cement-PFA slurries. The resulting flow curves were straight at high shear rates and convex towards the shear stress axis at low shear rates. Similar flow curves were obtained by Lapasin et al (1980) for cement slurries containing admixtures. They used the co-axial cylinder viscometer to perform a variety of constant shear rate experiments. The maximum and equilibrium shear stress were used to obtain shear stress versus shear rate flow curves. It must be noted that each point on the equilibrium flow curve represents a material with a different structural level. Moreover, the maximum shear stress versus shear rate flow curves are questionable because the recorded maximum shear stresses were probably affected by the inertia of both the viscometer and the recorder as shown in this thesis (Chapter 8). In addition, the exact shear rate in the gap at the start of shearing will not be known since the time to reach maximum torque will depend greatly on the stiffness of the measuring system in addition to the speed setting on the machine dial. Additionally the material takes some time to become completely sheared throughout the gap.
Finally, the papers cited here are only typical papers of a huge number of research publications in this field.

3.4: Factors Affecting the Rheology of Cement Slurries

The rheology of cement slurries can be affected, like other suspensions, by the physical and chemical factors discussed in Chapter 2. However, there exist a number of factors which affect cement slurries somewhat differently, the most important of these being discussed below.

3.4.1: Experimental Techniques and Conditions.

Perhaps these are the most important and critical areas determining the measured rheological characteristics of cement slurry of a given W/C ratio. The following quotation from Tattersall and Banfill (1983) gives an excellent summary of the importance of the experimental techniques and conditions and the real problems in equipment used to measure the rheology of cement slurries.

"A general feature of most of the research work published in this field is the failure of the results of successive authors to agree either on a qualitative or a quantitative basis. This is largely because the results obtained depend markedly on the detailed experimental technique used, a fact which seems not to be appreciated by many authors, who consequently give no more than a cursory description of their apparatus and methods. This lack of experimental information is a serious drawback and has delayed progress because subsequent workers, failing to reproduce earlier results, have either published their own data and emphasised the difference rather than the similarities or, worse, have been put off publishing and further research altogether."

Cement slurries are active systems and their rheological properties are continuously changing with time due to the chemical reaction, structural breakdown and/or thixotropic recovery. This makes cement slurries highly dependent on the shear history. The shear history of the material starts with the start of the mixing operation. Thus, the
results of any subsequent rheological measurement will to some extent be influenced by the mixing operation. A number of studies were concerned with the effects of mixing variables on the rheology of cement slurries. Banfill (1981) reported a decrease in yield stress and plastic viscosity with the increase in mixing time. He used a propeller turbine in a beaker. Roy and Asaga (1979) used nine different mixing procedures. They concluded the yield stress, plastic viscosity and hysteresis reduces with mixing intensity. This reduction was less pronounced if the mix contained superplasticisers. Jones and Taylor (1977) were able to produce a reproducible flow curve (i.e. no hysteresis loops) by the use of a vibration method of mixing. The time of mixing required to obtain these curves increased with the W/C ratio, suggesting that more concentrated slurries breakdown more rapidly.

Mixing is only a small part of the experimental technique and handling details. Equally important are many other variables, including viscometer type and geometries (see Banfill (1981) and Lapasin et al (1982) slurry age (Ish-Shalom and Greenberg (1960)), shear cycle and duration (see section 3.2), testing procedure (Massidda and Sanna (1982)). Hence it is vitally important for comparative work that the experimental conditions and details are closely matched.

3.4.2: Solids Concentration

As with other concentrated suspension (Chapter 2), particle concentration is one of the most influential factors determining the rheological properties of cement suspensions. Unlike other rheologists, the workers in the field of fresh cement slurries have generally, perhaps understandably, avoided attempting to predict the rheological properties from the original constituents. However, they have sometimes included particle concentration or water cement ratio in empirically established relationship e.g. Vom Berg (1979), Ivanov and Slaneova (1979), Ish-Shalom and Greenberg (1960), Jones and Taylor (1977) and Uchikawa et al (1982). Tattersall and Banfill (1983) have cited thirteen papers and plotted the results in the form of log yield stress and log plastic viscosity versus water cement ratio. They have found a huge variation between the results of the various workers. Expressing the 0.4 W/C ratio as an example, Tattersall and Banfill
reported 20-fold and 50-fold range for the yield stress and the plastic viscosity respectively. This huge variation spells out the complex nature of cement slurries and emphasises the importance of the experimental techniques and conditions and the variable nature and size of the cement particles themselves.

3.4.3: Cement Fineness.

In Chapter 2 it was noted that particle size and size distribution affect the rheology of suspensions. For cement slurries, the effect is even more pronounced since hydration rate is dependent on cement fineness (i.e. grading). It is well established in literature that increasing the surface area of cement particles increases both the yield stress and plastic viscosity of the material, see for example Papadakis (1959), White and Roy (1982) and Vom Berg (1979). White and Roy (1982) studied the rheological behaviour of cement slurries and mortars containing admixtures. The fluidity of the material was found to increase with the reduction in the surface area of the solids. This was found to be dependent on the age of the mix which perhaps reflects the difference in hydration rates. To avoid using cements of different composition, Vom Berg (1979) produced cements with different granulometry from a basic cement which was separated into different particle size fractions. These fractions were then combined to produce a continuous particle size distribution with varying maximum particle size. The yield stress and plastic viscosity were found to vary with specific surface area according to Equation 3.1 and 3.2

\[
\text{Yield stress} = K_1 S_v^{3.83} \quad \ldots \quad 3.1
\]
\[
\text{Plastic viscosity} = K_2 S_v^{2.47} \quad \ldots \quad 3.2
\]

where \( S_v \) is the specific surface area and \( K_1 \) and \( K_2 \) are constants dependent on the W/C ratio.

3.4.4: Cement Composition.

Cement is a mixture of inorganic compounds which are reactive with water. The rate of reactivity of these compounds varies from one compound to another. Hence, one would expect rheology of cement
slurries to be dependent on the surface nature, hydration rate, and percentage of the existing compounds. However, the chemical composition has been found by some authors to play a secondary role compared with the effects of particle concentration and surface area. Banfill (1981) and Ish-Shalom and Greenberg (1960) reported two to three fold variation in the yield stress and plastic viscosity of gypsum regulated cements of various compositions. In contrast, Odler et al (1978) obtained rheological measurements using 12 cements of different types and concluded that the chemical and mineralogical composition of the cement was of little importance except for the case of a regulated set cement which gave slurries of much higher viscosity than the others. The relative unimportance of chemical type is supported by Locher et al (1976) and Groves (1983) that when water is added to cement, the particles will be rapidly covered with a membrane of hydration products. Hence, the chemical nature of the particles is not important, the most influential factor coming from the nature of the membrane. It is well established that C$_3$A (Tricalcium aluminate) dominates the early hydration of cement leading to what is usually termed a flash set, where cement slurries lose their fluidity without gaining strength. The flash set is prevented or retarded by the addition of an appropriate amount of gypsum. Gypsum reacts with C$_3$A to form an ettringite ($3$CaO$\cdot$Al$_2$O$_3$.$3$CaSO$_4$.$32$H$_2$O) membrane which envelopes the surface of the particles and hence inhibits reaction, leading to the so called dormant period. While there is still a controversy about the cause of the dormant period and the mechanism of hydration, the membrane theory seems to offer explanation for some chemical and rheological phenomena (Tattersall and Banfill (1983)). Owing to the fast reaction and the modification of this reaction by calcium sulphate, the quantity of C$_3$A and calcium sulphate in cement are usually considered to be the most influential ingredients of cement in the early hours of hydration.

The variation of the rheological behaviour of cement slurries with C$_3$A content was studied by a number of authors (see for example, Ish-Shalom and Greenberg (1960) and Greenberg and Meyer (1963)). Ish-Shalom and Greenberg (1960) presented graphs showing the variation in the yield stress ($\tau_0$) and plastic viscosity ($\mu_p$) and hysteresis area ($A$) with C$_3$A percentage for varying slurry ages. All curves had nearly shown the same shape where $\tau_0$, $\mu_p$, and $A$ first reduce and then
increase as the C₃A content increases. Furthermore, the effects of C₃A content become more pronounced as the hydration age increased. An explanation for this form of behaviour of cement slurries is offered by Bombled (1980) with the hypothesis that the effect of C₃A on the rheology of cement slurries is the resultant of the effects of flocculation of the aluminate phases and their hydration. However, the assumption that flocculation of the aluminates reduces the yield stress is in contradiction with the general belief. Greenberg and Mayer (1963) performed a comprehensive study on the role of calcium sulphate on the rheology of cement slurries. They measured the yield stresses and the plastic viscosities for cements of various compositions, but since the surface area is also changed it is difficult to draw reliable conclusions. Nonetheless for cements of approximately the same surface area (2200 - 2280 cm²/g.) the yield stress and plastic viscosity have varied by more than six and three folds respectively depending on the calcium sulphate content. Moreover, the slurry which contained clinker only (no calcium sulphate) was too stiff to measure. Some of the most interesting data produced by Greenberg and Mayer is displayed by the experiment on the effects of changing the C₃A content in calcium sulphate - quartz - water systems. Their results are reproduced in Table 3.1. From this table it can be seen that a very small change in C₃A content leads to a dramatic change in the yield stress and plastic viscosity. Such is the behaviour with the relatively simple system and one would expect even more complex behaviour with cement slurries.

3.4.5: Effects of Additives.

In the following discussion we will use the word additive to refer to anything added to the basic ground cement clinker and this is taken to include materials classed as admixtures.

The use of additives in concrete technology is becoming more and more popular and in the past few decades has gained a wider acceptance. In the drilling industry, however, the use of additives is more of a necessity than a choice owing to the special needs of deep wells. They can be used to reduce or increase viscosity, accelerate or retard set, and reduce or increase density.
**TABLE 3.1: EFFECT OF C$_3$A ON RHEOLOGICAL PROPERTIES OF A QUARTZ-CALCIUM SULPHATE-WATER SYSTEM**

| Conc. C$_3$A (%) | 1st Cycle | | 2nd Cycle | | | | Over-all Behaviour |
|---|---|---|---|---|---|---|
| | Yield Value | Apparent Viscosity $f$ S/D (dynes/ sq cm) | Viscosity | Yield Value | Apparent Viscosity $f$ S/D (dynes/ sq cm) | $\Delta$(S/D) |
| Gypsum | | | | | | |
| 0 | 0 | 0.20 | 0 | 0.19 | -0.051 |
| 1 | 640 | 1.55 | 660 | 1.59 | +0.026 |
| 3 | 810 | 1.91 | 830 | 1.91 | 0 |
| 5 | 1,380 | 3.26 | 1,290 | 3.00 | -0.083 |
| Hemihydrate | | | | | | |
| 0 | 160 | 0.69 | 180 | 0.76 | +0.097 |
| 1 | 1,390 | 3.13 | 1,330 | 3.16 | +0.010 |
| 2 | 1,240 | 2.92 | 1,230 | 2.95 | +0.010 |
| 3 | 1,320 | 3.01 | 1,300 | 3.04 | +0.010 |
| 5 | 1,420 | 3.41 | 1,450 | 3.35 | -0.018 |
| Control | | | | | | |
| 2 | 100 | 0.43 | 100 | 0.44 | +0.023 |
The cement slurry used in this thesis contained 10% and 2% of the cement weight of calcium sulphate hemihydrate and calcium chloride respectively. It was noted in section 3.4.4 that calcium sulphate is usually used to inhibit or retard the flash set in the cement and water system. However, the high solubility of the hemihydrate compared with the gypsum, which is usually added to the clinker, produces a solution saturated with calcium sulphate and if sufficient quantity is added, a rapid crystallisation of gypsum may occur leading to what is termed plaster set.

In pure C₃S (Tricalcium silicate) hydration the concentration of Ca²⁺ and OH⁻ in the aqueous phase increases rapidly in the early stages of the hydration. In cement hydration the trend is similar but the process is complicated by the presence of gypsum where CaSO₄ which dissolves rapidly in water tends to reduce the solubility of Ca(OH)₂, as can be seen from the work of Thomas and Double (1981). This will have a marked effect on the OH⁻ ion concentration and, hence the pH of the solution. It is well established that the value of the pH determines the sign and magnitude of the surface charge of oxide surfaces which in turn has a strong influence on the physicochemical interaction forces between particles. Silica particles were shown (Ottewill (1983)) to have a negative charge over a wide range of pH, although it becomes increasingly positive as the pH is increased. The alumina particles, however, are positively charged at pH approximately below 9.0 and negatively charged above this. Moreover, this behaviour was found (Ottewill (1983)) to be greatly affected by the salt content of the system as shown in Figure 3.1 and Figure 3.2. Mobility in these figures is a measure of the particle charge and suspension fluidity. It can be seen from these figures that if the salt content is of adequate magnitude both the silica and the alumina particles have approximately zero electrical mobility, i.e. flocculated, at about pH value of 9.0. Comparing this with our system where pH value is 9.0 (measured at 15 minutes after mixing) and the calcium chloride content is 27 X10⁻² mol dm⁻³, could explain the rapid flocculation of the material. Moreover, since the electrical flocculation forces are of a weak nature, the formed gel structure can be readily broken down by shearing. Superimposed on this behaviour is the hydration of cement and the precipitation of the hemihydrate leading to a deposition of new crystalline structure and, hence, subsequent additional stiffening.
FIGURE 3.1: MOBILITY AGAINST pH (OTTEWILL (1983))

Δ, silica in 10^{-3} mol dm^{-3} sodium chloride solution;
○, alumina in 10^{-3} mol dm^{-3} sodium chloride solution;
●, alumina in 10^{-1} mol dm^{-3} sodium chloride solution.

FIGURE 3.2: MOBILITY AGAINST pH FOR SILICA PARTICLES IN VARIOUS CONCENTRATIONS (mol dm^{-3}) OF CALCIUM CHLORIDE (OTTEWILL (1983))

○, 10^{-4}; ▲, 10^{-3}; □, 10^{-3}; Δ, 5×10^{-4}; ●, 10^{-1}. 

In the graphs above, the decrease in mobility with increasing pH for silica and alumina particles in various concentrations of calcium chloride solution is shown. The curves reflect the increased adsorption of calcium ions on the particles, which somewhat reduces the hydration layers and affects the mobility.
CHAPTER 3

of the slurry.

3.4.6: Temperature and Pressure

Temperature and pressure are two important factors affecting the rheology of cement slurries in oil well cementing. Depending on the depth and various other factors, temperature and pressure can reach high values in deep wells. Consequently, knowledge of the material behaviour under these conditions is of a real importance for proper cementing operation.

Ish-Shalom and Greenberg (1960) examined the effects of hydration temperature, varying between 20 to 35°C, for a number of hydration times. The yield stress and the plastic viscosity generally increased with the increase in the hydration temperature. Similar effects of temperature were obtained by Greenberg and Mayer (1963). Bombled (1973) measured the yield value and plastic viscosity together with the Marsh cone flow time (an increase in flow time reflects a reduction in the material fluidity) of a mortar. Bombled found the yield stress to increase very slowly up to approximately 50°C and then started to increase much more rapidly. The plastic viscosity, however, was found to reduce with temperature to a minimum value at about 40°C and to increase rapidly above 50°C. The reduction in plastic viscosity could be reflecting the change in viscosity of water with temperature, which was superseded by the effects of the increased hydration rate at the higher temperatures. Marsh cone flow time, which is a measure of the combined effects of the yield stress and the plastic viscosity, gave a flow time-temperature curve which somewhat reflects these combined effects.

In deep wells temperature and pressure could reach much higher values than those investigated by the aforementioned authors. Figure 3.3 and Table 3.2, reproduced from Smith (1976), show the effects of high temperature and pressure on the thickening time (API (1986) standard test), which is a measure of the time for which the material remains pumpable. It can be seen from Figure 3.3 and Table 3.2 that both the temperature and pressure have a dramatic effect on the flow properties of the cement slurries.
FIGURE 3.3: EFFECT OF TEMPERATURE ON THICKENING TIME OF VARIOUS API CEMENTS AT ATMOSPHERIC PRESSURE (SMITH (1976))

TABLE 3.2: EFFECT OF VARYING PRESSURE ON THE THICKENING TIME OF API CLASS H CEMENT WITH RETARDER

<table>
<thead>
<tr>
<th>Depth (ft)</th>
<th>Temperature (°F)</th>
<th>Pressure (psi)</th>
<th>Thickening Time (hours:min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10,000</td>
<td>230  144</td>
<td>5,000</td>
<td>2:10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10,000</td>
<td>1:34</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15,000</td>
<td>1:18</td>
</tr>
<tr>
<td>14,000</td>
<td>290  206</td>
<td>10,000</td>
<td>8:35</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15,000</td>
<td>5:19</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20,000</td>
<td>1:14</td>
</tr>
<tr>
<td>16,000</td>
<td>320  248</td>
<td>10,000</td>
<td>4:11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15,000</td>
<td>3:39</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20,000</td>
<td>2:30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>25,000</td>
<td>2:08</td>
</tr>
</tbody>
</table>
3.5: Conclusion

Cement slurries are very complex mixtures, the rheological characteristics of which are highly dependent on a variety of factors. Unless these factors are specified, any rheological measurements obtained on the material are almost valueless. It seems from the rheological data presented in the literature that no mathematical model can accurately describe the rheological behaviour of all cement slurries, or even one particular slurry under all conditions.
EXPERIMENTAL METHODS AND APPARATUS

4.1: Introduction

As described in Chapter 3, section 3.3.1 the rheological behaviour of cement slurries is influenced to a large extent by the experimental techniques and apparatus and, therefore, it is important that these are reported. Thus, in this chapter a complete description of the apparatus and a number of standard techniques are given. A detailed description of the experimental procedure is given in the relevant sections throughout the thesis.

4.2: Instruments

4.2.1: Waring Commercial Blender

Mixing the cement, water and additives was carried out using a Waring Commercial Blender, Model 30-70, see Figure 4.1. The mixer was in accordance with API Specification for Materials and Testing for Well Cements (1986). The mixer is propeller-type capable of 18,000 r.p.m.. An electronic control box is incorporated with the basic mixer which contains a digital output from a tachometer, a timer and a constant speed control circuitry. The control box is supported on two columns as can be seen in Figure 4.1. The constant speed control circuitry is provided so that the speed of the propeller blade is independent of load. In addition to one operator-adjustable speed control, two preset speeds (usually 4000 and 12000 r.p.m.) are available at the press of a button.

Two mixing containers of 1 litre and 4 litre sizes were provided. These containers and the propeller blade are constructed of corrosion-resistant metal.

4.2.2: Atmospheric Pressure Consistometer.

This apparatus, Figure 4.2, consists essentially of two rotating cylindrical slurry containers which are equipped with a satisfactory paddle assembly. The containers are submerged in a stainless steel water bath which is fitted with a heating unit. The slurry containers
Figure 4.1: Warring commercial blender
are rotated at a constant speed of 150 r.p.m., by engaging pins on the lid in the slots of the rotors. The rotors are belt driven by a gear-head motor which also drives an impeller to keep the water path agitated. The heating unit consists mainly of a heating element, a thermocouple and a temperature rise controller.

The consistency of the cement slurry is measured by the amount of deformation of a standardised coil spring connecting the stirring paddle and a stationary head. The deformation of the spring is expressed in Bearden units of consistency, BC, where one Bearden unit is equivalent to $2.04 \times 10^{-3}$ Nm. The paddle, Figure 4.3, and the containers are made of corrosion-resistance metal.

About 0.6 Litre of material is required to fill the container to a specified level so that the sample will just cover the paddle.

The Atmospheric Pressure Consistometer is used in the drilling industry to measure the so-called "Thickening Time", the time taken for a cement slurry to reach a consistency of 100 BC. Also, it can be used to make determinations of free water content and fluid loss as defined by the API Spec. 10 (1986).

4.2.3: Crypto Peerless Mixer

The atmospheric consistometer was used to keep the small volume mixes (600 ml. or less) agitated. Larger mixes (0.6 - 8 L.) were agitated using a Crypto Peerless, EP 12F, mixer, Figure 4.4. This mixer has an approximately eight litre bowl, and a half horsepower motor. The beater (mixing blade) is connected eccentrically to the planetary gear (a disc holding the beater), so that when the planetary gear is rotated the beater will change position in the bowl. With the beaters own rotation superimposed on the planetary rotation the mixer will provide a very effective mixing mechanism. This is particularly useful for mixing two phase systems, such as cement slurries, where phase separation may occur. The mixer is equipped with three speed settings, slow, medium and fast. The slow setting was used throughout this work. At this setting, planetary and beater speeds are forty r.p.m. and a hundred r.p.m. respectively.
Figure 4.2: Atmospheric consistometer

Figure 4.3: Atmospheric consistometer's blade, spring and dial indicator
Figure 4.4: Crypto Peerless mixer
4.2.4: Fann V-G Meter

The Fann V-G meter model 35, shown in Figure 4.5 and Figure 4.6, is basically a co-axial cylinder viscometer in which the rotor sleeve (outer cylinder) rotates and the torque exerted on the bob (inner cylinder) is measured by a torsion spring. The instrument is equipped with a dual-speed synchronous motor and geared arrangement by which the rotor sleeve is driven from above. The bob is suspended on a torsion spring, which in turn, is secured to the viscometer head. The deflection of the spring is read on an indicating dial through a magnified eye piece. The dial is marked at one degree intervals from one to three hundred, the maximum permissible deflection. The spring measures $3.87 \times 10^{-5}$ Nm of torque per degree of rotation.

The bob is made from a hollow cylinder with thin walls to reduce inertia effects. The bottom of the bob is flat and the top is conical, Figure 4.6. The outer cylinder has no bottom and is simply a sleeve which is locked into place on the driving head. Filling the gap between the two cylinders is achieved by pushing up the cup (beaker), full of the sample, slowly to a predetermined height so that when in position the material fully covers the bob. The outer cylinder must be rotating whilst raising the cup to ensure a total gap filling.

There are six possible speeds on the basic instrument (3, 6, 100, 200, 300, 600 r.p.m.). However, since one of the two instruments available was equipped with a reduction gear box, there were six additional speeds. These were 0.9, 1.8, 30, 60, 90 and 180 r.p.m. Changing the rotation speed of the rotor sleeve is simple and fast and can be executed while the rotor sleeve and motor is running. This is not the case with the Weissenberg rheogoniometer for which speed change and stops can only be carried out when the motor is switched off. For this reason, and owing to the sensitivity of the material, the Fann V-G Viscometer was used to establish the basic flow curve of the cement paste used in this work.

4.2.5 Contraves Rheomat 15 Viscometer

This rotational viscometer, Figure 4.7, consists of two main
Figure 4.5: Fann V-G viscometer
Figure 4.7: Contraves Rheomate 15 viscometer
CHAPTER 4

components - a measuring head mounted on a stand, and a control cabinet. The control cabinet houses electrical components, an on/off switch on the front panel, and a fifteen-position frequency selector which may be set to produce fifteen different input signals to give a total of fifteen rotational speeds, ranging from 5.6 to 352 r.p.m.

Inside the outer case of the measuring head the synchronous motor, gears, speed-selector device, and a motor bearing are all suspended by a torsion wire which is securely attached at the top inside the outer case. The torque exerted on the bob when rotated in a fluid is measured, via the motor, by the twist of a precision spiral spring. The deflection of the spring is indicated by a pointer and dial indicator. The maximum deflection of the pointer is reached after a rotation of 344 degrees which is equivalent to the hundredth division on the scale. The spring measures $3.88 \times 10^{-5}$ Nm. of torque per scale division or $1.128 \times 10^{-5}$ Nm. per degree of rotation.

The instrument is supplied with a various combination of bobs and cups, the bobs are cylindrical with cone shaped ends.

The heavy weight of the solid bobs, gears and motor implies very high inertial effects which will complicate the measurements of the rheological properties of time-dependent fluids and may lead to false values of yield stress due to the torque overshoot. For this reason and since the cement slurry used in this work is a time-dependent material, the instrument was only used to obtain values of the gel strength of the cement slurry in order to compare them with the values obtained using other instruments.

4.2.6: Weissenberg Rheogoniometer

The basis of the Weissenberg Rheogoniometer is the simple cone and plate viscometer which has been developed over the years to offer a range of sensitivities and versatilities. The instrument was first developed to measure the stress-rate of shear dependences in the fluid under test in both the tangential and normal planes. The complete instrument is shown in Figure 4.8 in which the measuring head (Figure 4.9) is in the middle, the motor and gearbox on the left and the control panel on the right.
Figure 4.9: Weissenberg Rheogoniometer measuring head
The instrument is powered with three phase synchronous motor rated at one horse power. The motor drives a gearbox which covers nearly six decades of angular velocity in approximately sixty logarithmic steps, which allows the rotation speed of the bottom platen to be varied from 375 r.p.m. to $4.74 \times 10^{-4}$ r.p.m.. The gearbox is coupled to the measuring head via an electro-magnetic brake/drive unit. This brake/drive unit is provided so that extremely rapid starts and stops (less than ten milliseconds) can be made during testing, while the motor gearbox unit being left running. The electro-magnetic brake/drive unit is controlled by a switch mounted on the control panel. This switch is a forward/off/reverse switch which enables the motor to be run in each direction so that the symmetry of the sample can be tested.

The torsion head is a complete assembly mounted on a vertical precision slide (The rheogoniometer column) which is positioned at the back of the main drive box. The torsion head consists of a torsion bar firmly clamped at the top and clamped at the bottom to the rotor of an air bearing which in turn carries the upper platen at its lower end. When air pressure is applied, the rotor of the air bearing is held accurately in the centre with a high degree of rigidity and, since there is no metal contact, provides a virtually frictionless bearing. This yields an extremely sensitive torque measuring system. The clamp at the lower end of the torsion bar also carries a ten centimetre long arm, at the end of which is fitted the armature of the torsion head transducer. This transducer consists of the stator coils mounted side by side on the same axis, and the armature (cylindrical) is held within the coils. As the armature is moved axially, the increase in inductance of one coil and the decrease of the other is measured by transducer meter. The transducer meter is fitted with an output-jack socket and will drive any suitable external recorder or other equipment. A Bryans xy/t recorder, 2600 series, was used (see section 4.2.11). With this recorder the output of the transducer meter was found to contain an appreciable amount of noise, so a Double Chebycher Filter unit was used in conjunction with the recorder and successfully eliminated the noise. Calibration of the torsion bar was achieved by very low friction pulleys and weight system built in the laboratory and mounted on the rheogoniometer column. All the moving parts of the torque measuring system were designed to have a low
weight in order to reduce inertia effects.

The entire torsion head assembly, including the top platen, can be moved vertically along the rheogoniometer column, so that the distance between the top and bottom platens can be varied (0 - 30 cm.). In addition, the platens are equipped with three hollow bolts and countersunk screws, which allow cones, plates or cylinders to be attached to the top and bottom platens. This, consequently, produces an extremely versatile instrument, which is best displayed by its ability to accommodate any rotational viscometer arrangement (co-axial cylinder, cone and plate, etc.).

The instrument is equipped with a system for normal force measurement and temperature control. However, as neither of these facilities was used in the course of this work, they will not be discussed any further.

Although starts and stops of rotation can be performed extremely rapidly on this instrument, the common requirement of varying the speed of rotation without stopping rotation is not possible. This is why the Fann viscometer was used to establish the flow curve of the material in preference to the Weissenberg Rheogoniometer. Therefore, the Weissenberg Rheogoniometer was put in use only when a constant rotation speed was required, and only with the co-axial cylinder or vane test arrangements.

4.2.7 Haake Viscometer

The Haake viscometer, model RV2, is a co-axial cylinder viscometer in which the rotor (inner cylinder) is capable of constant rotation speed up to 1000 r.p.m.. The basic instrument is equipped with a xy-t recorder, a temperature controller and a speed programmer. The speed programmer (PG 142) starts with a hold time at zero speed selected for pre-warming or regeneration period, followed by a time linear speed increase up to the pre-set maximum which can be any percent value between 0.1 to 100% of 1000 r.p.m.. A hold time at the maximum speed can be chosen before the speed programme is automatically reversed. The whole programme can be repeated automatically.
Shear stress measured at the surface of the inner cylinder is measured by the amount of twist of a precision torsion spring. The twist in turn is measured by means of a potentiometer. The signal is recorded on a dial indicator which has a range between 0 to 100 or on the xy-t recorder. A wide range of measuring-drive-unit is available. The MK50 measuring-drive-unit has a maximum torque of $5 \times 10^{-3}$ Nm. and a maximum deflection of 0.5 degree.

The instrument was borrowed specifically to record the rate of build up or breakdown of structure of a fresh cement paste under a constant shear rate (constant rotor speed) and constant temperature conditions.

4.2.8: Gun Rheometer

The Gun Rheometer shown in Figure 4.10 is a special purpose extrusion viscometer. It operates under constant applied pressure and is used to measure the yield stress and storage properties of non-Newtonian fluids. The samples are stored in detachable horizontal tubes which are fitted to the air chamber. A solenoid operated valve controls the pressure and movement of the sample is derived from a photoelectric sensor which activates the timer. The pressure can be increased or decreased by turning a knob on the front panel. The pressure is displayed by a three digit indicator which operates at two ranges, from 0 - 0.2 and 0 - 2 bars.

Figure 4.10: Gun rheometer
4.2.9: Mono Pump

The Mono pump (Figure 4.12) used is a Mono Merlin moving cavity type pump. The pump and the complete pump line assembly is shown in Figure 4.11. The pump consists essentially of a single helix metal rotor which revolves eccentrically within a double helix resilient stator of twice the pitch length. The rotor, of constant circular cross section, makes an interference fit inside the stator and creates a continuously forming seal line and carrying the pumped material with it. The pump has a cast iron casing, soft natural rubber stator and a hard chrome plated steel rotor. The pump has a maximum capacity of 0.2 m³/hr. and a maximum delivery pressure of seven bars. A hydraulic infinitely variable speed drive unit is fitted to the pump with a combination of a handwheel and a speed indicator to give a manual speed control between 5 and 121 r.p.m. The speed of the pump is dependent on the delivery pressure. Thus, the reading of the indicator was not used to represent the actual discharge of the pump and other means were used to calculate the true pump discharge. A ten litre hopper was fitted to the flange at the feed end of the pump, Figure 4.12, while at the discharge end three different flange sizes were employed so that one of the three different sized tubes could be used with the pump. The tubes and the coupling were made of steel. Special attention was paid to the choice of the coupling so that there was no change in the internal diameter of the tube at the joints. The reason for this choice was to avoid interference with the flow when the material passes through the joint.

4.2.10: Intron Pump

The Intron, model 1122, Universal Testing Instrument, usually used for testing materials in tension, compression and flexure, was adapted to serve as a constant discharge pump. The crosshead on the load frame was used to push a plunger inside a vertical tube which is filled with cement paste, Figure 4.13. The vertical tube can be connected to any system (tube, orifice, etc.) so that when the plunger is pushed down, a constant speed pumping will be achieved. The crosshead has a vertical travel of 1000 mm and a constant speed which is precisely governed by a crystal controlled clock which commands a high performance positional servo drive system. This combination
Figure 4.11: Pump line
Figure 4.12: Mono pump
provides a constant rate of crosshead motion irrespective of load. The pushbutton control unit provides instantaneous electronic selection of crosshead speeds \((0/0.5, 0.1, 0.2, 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000 \text{ mm/minute})\) as well as the basic start/stop commands. Combining the constant speed of the crosshead with its high stiffness \((50 \text{ KN/mm})\) and the high stiffness of the tubes used in this work, yields a constant discharge pump which can be started/stopped and increased/decreased in discharge at a press of a button, whatever the load. This property of the Instron pump contrasts with the Mono pump which has a pressure dependent discharge and fluctuating pumping speed. Consequently, the Instron pump was used, in preference to the Mono pump, to measure the start up pressure in the pump line as explained in Chapter 7.

4.2.11: Pressure Transducers

Measurements of pressure were made using subminiature RDP, type "S", gauge pressure transducers which had a head diameter of only \(4.7 \text{ mm}\), Figure 4.14. These transducers utilise four bonded strain gauges arranged in a Wheatstone bridge configuration. The transducers have a thin flush diaphragm and heavy sidewalls which are made from one piece of stainless steel. The advantage of this design is that the diaphragm is rugged, but at the same time is thin enough to measure pressure of only a few centimetres of water. The diaphragm is welded to the transducer body to provide a perfect seal in order to prevent moisture from penetrating inside the transducer and causing the strain gauges to short to the metal case. Temperature in these transducers is accomplished by using temperature sensitive components located inside the transducers. The transducers have a pressure range from zero to seven bars, and a maximum input voltage of three volts. The output at the maximum input voltage is different for each transducer and it varies between 52 millivolts and 105 millivolts. The signals from the transducers are displayed by a Bryans xy/t recorder, see section 4.2.11.

Owing to the subminiature size of the transducer, very thin and fragile electrical connecting wires were supplied with it. To protect these wires, a rigid metal cylindrical casing was manufactured to
house them. The casing was designed so that the electrical socket could be fitted at one end and the transducer bolted at the other with all the fragile wiring protected inside the casing and the transducer protruding from the casing end. Figure 4.14 shows the transducer, the socket and the cylindrical casing or the transducer house. The transducer diaphragm can be installed to be flush with the inner surface of the tube of the pumping line by screwing the protective cylindrical casing into a carefully cut adapter which is welded to the tube surface. Figure 4.15 shows the transducer casing, the adapter and an electrical cable which is used to connect the transducer to the power supply and the xy/t recorder.

A perfect seal was achieved by inserting two rubber O-rings, Figure 4.16, one between the transducer and the transducer casing, and another between the transducer casing and the outer surface of the tube of the pumping line.

The use of this subminiature welded flush diaphragm pressure transducer is convenient in this work for two reasons: Firstly, the welded diaphragm leads to a direct measurement of pressure and no intermediate medium is needed to transfer the pressure between the cement slurry and the transducer. Secondly, the small diameter of the diaphragm (4.7 mm) results in little interference to flow when the transducer is fitted flush with the inner surface of the tube.

### 4.2.12: Bryans xy/t Recorder

The output signals from the pressure transducer and the Weissenberg Rheogoniometer were read by Bryans xy/t recorders. These recorders use A3 size graph paper. They are used in the y-t mode, where y and t were taken to represent the output signal and time respectively. Seventeen basic sensitivity switch settings for the input signal (from 0.05 millivolts to 10 volts per centimetre) and seventeen for time (from 0.1 second to 20 seconds per centimetre) are provided by the 2600 series. In addition each setting can be modified to increase the sensitivity up to 2.5 times the set values.
Figure 4.14: Pressure transducer and the cylindrical casing

Figure 4.15: Pressure transducer - complete assembly
4.3: Experimental Techniques

In this work, numerous experimental techniques and methods were employed to suit the requirements and conditions of each test. To avoid confusion and to help the reader to relate the methods and techniques to the test in question, they have been outlined in the relevant sections throughout this thesis.

The materials proportions and the mixing procedure, which were maintained in this work are given below. The standard agitation procedure and the standard Fann viscometer tests are also given below.

4.3.1: Materials
For all the work described in this thesis the same mix proportions were used as shown in Table 4.1. However, the total mix quantity has varied according to the volume of sample needed in any particular test.
CHAPTER 4

Table 4.1: Table of Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>% of Cement Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class G Cement</td>
<td>100</td>
</tr>
<tr>
<td>Distilled Water</td>
<td>65</td>
</tr>
<tr>
<td>Gypsum - Calcium Sulphate hemihydrate</td>
<td>10</td>
</tr>
<tr>
<td>Calcium Chloride</td>
<td>2</td>
</tr>
</tbody>
</table>

The class G oil well cement was supplied by Blue Circle Industries PLC. The material was packed in either sealed drums or in double polythene bags and stored in a dry room. The typical chemical composition and particulate properties are given in Table 4.2 and 4.3. The gypsum is a commercial product marketed by Dowell Schlumberger. It consists mainly of calcium sulphate hemihydrate. The calcium chloride used was fused granular type of 1-2 mm particle size.

Table 4.2: Typical Chemical Composition of Class G Oil Well Cement.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₃S</td>
<td>57.4</td>
</tr>
<tr>
<td>C₃A</td>
<td>2.2</td>
</tr>
<tr>
<td>C₄AF+2C₃A</td>
<td>20.5</td>
</tr>
<tr>
<td>MgO</td>
<td>0.8</td>
</tr>
<tr>
<td>SO₃</td>
<td>1.9</td>
</tr>
<tr>
<td>L.O.I.</td>
<td>0.9</td>
</tr>
<tr>
<td>I.R.</td>
<td>0.25</td>
</tr>
<tr>
<td>Total alkali as NA₂O</td>
<td>0.41</td>
</tr>
</tbody>
</table>
Table 4.3: Typical Particulate Properties of Class G Oil Well Cement

Apparent particle density = 3180 Kg./m³
Surface area = 310 m²/Kg.

Particle Grading

<table>
<thead>
<tr>
<th>Size (micron)</th>
<th>% finer than</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>100</td>
</tr>
<tr>
<td>500</td>
<td>99.99</td>
</tr>
<tr>
<td>250</td>
<td>99.98</td>
</tr>
<tr>
<td>125</td>
<td>99.3</td>
</tr>
<tr>
<td>90</td>
<td>97.2</td>
</tr>
<tr>
<td>63</td>
<td>90.2</td>
</tr>
<tr>
<td>45</td>
<td>80.6</td>
</tr>
<tr>
<td>40</td>
<td>74</td>
</tr>
<tr>
<td>25</td>
<td>60</td>
</tr>
<tr>
<td>20</td>
<td>52</td>
</tr>
<tr>
<td>15</td>
<td>42</td>
</tr>
<tr>
<td>10</td>
<td>32</td>
</tr>
<tr>
<td>5</td>
<td>18</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
</tr>
</tbody>
</table>
CHAPTER 4

4.3.2: Mixing Procedure

The Procedure used for mixing was that recommended by the API Spec 10 (1986). The required amount of water, Table 4.1, was placed in the mixing container (1 or 4 litre size depending on the size of the mix) of the Waring Blender which was then turned on at low speed (4000 r.p.m.). The weights of solids calcium chloride, gypsum and finally cement were added to the water as fast as possible. The mixer was then turned on at high speed (12000 r.p.m.) for a further 35 seconds.

4.3.3: Standard Agitation Procedure

After mixing the sample was transferred to the atmospheric consistometer container. The material was then stirred in a water bath at 20°C for 20 minutes.

4.3.4: Standard Fann Viscometer Test

After stirring in the consistometer the sample was transferred to the Fann viscometer cup, and it was sheared at apparent rate of shear of $10225^{-1}$ for 60 seconds, the dial reading was taken at the end of 60 seconds, and the rate of shear was reduced to zero in six steps. The time between each step was 20 seconds and the dial reading was taken at the end of each 20 seconds.

To obtain consistent results, all tests were performed in a constant temperature room at 20°C.
APPRAISAL OF THE USE OF THE CO-AXIAL CYLINDER VISCOMETER TO MEASURE THE RHEOLOGICAL CHARACTERISTICS OF THE CEMENT SLURRY USED IN THIS WORK.

5.1: Derivation of Basic Equations

For the purposes of the following discussion we shall consider a co-axial cylinder viscometer of dimensions shown in Figure 5.1, and of a sufficient height for the end effects to be neglected. For simplicity we shall also consider that the torque is measured on a stationary inner cylinder and the outer cylinder is rotating at constant angular velocity \( \Omega \).

![Figure 5.1: Co-Axial Cylinder Viscometer](image-url)
CHAPTER 5

5.1.1: Assumptions

For the following derivation to hold, the following assumptions must be true.
1) Flow occurs only in circular streamlines, on a horizontal plane perpendicular to the axis of the cylinders.
2) The liquid is incompressible, homogeneous and isotropic.
3) The motion is steady.
4) No slippage at the surface of the cylinders.
5) The system is isothermal.
6) No plug flow for material with yield stress.

5.1.2: Basic Equations

From the above assumption it can be deduced from the equations of motion (see Van Wazer et al (1963) that:

\[
\frac{d}{dr}(r \tau) = 0 \quad \text{....(5.1)}
\]

where \( \tau \) is the shear stress at radius \( r \) in the fluid in the annulus.

Integration of Equation 5.1 gives:

\[
r \tau = C \quad \text{....(5.2)}
\]

Where \( C \) is constant and can be found from the boundary conditions. If the measured torque at the surface of the inner cylinder is \( T \) then the shear stress at the surface of the bob is given by:

\[
\tau_b = \frac{T}{2 \pi h R_b^2} \quad \text{....(5.3)}
\]

Putting \( \tau = \tau_b \) at \( r = R_b \) in Equation 5.2 and substituting in Equation in 5.3 yields:

\[
C = \frac{T}{2 \pi h} \quad \text{....(5.4)}
\]

Substituting in Equation 5.2 gives:
The non-zero shear rate in the co-axial cylinder viscometer can be shown (see for example Van Wazer et al (1963), to be equal to:

$$\gamma = r \frac{d\omega}{dr} \quad \ldots (5.6)$$

Where $\omega$ is the angular velocity at radius $r$.

Rearranging Equation 5.6 and integrating gives:

$$\int_{\frac{1}{R_b}}^{\frac{1}{R_c}} \Omega = \int_{\frac{1}{R_b}}^{\frac{1}{R_c}} \frac{1}{r} \gamma \, dr \quad \ldots (5.7)$$

Using Equation 5.5 Equation 5.7 can be rearranged to give:

$$\frac{\tau_c}{\tau_b} = \frac{\Omega}{\frac{1}{R_b} \int_{\frac{1}{R_b}}^{\frac{1}{R_c}} \gamma \frac{dt}{t}} \quad \ldots (5.8)$$

For time independent fluids

$$\gamma = f(\tau)$$

$$\frac{\tau_c}{\tau_b} = \frac{\Omega}{\frac{1}{R_b} \int_{\frac{1}{R_b}}^{\frac{1}{R_c}} f(\tau) \frac{dt}{t}} \quad \ldots (5.8)$$

Equation 5.8 is the general equation of flow in the co-axial cylinder viscometer for time independent fluids. If the form of $f(\tau)$ is known, Equation 5.8 may be integrated to derive an expression relating the angular velocity to the torque, material rheological parameters and
CHAPTER 5

instrument geometries. On the other hand, a number of authors (see Whorlow (1980)) have obtained a general solution for the shear rate in annulus of the co-axial cylinder viscometer by differentiating Equation 5.8 with respect to shear stress and expressing the results in a series form. However, the analysis of results using this procedure is laborious and much more involved than the exact solution derived for the simple fluids, section 5.1.3 and 5.1.4.

In section 5.1.3 and 5.1.4 expressions for the most common flow models, viz. the Newtonian and Bingham fluid flow models will be given. Expressions for other models have also been derived (see Van Wazer et al (1963)), but in most cases the resulting expressions are at least as difficult to handle as the general approach mentioned above.

5.1.3: Newtonian Fluids

The relation between shear stress and shear rate for Newtonian fluids was given in Chapter One as:

\[ \tau = \mu \dot{\gamma} \]  
\[ \text{...(5.9)} \]

Substituting for \( \tau \) and \( \dot{\gamma} \) from equations 5.5 and 5.6 yields

\[ d\omega = (T/2\pi h \mu) \frac{dr}{r^3} \]
\[ \text{...(5.10)} \]

Integrating Equation 5.10 between the limits \( \omega = 0 \) and \( \Omega \), and \( R_b \) and \( R_c \)

\[ \int_{0}^{R_c} d\omega = \frac{T}{2\pi h \mu} \int_{R_b}^{R_c} \frac{dr}{r^3} \]

which yields

\[ \Omega = \frac{T}{4\pi h \mu} \cdot \left( \frac{1}{R_b^2} - \frac{1}{R_c^2} \right) \]
\[ \text{...(5.11)} \]
CHAPTER 5

Substituting for $T$ from Equation 5.5

$$\Omega = \frac{(r^2 \tau)}{(2 \mu)} \cdot \left( \frac{1}{R_b^2} - \frac{1}{R_c^2} \right)$$  \hspace{1cm} \ldots (5.12)

Using Equation 5.9 and 5.12 it can be shown that

$$\dot{\gamma} = \frac{2\Omega}{(r^2 \cdot \left( \frac{1}{R_b^2} - \frac{1}{R_c^2} \right))}$$  \hspace{1cm} \ldots (5.13)

Using Equation 5.13 it can be shown that the shear rate at the surface of the bob and cup are

$$\dot{\gamma}_b = \frac{(2R_c^2)}{(R_c^2 - R_b^2) \Omega}$$  \hspace{1cm} \ldots (5.14)

$$\dot{\gamma}_c = \frac{(2R_b^2)}{(R_c^2 - R_b^2) \Omega}$$  \hspace{1cm} \ldots (5.15)

and the mean shear rate in the gap is

$$\dot{\gamma}_m = \frac{(R_b^2 + R_c^2)}{(R_c^2 - R_b^2) \Omega}$$  \hspace{1cm} \ldots (5.16)

5.1.4: Bingham Fluids

For Bingham fluids

$$\tau = \tau_0 + \mu_p \dot{\gamma}$$  \hspace{1cm} \ldots (5.17)

Similar treatment to that given above with the Newtonian fluids yields

$$\Omega = \frac{(\dot{\gamma}/(4\pi\mu_p))}{(1/R_b^2 - 1/R_c^2)} = (\tau_0/\mu_p) \ln \left( \frac{R_c}{R_b} \right)$$  \hspace{1cm} \ldots (5.18)

Putting $\tau = \tau_b$ at $r = R_b$ in Equation 5.5 and substituting for $T$ in Equation 5.18 yields

$$\tau_b = \left[ \frac{(2\mu_p R_c^2)}{(R_b^2 - R_c^2)} \right] \Omega + \left[ \frac{(2\tau_0 R_c^2)}{(R_b^2 - R_c^2)} \right] \ln \left( \frac{R_c}{R_b} \right)$$  \hspace{1cm} \ldots (5.19)
CHAPTER 5

Hence, a plot of $\tau_b$ versus $\Omega$ gives a straight line of slope

$$
(2\mu_p \frac{R_c^2}{(R_b^2 - R_c^2)} \text{ and intercept the shear stress axis at}
$$

$$
\left(\frac{2\tau_b R_c^2}{R_b^2 - R_c^2}\right) \ln\left(\frac{R_c}{R_b}\right)
$$

Equation 5.18 and 5.19 are only applicable if all the material in the gap is undergoing shearing. If the shear stress at any point in the annulus falls below the yield stress ($\tau_y$) plug flow will occur. The equation relating the shear stress $\tau_b$ to the angular velocity can be deduced from Equation 5.19 by substituting $R_c$ by $r_p$ the inner radius of the plug.

5.1.5: Time Dependent Materials

Unfortunately the equations derived in sections 5.1.3 and 5.1.4, or other expressions derived in literature including the general approach, do not give exact solutions for time dependent material of the type used in this thesis. With such materials the best solution is to keep the gap width to a minimum in order to reduce the variation in shear rate across the gap. With small gap widths the shear rates calculated for Newtonian fluids, section 5.1.3, can be used with any fluid without a great loss of accuracy, and this is often called 'apparent' shear rate. However, this is not always true and care must be taken when analysing results.

5.2: Factors Affecting Rheological Measurements in the Co-axial Cylinder Viscometer.

Because the problems detailed in the previous section are often not appreciated and corrected for by workers in cement rheology, there are many false results in literature where the published data does not reflect the real rheological characteristics of the material. Deviation from the assumptions in section 5.1.1 are not the only source of errors, other problems being associated with the instruments and the materials.
5.2.1: Errors Related to Instruments

These errors could arise from a variety of causes. These could be:-

i) Dimensional errors: For reputable manufacturers these are assumed to be minimal.

ii) Setting errors: The cylinders must ideally be set in a concentric position, but due to age or misuse some eccentricity may occur.

iii) Calibration errors: The torque measuring system and rotation speeds of the instrument must be checked and calibrated regularly to ensure a correct calculation of shear stress and shear rate.

iv) End effects: In the derivation of the equations in section 5.1 the cylinders were assumed to be long so that end effects can be neglected. In practice, cylinders have a finite height and end effects could be quite significant (as high as 30%). End effects are dependent on numerous factors such as the height and radius of the cylinders, gap width, instrument design and material rheological behaviour.

Experimental determination of end effects is explained in Van Wazer et al (1963) and Whorlow (1983). In these methods, correction for end effects is obtained by assuming that the bob height is equal to \( h + h_0 \) where \( h \) is the actual height of the bob and \( h_0 \) is the additional height due to the end effect. \( h_0 \) can be obtained from a plot of the experimental data taken with several values of \( h \). However, this is a laborious and time consuming operation particularly because of the shear history dependence of time dependent materials. Consequently, ingenious designs have been adopted by research workers and instrument manufacturers to reduce but not eliminate end effects, see Whorlow (1980) for details.

Bottom end effects have been reduced in the Fann viscometer by rotating the outer cylinder while the torque is measured on the bob (see Chapter 4) and by co-terminous inner and outer cylinders, Figure 5.2. The effect of the cone shaped top end being accounted for in the calculation of shear stress. With the Weissenberg Rheogoniometer and
the Haake viscometer the effects of the top and bottom ends are reduced by manufacturing the bob with a hollow bottom so that an air bubble will be trapped under it, consequently, with the top end of the bob not covered with fluid, the end effects could be very small.

![Diagram of Haake viscometer](image)

**FIGURE 5.2**: CO-TERMINOUS INNER AND OUTER CYLINDERS USED WITH THE FANN VISCOMETER TO REDUCE BOTTOM END EFFECT

v) Turbulence: At very high rotation speeds the stream line flow field will be disturbed and the flow will be too complex to enable the material rheological behaviour to be calculated from the co-axial cylinder viscometer torque readings. However, this was not a problem with the speeds and materials described in this thesis.

vi) Viscous heating: The viscosity of most fluids is affected to some extent by temperature. In the co-axial cylinder viscometer, the material stays in the gap, hence, the heat produced by shearing could present a problem, although with suspensions the change in viscosity of the suspending fluid has less effect. However, with cement slurries temperature rise could also alter the viscosity by influencing the hydration process (see Chapter 3 section 3.3.6), which makes the task of accounting for temperature effects very difficult.
Consequently, long time measurements should be avoided, but, if this is not possible an instrument with a constant temperature bath should be used.

5.2.2: Errors Related to Materials

Apart from the time dependency and the real problems associated with it, as discussed in Chapter 3, phase separation presents serious complications which may have a significant influence on the results of cement slurries. In section 5.1.1 it was assumed that the material is homogeneous. Although suspensions are not ideal homogeneous materials, they can be assumed to behave like them, if the dimensions of the instrument are much larger than the particle size in the suspension. However, if phase separation occurs leading to a differential particle concentration, the assumption of homogeneity will collapse.

The degree to which this deviation from the ideal homogeneous fluid will affect the measurements is dependent on the type and the degree of separation. Wesche et al (1973) defined the various possible types of separation which can occur in the co-axial cylinder apparatus. These include separation due to gravitational and centrifugal force, slippage at the surfaces of the cylinders and differential structural level due to the variation in shear rate across the gap.

5.2.2.1: Separation Due to Sedimentation and Centrifugal Force

Wesche et al (1973) have shown experimentally the existence of separation, but, they were unable to produce a conclusive explanation of the problem. Bhatty and Banfill (1982) studied the sedimentation behaviour of cement slurries in two rotational viscometers of different geometries. They found a significant sedimentation in the co-axial cylinder viscometer and, concluded that, if errors are to be kept small, only cement slurries of W/C ratio of less than 0.35 should be used with this geometry. They also found the interrupted helical impeller, which imparts an upward component of motion to cement particles in addition to the radial shearing, largely reduces the sedimentation of cement particles.
Sedimentation problems could perhaps be solved by making the duration of the test as short as possible. On the other hand, separation due to centrifugal force could be eliminated or reduced by the use of low rotation speed for a given shear rate, i.e. by the use of cylinders of large radii and small gap width.

5.2.2.2: Separation Due to Slip and Wall Effects

When the co-axial cylinder viscometer is used to measure the rheological properties of cement slurries in particular and suspensions in general, three questions are often asked. Firstly, does slip occur on the surface of the smooth cylinders? Secondly, if slip occurs, how does it affect the measurements? Thirdly, can slip be eliminated by the use of roughened cylinders? Although it is highly unlikely that suspensions, including cement slurries exhibit a true slip, apparent slip can be observed owing to variation in particle concentration near the wall of the cylinders. Dimond (1975) was probably the first person to visually observe the slip phenomenon in the co-axial cylinder viscometer. Dimond (1975) used a specially built instrument in order to allow for an uninterrupted view of the flow in the annulus. The flow patterns produced at constant speed of rotation were observed with a cine camera and two distinct flow regions were produced with smooth cylinders:

i) A sheared layer adjacent to the inner cylinder.

ii) A solid plug adjacent to the outer cylinder which slides at some rotation velocity. After a given time, this plug breaks and shearing occurs throughout the gap.

With serrated cylinders a similar pattern was observed except that the outer plug did not slide but remained intact and connected to the outer cylinder to the end of the experiment. Lapasin et al (1983) used co-axial cylinder viscometers of different geometric characteristics i.e. four with smooth cylinders of 0.96, 1.41, 2.6 and 5.8mm gap width, and one with rough cylinders of 2.6mm gap width.

The largest three gap widths resulted in similar shear stress - shear
CHAPTER 5

rate flow curves, while the curve obtained with the smallest gap width considerably deviated towards lower shear stresses at a given shear rate. Lapasin et al attributed this deviation to the small gap size compared with the particle size, hence, upsetting the condition of laminar flow. Also, with the largest three gap width and smooth cylinders, a discontinuity in the flow curve was observed. This discontinuity did not exist with the grooved cylinders, an observation attributed by Lapasin et al to the slippage and plug flow. Jones et al (1976) using the cone and plate arrangement, observed on the surface of the cement slurry a water layer which they related to a plug flow behaviour, a phenomenon usually associated with the co-axial cylinder viscometer. Jones and Taylor (1978) demonstrated experimentally the existence of such a layer by monitoring the electrical current passing through a copper wire imbedded in the top perspex cone.

The papers referred to above confirm the existence of slip with the smooth surfaces. Nonetheless, only few researchers, Higgs (1974) and Mannheimer (1983) have corrected their co-axial cylinder viscometer data to account for slippage.

5.3.1: Methods for the Calculation of the Slip Effect

Many methods have been proposed in the literature for the calculation of the slip effect. (Mooney (1931) derived a method which corrected the experimental data for the slip effect, but this method required three combinations of bob and cup radii. Oldroyd (1956) discussed another method to calculate slip velocity in the co-axial cylinder viscometer. The method is based on the assumption that slip velocity is only dependent on the shear stress. But, again various cup sizes are required. In recent years Cheng and Parker (1976) and Mannheimer (1983) have introduced further methods to calculate the slip velocity. These methods are described briefly below.

i) Cheng and Parker (1976)

Cheng and Parker presented four methods for the determination of wall slip in the co-axial cylinder viscometer. Two of these methods, suitable for a finite cup (outer cylinder) radius, will be described
here. Cheng and Parker assumed that slip does not occur with rough surfaces and that the slip velocity \((V_s)\) is a function of the wall shear stress \((\tau_w)\), i.e.

\[
V_s = f(\tau_w) \quad \ldots \quad (5.20)
\]

However, it is shown in section 5.4 that Cheng and Parker's assumptions are not true for the slurries described in this thesis and hence the derivation of their equations for slip velocity is not given, only the final result for \(V_s\).

**Method A** is based on the use of one smooth bob and one rough bob of the same diameter, and one rough cup. The final expression for the slip velocity at the surface of the bob is:

\[
V_{sb} = R_b (\omega_s - \omega_r) \quad \ldots \quad (5.21)
\]

where \(\omega\) is the rotation speed at a given shear stress and \(s\) and \(r\) indicate smooth and rough surfaces.

**Method B** is based on the use of a smooth bob, a smooth cup and a rough cup, the smooth and rough cups are of equal diameter. The final expression for slip velocity at the surface of the bob is:

\[
V_{sb} = R_c (\omega_s - \omega_r) \quad \ldots \quad (5.22)
\]

**ii) Mannheimer (1983)**

Assuming that slip is a function of shear stress, Mannheimer derived an expression for the slip velocity in the co-axial cylinder viscometer by analysing the data obtained with two different gap widths \(H_1\) and \(H_2\). Mannheimer's expression is:

\[
V_s = \left[ \dot{\gamma}_{a1} - \dot{\gamma}_{a2} \right] \frac{(H_1 H_2)}{(H_1 - H_2)} \quad \ldots \quad (5.23)
\]

where \(\dot{\gamma}_{a1}\) and \(\dot{\gamma}_{a2}\) are the apparent shear rate at a given shear stress in \(H_1\) and \(H_2\) respectively.
5.4: Correction of the Fann Viscometer Readings to Allow for the Presence of Slip

5.4.1: Experimental investigation

5.4.1.1 Procedure

All apparatus, standard proportions and standard procedures referred to below are described in Chapter 4.

Approximately 600 ml. of material was mixed, agitated and was then transferred to the Fann viscometer cup. Thereafter a down curve was obtained according to the standard procedure.

5.4.1.2: Results and Discussion

The Cheng and Parker (1976) theory was used to investigate the presence of slippage. The results between five and seven tests performed with bob/rotor combinations 1, 2, 3 and 4, see Table 5.1, are summarised in Table 5.2 and plotted in Figure 5.3. This data shows that there is no statistical difference above zero r.p.m. even at 80% confidence level using the student's 't' test between the various bob/rotor combinations.

The implication from the statistics is either that the slip layer effect is very small and the accuracy of the experimental technique is not sufficient to detect it, or that the slip layer is still relatively large even with the roughened cylinders and similar slip occurs in all bob/rotor combinations. Regarding experimental accuracy, the relatively low standard deviation (Table 5.2) imply excellent control procedure.

The results presented above are insufficient to draw a decisive conclusion regarding slip effects. Consequently thirty two tests (Table 5.3) were carried out using bob/rotor combinations 1, 5, 6 and 7 described in Table 5.1. The experimental procedure used was the same as that described above except that the top shear rate was 511 s⁻¹.
### Table 5.1: Details of the Various Combinations of Cylinders Used with the Fann Viscometer

<table>
<thead>
<tr>
<th>Bob/Rotor Combination Number</th>
<th>Radius mm</th>
<th>Surface Nature</th>
<th>Gap width mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 bob</td>
<td>17.25</td>
<td>smooth</td>
<td>1.17</td>
</tr>
<tr>
<td>cup</td>
<td>18.42</td>
<td>smooth</td>
<td></td>
</tr>
<tr>
<td>2 bob</td>
<td>17.25</td>
<td>smooth</td>
<td>1.17</td>
</tr>
<tr>
<td>cup</td>
<td>18.42</td>
<td>serrated</td>
<td>0.1/2 *</td>
</tr>
<tr>
<td>3 bob</td>
<td>17.25</td>
<td>serrated</td>
<td>0.1/2</td>
</tr>
<tr>
<td>cup</td>
<td>18.42</td>
<td>smooth</td>
<td>1.17</td>
</tr>
<tr>
<td>4 bob</td>
<td>17.25</td>
<td>serrated</td>
<td>0.1/2</td>
</tr>
<tr>
<td>cup</td>
<td>18.42</td>
<td>serrated</td>
<td>0.1/2</td>
</tr>
<tr>
<td>5 bob</td>
<td>17.25</td>
<td>serrated</td>
<td>1 /1</td>
</tr>
<tr>
<td>cup</td>
<td>18.42</td>
<td>serrated</td>
<td>1 /1</td>
</tr>
<tr>
<td>6 bob</td>
<td>17.25</td>
<td>smooth</td>
<td>0.754</td>
</tr>
<tr>
<td>cup</td>
<td>18.004</td>
<td>smooth</td>
<td></td>
</tr>
<tr>
<td>7 bob</td>
<td>17.25</td>
<td>smooth</td>
<td>2.616</td>
</tr>
<tr>
<td>cup</td>
<td>19.866</td>
<td>smooth</td>
<td></td>
</tr>
</tbody>
</table>

* 0.1/2 indicates a serration depth of 0.1mm and spacing of 2mm.*
Table 5.2: Results of Fann Viscometer Tests Using 1.17mm. Gap Width

<table>
<thead>
<tr>
<th>Bob/Rotor Combination Number</th>
<th>Smooth Bob/Smooth Rotor</th>
<th>Rough Rotor 1433</th>
<th>Rough Rotor</th>
<th>Rough Rotor</th>
<th>Smooth Bob</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotation Speed (r.p.m.)</td>
<td>Shear stress at surface of the bob (Pa)</td>
<td>mean S.D.</td>
<td>mean S.D.</td>
<td>mean S.D.</td>
<td>mean S.D.</td>
</tr>
<tr>
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<tr>
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<td></td>
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</tbody>
</table>

Number of Readings: 7 7 5 6

S.D. = Standard Deviation
RATE FOR FOUR COMBINATIONS OF ROUGH AND SMOOTH CYLINDERS

FIGURE 5.3 : SHEAR STRESS AT THE SURFACE OF THE BOB AGAINST APPARENT SHEAR

HOTATION SPEED (R.p.m.)

0 100 200 300 400 500 600 700

APPEARANT SHEAR RATE (1/s)

0 170 340 510 680 850 1020 1190

Each point represents the mean of a minimum of five results.

Combination

\[
\begin{align*}
0 \\
\square \text{Combination 2} \\
\bigtriangleup \text{Combination 4} \\
\times \text{Combination 1}
\end{align*}
\]

SHEAR STRESS AT THE SURFACE OF THE BOB (Pa)
Table 5.3: Results Obtained with the Fann Viscometer.

Each result is the mean of 8 readings for each rotation speed.

<table>
<thead>
<tr>
<th>Bob/Rotor Combination Number</th>
<th>Rotation Speed r.p.m.</th>
<th>Average Apparent Shear Rate S⁻¹</th>
<th>Average Shear Stress at Bob Surface (Pa)</th>
<th>Average Shear Stress in Gap (Pa)</th>
<th>Standard Deviation</th>
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<tr>
<td></td>
<td>200</td>
<td>511</td>
<td>96.3</td>
<td>92.3</td>
<td>7.32</td>
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<td>255.5</td>
<td>75.7</td>
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<td>6</td>
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<td></td>
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<td>96.5</td>
<td>90.6</td>
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<td></td>
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<td>340.7</td>
<td>81.6</td>
<td>76.7</td>
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<td>66.7</td>
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<td>0</td>
<td>20.8</td>
<td>19.5</td>
<td>2.36</td>
</tr>
</tbody>
</table>
CHAPTER 5

The surface roughness used with combination 5 is significantly more than that used previously with combinations 2, 3, and 4. The aim of this was to find out whether or not the increase in surface roughness would reduce the slip velocity. Moreover, further assessment of slip velocity can be achieved using the Mannheimer method, described in section 5.3.1, from the data obtained with bob/rotor combinations 1, 6 and 7. The results from this investigation are described below.

5.4.1.3: Effects of Roughening the Cylinders

Figure 5.4 shows a plot of the shear stress on the surface of the bob versus the apparent average shear rate in the gap of bob/rotor combination 5 (serrated cylinders, 1.17mm gap width) and combination 1 (smooth cylinders, 1.17 gap width). At high apparent shear rates, (larger than 170 S⁻¹) it can be seen from Figure 5.4 that the difference between the shear stresses obtained with the serrated cylinders and those obtained with the smooth cylinders is nil or very small. At low apparent shear rates (less than 170 S⁻¹), however, the curve corresponding to the serrated cylinders starts to deviate from that obtained using the smooth cylinders, as the apparent shear rate is reduced. The difference between the shear stresses is statistically significant as can be seen in Figure 5.5 which is an enlarged view of part of Figure 5.4. Comparing these results with the previous investigation (Figure 5.3), it can be seen that the increase in surface roughness seems to reduce the slip effect and increase the yield stress. Hence, one may conclude that a serration depth of 0.1mm on the surface of the cylinders may not totally eliminate the slippage with the co-axial cylinder arrangement. Consequently, Cheng and Parker's (1976) assumption that slippage does not occur with the roughened cylinders will depend on the roughness chosen which sheds some doubt on the method they derived to calculate slip velocities.

5.4.1.4: Effects of Varying the Gap Widths

Figure 5.6 shows a plot of the measured shear stress at the surface of the bob versus apparent average shear rate in the gap for bob/rotor combinations 1, 6 and 7 (see Table 5.1). As can be seen from Figure 5.6 the results obtained with 0.745mm gap width (combination 6) and
Figure 5.4: Shear stress at the surface of the bob against apparent shear rate (1/s)

Shear stress at the surface of the bob (Pa)

Apparent shear rate (1/s)

Combination 5
Combination 1

Rate for two combinations of rough and smooth cylinders.
Figure 5.5: Shear Stress at the Surface of the Bob Against Apparent Shear Rate

Bars indicate 95% confidence limits.
Figure 5.6: Shear Stress at the Surface of the Bob Against Apparent Shear Rate (1/s)

Shear Stress at the Surface of the Bob (Pa)

Apparent Shear Rate (1/s)

- Combination 1
- Combination 2
- Combination 3
1.17mm gap width (combination 1) yielded almost the same curve whilst the curve obtained using the 2.616mm gap width (combination 7) deviated considerably from the other two curves. This deviation can be partly attributed to the use of the shear stress at the surface of the bob (provided in the manufacturer's literature) instead of the more representative average shear stress.

Figure 5.7 shows a plot of the mean values of the average shear stress versus the apparent average shear rate which shows that the inconsistency between the results of the various combinations has been somewhat reduced. However at low shear rates the mean value of the average shear stresses, at a given average shear rate, are still statistically different, as can be seen from Figure 5.8. This difference can be related to plug flow, with or without slippage on the surface of the cylinders.

The implication of plug flow is that the curves of shear stress at the surface of the bob versus the rotation speed (Figure 5.9) will be independent of the radius of the outer cylinder. Moreover, the curves start to diverge only when the plug flow in the annulus of one or all the combinations is totally eliminated. Figure 5.9 shows a plot of shear stress at the surface of the bob versus rotation speed which shows, as predicted by the plug flow hypothesis, that shear stress measured at the surface of the bob is independent of the outer cylinder radius at low rotation speeds. The results at low rotation speeds presented in Figure 5.9 are re-plotted with an expanded rotation speed axis in Figure 5.10. From Figure 5.9 and Figure 5.10 it can be seen that one line can satisfactorily fit the data measured with the three gap widths up to rotation speed of 30 r.p.m. and up to rotation speed of 200 r.p.m. for 1.17 and 2.616mm gap widths. Consequently, one may deduce that plug flow has occurred in the annuli of the 0.745 and 1.17mm gap widths up to rotation speeds of 30 and 200 r.p.m. respectively. From the cross-over point on Figure 5.7 it can be deduced that plug flow is eliminated in the 2.616mm gap at an apparent average shear rate of about 400 S\(^{-1}\) i.e. a rotation speed which lies between 300 to 600 r.p.m. It follows from the discussion of the results shown in Figure 5.7 that satisfactory correlation between different gap widths is obtained when plug flow is totally eliminated in the annuli. Consequently, applying the Mannheimer
Figure 5.7: Average shear stress in the gap against apparent shear rate for three combinations of smooth cylinders.
AVERAGE SHEAR STRESS IN THE GAP (Pa)

Figure 5.8: Average shear stress in the gap against apparent shear rate for three combinations of smooth cylinders.

Bars indicate 95% confidence limits:
- Combination 1
- Combination 6
- Combination 7

Rate for three combinations of smooth cylinders.
Figure 5.9: Shear stress at the surface of the bob against rotation speed.

Shear stress at the surface of the bob (Pa)

Rotation speed (r.p.m.)

Combination 1
Combination 2
Combination 3
Combination 4
Combination 5
Combination 6
FOR THREE COMBINATIONS OF SMOOTH CYLINDERS

FIGURE 5.10: SHEAR STRESS AT THE SURFACE OF THE BOB AGAINST ROTATION SPEED

SHEAR STRESS AT THE SURFACE OF THE BOB
(\( \text{Pa} \))

ROTATION SPEED (r.p.m.)

0 200 400 600 800 1000

Combination 1
Combination 6
Combination 7
(1983) method, discussed in section 5.3.1, one may conclude that when plug flow is eliminated, slippage at the surfaces of the smooth cylinders is non-existant or its effects are negligible. This finding is supported by the results presented in section 5.4.1.3 and displayed in Figure 5.4, since only at high shear rate, where plug flow is expected to vanish, the shear stresses measured with the smooth cylinders were closely matched with those measured with the roughened cylinders.

5.4.1.5: Yield stress

Figure 5.4 and Figure 5.7 show that at high shear rates, where plug flow is eliminated, the material appears to behave in accordance with the Bingham flow model, the yield stress and plastic viscosity of which are 50.0 Pa and 0.081 Pa S respectively. A comparison can be made between this yield stress and those obtained using the stress relaxation method, i.e. the shear stresses measured at the zero rotation speeds on the surfaces of the smooth and roughened cylinders listed in Table 5.3 and reproduced in Table 5.4. This table shows a marked difference between the yield stress measured on the surface of the smooth bobs, rough bob and extrapolated Bingham yield stress. The difference between the yield stresses measured with the smooth cylinders and roughened cylinders can simply be related to the slippage on the surfaces of the smooth cylinders. On the other hand, the difference between the yield stress measured with the roughened cylinders and the Bingham yield stress can be attributed to one of two reasons. Firstly, slippage could still occur with the roughened cylinders, hence, resulting in a lower yield stress than the real value. Secondly, the material does not obey the Bingham model at low shear rates, so that the extrapolated Bingham yield stress may not reflect the true yield stress of the material.

A further assessment of the yield stress can be obtained from the measured shear stress at the point where plug flow is eliminated ($\tau_p$) in a given geometry. From Equation 5.5, section 5.1.2, it can be shown that the yield stress ($\tau_o$) is given by:

$$\tau_o = R_b^2 \frac{\tau_p}{R_c^2} \quad ...(5.24)$$
The shear stresses at which plug flow vanishes in the three annuli used (0.754, 1.17 and 2.616 mm gap width) can be found from Figure 5.9 using the corresponding rotation speeds deduced in section 5.4.1.4. The resulting yield stresses are listed in Table 5.4. It must be stressed that the rotation speed at which $\tau_p$ were measured are ill defined, hence, the yield stresses calculated from Equation 5.2.4 are not exact. Nonetheless, these yield stresses show the following: Firstly, their magnitude is closer to the extrapolated Bingham yield stress than the yield stresses determined with the stress relaxation methods, hence suggesting that slippage occurred even with the roughened cylinders at low rotation speeds. Secondly, the yield stress appears to increase with the increase in gap width which is not possible since the same material was used with all gap widths.

Table 5.4: Yield Stress Obtained with Various Methods

<table>
<thead>
<tr>
<th>Gap width (mm)</th>
<th>0.754</th>
<th>1.17</th>
<th>2.616</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Extrapolated Bingham model</td>
<td>50</td>
<td>50</td>
<td>-</td>
</tr>
<tr>
<td>Stress relaxation method - smooth cylinders</td>
<td>2.4</td>
<td>2.6</td>
<td>2.0</td>
</tr>
<tr>
<td>Stress relaxation method - roughened cylinders</td>
<td>-</td>
<td>20.8</td>
<td>-</td>
</tr>
<tr>
<td>Equation 5.24</td>
<td>44.5</td>
<td>58.3</td>
<td>67.4 - 72.6</td>
</tr>
</tbody>
</table>
Consequently, one may conclude that the shear stress at which plug flow was eliminated was dependent on factors other than the yield stress and cylinders radii such as gap width. A possible explanation for this is that in small gap widths the plug is thin and breaks at relatively small shear stress. However, further work is required to substantiate this conclusion.

5.5: Measurement of the Rheological Behaviour of the Material used in this Thesis

5.5.1: Preliminary work

As a starting point the API (1982) standard procedure for the determination of the rheological properties of cement slurries described in Chapter 4, was adopted to obtain the flow curve for the material used in this work. Figure 5.3 shows a typical flow curve obtained using this procedure. From this figure it appears that the Bingham flow model can satisfactorily fit the results at high shear rates. The power law model, as shown in Figure 5.11, does not give a much better fit over the whole range of shear rates. According to the API (1982) specification, the resulting Bingham, or power low curves can then be used to predict the pressure drop in pipes or annuli. However, further investigation into the material rheological behaviour revealed, as will be shown later, that the flow curves obtained above are only two curves amongst a large number of curves which the material may exhibit depending on the experimental procedure and slurry age. This behaviour is hardly surprising with a material which appears to be thixotropic in nature. This is demonstrated by the material's ability to become self supporting within few minutes of standing time and, only a gentle agitation is needed to transform the material back to a runny fluid. However, it was initially thought that the 60 seconds pre-conditioning time at the top shear rate of $1022 \text{ S}^{-1}$ (Chapter 4) would break this thixotropic structure. Hence, the equilibrium flow curve should have been obtained whatever the duration and the experimental technique of the down curve cycle. This assumes a negligible structural build up due to hydration reaction during the short time of the experiment.

To clarify this point, two tests were carried out at a constant shear
FIGURE 5.11: LOGARITHMIC PLOT OF SHEAR STRESS AGAINST APPARENT SHEAR RATE

LOG SHEAR STRESS (Pa)

LOG APPARENT SHEAR RATE (1/s)

(1) OBSERVED WITH SMOOTH CYLINDERS (COMBINATION 1)
rate of 511 S\(^{-1}\) (300 r.p.m.), the results are shown in Figure 5.12. In both tests, the material was preconditioned at a shear rate of 1022 S\(^{-1}\) for 60 seconds. In the first test, the shear rate was reduced suddenly to shear rate of 511 S\(^{-1}\), whilst in the second test the sample was allowed to rest for 10 seconds before the 511 S\(^{-1}\) shear rate was applied. The sudden step change in shear rate employed with the first test resembles the API standard test, used above. Figure 5.12, test 1, shows that no equilibrium shear stress was reached even after 11 minutes of shearing. Similar results were displayed by the material in test 2 after a short time needed to break the structure which appears to have formed during the course of 10 seconds standing time. These results clearly illustrate why different flow curves were obtained with different shearing cycles. This behaviour of the material was investigated further below.

5.5.2: Measurement of Structural Change under the Condition of Constant Shear Rate

5.5.2.1: Fann Viscometer

It has been shown above that when the material used in this work was sheared at constant shear rate, the recorded shear stress at the surface of the bob increased to the end of the experiment, after an initial rapid drop, without any tendency to reach an equilibrium value. With a material which behaves in such a manner, the standard type of shear stress-shear rate flow curve is almost valueless since it is highly dependent on the type and duration of the shear cycle. It was decided that it would be much more productive to study the material behaviour under the condition of constant shear rate. To that end tests were performed at constant apparent average shear rates of 170 S\(^{-1}\) (100 r.p.m.) (3 tests), 511 S\(^{-1}\) (300 r.p.m.) (2 tests) and 1022 S\(^{-1}\) (600 r.p.m.) (3 tests) using the Fann viscometer with cylinder combination 1, see Table 5.1.

The samples were prepared using the standard mixing and agitation procedure described in Chapter 4. After agitation in the atmospheric consistometer, the sample was preconditioned at an apparent shear rate of 1022 S\(^{-1}\) for 60 seconds, during which time the shear stress was not recorded. Thereafter, the sample was sheared at the specified constant apparent shear rate for one hour with the shear stress being
USING THE FANN VISCOMETER AT RATE OF SHEAR OF 511.1/S
figure 5.4.2: shear stress against shearing time obtained

shearing time (min)

shear stress (Pa)

Test number 2

Test number 1

X X

X X

X X

X X
recorded at time intervals of 0.25 minutes and 4 minutes depending on the rate of change of shear stress. The results are shown in Figure 5.13, 5.14, 5.15. These results are not very consistent but, nonetheless generally show a continuous increase in shear stress until the end of the experiment. This general trend is overlaid by apparently random breakdown and rapid build up for short periods. Furthermore there exists no value of shear stress which can be considered as the equilibrium shear stress. This indicates that the material is not thixotropic as commonly defined, but rather more complex with some thixotropic properties, viz., reduction in the apparent viscosity with shearing after rest followed by a build up in structure due to chemical reaction.

The drawbacks of the tests presented above are: firstly, the material behaviour may have been influenced by the viscous heating produced by the prolonged shearing. Secondly, the 20 minutes agitation prevents assessment of the material behaviour in this important period. Consequently the following tests were performed to overcome these shortcomings.

5.5.2.2: Haake Viscometer

The Haake viscometer (Chapter 4), used for this investigation was provided with a temperature controlled water bath and a chart recorder for continuous torque (shear stress) measurements. The MVSt cup and MVISt bob (rotor) were used in all tests. These have diameters of 42mm and 40.08mm respectively giving a gap width of 0.96mm. The samples were prepared using the mixing procedure described above. Straight after mixing, the viscometer cup was loaded with material which was then tested at constant apparent shear rate of 511 and 1022 S\(^{-1}\) with the shear stress being recorded continuously using the chart recorder. Water at a constant temperature of 20°C was kept circulating around the cup (outer cylinder) to reduce or eliminate the temperature rise produced by the prolonged shearing. To prevent or reduce evaporation the top of the sample was covered throughout the test. Relatively high apparent shear rates were employed to prevent the formation of plug flow and while it is difficult to check the realism of this assumption, a tactile examination of the sample at the end of the test indicated a uniform consistency across the annulus.
Using the Fann Visccometer at a Rate of Shear of 170 r/min
Figure 5.13: Shear Stress against Shearing Time Obtained

Shearing Time (min)

Shear Stress (Pa)
Figure 5.15: Shear Stress against Shearing Time Obtained Using the Fann Viscometer at Rate of Shear of 1022 1/s
Hence, it may be said that plug flow probably did not take place under conditions of the experiment. The results from eight tests are shown in Figures 5.16 and 5.17. As can be seen from these figures the shear stress has generally increased, after an initial rapid drop, continuously to the end of the experiment (maximum of 3 hours) without any tendency to reach an equilibrium value. The rate of increase in shear stress is greatest in the first 30 minutes of the slurry life. These results indicate that the increase in shear stress is a property of the material and is not caused by the viscous heating.

Several further tests were performed with the Haake viscometer using exactly the same procedure as described above, except that a down curve was run after 9 minutes of shearing and every 7 minutes thereafter. These down curves were obtained by linearly reducing the apparent shear rate from the top value of 1022 S\(^{-1}\) to zero in one minute. Thereafter, the apparent shear rate was restored to its original top value by linearly increasing the shear rate in one minute. Figure 5.18 shows the Bingham model used on a typical down curve obtained after 51 minutes of shearing. The yield stresses and plastic viscosities obtained every 7 minutes from similar curves are illustrated in Figure 5.19. This figure shows that in the first 30 minutes of slurry life both the yield stress and plastic viscosity experienced a rapid increase in magnitude. Subsequently, the plastic viscosity showed little variation with time, whilst the yield stress continued to increase until the end of the experiment.

5.5.2.3: Low Shear Rates Experiments

As noted above, relatively high shear rates were used in the previous investigation in order to avoid the occurrence of plug flow. Plug flow where the outer segment of the material in the annulus remains intact, i.e. without shearing taking place, is a property of the co-axial cylinder viscometer at low rotation speeds with viscoplastic materials such as most cement slurries. Hence it is difficult to produce meaningful results at low shear rates with the co-axial cylinder arrangement. Instead the atmospheric consistometer and the Mono pump with a tapered tube arrangement shown in Figure 5.20 were used to study the material behaviour in the low shear rate region. A
FIGURE 5.16: SHEAR STRESS AGAINST SHEARING TIME OBTAINED USING THE HAAKE VISCOMETER AT RATE OF SHEAR OF 511 1/S.

Test number 4
Test number 3
Test number 2
Test number 1

SHEAR STRESS (Pa)

SHEARING TIME (min)
Fig. 5.17: Shear Stress against Shearing Time Obtained Using the Haake Viscosimeter at a Rate of Shear of 1022 1/s
FIGURE 5.18: TYPICAL SHEAR STRESS - SHEAR RATE CURVE OBTAINED WITH THE HAHE VISCOMETER AFTER 5 MINUTES OF SHEARING.

APPEARANT SHEAR RATE (1/s) vs. SHEAR STRESS (Pa)
FIGURE 5.19: CHANGE IN YIELD STRESS AND PLASTIC VISCOSITY WITH TIME

![Graph showing the change in yield stress and plastic viscosity with time. The graph includes data points for yield stress and plastic viscosity over a range of shear time (min).]
full description of the atmospheric consistometer and the Mono pump is given in Chapter 4.

Unfortunately, unlike the co-axial cylinder viscometer, the flow field generated and the shear rate produced in these geometries are not very well defined and almost certainly vary from place to place. Hence, it is difficult, or could be impossible, to calculate the exact shear rate with these geometries. However, a tentative calculation of the rate of shear shows that under the flow and rotation speeds used, the average or equivalent shear rate generated was typically less than 20 s$^{-1}$.

Figure 5.20: Sketch of the Mono Pump-Tapered Tube Pumping Loop

Flow rate = 48 ml/s

Pressure transducer

Mono pump feed pipe

Mono pump
5.5.2.3.1: Experimental Procedure

Approximately 2 litres of material was mixed according to the standard procedure given in Chapter 4. The slurry was then de-aired in a vacuum flask for 5 minutes before transferring to the consistometer container and the Mono pump hopper. The pressure to maintain flow in the tapered tube section was recorded continuously using the Bryans xy/t recorder. Simultaneously, the torque (in Bc units, see chapter 4) exerted on the shaft of the consistometer paddle was recorded at time intervals between 3 and 10 minutes depending on the rate of change of torque.

Two tests were performed with the consistometer and with the tapered tube, the results from these tests being virtually identical for each test set up. Figure 5.21 displays the results from one set of tests.

5.5.2.3.2: Results

Figure 5.21 illustrates the variation in the material consistency with time. Apart from a dormant period (between 25 and 70 minutes) the material has shown, similar to the high rate of shear experiments, an increase in consistency with time, with the rate of increase being highest in the first 30 minutes of the slurry life. The most noticeable feature of the results shown in Figure 5.21 is that both geometries appear to reflect the same variation in material consistency which suggests a similar shearing action in both geometries.

5.5.2.4: Static or no Shear Experiments

To complete the assessment of rheological properties, the material behaviour was investigated under static, i.e. no shear, conditions. This was achieved by means of the shear vane test, described in Chapter 8.

5.5.2.4.1: Experimental Procedure

Approximately 600 ml of material was mixed according to the standard mixing procedure described in Chapter 4. In the first series of tests
(fresh sample) the material was then transferred to the Weissenberg Rheogoniometer cup (C2, see Table 8.2, Chapter 8) and the vane (V4, see Table 8.2) was immersed 13mm below the surface. Thereafter, the material was allowed to stand for a given time after which it was tested at a rotation speed of 0.6 r.p.m.

In the second series of tests agitated samples were used. The procedure used to prepare and test the sample was identical to that used with the fresh samples except that the material was agitated after mixing for 20 minutes in the atmospheric consistometer.

5.5.2.4.2: Results

Figure 5.22 shows the results from eighteen and fifteen test obtained with the fresh and agitated samples respectively. From this figure it appears that some differences exist between the gel strength measured with the fresh and agitated samples, which perhaps reflects the difference in experimental procedure and slurry age. The results displayed here illustrate the spectacular behaviour of the material where gel strength has increased from few Pa to thousands of Pa in relatively short time. This dramatic increase in consistency under the static conditions was not matched by the dynamic, i.e. continuous shear, conditions, even when the shear rates were of small magnitude, as seen in section 5.5.2.3.

5.5.3: Summary

All the results obtained in the various investigations described above clearly illustrate the strong time dependent behaviour of the material. These results also indicate that the rate of change in the material consistency is dependent on time, shear rate and the experimental procedures. The time dependent behaviour exhibited by the material is much more complex than that associated with normal thixotropic and rheopectic materials. This is because the change in the material consistency is not caused only by shearing, as is the case with thixotropic and rheopectic fluids, but, is also caused by the continuous change in the slurry constituents and the forces associated with them, which are brought about by the hydration of the cement and the precipitation of the calcium sulphate hemihydrate. The
FIGURE 6.22: VARIATION IN GEL STRENGTH WITH TIME
implies that any shear stress-shear rate flow model obtained for the material is almost valueless for predictive purposes of flow behaviour of the material in tubes and other geometries. For such models to be of value other factors such as time and material pre-conditioning procedures have to be included.

A number of equations exist in literature (Chapter One) to describe the time dependent behaviour of the thixotropic and rheopectic materials and such equations have been used by some authors, such as Lapasin et al (1980) and (1983b), to describe the flow behaviour of cement slurries. Nevertheless, these equations cannot be used with our material and much more complex equations are needed. Moreover, the strong dependence of the material on the pre-conditioning procedure makes any empirically established equation suitable for predictive purposes in pipelines only if the conditions of the test are closely matched in the field.

5.6: Conclusions
5.6.1: Conclusions Related to the Co-axial Cylinder Viscometer

1) The co-axial cylinder viscometer can be a very useful instrument to assess the flow behaviour of cement slurries. However, misleading results may be obtained if plug flow and phase separation of the material are ignored.

2) Slippage on the surfaces of the cylinders may not be totally eliminated by the use of roughened cylinders.

3) Rotation speed at which plug flow is eliminated in the annulus cannot easily be predicted by theory. Hence a minimum of two gap widths should always be used, and the results should be accepted only if the results from both gap widths are in agreement.

5.6.2: Conclusions Related to the Material

1) With complex materials such as the slurry used in this work, the API (1982 and 1986) standard procedures, for the determination of the rheological properties of cement slurries, are too simplistic and may lead to misleading results.
CHAPTER 5

2) The material resistance to flow increases with time under shearing or when at rest. Nonetheless, an initial rapid reduction in the material resistance to flow can be seen if the material is sheared after some rest.

3) The rate of increase or reduction in the materials resistance to flow is dependent on many factors, such as shear rate, time, slurry age and experimental procedures.

4) A general equation to describe the flow behaviour of the material has proven to be difficult and could be impossible to establish.
6.1: Introduction

In the cementing operation, the cement slurry travels down the casing and up the annulus. Hence accurate determination of flow behaviour of the material in pipes and annuli is necessary for a proper cementing job. It is equally necessary for the engineer to be able to predict the pressure to start flow in case of a shutdown.

Dense suspensions are very complex materials, they include all forms of non-Newtonian fluids such as shear thinning, shear thickening, viscoplastic and thixotropic. Slippage at the smooth surfaces of equipment in contact with the material often adds to the complication when attempting to predict the flow behaviour of the material in pipes and other geometries. A wealth of theories and methods have been developed to predict the flow behaviour of non-Newtonian materials in pipes, but most, if not all of these theories and methods treat the material as a stable system which obeys a unique flow model whatever the conditions or length of the pumping operation. Cement slurries, particularly the mix used in this work, often exhibit a variety of flow models, see Chapter 5. Therefore, special attention must be paid to such systems.

6.2: Classification of Slurries

Solid particles in a fluid have the tendency to settle under the action of gravity. In designing for pipe flow of suspensions it is important to distinguish between settling and non-settling systems or as some authors prefer (Wasp et al. (1977)) homogeneous or heterogeneous systems. The settling and non-settling terminology is preferred here since, although the slurry may be homogeneous or semi homogeneous, a phase separation may occur in the vicinity of the tube wall.

The velocity of settlement of the solid particles in the fluid is dependent on numerous factors such as fluid viscosity, particle density, electrical forces and the rheological properties of the
suspending fluid. These factors and their influences on the settling velocity have been reviewed by Wasp et al (1977) and in more details by Govier and Aziz (1972).

6.2.1: Settlement of Single Particles

For the simplest case of a spherical particle settling in a Newtonian fluid under the influence of the gravitational force, the settling velocity (Vst) of the particle may be calculated from Equation 6.1, 6.2 and 6.3 taken from Govier and Aziz (1972).

For laminar motion, \( \frac{d \text{Vst} \rho_s}{\mu} < 1 \)

\[
Vst = \frac{g(\rho_p - \rho_s) \, d^2}{18 \mu} \quad \ldots(6.1)
\]

For Transition motion, \( 1 < \frac{d \text{Vst} \rho_s}{\mu} < 1000 \)

\[
Vst = 0.2 \left[ \frac{g ( \rho_p - \rho_s )}{\rho_s} \right]^{0.72} \left( \frac{d}{\mu/\rho_s} \right)^{0.45} \quad \ldots(6.2)
\]

For Turbulent motion, \( \frac{d \text{Vst} \rho_s}{\mu} > 800 \)

\[
Vst = 1.74 \left[ \frac{g ( \rho_p - \rho_s )}{\rho_s} \right]^{0.5} \, d^{0.5} \quad \ldots(6.3)
\]

For a particle of irregular shape the settling velocity will be smaller than that given by the above equations. Hence the settling velocities given by Equation 6.1 - 6.3 have to be modified by a factor depending on the degree of irregularity. Additionally, the tube wall will further influence the settling velocity of the particle depending on the magnitude of \( d/D \) where \( d \) and \( D \) are the particle diameter and the tube diameter respectively.
6.2.2: Settlement of Aggregate of Particles

In the above discussion the concentration of solid particles has been assumed to be small so that the particles do not interact hydrodynamically or as a colloid. However, as the concentration is increased the hydrodynamic and other interactions will increase further, which may increase or reduce the settling velocity depending whether or not the particles aggregate. An aggregate may contain a number of particles and in addition occluded fluid within the particle cluster. These clusters have a settling velocity much different from the velocity of a single particle, and the velocity will depend on the resultant size, shape and effective density of the cluster. Suspensions of mixed size particles will settle at different speeds leading to particle collision which in turn leads to increase or reduction in the velocity of the colliding particle. Hence if the concentration of particles is high so that particle collisions and interactions are common, the particle will settle in bulk at a constant speed termed in literature as hindered settling velocity. The hindered settling velocity of a particle is much lower than the free settling velocity of the same particles.

Hindered settlement is usually encountered with most, if not all, the practical mix proportions of cement slurries because the interaction forces are comparable with the settling gravitational forces. This results from the small size of the cement particles where approximately 90% of the particles are smaller than 63 microns, see Table 4.3, and the particle size varies between 2 and 100 microns. Under these conditions the empirical and theoretical equations developed to predict the hindered settlement of particles will not be adequate because only the viscosity of the medium, and particle size, shape, density and concentration are included. The additional factors influencing the hindered settlement velocity of cement particles are many and complex and include all the factors affecting suspension rheology discussed in Chapter 2 and consequently it may be said that no single empirical or theoretical equation can adequately predict the hindered settling velocity of cement slurries, hence, one has to rely on experiment to measure the settling velocity of cement slurry in any given practical situation.
The method used by Raffle (1970) could be useful in estimating the settling velocity of cement slurries. He measured the hindered settling velocity for cement slurries of W/C ratio varying from 0.35 to 0.65. The measured hindered settling velocity has varied between 0.008 to 0.01 cm/min. It must be stressed that the above measured velocities are only valid under the specific conditions of the test.

For the slurry described in this thesis where the W/C ratio used was 0.65 plus additives, see Table 4.1, no settlement was noticed when the material was allowed to stand in a cylinder for a few hours after agitating for say 10 minutes to condition the slurry after mixing. Bleed water depth of less than a millimetre was observed even without conditioning after mixing.

Obviously the settling velocity will be different under flow conditions and will depend on the type and rate of flow. Wasp et al (1970) measured the concentration gradient of coal suspension flowing in horizontal pipes. He recorded little diameter effect and little or no settlement for particle sizes of less than 800 microns. For the cement slurry used in this work one could deduce that little or no settlement will take place and the material can be taken as non-settling material particularly with vertical flow where the effects of small particle settlement will be smaller than with horizontal flow.

6.3: Classification of Flow

Newtonian fluids flowing in pipes may do so in laminar conditions where the material flows in stream lines along the pipe axis or in turbulent conditions which is characterised by fluctuating components of velocity in all directions depending on the value of Reynold's number (Re). For Newtonian fluids Re is given by:

\[ Re = \frac{\rho Vm D}{\mu} \]  

...(6.4)

The critical value of Re, where the transition from laminar to turbulent flow occurs is usually taken as 2300 to 3000, Wasp et al (1977). Strictly speaking the value of the critical Re is not very well defined and much higher or lower values can be encountered.
For non-Newtonian fluids the transition from laminar to turbulent flow is also governed by Re. Re, defined in Equation 6.4, however, has to be modified to accommodate for the changing viscosity term. Various expressions for the prediction of transition velocity in non-Newtonian fluids have been proposed in literature, such as those of Hedstrom (1952) and Hanks (1963). However, since only laminar flow was present in this investigation, these will not be discussed any further.

6.4: Laminar Flow of Suspensions in Pipes
6.4.1: Theory

For time independent fluid flowing in a pipe the rheological equation relating shear stress to shear rate can be written in a general form as:

\[ \frac{dv}{dr} = f(\tau) \]  ....(6.5)

Where \( \tau \) is the shear stress at radius \( r \). Resolving the forces acting on a cylindrical element of radius \( r \) as shown in Figure 6.1 yields:

\[ 2\pi r \Delta \tau = \pi r^2 \Delta p \]

or \( \tau = \frac{(r \Delta p)}{(2\ell)} \)  ....(6.6)

at the wall of the tube \( \tau = \tau_w \) and \( r = D/2 \), then

\[ \tau_w = \frac{(D \Delta p)}{(4\ell)} \]  ....(6.7)

From Equation 6.6 and 6.7

\[ \tau = \frac{(2\tau_w r)}{D} \]  ....(6.8)
The flow rate of fluid flowing in a pipe can be obtained by the general equation:

$$Q = \int_{0}^{\frac{D}{2}} 2\pi rv \, dr$$

Integrating Equation 6.9 by parts yields:

$$Q = \pi \left[ r^2 v - \int_{0}^{\frac{D}{2}} r^2 \frac{dv}{dr} \, dr \right]$$

For the condition of no slip at the wall of the tube, Equation 6.10 reduces to:
Substituting Equation 6.5 in 6.11

\[ Q = \pi \int_0^{D/2} r^2 f(r) \, dr \]  

...(6.12)

From Equation 6.8

\[ r = \frac{D \tau}{2 \tau_w} \]  

...(6.13)

and

\[ dr = \frac{d\tau \, D}{2 \tau_w} \]  

...(6.14)

Substituting Equation 6.13 and 6.14 in 6.12 gives:

\[ Q = \left( \pi \frac{D^3}{(8 \tau_w^3)} \right) \int_0^{D/2} \tau^2 f(\tau) \, d\tau \]  

...(6.15)

This is a general equation which is valid for any time independent fluids which display no slip at the surface of the tube.

For Newtonian fluids

\[ f(\tau) = \frac{\tau}{\mu} \]  

...(6.16)

Putting Equation 6.16 in 6.15 gives:

\[ Q = \left( \pi \frac{D^3 \tau_w}{32 \mu} \right) \]  

...(6.17)

substituting Equation 6.7 in 6.17 gives:

\[ Q = \left( \frac{\pi D^4 \Delta P}{128 \mu \ell} \right) \]  

...(6.18)
which is the Poiseuille equation for Newtonian laminar flow.

For Bingham fluids similar treatment will yield:

\[ Q = \frac{\pi D^3 \tau_w}{(32 \mu_p) \left[ 1 - 4/3 \left( \frac{\tau_0}{\tau_w} \right) + 1/3 \left( \frac{\tau_0}{\tau_w} \right)^4 \right]} \]  

...(6.19)

Substitution for \( \tau_w \) can be made from Equation 6.7. This is the so-called Buckingham-Reiner equation. This equation can not be solved explicitly for the pressure loss and a trial and error or other procedure must be employed to obtain the pressure loss for a given flow rate. Hedstrom (1952) has proposed a solution procedure based on curves and a nomogram which simplifies and produces an accurate calculation of the pressure loss in a simple and straightforward manner.

For the power law model, integration of Equation 6.15 yields:

\[
\frac{\pi D^3}{32} \left( \frac{4n}{3n+1} \right) \frac{\tau_w^{1/n}}{k} \left( \frac{D \Delta p}{4\pi k} \right)^{1/n}
\]

or

\[
Q = \frac{\pi D^3}{32} \left( \frac{4n}{3n+1} \right) \left( \frac{D \Delta p}{4\pi k} \right)^{1/n}
\]

...(6.20)

Cheng (1970) used the generalised Bingham model to obtain a general equation for the flow rate which includes all the above cases as a special case, his equation is:

\[
Q = \frac{\pi D^3}{32} \cdot \frac{4n}{3n+1} \left( \frac{\tau_w}{K} \right)^{1/n} \left( 1 - \frac{\tau_0}{\tau_w} \right) \cdot \left( 1 - \frac{\tau_0/\tau_w}{2n+1} \left( 1 + \frac{2n}{n+1} \left( \frac{\tau_0}{\tau_w} \right) \left( 1 + n \frac{\tau_0}{\tau_w} \right) \right) \right)
\]

...(6.21)

\( \tau_w \) is again given by Equation 6.7.
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Many more equations for the rate of flow have been proposed in literature, see Govier and Aziz (1970) and Wilkinson (1960), other new ones are still being proposed, see for example, Hanks (1984).

A general approach for scaling up a capillary tube viscometer data was introduced by Metzner and Reed (1955). They used the expression for the rate of shear developed by Rabinowitsch (1929) and Mooney (1931). The Rabinowitsch and Mooney expression, Equation 6.22, is entirely independent of the flow model provided that the fluid under consideration is time independent.

\[- \left( \frac{dv}{dr} \right)_w = \frac{3(8Q)}{\pi D^3} + \frac{4 \Delta P}{D} \cdot \frac{d(8Q/\pi D^3)}{d(D \Delta P/4 \ell)} \tag{6.22} \]

Since the mean velocity V_m is equal to \(4Q/\pi D^2\), Equation 6.22 can be rearranged as:

\[- \left( \frac{dv}{dr} \right)_w = \frac{3V_m}{4 D} + 1 \cdot \frac{V_m}{4 D} \cdot \frac{d(\ln(8V_m/D))}{d(\ln(D \Delta P/4 \ell))} \tag{6.23} \]

If \(d(\ln(8V_m/D)) \over d(\ln(D \Delta P/4 \ell))\) is made to be equal to \(\frac{1}{n'}\),

then Equation 6.23 can be written as

\[- \left( \frac{dv}{dr} \right)_w = \frac{(3n'+1)(8V_m)}{4n' D} \tag{6.24} \]

Equation 6.24 is another form of the Rabinowitsch and Mooney equation.

From \(\frac{1}{n'} = \frac{d(\ln 8V_m/D)}{d(\ln(D \Delta P/4 \ell))}\) and assuming \(n'\) is constant over a range of shear stress, then it can be seen that

\[\frac{D \Delta P}{4 \ell} = K' \left( \frac{8V_m}{D} \right)^{n'} \tag{6.25} \]
or using Equation 6.7

\[ \tau_w = K' \left( \frac{8V_m}{D} \right)^{n'} \]  

...(6.26)

where \( K' \) is constant

Metzner and Reed found that \( n' \) and \( K' \) were constant over a wide range of \( 8V_m/D \) or \( \tau_w \). For some fluids this is not the case, and care must be taken to ensure that the range of integration is small. On substitution for \( 8V_m/D \) in Equation 6.26 from Equation 6.24 one obtains:

\[ \tau_w = K' \left( \frac{4n'}{3n'+1} \right)^{n'} \left( \frac{dV_m}{d} \right)_w^{n'} \]  

...(6.27)

or \( \tau = K' \left( \frac{4n'}{3n'+1} \right) \left( \frac{dV_m}{dr} \right)^{n'} \)  

...(6.28)

This equation is similar to the power law model and is usually referred to as the Metzner-Reed power law model for fluid flowing in a tube. If \( n' \) is equal to \( n \) and

\[ K' \left( \frac{4n'}{3n'+1} \right)^{n'} \]

is equal to \( K \) then Equation 6.28 reduces to the power law model.

Bowen (1961) has presented a complete design procedure for scaling up tube viscometer results to large pipes in both the laminar and turbulent regimes. In the laminar regime, Bowen used the method of Metzner and Reed described above.

6.4.2: Wall Effect

In the analysis of the above equations the assumption was made that
slip does not occur at the wall of the tube and hence the velocity at the wall is zero. If that is true then, for a time independent fluid flowing in tubes in laminar condition and with no end effect, the data derived for a series of tubes of varying radius and length must fall on a unique curve when \( \frac{8Q}{\pi D^3} \) is plotted against \( \frac{\Delta P D}{4L} \). However, numerous studies on suspensions flowing in tubes have reported a large diameter effect (see for example Dix and Scott Blair (1940) and Jastrzebski (1967). To explain this phenomenon the suspended particles near the wall were assumed to migrate towards the centre leaving a layer, usually termed as slip layer, of low particle concentration and consequently low consistency near the wall of the tube. A result of the migration of the particles towards the centre of the tube is that the mean velocity of the particles will be greater than that of the suspending fluid. Therefore the particle on average will spend less time in the tube than the bulk fluid leading to a reduction in particle concentration in the tube. A reduction in particle concentration of suspensions flowing in tubes was measured by many workers (see for example, Moude and Whitmore (1956), Seshadri and Sutera (1968), Segre and Silberberg (1962), and Karnis et al. (1963). According to Vand (1948) the origin of this layer is mechanical and comes from the fact that the particle centres cannot approach the tube wall to a distance less than a half of the effective diameter of the particles, and hence a reduction in concentration must result adjacent to the tube wall. Moude and Whitmore (1956), and Seshadri and Sutera (1968) used this approach with some success. Segre and Silberberg (1961,1962) proposed that the cause of the particle movement is a centripetal force of hydrodynamic origin. They supported their hypothesis by theory and experiment. Brenner (1966) presented a review of many workers both of theoretical and experimental nature. Brenner concluded that none of the theories presented furnish a satisfactory fundamental explanation of the radial migration phenomenon in tubes. Cox and Brenner (1968) proposed a theory in which they claimed to provide what is lacking in the others, but, since their theory incorporated some numerical coefficients which have not been evaluated, a complete quantitative comparison with experimental data is not possible.

The origin of the particle migration or slip layer should not concern us here as much as its existence, particularly since in flocculated
suspensions in plug flow such as cement slurries, the slip phenomenon may be caused by more than one factor. What is important from the engineering point of view is how the slip layer would affect the flow behaviour of suspensions in a tube.

Re-writing Equation 6.10

\[
Q = \pi \left[ \frac{r^2}{2} - \int_0^D r^2 \frac{dv}{dr} \, dr \right]
\]

If there were no slip the first part of the equation would disappear. However, this is a special case and generally there will be a slip velocity, \( v_s \), then

\[
Q = \pi (D/2)^2 v_s - \pi \int_0^D r^2 \frac{dv}{dr} \, dr \quad \ldots (6.29)
\]

Using Equations 6.5, 6.8 and 6.29 we obtain

\[
Q = \pi (D/2)^2 v_s + \pi \int_0^D \left(2\tau D/\tau_w\right)^2 f(\tau) \, d\tau \quad \ldots (6.30)
\]

Substituting for \( d\tau \) from Equation 6.14 in Equation 6.30

\[
Q = \pi (D/2)^2 v_s + \left(\frac{\pi D^3}{8 \tau_w^3}\right) \int_0^D f(\tau) \, d\tau \quad \ldots (6.31)
\]

Oldroyd (1949) suggested that \( v_s \) is a function of the shear stress at the wall of the tube. By introducing a coefficient of slip, \( S \), defined as \( v_s/\tau_w \).

Substituting for \( v_s \) in Equation 6.31,

\[
Q = \pi (D/2)^2 S \tau_w + \left(\frac{\pi D^3}{8 \tau_w^3}\right) \int_0^D f(\tau) \, d\tau
\]
Hence by plotting \( \frac{8Q}{\pi D^3 \tau_w} \) against \( 2/D \) for constant values of \( \tau_w \),
the relation between \( S \) and \( \tau_w \) can be found. However, Jastrzebski (1967) found that \( S \) is not only dependent on \( \tau_w \) but varies inversely
with the tube radius. Thus if one defines \( S \) as \( (2Sc)/D \) where \( Sc \) is
the corrected slip coefficient which depends only on \( \tau_w \). Then
substituting for \( S \) in Equation 6.32 results in

\[
\frac{8Q}{\pi D^3 \tau_w} = \frac{25}{D} + \frac{1}{\tau_w^4} \left( \int_0^{\tau_w} f(\tau) \, d\tau \right) \quad \ldots (6.33)
\]

Then for a constant value of \( \tau_w \), the corrected slip coefficient can be
found from the slope of the line obtained from plotting \( \frac{8Q}{\pi D^3 \tau_w} \)
against \( 4/D^2 \).

This coefficient can then be used in flow calculations.

The scale up procedure presented by Metzner and Reed (1955) will not
be valid if slip occurs at the surface of the tube. Bannister (1980)
obtained rheological measurements for a series of cement slurries and
drilling fluids. He found the data to follow the Metzner-Reed power
law model, Equation 6.26. However, he also found the power law
parameter \( K' \) to be dependent on pipe diameter. Re-writing the
Metzner-Reed power law model, Equation 6.26, with \( K' \) equal to \( K'D' \),
where \( K'D' \) is the consistency index at constant pipe diameter.

\[
\frac{8V_m}{D} = \left( \frac{\tau_w}{K'D} \right)^{1/n'} \quad \ldots (6.34)
\]

If the slip velocity is assumed to be equal to \( V_s \), then the velocity
without slip in the Metzner-Reed equation is now equal to \( (V_m - V_s) \).
Hence, from Equation 6.26:

\[
\frac{8V_m}{D} = \frac{8V_s}{D} \left( \frac{\tau_w}{K'} \right)^{1/n'} \quad \ldots (6.35)
\]
Substituting Equation 6.34 in 6.35 yields:

\[
\left( \frac{1}{K_D'} \right)^{1/n'} = \left( \frac{1}{K'} \right)^{1/n'} + \frac{8/(D(T_W)^{1/n'})}{D}
\]  

...(6.36)

Bannister (1980) introduced coefficient of slip, S, defined as

\[
V_s = S(T_W)^{1/n'}
\]  

...(6.37)

Substituting Equation 6.37 in Equation 6.36 gives:

\[
\left( \frac{1}{K_D'} \right)^{1/n'} = \left( \frac{1}{K'} \right)^{1/n'} + \frac{(8S)}{D}
\]  

...(6.38)

Substituting for Vs from Equation 6.37 in Equation 6.36 yields:

\[
\frac{8V_m}{D} = \left( \frac{1}{K_D'} \right)^{1/n'} + \frac{8S}{D} (T_W)^{1/n'}
\]  

...(6.39)

Equation 6.39 represents the modified Metzner-Reed equation. The parameter \( K', n' \) and S can be found from the slopes and intercepts of the straight lines obtained by plotting \( \log (T_W) \) versus \( \log (8V_m/D) \) Equation 6.34,

and \( \left( \frac{1}{K_D'} \right)^{1/n'} \) versus \( \frac{1}{D} \), Equation 6.38,

### 6.4.3 Experimental Investigations

While there exists a large number of published papers on the theoretical analysis of non-Newtonian fluids, surprisingly few papers have implemented the equations proposed to predict the flow behaviour of suspensions in pipes from data obtained using a laboratory viscometer. Most of the work involving flow of slurries in pipes has concentrated on the flow of settling slurries and/or turbulent flow conditions. Amongst the small number of papers published on the non-settling slurries in laminar flow, only a few have attempted a
theoretical prediction of flow. A number of authors (Sauermann (1982) and Verkerk (1982)) have just presented their results without attempting to make any prediction, whereas others, such as Loken et al (1982), and Kazanskij and Mathias (1978), have used empirical equations to scale up their pipe results to larger diameter systems. Loken et al (1982) used an equation in the form given in Equation 6.40 to correlate their results.

\[ \Delta P/l = A V_m^x D^y C_v^z \]  

...(6.40)

where \( A, x, y, \) and \( z \) are empirical constants  
\( C_v \) is the solids volumetric concentration.

A good correlation of results was obtained between the various diameter tubes. Kazanskij and Mathias (1978) presented experimental and theoretical analysis concerning the pressure gradient of a metalliferous mud in a pumping loop. The co-axial cylinder viscometer data fitted closely a power law model, although the authors only presented the data at high shear rates. In the laminar region they found by linear regression analysis that:

\[ \Delta P/l = (\lambda V_m^2 \rho) / 2D \]  

...(6.41)

where \( \lambda = 64/Re \)

\[ \text{and } \text{Re} = \left( V_m^{2-n} D^2 \right) / \left( 3K^{0.75} (2+6n)/n \right) \]

\( n \) and \( K \) are the power law constants. Kazanskij and Mathias emphasised the importance of the viscometric. They mentioned that the material exhibited some thixotropic characteristics but they did not explain how the flow curves were obtained or their relation to the pumping loop results.

Horsley (1982) used the co-axial cylinder viscometer to provide data for gold slime slurries in pipelines. He used the least square method to determine a parabolic law describing the shear stress as a function of shear rate which he used to predict pressure drop in the pipes. The predicted pressures generally agreed with the measured ones, although in Horsley's work he excluded the thixotropic effect of
the gold slime and used the equilibrium flow curve. Want et al (1984) have carried out rheological measurements on red mud residue. They compared measured pressure gradients (shear stresses) at the wall of the full scale pipe lines with those measured in a tube rheometer. Want et al found that while there was a good correspondence at shear rates higher than the apparent shear rate \((8\text{Vm/D})\) of 20 S\(^{-1}\), the full scale data diverged considerably below an apparent shear rate of 10 S\(^{-1}\). These anomalous results were attributed to a plug flow condition which occurred only in the smooth tube rheometer at low shear rates. Kenchington (1978) performed an experimental investigation to measure the pressure gradient for a dense phase medium (40% by weight clay suspension) transporting settling particles (750 microns sand particle) for a range of flow regimes. For the clay suspension alone Kenchington fitted the generalised Bingham equation to the data obtained from a rotational viscometer. In the laminar regime Cheng's (1970) expression, Equation 6.21 for the generalised Bingham model was used to predict pressure loss for three tubes (13, 25 and 51 mm diameter). The predicted pressure losses were compared with the measured one. For the 25 mm tube agreement was generally good. However, the predicted pressure loss was much higher than the measured one for the 13 mm diameter tube and vice versa for the 51 mm diameter tube. The difference between the predicted and measured values for the 13 mm tube was attributed to laminar slip, but no explanation was given for the anomalous situation for the 51 mm diameter tube. Perhaps it is worth while noting that Cheng's expression stated in the Kenchington paper was in error. Therefore the predicted values would be slightly different from the stated values had the correct expression been used, but that would not account for the difference between the predicted and measured values. Also the scale on the published figure relating the pressure loss to the average velocity is in error by a factor of ten.

In an earlier investigation Kenchington (1974) provided a comprehensive study of the flow of clay and kiln feed slurries. Again Kenchington used the co-axial cylinder viscometer to establish a relationship between the shear stress and the shear rate. The uncorrected data was best fitted by the generalised Bingham model which gave a reasonably good prediction of pressure loss in the pilot plant test loops (27, 39, 53 and 82 mm diameter pipes). However, when a Bingham model representing data corrected for the relatively large gap width was
utilised, a less satisfactory correlation between the predicted and measured pressure in the pipes was obtained. Kenchington attributed this behaviour to the errors in the results of the co-axial cylinder viscometer which cancel each other with the uncorrected data. The results from the co-axial cylinder viscometer and the pilot plant loops were used to evaluate the pressure drop in the full scale plant pipes. Cheng's (1970) equation and Bowen's (1961) method (based on the Metzner-Reed equation) were used to scale up the co-axial cylinder viscometer data and the pilot plant results respectively. Both methods provided a considerable over estimation of the pressure drop for the clay suspension.

Windhab and Gleissle (1984) obtained rheological measurements on an aqueous Kaolin suspension of 35% water by weight. A plot of shear stress versus shear rate for data collected with the co-axial cylinder; cone and plate and capillary viscometers resulted in a completely different flow curve for each instrument. Even changing the tube diameter of the capillary viscometer had a large influence on the resulting flow curve. The slip velocity, which was assumed to be dependent only on the wall shear stress, was measured by a twin capillary viscometer, designed by the authors in order to speed up and simplify data acquisition.

Cement slurries of water cement ratio of 0.4, 0.5, and 0.6 were pumped through pipes of various diameters by Raffle (1972). He presented the results in the form of plots of wall shear stress ($\tau_w$) versus apparent shear rate ($8V_m/D$) and pressure loss versus time. In addition, Raffle presented a start up wall-shear stress against time for a number of pipes and cement slurries. No prediction was attempted.

Bannister (1980) carried out experimental investigation on numerous cement slurries and drilling muds. He found that the data, obtained using a capillary viscometer, is best fitted by a Metzner-Reed type flow model, (Equation 6.26). However, he found that $K'$ is a function of the tube diameter which he corrected for by introducing a slip velocity, see section 6.4.2. Bannister used the corrected flow model to predict the pressure drop in a large diameter pipe and good results were achieved. The co-axial cylinder viscometer was also used by Bannister to obtain the flow curve of a cement slurry. In this case, the material
obeyed a Bingham type flow model which was employed to predict the pressure loss in the large diameter pipes. The predicted pressures did not agree with the experimentally measured ones. Perhaps this is not surprising, since materials which slip in tubes will probably slip in the co-axial viscometer. Hence, correction for slip must be made before prediction should be attempted.

6.4.4: Summary

In the above review it was seen that if the material is time independent and the relation between the shear stress and shear rate is known, a solution of the flow behaviour is possible by integrating Equation 6.15. If slippage occurs at the surface of the tube then the resulting equations may be modified to account for the effects of the slip. But, since the slip layer is often very thin it is difficult, or may be impossible, to measure its thickness or its properties and therefore the modified equations may have to rely on unconfirmed assumptions. The picture which one gets from the literature on experimental results is confusing. While some authors have predicted the flow behaviour successfully others were not so successful. Many of those who have been successful had to correct their results empirically before achieving the correct prediction. Perhaps this is not so surprising given the complex nature of the material we are dealing with. However, it is somewhat disappointing that no general formulation or method has emerged from the huge amount of research work undertaken on the subject.

The main reason behind this confused state lies in the multi-phase nature of suspensions. Suspensions may undergo phase separation and/or structural change when subjected to shearing action which can be strongly dependent on the flow conditions. The majority of workers have chosen to ignore or to eliminate the effects of structural change such as thixotropy by working on the completely broken down structure, viz. the equilibrium structural level. If the material has no yield stress and it undergoes a complete structural breakdown in a short time in comparison with the time the material takes to flow in the tube, then the use of the equilibrium structural level rheological parameters in the calculation of flow problem will present no significant practical errors. However, if the material possesses a yield stress and/or the time taken to reach the equilibrium structural level is relatively long
or is never reached then the use of the equilibrium flow parameters may lead to serious errors in the prediction of the flow behaviour of the material. Materials which exhibit a structural change or time dependency have not been studied to the same depth as time stable materials. The cause of the lack of exploration of this field stems from the fact that none of the existing models which describe the behaviour of time dependent fluids has received a general acceptance from the workers in this field of rheology. This difficulty arises from the inadequacy and/or the complexity of the rheological equations proposed. Nevertheless, some attempts have been presented for the solution of flow problems of time dependent material in pipes such as the method of Govier and Ritte, and Ritter and Batycky described in Govier and Aziz (1972). Parker (1975) and Cheng and Whittaker (1972) have used a computer analysis to solve the resulting equations. Cheng (1979) has obtained solutions for some special cases of start up problems of thixotropic fluid in pipes. Apart from the Cheng method (1979), the resulting calculation of the flow problem is laborious and requires a comprehensive determination of the rheological characteristics of the material which is itself laborious and in some cases difficult to achieve due to the sensitivity or the instability of the material. If the material possesses a slip at the solid surfaces of the measuring instrument and/or on the surface of the pipe, the task of determining the rheological characteristics and the flow behaviour will be many times harder and could be impossible to achieve. The material described in this thesis (see Chapter 5) is a reacting suspension which possesses a variable time dependent behaviour which is highly dependent on shear history and flow conditions. In addition the material exhibits slippage at smooth solid surfaces. It was found that no unique flow model, however complex, can adequately describe the flow behaviour of this material. As a result of these difficulties a technique which gives an approximate prediction of the flow problem of a complex material in pipes was developed and is described in Chapter 7. Prediction is based on data obtained from a simple test performed with the co-axial cylinder viscometer. The technique is suitable for any material, time dependent or time independent, as long as the material exhibits a yield stress. The technique is particularly suitable for time dependent materials which possess a slip at the surface of the pipe and which exhibit a relatively high yield stress. These requirements are properties of the material used in this work.
7.1: Introduction

It was pointed out in section 6.4.2 that when a suspension flows through a pipe, a slip layer (layer of low particle concentration relative to the bulk concentration) may develop adjacent to the pipe wall. Hence when a material with a yield stress flows through a pipe, the flow pattern is generally categorized by three distinct regions, viz: a slip layer near the wall, a plug in the centre and sheared layer of material in between. Depending on the material rheological behaviour, each layer may exhibit completely different rheological characteristics. Consequently, to achieve an accurate prediction of the flow of suspensions in pipes the rheological and time dependent behaviour of each layer must be established. This makes the task of determining their rheological characteristics and the calculation of their flow behaviour very difficult and complex.

In the proposed technique the disadvantages of these materials, viz, yield stress, thixotropy and slippage are turned to an advantage. As a result only a simple co-axial cylinder viscometer test was required to obtain a simple solution of the problem of predicting the pressure required to pump these materials in pipes. For this method to be valid, the following assumption must be true:

1). The material possesses a yield stress.
2). The material flows in a laminar condition.
3). There is plug flow over a substantial portion of the tube and in the co-axial cylinder viscometer, so that the thickness of the sheared layer in both systems is small compared with the tube diameter and with the diameter of the inner cylinder of the co-axial cylinder viscometer.

7.2: Hypothesis to Predict Pressure Gradient in Steady Plug Flow in Parallel Sided Pipes

If the above assumptions are true, then the material will be
effectively sheared between the surfaces of a moving solid central plug and a stationary tube. The resulting sheared layer, can be reproduced in the co-axial cylinder viscometer if the outer cylinder (rotor) is large enough so that plug flow will always exist. Thus, if the speed of the plug in the co-axial cylinder viscometer is equated to the speed of the plug in the pipe, the sheared layer in both the tube and the co-axial cylinder viscometer should be similar. The similarity should include the properties of the slip layer, thickness of the sheared layer, the shear rate, the rate of build up or breakdown of structure for time dependent material, etc.

This analogy implies that the shear stress at the surface of the bob will be equal to that on the tube surface. Hence, the shear stress measured at the bob surface can be used to calculate the pressure drop in tubes. The main advantage of this technique is that all the information required to predict the pressure in a pipe is obtained from one simple test. Moreover the calculation involved is very simple and short as will be seen in the following section.

The above assumptions are not entirely without foundation, but have some theoretical backing because if one imagines the pipe diameter and the inner cylinder (bob) of the co-axial cylinder viscometer to be large and the thickness of the sheared layer to be small, then the material will be effectively shearing between two parallel plates, where the wall of the pipe or the surface of the bob acts as one plate and the plug as the other. The direction of shearing should not make a significant difference as long as the centrifugal force does not cause the particles in suspension to migrate outwards in the gap of the co-axial cylinder viscometer.

The hypothesis of parallel plates diminishes in accuracy as the radius of the pipe (Rp) and/or the radius of the bob (Rb) reduces, and the thickness of the sheared layer (x) increases. Nevertheless, if the ratio of x/(Rb) and x/(Rp) are kept small, this hypothesis should still be applicable. The thickness of the sheared layer is dependent on the relative magnitudes of wall shear stress and the yield stress (gel strength) of the material, i.e. on the ratio of τw/τ0 a small ratio implying a thin sheared layer. Thixotropy and slippage on the wall of the pipe and the surface of the bob lead to a low τw, thus resulting
in a reduction in the magnitude of this ratio, for a given material and conditions. Consequently this technique is best used with materials such as cement slurries which exhibit a thixotropic behaviour and/or promote slippage at the surfaces of the pipe and the bob.

It must be stressed that the technique should be applicable for any material, and not only for thixotropic materials or when the slippage on the surface of the pipe occurs, as long as the assumptions in section 7.1 hold.

7.3: Mathematical Interpretation of Plug Flow Hypothesis

7.3.1 Co-axial Cylinder Viscometer

i) Shear Stress

The shear stress at the surface of the inner cylinder (bob) can be obtained by the balance of torque exerted on the bob by the material and the torque measured on the bob shaft. A full description of the bob is given in section 7.4.3, but it is important, at this stage, to note that the bob was designed so that no material is sheared at its top and bottom ends. With this design, the balance of torque yields:

\[ \tau_b = \frac{T}{2\pi H R_b^2} \]  

...(7.1)

Where \( \tau_b \) is the shear stress at the surface of the bob and \( T \) is the torque measured at the bob's shaft and \( H \) and \( R_b \) are the bob's height and radius respectively.

If the material is time dependent, then the shear stress at any time \( t \) will be \( \tau_b = \tau_b(t) \) and hence the torque \( T = T(t) \). Substituting in Equation 7.1 produce

\[ \tau_b(t) = \left[ (T(t)) / (2\pi H R_b^2) \right] \]  

...(7.2)

ii) Plug Linear Speed

The linear speed of the plug \( (V_p \ m/min.) \) can be easily calculated
from the rotation speed of the outer cylinder. If the rotation speed of the cylinder is $\Omega$ r.p.m and the thickness of the sheared layer is $x$, then

$$V_p = 2\pi\Omega(R_b + x) \quad \ldots(7.3)$$

If $x$ is small compared with $R_b$, then

$$V_p = 2\pi\Omega R_b \quad \ldots(7.4)$$

7.3.2: Pipe

i) Shear Stress

Consider a cylindrical element, Figure 7.1, of radius $R_p$, where $R_p$ is the radius of the pipe. If we consider the general case of a fluid in which the shear stress depends on the duration of shearing then over a length $\Delta l$ of pipe there will be a change of shear stress from say $\tau$ to $\tau + \Delta \tau$. Balance of forces acting on this element produce:

$$\Delta P = \pi R_p^2 = 2\pi R_p \Delta l \left(\tau + \tau + \frac{\Delta \tau}{2}\right)/2$$

$$\Delta P = (2\Delta l/R_p)(\tau + (\Delta \tau/2)) \quad \ldots(7.5)$$

In the majority of practical situations the mean flow velocity $V_m$ is constant then

$$\Delta l = V_m \Delta t \quad \ldots(7.6)$$

where $\Delta t$ is the time taken to travel distance $\Delta l$.

Substituting Equation 7.6 in Equation 7.5 yields:

$$\Delta P = 2V_m \Delta t \left(\tau + (\Delta \tau/2)\right)/R_p \quad \ldots(7.7)$$

In the limits Equation 7.7 can be reduced to:

$$\Delta P = 2V_m \Delta t \tau/R_p \quad \ldots(7.8)$$
For time dependent material
\[
\tau = \tau(t) \quad \ldots (7.9)
\]

Then from Equation 7.9 and Equation 7.8 we get
\[
\Delta p = 2V_m \Delta t \frac{\tau(t)}{R_p} \quad \ldots (7.10)
\]
on integration of Equation 7.10
\[
\begin{align*}
P &= \left[ \frac{2V_m}{R_p} \right] \int_{t_1}^{t_2} \tau(t) \, dt \\
&= \left[ \frac{2V_m}{R_p} \right] \left[ \frac{\tau(t)}{R_p} \right]_{t_1}^{t_2} \\
&= \left[ \frac{2V_m}{R_p} \right] \left[ \tau(t_2) - \tau(t_1) \right]
\end{align*}
\quad \ldots (7.11)
\]  
where \( P \) is the total pressure drop over the integrated pipe length.

Inspection of Equation 7.11 reveals that the integral
\[
\int_{t_1}^{t_2} \tau(t) \, dt
\]
is in fact the area under the curve of shear stress versus time plot, Figure 7.2. If we call this quantity \( A \) then Equation 7.11 can be written as:
\[
P = \frac{2V_m A}{R_p} \quad \ldots (7.12)
\]

For material which is independent on the duration of shearing \( \tau(t) \) will be constant and equal to, say, \( \tau \), then from Equation 7.11 we obtain:
\[
\begin{align*}
P &= (2V_m \tau/R_p)[t] \\
&= (2V_m \tau/R_p)[t_2 - t_1]
\end{align*}
\quad \ldots (7.13)
\]
or
\[
P = (2V_m \tau/R_p)(t_2 - t_1) \quad \ldots (7.14)
\]
FIGURE 7.1: FORCES ACTING ON A CYLINDRICAL ELEMENT OF FLUID FLOWING IN A PIPE

FIGURE 7.2: SHEAR STRESS VERSUS TIME CURVE FOR A GENERAL FLUID
but \( V_m(t_2 - t_1) = \ell \) the distance travelled,

Substituting in Equation 7.14 then

\[
P = 2\tau \ell / R_p \quad \ldots (7.15)
\]

If we compare Equation 7.15 with equation 7.12, it can be seen that Equation 7.12 can be written in the form of Equation 7.15 if \( \tau \) is equal to \( V_m A / \ell \). In this case \( \tau \) is the average shear stress over the length of the pipe considered. Rewriting Equation 7.12 in the form of Equation 7.15 using \( \tau a \) the average shear stress, a general equation for duration of shear dependent and independent fluids will result.

Pressure drop, \( P = 2\ell \tau a / R_p \quad \ldots (7.16) \)

where \( \tau a = \tau \) for shear duration independent fluids and

\[ \tau a = V_m A / \ell \quad \ldots (7.17) \]

for shear duration dependent fluids.

Hence the average shear stress \( \tau a \) or pressure drop \( P \) for any given length of pipe (\( \ell \)) can be obtained from Equation 7.16 if \( P \) or \( \tau a \) respectively are known. \( \tau a \) can also be calculated from Equation 7.17 if \( V_m, A \) and \( \ell \) are known where \( A \) is the area under shear stress versus time curve between \( t_1 \) and \( t_2 \), for a given velocity \( V_m \) as shown in Figure 7.2. \( t_1 \) and \( t_2 \) represent the minimum and maximum shearing times respectively.

ii) Plug Speed

Figure 7.3(a) gives the velocity profile of a material with a yield stress flowing through a tube. \( V_m \) and \( V_p \) are the mean and the plug speed respectively. Over the thickness of the sheared layer (\( \ell \)), the velocity varies from zero at the wall of the tube to \( V_p \) at the surface of the plug. The exact shape of velocity profile over the sheared layer is dependent
FIGURE 7.3: VELOCITY PROFILE OF A YIELD STRESS MATERIAL FLOWING THROUGH A PIPE IN PLUG FLOW
(a) TRUE VELOCITY PROFILE; (b) ASSUMED VELOCITY PROFILE

on the rheological characteristics of the sheared layer, including the slip layer. However, if the thickness of the sheared layer is small the variation in velocity, throughout the thickness of the sheared layer, can be assumed to be linear, as shown in Figure 7.3(b), without a significant loss of accuracy.

Referring to Figure 7.3(b) it can be seen that

\[ V_m \pi R_p^2 = (V_p \tau (R_p^2 - (R_p - x)^2)/2) + V_p \tau (R_p - x)^2 \] ... (7.19)

Equation (7.19) can be reduced to:

\[ V_p = V_m/(1 - (x/R_p) + 0.5(x/R_p)^2) \] ... (7.20)

If \( x \) is small compared with \( R_p \)

\[ V_p = V_m/(1 - (x/R_p)) \] ... (7.21)
If $x$ is very small compared with $R_p$ then $V_p = V_m \quad \ldots (6.74)$

Therefore if the thickness of the sheared layer is known and the mean speed of flow is also known then plug speed can be calculated using Equation 7.20, 7.21 or 7.22 dependent on the relative magnitude of $x$ and $R_p$, and the degree of accuracy required.

7.4: Experimental Techniques

A full description of the apparatus is given in Chapter 4, only the test procedure is described here.

7.4.1: Pumping Line

To test the hypothesis given in section 7.2 that the co-axial cylinder viscometer with a very large gap width could be used to predict the pressure drop in pipes, several tests were carried out using vertical and inclined ($22^\circ$ to the horizontal) pumping lines. Each pumping line consisted of a Mono pump and a straight pipe section as shown in Chapter 4, Figure 4.11.

The straight sections of the pipe were 4.5m and 13.5m for the vertical and inclined pipes respectively. The vertical pumping line had two pressure transducers at distances of 1.785m and 3.785m from the discharge end, while the inclined pumping line had three pressure transducers placed at 1.785m, 3.785m and 13m from the discharge end. Pipe diameters of 19.54mm and 34.6mm were used with the vertical line, but only the larger of the above two pipes was used with the inclined line.

The mean flow velocity $V_m$ in the pipes was measured by recording accurately the time taken to fill a known length of pipe. This was obtained from the plot of pressure against time curve recorded from the output of the pressure transducer while the pipes were being filled. A typical pressure-time curve is shown later in Figure 7.7.
7.4.2: Mixing Procedure

Approximately four litres of material were mixed using the four litre container of the Waring commercial blender, the constituent proportions and mixing procedure being given in Chapter 4, section 4.3. Six identical mixes were prepared for each test run for the inclined line and two mixes for each run in the vertical line. Fresh and agitated materials were used in separate test runs, the purpose of the agitation being to produce a homogeneous material with different rheological characteristics from that of the fresh material. The fresh material was transferred to the pump hopper straight after mixing. However, because of the large quantities of slurry required to fill the pumping line the pump was not turned on until the second mix was ready with the vertical pumping line and until the third quantity had been mixed with the inclined pumping line. In the case of agitated material, the material was transferred after mixing to the container of the Crypto Peerless mixer and stirred for forty eight minutes from the start of the first mix. The material was then transferred to the pump hopper and the pump was turned on at predetermined speed.

7.4.3: Tests on the Co-axial Cylinder Viscometer

The Weissenberg Rheogoniometer container (outer cylinder) was filled with material at the same time as the slurry was placed into the hopper of the pump. The container was then placed in position on the Rheogoniometer's bottom platen and the bob (inner cylinder) was lowered down until the top surface of the bob was level with the surface of the material in the container (see Figure 7.4). When the bob was in position and the pump about to be started on the pumping line the outer cylinder was rotated at a constant rotation speed simultaneously with pump start up. This procedure ensured that the material in both the pump and the co-axial cylinder viscometer were subject to as similar a shear history as possible.
In order to ensure plug flow throughout most of the slurry a large outer cylinder of 97mm diameter and 110mm height was used. The bob was manufactured from a cylinder taken from the 34.6mm internal diameter pipe the outer surface of which appeared to have a similar roughness to the inner surface. The final dimensions of the bob were 43mm diameter by 48.5mm height. The bob had a hollow bottom end, as shown in Figure 7.4, so that when it was lowered into the slurry an air bubble was trapped under it. Hence, if the top end of the bob was level with the surface of the material in the container, the contribution of both ends to the measured torque on the bob shaft was negligible. Consequently, in all of the subsequent calculations the measured torque was assumed to be produced only by the shear stress at the circumference of the bob.

The torque measured at the shaft of the bob and the output from the pressure transducer in the pumping line were recorded on separate Bryan's x-y/t recorders to ensure a continuous trace of the results throughout the test. A typical output from both the pressure transducer and the Weissenberg Rheogoniometer are shown in section 7.5.
Chapter 7

7.5: Results and Discussion for Slip Plug Flow

7.5.1: Agitated Material in the Vertical Pumping Line and in the Co-axial Cylinder Viscometer

Eighteen tests were carried out using the Weissenberg Rheogoniometer and eighteen tests using the vertical pumping line. Nine of the pumping line tests were performed with the 19.54mm diameter tube and nine using the 34.6mm diameter tube. Figures 7.5 and 7.6 show two typical shear stress versus time curves measured at the surface of the bob in the Weissenberg Rheogoniometer as described in section 7.4.3. These two figures show clearly the effect of changing rotation speed on the shear stress. Figure 7.7 shows typical pressure-time curves recorded by transducer 1 (1.785m from the discharge end) for the 19.54mm diameter pipe. A summary of all the results is given in Tables 7.1 to 7.5. Tables 7.1 to 7.4 show the results recorded by transducers 1 and 2 in the 19.54mm and 34.6mm diameter pump line. These tables also include the average shear stress calculated using Equation 7.16. Table 7.5 shows the average shear stress for transducers 1 & 2 calculated using Equation 7.17. 'A' in Equation 7.17 represents the area under the shear stress versus time curve measured at the surface of the bob (see Figure 7.5) where $t_1$ and $t_2$ in Figure 7.5 correspond to the minimum and maximum shearing times respectively of a material flowing in the pipe between transducer number 2 and the discharge end.

The average shear stresses presented in Table 7.1 to Table 7.5 are plotted against plug speed in Figure 7.8 and Figure 7.9. Plug speeds listed in Table 7.1 to 7.4 were calculated from Equation 7.22 and plug speeds in Table 7.5 from Equation 7.4. These two equations were used instead of Equation 7.20 and Equation 7.3 because the thickness of the sheared layer was too small to measure. However at the maximum speed, where the thickness of the sheared layer was the largest a sheared layer of thickness of less than two millimetres was recorded in comparison with pipe and bob diameter of 19.54mm and 43mm respectively. Hence, in the most extreme cases, the errors involved would alter the relative position of the points in Figure 7.8 and 7.9 by less than 10% with the results from the 19.54mm diameter pipe being shifted to the right relative to the other points. Nevertheless, the results of the Figure 7.8 and Figure 7.9, as they
Figure 7.5: Typical shear stress - time curve for the agitated material obtained.
Figure 7.6: Typical shear stress - time curve for the agitated material obtained with the Weissenberg Rheogoniometer at rotation speed of 3.75 R.P.M.

Shear stress at the surface of the bob (Pa)

Time (min)
WITH TRANSUDER 1, PUMPING SPEED = 3.83 m/minute, PIPE DIAMETER = 19.54 mm

FIGURE 7.7: TYPICAL PRESSURE - TIME CURVE FOR THE ACTUAL MATERIAL OBTAINED
Table 7.1: Results from Transducer 1 with the 19.54 mm Diameter Vertical Pipe

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Plug Speed (m/minute)</th>
<th>Measured Pressure (K Pa)</th>
<th>Average Shear Stress ((\tau_a)) (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.97</td>
<td>23.31</td>
<td>63.8</td>
</tr>
<tr>
<td>2</td>
<td>1.84</td>
<td>11.86</td>
<td>32.5</td>
</tr>
<tr>
<td>3</td>
<td>0.74</td>
<td>4.91</td>
<td>13.4</td>
</tr>
<tr>
<td>4</td>
<td>3.825</td>
<td>20.04</td>
<td>54.8</td>
</tr>
<tr>
<td>5</td>
<td>1.0</td>
<td>7.36</td>
<td>20.1</td>
</tr>
<tr>
<td>6</td>
<td>2.97</td>
<td>18.0</td>
<td>49.3</td>
</tr>
<tr>
<td>7</td>
<td>2.97</td>
<td>16.8</td>
<td>46.0</td>
</tr>
<tr>
<td>8</td>
<td>2.97</td>
<td>18.0</td>
<td>49.0</td>
</tr>
<tr>
<td>9</td>
<td>2.97</td>
<td>19.6</td>
<td>53.6</td>
</tr>
</tbody>
</table>

Note: (i) Tables 7.1 to 7.4 inclusive the "measured pressure" is in fact a "pressure difference" between the open end of the pipe at atmospheric and the transducer.

(ii) Weight pressure has been deducted before the value was included in the Tables.

(iii) \(\tau_a = (P - R_p) / (2 \ell)\)

Table 7.2: Results from Transducer 2 with the 19.54 mm Diameter Vertical Pipe

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Plug Speed (m/minute)</th>
<th>Measured Pressure (K Pa)</th>
<th>Average Shear Stress ((\tau_a)) (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.97</td>
<td>52.1</td>
<td>67.2</td>
</tr>
<tr>
<td>2</td>
<td>1.84</td>
<td>28.66</td>
<td>37.0</td>
</tr>
<tr>
<td>3</td>
<td>0.714</td>
<td>10.42</td>
<td>13.4</td>
</tr>
<tr>
<td>4</td>
<td>3.825</td>
<td>43.24</td>
<td>55.8</td>
</tr>
<tr>
<td>5</td>
<td>1.0</td>
<td>17.2</td>
<td>22.2</td>
</tr>
<tr>
<td>6</td>
<td>2.97</td>
<td>39.6</td>
<td>51.1</td>
</tr>
<tr>
<td>7</td>
<td>2.97</td>
<td>43.8</td>
<td>56.5</td>
</tr>
<tr>
<td>8</td>
<td>2.97</td>
<td>41.7</td>
<td>53.8</td>
</tr>
<tr>
<td>9</td>
<td>2.97</td>
<td>39.1</td>
<td>50.5</td>
</tr>
</tbody>
</table>
### Table 7.3: Results from Transducer 1 with the 34.6 mm Diameter Vertical Pipe

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Plug Speed (m/minute)</th>
<th>Measured Pressure (K Pa)</th>
<th>Average Shear Stress (τa) (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.05</td>
<td>7.36</td>
<td>35.7</td>
</tr>
<tr>
<td>2</td>
<td>2.55</td>
<td>8.59</td>
<td>41.6</td>
</tr>
<tr>
<td>3</td>
<td>0.303</td>
<td>2.45</td>
<td>11.9</td>
</tr>
<tr>
<td>4</td>
<td>0.805</td>
<td>3.27</td>
<td>15.8</td>
</tr>
<tr>
<td>5</td>
<td>2.05</td>
<td>9.0</td>
<td>43.6</td>
</tr>
<tr>
<td>6</td>
<td>2.05</td>
<td>8.4</td>
<td>40.7</td>
</tr>
<tr>
<td>7</td>
<td>2.05</td>
<td>10.2</td>
<td>49.4</td>
</tr>
<tr>
<td>8</td>
<td>2.05</td>
<td>8.2</td>
<td>39.7</td>
</tr>
<tr>
<td>9</td>
<td>2.05</td>
<td>7.4</td>
<td>35.9</td>
</tr>
</tbody>
</table>

### Table 7.4: Results from Transducer 2 with the 34.6 mm Diameter Vertical Pipe

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Plug Speed (m/minute)</th>
<th>Measured Pressure (K Pa)</th>
<th>Average Shear Stress (τa) (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.05</td>
<td>17.71</td>
<td>40.5</td>
</tr>
<tr>
<td>2</td>
<td>2.55</td>
<td>21.88</td>
<td>50.0</td>
</tr>
<tr>
<td>3</td>
<td>0.303</td>
<td>4.17</td>
<td>9.5</td>
</tr>
<tr>
<td>4</td>
<td>0.85</td>
<td>8.3</td>
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</tr>
<tr>
<td>5</td>
<td>2.05</td>
<td>17.7</td>
<td>40.5</td>
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<tr>
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<td>2.05</td>
<td>19.3</td>
<td>44.1</td>
</tr>
<tr>
<td>7</td>
<td>2.05</td>
<td>18.8</td>
<td>43.0</td>
</tr>
<tr>
<td>8</td>
<td>2.05</td>
<td>17.7</td>
<td>40.5</td>
</tr>
</tbody>
</table>
Table 7.5: Average Shear Stresses for Transducers 1 and 2 Calculated from the Shear Stress Time Curve Measured at the Surface of the Bob in the Weissenberg Rheogoniometer.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Plug Speed r.p.m.</th>
<th>Plug Speed m/min.</th>
<th>Average Shear Stress Transducer 1 (Pa)</th>
<th>Average Shear Stress Transducer 2 (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>23.7</td>
<td>3.3</td>
<td>50.0</td>
<td>53.2</td>
</tr>
<tr>
<td>2</td>
<td>11.9</td>
<td>1.61</td>
<td>33.0</td>
<td>24.5</td>
</tr>
<tr>
<td>3</td>
<td>29.8</td>
<td>4.0</td>
<td>59.0</td>
<td>63.0</td>
</tr>
<tr>
<td>4</td>
<td>15</td>
<td>2.0</td>
<td>37.7</td>
<td>41.3</td>
</tr>
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<td>5</td>
<td>7.49</td>
<td>1.01</td>
<td>17.8</td>
<td>18.8</td>
</tr>
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<td>6</td>
<td>3.75</td>
<td>0.51</td>
<td>12.1</td>
<td>12.1</td>
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<tr>
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<td>9.43</td>
<td>1.27</td>
<td>25.9</td>
<td>26.4</td>
</tr>
<tr>
<td>8</td>
<td>11.9</td>
<td>1.27</td>
<td>24.1</td>
<td>24.1</td>
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<tr>
<td>9</td>
<td>11.9</td>
<td>1.61</td>
<td>23.1</td>
<td>23.4</td>
</tr>
<tr>
<td>10</td>
<td>29.8</td>
<td>4.0</td>
<td>59.3</td>
<td>63.9</td>
</tr>
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<td>11</td>
<td>18.8</td>
<td>2.54</td>
<td>45.4</td>
<td>49.7</td>
</tr>
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<td>12</td>
<td>18.8</td>
<td>2.54</td>
<td>41.2</td>
<td>45.8</td>
</tr>
<tr>
<td>13</td>
<td>1.88</td>
<td>0.25</td>
<td>14.2</td>
<td>10.65</td>
</tr>
<tr>
<td>14</td>
<td>18.8</td>
<td>2.54</td>
<td>46.9</td>
<td>48.46</td>
</tr>
<tr>
<td>15</td>
<td>29.8</td>
<td>55.0</td>
<td>59.5</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>29.8</td>
<td>51.5</td>
<td>52.4</td>
<td></td>
</tr>
</tbody>
</table>

Note: $\tau_a = (V_m \cdot A) / \ell$
FIGURE 7.8: AVERAGE SHEAR STRESS AGAINST PLUG SPEED FOR TRANSUDER 1 (1.785 m FROM THE DISCHARGE END)
FIGURE 7.9: AVERAGE SHEAR STRESS AGAINST PLUG SPEED FOR TRANSDUCER 2 (3.785 m FROM THE DISCHARGE END)
stand or if they were corrected for the small error introduced by assuming a very small sheared layer, support the hypothesis of section 7.2.

One distinct feature of Figure 7.7 is that a considerable reduction in peak pressure occurred before the pressure gradient/time curve reached an equilibrium state when the pipe was full. This phenomenon was thought to be caused by the leading portion of the material in the pipe being drier than the bulk material. Hence, with resulting higher shear stresses a higher pressure gradient was required to push it through the pipe. The leading edge of the material was expected to be drier than the bulk material because water would be drawn from it to wet the inner surface of the dry pipe used. To test this argument, a few tests were carried out with a small amount of water in front of the cement slurry. A typical result is shown in Figure 7.10, which shows in contrast with Figure 7.7, that the water lubrication had eliminated the reduction in peak pressure gradient.

7.5.2: Fresh Material in the Vertical Pumping Line and in the Co-axial Cylinder Viscometer

Seven tests were carried out using the Weissenberg Rheogoniometer, and seven using the vertical pumping line, three of which were performed with the 19.54mm diameter pipe and four with the 34.6mm diameter pipe. Figure 7.11 shows a typical shear stress-time curve obtained using the Weissenberg Rheogoniometer, the shear stresses being determined at the bob surface as in section 7.5.1. Figure 7.11 is significantly different from Figure 7.5 for the agitated material, the difference being due to the rapid build up of structure at early ages after mixing. Figure 7.12 shows a typical pressure-time curve for the 19.54mm diameter pipe. It can be seen that most of the pressure in this figure is due to the head of the slurry and little to wall friction in comparison with the agitated material (Figure 7.7). Plug speed and the average shear stresses were calculated using the procedure described in section 7.5.1. The results are plotted in Figure 7.13 and 7.14 in which the pipe shear stresses are generally greater than those from the Weissenberg Rheogoniometer. Although the percentage errors are relatively large the numerical difference between the different techniques of measuring shear stresses are small
SMALL AMOUNT OF WATER WAS PUMPED IN FRONT OF THE CEMENT PLUG WITH TRANSDUCER 1. PUMPING SPEED = 3.0 m/minute. PIPE DIAMETER = 19.54 mm.

FIGURE 7.40: TYPICAL PRESSURE - TIME CURVE FOR THE ACTIVATED MATERIAL OBTAINED

Weighgt pressure

TIME (s)

PRESSURE (kPa)
Figure 7.44: Typical shear stress - time curve for the fresh material obtained with the Wessex Rhesus Rheogoniometer at rotation speed of 15 r.p.m.

Shear stress at the surface of the bob (Pa)

Time (min)
WITH TRANSDUCER 1, PUMPING SPEED = 4.2 m/minute, PIPE DIAMETER = 19.54 mm

FIGURE 7.12: TYPICAL PRESSURE - TIME CURVE FOR THE FRESH MATERIAL OBTAINED

<table>
<thead>
<tr>
<th>TIME (s)</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>PRESSURE (kPa)</td>
<td>0</td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>20</td>
<td>25</td>
<td>30</td>
</tr>
</tbody>
</table>

Weight pressure
FIGURE 7.13: AVERAGE SHEAR STRESS AGAINST PLUG SPEED FOR TRANSDUCER 1 (1.785 m FROM THE DISCHARGE END) USING THE FRESH MATERIAL
FIGURE 7.14: AVERAGE SHEAR STRESS AGAINST PLUG SPEED FOR TRANSDUCER 2 (3.785 m FROM THE DISCHARGE END) USING THE FRESH MATERIAL
CHAPTER 7

when the low magnitude of shear stress is considered. The pressure drops produced are particularly small when compared with the hydrostatic pressure produced by the material weight (see Figure 7.12) which increases the percentage error in the frictional pressure drop measurement. Another source of error could have been the end effect which could be large compared with the small frictional pressure drops. These two sources of errors would be reduced for larger pipe lengths.

7.5.3: Fresh Material in Inclined Pumping Line and in the Co-axial Cylinder Viscometer

The inclined pumping line was used to increase the pipe length since the vertical pipe length was limited to the laboratory height. Pressure transducers were placed at 1.785m (Transducer 1), 3.785m (Transducer 2) and 13.0m (Transducer 3) from the discharge end. Four tests were carried out at speeds of 2.6m/min using the 34.6mm diameter pipe, and four using the Weissenberg Rheogoniometer at a plug speed of 2.6m/min. The results are plotted in Figure 7.15. Again the results from transducers 1 and 2 which were nearest to the open end produced values of shear stress which were slightly higher than those obtained from the Weissenberg Rheogoniometer. Transducer 3, however, produced slightly lower values of shear stress to that obtained with the Weissenberg Rheogoniometer. These results give support to the argument in section 7.5.2, but, as can be seen from Figure 7.15, none of these values of shear stress are statistically different which suggests that the rather wide confidence limits in the values of shear stress are due to random variability of the material or small experimental errors.

7.5.4: Results from Literature

Average velocities did not exceed 4m/min in this work and in order to test the hypothesis of section 7.2 at higher speeds, data from literature was employed. Unfortunately, the literature did not contain data from co-axial cylinder viscometers where the bob had been rotated in a sea of fluid simultaneously with pipe flow experiments. However, results from varying pipe sizes were obtained and utilised to test the hypothesis. Figures 7.16, 7.17 and 7.18 are
inclined pump line X
co-axial cylinder viscometer 0

bars indicate 95% confidence limits

FIGURE 7.15: AVERAGE SHEAR STRESS FOR TRANSDUCER 1, 2 AND 3 OBTAINED WITH THE INCLINED PUMP LINE AND THE CO-AXIAL CYLINDER VISCOMETER. PLUG SPEED = 2.6 m/min.
FIGURE 7.16: WALL SHEAR STRESS AGAINST MEAN FLOW VELOCITY FOR CEMENT ROCK SLURRY OF 54.3% ROCK BY WEIGHT (WILHELM et al (1939))
FIGURE 7.17: WALL SHEAR STRESS AGAINST MEAN FLOW VELOCITY FOR CLAY SLURRY OF 40% BY WEIGHT SOLIDS (KENCHINGTON [1978])
FIGURE 7.18: WALL SHEAR STRESS AGAINST MEAN FLOW VELOCITY FOR CLAY SLURRY OF 34% BY WEIGHT SOLIDS (KENCHINGTON (1974))
plots of shear stress against mean speed from three different papers. The mean speed was used instead of plug speed because plug speed cannot be estimated from the information given. However, as pointed out in section 7.5.1 the average speed should give a good approximation. Figure 7.16 gives a very good comparison between wall shear stress and flow velocity for various pipe diameters, even in the region where the material is thought (Hedstrom (1952)) to be in the turbulent flow. The comparison between wall shear stress and flow velocity in Figures 7.17 and 7.18 is not as good as in Figure 7.16 and the scatter of values was much larger. Close inspection of the data shows that no trend with pipe size can be found and the points spread randomly, which suggests a large experimental or material variability. These references therefore further support the hypothesis presented in section 7.2.

7.6: Pressure to Start or Re-start Flow after a Stoppage

One of the most critical problems in cementing is the problem of starting flow after a deliberate or accidental shutdown. It is important to be able to predict the pressure needed to re-start flow so that a suitable pump can be provided. Prediction of this pressure is particularly important with highly thixotropic materials owing to the rapid build up of gel structure. The problem involves not only the flow of material in parallel sided pipes and concentric annuli but also shearing at section changes and in eccentric annuli. However, only the prediction of minimum pressure to start flow in parallel sided pipes will be discussed here, although the technique could be easily adapted to concentric annuli.

7.6.1: Theory

The method described in section 7.3 should be still valid in this case. The average shear stress over the length of the pipe will be equal to the shear stress at start-up (gel strength) ($\tau_0$), $\tau_0$ is also equal to the value of shear stress at time equal to zero on the shear stress-time curve recorded at the surface of the bob in co-axial cylinder viscometer. Substituting $\tau_0$ in Equation 7.16 gives the pressure to start flow ($P_s$) as:
The pressure predicted by Equation 7.23 is correct only if the inertia effects in both the co-axial cylinder viscometer and the pipe are negligible, and assuming that the flow in the pipe length occurs simultaneously at all points. The inertia effect can only be ignored if flow is slow and if the co-axial cylinder viscometer's torque measuring system is sufficiently stiff. If this is not the case then inertia effects must be included. Inertia effects in the pipe are dependent on speed of flow, weight of material, and time taken to initiate flow which is dependent on the rigidity of the material. A typical calculation for the effect of inertia on start-up pressure in a tube is given in Appendix 7.1 from which it can be seen that in this case the effect was negligible. With the co-axial cylinder viscometer the effect of inertia is limited to that of the bob and torque measuring system, the effect of the inertia of the relatively heavy bob tending to delay the build-up of maximum torque with the possibility of overshoot. However, the Weissenberg Rheogoniometer is designed, as described in Chapter 4, to have small inertia effects and they were therefore ignored.

7.6.2: Experimental Techniques to Measure Start-up Pressures

All the material used was mixed according to the mixing procedure described in Chapter 4. The mix proportions have remained constant throughout the investigation but the quantities and the experimental procedures have varied with the needs of each set of tests. The tests were divided into three sets.

7.6.2.1: Gun Rheometer

For this set of tests, approximately 600ml of material was mixed in the 1l container of the Waring Commercial Blender. Tests were carried out in two stages.
Stage 1.

Smooth glass tubes of four different diameters (3.25mm, 4.9mm, 7.8mm and 10.1mm) and 700mm length were used. One minute after mixing the tubes were filled with cement slurry by suction using a vacuum pump. The tubes were stored vertically for times of 30 and 54 minutes after mixing. After storage, they were attached to the gun rheometer pressure vessel, starting from zero, the pressure was increased slowly until the slurry in the tube started to flow at the downstream end of the tube.

Stage 2

An internally threaded (1mm deep by 1mm spacing) steel tube of 5.2mm diameter and 550mm length was filled with cement slurry straight after mixing. The tube was then stored vertically for 30 minutes and then tested as in stage 1.

7.6.2.2: Mono Pump

Four mixes of approximately four litres were prepared sequentially using the four litre container of the Waring Commercial Blender. The slurry was then transferred to the Mono pump hopper, the pump being turned on after the third mix was ready which was approximately 10 minutes from the start of the mixing operation. A speed of 2.6 m/min. was used to fill the 13.5m length inclined pipe described in section 7.4 and at least 3m of material was allowed to discharge before the pump was turned off. Exactly 30 minutes from the start of mixing, the pump was turned on again at speed of approximately 0.5m/min. The pressure to start flow was then recorded at 1.785m (transducer 1), 3.785m (transducer 2) and 13.0m (transducer 3) from the discharge end. The test was repeated four times.

7.6.2.3: Instron Piston Pump

Two steel tubes of 10.55mm and 19.54mm diameter were used in this set of tests. The test section of the tube was attached horizontally to the vertical plunger section (see Figure 4.13). Filling the tube was achieved by pouring the material into the plunger section until
the required length was full. The plunger was then pushed down by the Instron cross-head, until at least a half-metre of cement slurry was discharged. Thereafter the Instron head was stopped and the material was left to stand for 20 minutes before the Instron was switched on again to push the plunger down. Meanwhile the Weissenberg Rheogoniometer was filled from the same material, the bob, cup and procedure being described in section 7.4.3. The material near the surface of the bob was preconditioned by rotating the outer cylinder at the equivalent linear speed used to fill the tubes and for a time equal to that used to fill half of the test section of the tube. The material was then left to stand for 20 minutes before the outer cylinder was rotated so that the plug linear speeds in both the co-axial cylinder and the tube were equal. For this set of tests, approximately two litres of material was mixed and agitated after mixing to eliminate the small amount of bleed water which would otherwise have been present. For some tests (non-de-aired material) the slurry was agitated in the Crypto Peerless mixer for 25 minutes. For other tests (de-aired material) the slurry was transferred to a vacuum pump flask (-700mm of mercury) and de-aired for 5 minutes before being agitated in the Crypto Peerless mixer for 10 minutes. Further de-airing for 8 minutes was carried out, the flask of the vacuum pump being continually tapped in an attempt to remove any air retained during the agitation process. However, it was found that re-agitation of the material was necessary to break down the gel structure formed during the de-airing time so that the material could be poured into the plunger section. Thus the material was re-agitated for a further 2 minutes. The pumping speed and tube lengths used will be given later with the results.

7.6.3: Results and Discussion on Pressure to Start Flow in Pipes

7.6.3.1: Gun Rheometer

Figure 7.19 shows a plot of the measured gel strength against tube diameter for standing times of 30 min. and 54 min. after mixing (A full summary of the results is given in Appendix 7.2 and 7.3). Although the means of the gel strength for for standing times of 30 min. measured with the different tube radii has increased with the increase in tube diameter, none of these gel strengths are statistically different, calculated at a 95% confidence level.
Internally threaded steel tube _ 30 min. standing time X
Smooth glass tube _ 30 min. standing time Δ
Smooth glass tube _ 54 min. standing time 0

FIGURE 7.19: MEAN OF GEL STRENGTH AGAINST TUBE DIAMETER MEASURED WITH THE GUN RHEOMETER
However, for the 54 minutes standing time, the mean of $\tau_0$ corresponding to the 3.25mm diameter tube is found to be statistically different from all the $\tau_0$ measured with other tubes at a confidence level of 95%.

The mean of gel strength for 54 minutes standing time measured with the smallest (3.25mm diameter) and the largest (10.1mm diameter) tubes were chosen for further statistical analysis since these two tubes provide the biggest difference between the measured mean gel strength. A student "t" test was carried out, in which the calculated $t$ value was 3.482 against 2.977 for a 99% confidence level. This shows that the mean gel strength $\tau_0$ measured with the 3.25mm and the 10.1mm diameter tubes are statistically different at a confidence level higher than 99%. The reason for this difference may be related to two factors:

a) Compression of Air Bubble in the Slurry

While increasing the pressure, the upstream end of the slurry in the tubes was observed to move while the downstream end was stationary. This movement was attributed to the compression of the air bubbles in the system by the applied pressure. The compression effect would be increased with the smaller diameter tubes which theoretically required a higher pressure to initiate flow (Equation 7.23) if all the slurry slipped simultaneously. Consequently, since the shear strain required to break the structure of cement slurry is small, probably less than $10^{-4}$ radians, (Hannant and Keeting (1985)), flow in the tube was probably initiated locally at the higher pressure end of the tube by progressive failure due to the local straining which occurred as the air bubbles compressed in the slurry. Calculation of gel strength (Equation 7.24), however, is based on the assumption of simultaneous slip over the entire tube length. This observed effect of local failure was the most likely explanation for the difference between tube sizes and was further examined in section 7.6.3.2.

b) Slippage Due to Wall Anomaly at the Smooth Surface of the Glass Tubes

This is a very likely problem with cement slurries but there is no reason why the material should slip at different shear stresses in
different size tubes. In order to check on the existence of a slip layer, tests with the internally threaded tube described in section 7.6.2.1 were carried out. The results from this tube are tabulated in Appendix 7.4 and plotted on Figure 7.19 for comparison with the smooth tube results. As can be seen from Figure 7.19 the average gel strength measured with the threaded tube are more than twice those measured with the smooth tubes which could be explained by the existence of greater slippage at the surface of the smoother tubes. However, a shorter length tube was used with the threaded tube (550mm compared with 700mm) which could account for a small part, but not all of the differences in the measured gel strength.

7.6.3.2: Test Using De-aired and Non-de-aired Mixes in the Mono and Instron Piston Pumps

Progressive failure due to compression of failure was seen to exist visually, but, a more controlled test, where the pressure and flow rate could be monitored or fixed, was needed to quantify the problem. The Mono pump with continuous pressure monitoring was first used to pump the slurry at a fixed pump discharge rate. However, after performing four tests, it became apparent that the discharge from the pump was fluctuating and not steady as was required. Figure 7.20 shows typical pressure-time curves from transducer 1 and 2 (1.785m and 3.785m from the discharge end). These curves demonstrate clearly that the material has broken down in steps corresponding to the pump delivery stages implying progressive failure initiated near the pump. Although this form of breakdown demonstrates clearly the existence of progressive failure along the tube length, the fluctuating output of the pump was thought to be unsuitable for a comprehensive study of the progressive failure phenomenon. Consequently, the Instron piston pump was designed to provide a constant flow velocity as described in section 7.6.2.3.

The detailed results obtained with the piston pump are shown in Appendix 7.5 to 7.7 and are plotted in Figure 7.21 and 7.22. These figures also include the results obtained with the co-axial cylinder viscometer for comparison, see section 7.6.3.3. Figure 7.23 and Figure 7.24 show typical pressure time curves for the non-de-aired and de-aired materials recorded at various tube length. From Figure 7.23
PRESSURE (TRANSDUCER 1) (kPa)

Pumping speed = 0.5 m/minute

Figure 7.20: Typical pressure-time curve recorded by transducer 1 and 2 using the inclined pumping line and with the mono pump
Bars indicate 95% confidence limits

10.55 mm diameter tube X
19.54 mm diameter tube △
Co-axial cylinder viscometer ○

FIGURE 7.21: MEAN OF GEL STRENGTH AGAINST TUBE LENGTH MEASURED WITH THE PISTON PUMP USING NON-DE-AIRED MATERIAL. THE FIGURE ALSO SHOWS THE MEAN OF GEL STRENGTH MEASURED WITH THE CO-AXIAL CYLINDER VISCOMETER. Plug speed = 100 mm/minute
FIGURE 7.22: MEAN OF GEL STRENGTH AGAINST TUBE LENGTH MEASURED WITH THE PISTON PUMP USING DE-AIRED MATERIAL. THE FIGURE ALSO SHOWS THE MEAN OF GEL STRENGTH MEASURED WITH THE CO-AXIAL CYLINDER VISCOMETER.

Plug speed = 100 mm/minute

Bars indicate 95% confidence limits

10.55 mm diameter tube X
Co-axial cylinder viscometer O
(A) Tube length = 0.515 m: Maximum pressure = 36 kPa
(B) Tube length = 1.630 m: Maximum pressure = 58 kPa
(C) Tube length = 3.785 m: Maximum pressure = 55 kPa

FIGURE 7.23: TYPICAL PRESSURE - TIME CURVES FOR FLOW START UP OBTAINED WITH THE NON-DE-AIRED MATERIAL AND USING THE PISTON PUMP. FLOW SPEED = 100 mm/min. TUBE DIAMETER = 10.55 mm
(A) Tube length = 0.515 m : Maximum pressure = 10.5 kPa  
(B) Tube length = 1.630 m : Maximum pressure = 34.5 kPa  
(C) Tube length = 3.630 m : Maximum pressure = 68 kPa

FIGURE 7.24: TYPICAL PRESSURE - TIME CURVES FOR FLOW START UP OBTAINED WITH THE DE-AIRED MATERIAL AND USING THE PISTON PUMP
FLOW SPEED = 107 mm/min. TUBE DIAMETER = 10.55 mm
it can be seen that for the non-de-aired material the time interval between zero and peak pressure has varied from about 3 seconds in Figure 7.23A to 16 seconds in Figure 7.23C while the maximum pressure has only doubled with an increased tube length of more than seven times. The curve corresponding to the 0.515m tube length has shown approximately a linear increase in pressure while with the other tube lengths the pressure-time curves have tended to curve towards the time axis. These effects could be explained if progressive failure is the dominant mode. In contrast in Figure 7.24, the pressure-time curve shape for de-aired material has shown little dependency on the tube length with all tubes reaching the maximum pressure in less than 2 seconds and with the maximum pressure increasing approximately in proportion to tube length. This indicates that progressive failure has been eliminated by the removal of air. The large difference between the pressures displayed in Figure 7.23 and 7.24 emphasise the importance of the experimental procedure.

The progressive failure phenomenon is further supported by the results displayed in Figure 7.21 and 7.22. Figure 7.21 shows a reduction in the gel strength ($\tau_0$) with the increase in tube length and the reduction in tube diameter. In contrast, in Figure 7.22, tube length has shown little influence on the gel strength for the de-aired material. These results could again be explained if progressive failure is the dominant mode.

The above results prove conclusively the existence of progressive failure with the non-de-aired material and indicate that the problem can be eliminated with the de-aired material. Thus, the practical conditions in the borehole where the air bubbles would most likely collapse under the high hydrostatic pressure, can be simulated in a low pressure pumping line if de-aired material is used. However, in this investigation relatively short steel tubes were used. In practical situations, much longer pipe lines are normally utilised and, hence, the pressure to start-up flow would be much higher than those encountered in this investigation. Consequently, progressive failure may still occur if the applied pressure is high enough to cause adequate compression of the cement slurry for progressive failure to occur. Furthermore, in some other practical situations, flexible tubes are used. These tubes expand under pressure and may therefore cause progressive failure.
7.6.3.3: Prediction of Start-up Pressure from Co-axial Cylinder Viscometer Results

A typical shear-stress-time curve for the co-axial cylinder viscometer is shown in Figure 7.25. The peak value of torque was used to estimate the average shear stress (gel strength) ($\tau_o$) at pumping start-up as shown in Appendix 7.8 and 7.9. $\tau_o$ is compared in Figure 7.21 and 7.22 with those measured with the various tubes. For the non-de-aired material, Figure 7.21, $\tau_o$ measured with the co-axial cylinder viscometer is statistically different from those measured with 1.785m and 3.785m length of tubes. This difference can be explained by the progressive failure phenomenon described above. For de-aired material, Figure 7.22, the mean values of $\tau_o$ measured with the tubes are not statistically different from the value obtained with the co-axial cylinder viscometer which indicates that the viscometer results could be used to predict start-up pressures.

It can be concluded that the Weissenberg Rheogoniometer could be used to provide a prediction of the minimum pressures required to start-up the flow in pipes as long as the flow is slow so that inertia effects can be neglected and assuming that the material does not fail progressively in the tubes.

7.7: Conclusions

1) Plug flow in pipes can be predicted from similar flow in the co-axial viscometer based on the assumption that the sheared layers in both systems are similar. The experimental and analytical work involved are simple and the accuracy of the prediction is adequate for most engineering applications.

2) The minimum pressure to start flow in pipes can be predicted from the shear stress measured at the surface of the inner cylinder in the co-axial cylinder viscometer only, if the inertia effects in both systems are negligible and assuming that the material does not fail progressively in the pipe.
SHEAR STRESS AT THE SURFACE OF THE BOB (Pa)

**Figure 7.25:** Typical shear stress - time curve for the de-aired material.
CHAPTER 8

THE SHEAR VANE TEST AND THE MEASUREMENT OF GEL STRENGTH OF CEMENT SLURRIES

8.1: Introduction

Gel strength is a parameter sufficiently important to industry to warrant attention and study. Nevertheless, industry has yet to find a reliable technique to measure it. The pumpability of cement slurries is usually assessed in the drilling industry, by the so called thickening time test (API Spec. 10 (1986)). This test is a dynamic test which measures the properties of the material under simulated flow conditions. However, if pumping ceases by accidental or deliberate shutdown, most, if not all cement slurries develop a gel structure which has to be broken down before pumping can resume again. In a series of papers, Sabins and co-workers (1980), (1982), (1984) and (1986) have found no relationship between the thickening time and the development of gel strength. However, their methods for the determination of gel strength are questionable. The yield stress (a general term which includes gel strength as a special case) of viscoplastic fluids is often measured at a solid fluid interface, but, as was shown in Chapters 5, 6 and 7, a slip layer may exist at the solid surfaces. Consequently, yield stresses measured at these surfaces may not reflect the real material yield stress, but, rather the properties of the slip layer. To measure the yield stress more reliably, a technique which forces the material to shear within itself and not on solid surfaces is needed. The shear vane test commonly used in soil mechanics to measure shear strength of clays is thought to fulfil this objective, particularly for the material described in this work which was designed to be highly thixotropic through the use of special additives.

8.2: Yield Stress

8.2.1: Definition

Yield stress of a Bingham body is defined (BS5168:1975) as that stress below which the material is an elastic solid and above it a liquid with plastic viscosity up so that

\[ \tau = \tau_y + \mu \dot{\gamma} \]
where $\tau$ and $\dot{i}$ are the shear stress and shear rate respectively.

However, this definition is not adequate for thixotropic materials which breakdown after yield so that $\tau < \tau_y$.

In general this definition of yield stress is only an idealisation of more complex material behaviour below and above the yield point. In fact there is still a great deal of argument among rheologists about the nature of yield stress and how to measure it. Some authors, Barnes and Walters (1985), even argue that yield stress is not a real material parameter and it is only found due to the inaccuracy of the experimental procedure and apparatus. Hence, given accurate measurements, no yield stress exists. Lang and Rha (1980) and Cheng (1985), on the other hand, argue that yield stress is a time dependent quantity and its magnitude is dependent on the time allowed to determine whether or not the material has ceased flowing under the action of the present stresses. Practically, this is not a straightforward problem and, as Cheng (1985), explained, is often complicated by creep, stress growth and thixotropic breakdown and recovery. In the case of cement slurries which change their behaviour from liquid to solid in a few hours the above difficulties are increased. Whether the yield stress actually exist or not, it is still too important an industrial and engineering parameter to disregard. However, one should realise that it is not an absolute value and it's magnitude may vary according to the experimental or practical conditions.

Some materials including most cement slurries develop a delicate and fragile gel structure on standing. This structure is readily broken down by shearing. The shear stress measured after shearing (dynamic yield stress) is usually much smaller than that measured after prolonged rest (static yield stress). In this Chapter we will be concerned with the latter yield stress which we will refer to as gel strength to signify that it is a property of the gel structure.
Numerous methods and techniques have been implemented to measure the yield stress of viscoplastic materials. Cheng (1985) has discussed the bases of the majority of these techniques. Most techniques make use of the various types of the existing viscometers, with either the constant shear stress or constant shear rate modes. With the constant shear stress experiment the yield stress can be found by creep tests with the shear stress being increased step by step until the material response changes from a constant strain to constant strain rate. This change marks the point at which the material response changes from 'solid like' to 'fluid like behaviour (see Chapter 1, section 1.1 for definition of solids and fluids). The yield stress is the shear stress present at the point of change in the material response, and can be determined accurately by making the shear stress increment small. However, there exist practical difficulties in establishing accurately when the material response has changed from constant strain to constant strain rate, because this is dependent on the time of observation, as Cheng (1985) illustrated. With the constant shear rate experiment the shear stress-shear rate flow curve can be established which gives the yield stress as the shear stress at zero shear rate. In practice, each viscometer has a minimum shear rate below which measurements are not possible, or are inaccurate. Hence, the shear stress at zero shear rate or the yield stress can only be obtained by extrapolating the flow curve to zero shear rate. The accuracy of this technique, obviously, increases as the value of the lowest shear rate used in the experiment tends to zero. Barnes and Walters (1985) have shown that the yield stress measured with this method changes significantly as the lowest shear rate used in the experiment is reduced.

The stress relaxation method is regularly used to measure the yield stress of viscoplastic material by, for example, the analysis of the torque decay curve obtained with the rotational viscometers. The yield stress can be found by calculating the stress corresponding to the residual torque remaining after stoppage of some rotation speed. However, as Vocadlo and Charles (1971) noted, the torque decay curve may be influenced by the sum of the kinetic and potential energy of the sensing system. Various other techniques have been proposed to
measure the yield stress of viscoplastic materials using rotational viscometers, see Cheng (1985). In addition, there exist a number of techniques specifically designed to measure the yield stress such as the inclined plane, Uhlherr et al (1984), and moving objects (balls, cylinders, cones and others) in translation movement in the fluid. These devices include various kinds of penetrometers such as the Vicat apparatus and other cylindrical and conical devices.

The principal problem in the majority of the methods stated above is inherited in the design of the apparatus. The shear stress in these devices is often measured at a solid-fluid interface which may lead to apparent slippage of two phase fluids (see Chapter 5, section 5.2.2.2), particularly in the low shear rate region from which the yield stress is often extracted. Moreover, the shear strains induced in the course of the experiment are often large compared with the fracture strains of the highly delicate and fragile structure which exists in many complex suspensions. In cement paste, for instance, strains of less than $10^{-4}$ radians may be required. To eliminate or reduce slip effects, roughened surfaces have been used by many authors, but it has not yet been proved that slippage does not still occur with these surfaces. A method was proposed by Vocadlo and Charles (1971) to solve the slip problem in the co-axial cylinder arrangement. They used ribbed cylinders of various ratios of $A_s/A_g$, where $A_s$ and $A_g$ are the surface area of the ribs and grooves respectively. The true yield stress (no slip) was obtained by extrapolating the measured shear stress versus $A_s/A_g$ curve to zero $A_s/A_g$. The results from this method should correlate well with the shear vane test (described below), if one assumes that the surface area of the edges of the blades of the vane is negligible compared with the surface area of the circumscribing cylinder. The advantage of the shear vane test method over that of Vocadlo and Charles is that while one test suffices with the vane, several are required in the latter method.

8.3: Shear Vane Test

The shear vane test is simply a vane (Figure 8.1) with two or more blades immersed in the material to be tested so that when it is rotated the sheared surface of rotation is usually cylindrical. The
vane can be rotated at constant speed while the torque is monitored on the shaft holding the vane. The maximum torque recorded on the vane shaft can be used to calculate the shear strength (gel strength) of the material by using a suitable stress distribution around the vane (see section 8.3.1). A typical torque time curve obtained with the vane with a cement slurry used in this work is shown in Figure 8.2 in comparison with a smooth bob in similar slurry. It can be seen from Figure 8.2 that the maximum torque for the vane is considerably larger than for the bob even though both have the same diameter. In this work the bob in the co-axial cylinder viscometer has been replaced by a vane, so that depending on the instrument characteristics either the vane or the outer cylinder is rotated at constant speed.

A considerable amount of research has been carried out on the shear vane test in soil mechanics (see for example Skempton (1948), Cadling and Odenstad (1950), Flaate (1966), Wiesel (1973) and Merrifield (1980)). Only few papers (Keentock (1982), Keentock et al (1985), Dzuy and Boger (1983), Dzuy and Boger (1985)) have been published on the use of the vane with other gel like materials. Keentock (1982) used the vane to measure the yield stress of greases and found a good correlation between the vane results and the yield stress obtained by
ARRANGEMENT ON THE WEISSFLEBERG PHOTODIONOMETER

FIGURE 8.2: BREAKDOWN CURVE FOR CEMENT SLURRY USING THE VANE AND THE BOB

Rotation Speed = 0.1 r.p.m.
Standing Time = 15 minutes

Rotation (degrees)  7.5  5  2.5  0
Time (s)  13.5  9.0  4.5  0

Torque (N.m)

-400.0
-200.0
0
200.0
400.0
600.0
800.0
1000.0

Bob

Vane
extrapolating shear stress/shear rate curve from a cone and plate viscometer data. Keentock concluded that cone penetration and stress relaxation methods should not be used to measure the yield stress of greases, unless slip is eliminated on the surface of the cone and plate. The yield stress measured with the vane was also compared with those obtained using more traditional methods by Dzuy and Boger (1983) and (1985) for red mud suspensions. They found that the yield stress obtained using the vane is identifiable with the true yield stress, particularly in the highly concentrated systems. As described above in section 8.2.2 and noted by Keentock and Dzuy and Boger, the traditional techniques to measure the yield stress suffer from serious problems and therefore little can be achieved by comparing the yield stresses measured by these techniques and that obtained with the vane test which was introduced to overcome the problems inherent in the traditional techniques.

Direct observation made in this work and others (see Cadling and Odenstad (1950)) shows that the material shears near to the circumscribing surface of the vane. However, to find out what the vane actually measures, we must study the following:

i) The stress distribution around the vane.
iii) The effect of nominal rotation speed and measuring system stiffness.
iv) The effect of vane dimensions and shape.

8.3.1: Stress Distribution and Progressive Failure Effects

Progressive failure is a term used to describe the progressive breakdown of a shear sensitive material when subjected to differential shear stress or strain and, as this is closely related to the stress distribution, these factors are discussed together. Two forms of progressive failure may arise with the vane when rotated in a shear softening material. Firstly, progressive failure due to the strain at the horizontal edge of the vane differing from that at the vertical. Secondly, progressive failure due to the progressive breakdown of the material in front of the blades of the vane.
CHAPTER 8

The work of Cadling and Odenstad (1950) has shown that up to failure a uniform distribution of strain was present around the vane and the surface of rupture is cylindrical. Hence, one could conclude that progressive failure of the material in front of the vane is non-existent. Nevertheless, with the cement slurry used in this work where approximately 5% of air may be retained in the mix, a gradual breakdown of the material may occur due to the compression of the air bubbles, (see section 8.5.2).

The classical shape of the stress distribution around the vane is the rectangular stress distribution, Figure 8.3a, which describes the behaviour of an ideal plastic material i.e. the material does not undergo any strain until the yield point is reached, and when yielded the material undergoes no strain hardening or strain softening characteristics. This ideal behaviour of the material seldom exists in practice although some materials may be approximated to behave in this manner. The relationship between maximum torque $T$ on the shaft of the vane or bob and the gel strength $\tau$ is conventionally given by Equation 8.1 (Duzy and Boger (1985)).

$$T = \left(\pi D^3/2\right) (H/D + 1/3) \tau$$

...(8.1)

where $D$ and $H$ are the diameter and the height of the vane respectively.

Other types of stress distribution have been used such as triangular (Figure 8.3b) or parabolic or exponential but the most appropriate distribution to use in each case will depend on the individual material characteristics. To explain this point, consider the idealised stress-strain models shown in Figure 8.4. Materials of types (1) and (2) in Figure 8.4 would mobilise approximately the same stress after initial yield whatever the strain, hence the rectangular stress distribution is the one to use.

For material of type (3) the stress distribution is slightly more complicated. When the stress at the vertical edge reaches its maximum value, the stress at the horizontal one would be triangular (Figure 8.3b), and further straining would reduce the stress at the vertical
(a) Rectangular stress distribution
(b) Triangular stress distribution

FIGURE 8.3: STRESS DISTRIBUTION ASSUMED TO MOBILISE AROUND THE VANE

FIGURE 8.4: IDEALISED STRESS-STRAIN MODELS
edge. However, at the horizontal edge the stress at any radius will continue to increase up to the yield value after which it will reduce. This will produce a stress distribution which lies between the rectangular and triangular stress distribution. The closeness of fit to either of them will depend on the relative slopes of strain hardening-strain softening lines, $K_1$ and $K_2$ in Figure 8.5a.

Merrifield used the rectangular stress distribution and produced correction factors for progressive failure based on the idealised stress-strain model of Figure 8.5a. For a cylindrical vane he plotted the progressive failure correction factor against the stiffness ratio $K_1/K_2$ as shown in Figure 8.5b. It can be seen from Figure 8.5b that the effect of progressive failure reduces with the increase of $H/D$ ratio. For most sensitive materials, the effect of progressive failure is about 4% for $H/D$ ratio of 2 (recommended in soil mechanics). This effect can be reduced to 2% if the $H/D$ ratio of 4 is used. The effect of using vanes with the same diameter but different height is given in section 8.5.2.

Material of type (4), Figure 8.4, would produce a stress distribution which lies between that of type (2) and type (3), so that the maximum error produced by assuming a rectangular stress distribution is smaller than 4%. For cement paste used in this work which has a material behaviour like that shown earlier in Figure 8.2 the model can be approximated to types (2) and (4), Figure 8.4. It can be shown that if the $H/D$ ratio is equal to or greater than 2 the assumption of a rectangular stress distribution would produce a very small error (less than 2%) which is well within the variability of the material.

To prove this point Dzuy and Boger (1985) have resorted to a comprehensive experimental investigation where values of $H/D$ between 0.95 and 3 were used. However, using the curve of torque versus angle of rotation presented in their paper and, following the argument presented above, it can be shown that the error of assuming a rectangular stress distribution will be very small (a few percent in the most extreme cases) which, most likely, lies within the material variability. This makes questionable the merits of performing such an investigation.
FIGURE 8.5: (a) IDEALISED STRESS-STRAIN CURVE FOR STRAIN SOFTENING MATERIAL
(b) PROGRESSIVE FAILURE FACTOR VERSUS $K_1/K_2$ FOR CYLINDRICAL VANE (MERRIFIELD (1980))
It must be emphasised that the stress distributions inferred are hypothetical and in practice one would expect the stress distribution to vary along both the vertical and horizontal edges of the surfaces of the vane. This was shown to be true by Merrifield (1980). Merrifield used a specially designed vane to measure the shear stress at different points around the vane as function of time while the vane is rotated at constant speed. Merrifield's work has shown a nearly uniform stress distribution along the vertical edge of the vane with a stress concentration in the vicinity of the blade corners. On the horizontal ends of the vane, the stress distribution has been found, as expected, to be dependent on the type of the material used and to vary from a maximum value to zero along the edge.

8.3.2: Effects of Vane Rotation Speed and Measuring System Stiffness

It is well established, in soil mechanics, that rotational speed affects the measured shear strength of soils. The results of Cadling and Odenstad (1950) give roughly 20% increase in shear strength with an increase in rotation speed from 0.1°/s to 1°/s. Skempton (1948) found the increase in shear strength with the increase in rate of straining (rotation speed) to vary in the same manner as the compression test results. The results of Skempton (1948), and Cadling and Odenstad (1950) were confirmed by Wiesel (1973) who found the shear strength to vary with rotation speed according to a power law function. Consequently a standard speed of 0.1°/s has been adopted to obtain comparative results in the industry. For other materials the effect of rotation speed on the gel strength has not been studied to the same degree as that for the soils. Dzuy and Boger (1983) and (1985) carried out vane tests with the Haake viscometer on red mud suspensions. They used rotation speeds of 0.1 r.p.m. to 256 r.p.m. and found the measured gel strength (yield stress in their terminology) to be constant for rotational speeds of 0.1 r.p.m. to 8 r.p.m. These results seem to contradict the soil mechanics data and the findings of Barnes and Walters (1985). However, Dzuy and Boger did not appreciate that the actual rotation speed of the vane is not necessarily determined only by the setting on the dial of the instrument but, could be influenced by many other factors such as the instrument stiffness and material rheological behaviour. In order to measure torque, most rotational viscometers use a spring system which
in itself has to be rotated in order to increase and measure the torque. This together with the material deformation before flow makes the true or effective rotation speed of the vane through the material before yielding quite different from the set rotation speed of the motor. The implication of these remarks when considering the three instruments used in this investigation are as follows:

A relatively soft system such as a Fann viscometer with a stiffness of $38.7 \times 10^{-6} \text{ Nm/degree}$ would require a rotation of $51.7^\circ$ before reaching a torque of $2000 \times 10^{-6} \text{ Nm}$ — a typical value for peak torque in thixotropic cement. This would take $1.44$ seconds at $6 \text{ r.p.m}$ nominal rotation speed. A similar calculation for the Contraves Rheomat 15 and the Weissenberg Rheogoniometer with measuring system stiffnesses given in Table 8.1 gives the time to failure as $4.25$

Table 8.1: Effect of Instrument Torque Measuring System Stiffness on Time Before Failure.

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Rheomat 15</th>
<th>Fann Viscometer</th>
<th>Weissenberg Rheogoniometer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measuring system stiffness</td>
<td>$13.08 \times 10^{-6}$</td>
<td>$38.7 \times 10^{-6}$</td>
<td>$167200 \times 10^{-6}$</td>
</tr>
<tr>
<td>Twist before failure (degree)</td>
<td>$152.9$</td>
<td>$51.7$</td>
<td>$0.012$</td>
</tr>
<tr>
<td>Time to achieve required twist (s)</td>
<td>$4.25$</td>
<td>$1.44$</td>
<td>$0.00033$</td>
</tr>
<tr>
<td>Total time before failure (s)</td>
<td>$4.2$</td>
<td>$1.46$</td>
<td>$0.02$</td>
</tr>
</tbody>
</table>
seconds and 0.00033 seconds respectively. It must be stressed that the times to failure calculated above are only the times taken to rotate the vane by the required amount to generate the failure torque. Hence, these times are true only if the material is perfectly rigid. In practice, all viscoplastic materials will show some deformation before yielding. Hence the real time to failure is the time calculated above plus an extra time to produce the rotation required to achieve the yielding strain. A total time to failure of 0.02 second was measured with the Gould Digital Storage Oscilloscope, Type 4035, which was connected to the Weissenberg Rheogoniometer for the material used to compare the results from the aforementioned instruments. This 0.02 seconds can be considered as the time taken to deform the material to the yielding strain, since the time to turn the vane to produce the failure torque is very small with the Weissenberg Rheogoniometer, as can be seen in Table 8.1. Therefore the total times to failure with the Weissenberg Rheogoniometer, Fann Viscometer and the Contraves Rheomat 15 are 0.02, 0.02 + 1.52, 0.02 + 4.33 second respectively.

The exact rate of straining cannot be calculated easily with the vane geometry because the thickness of the sheared layer is not well defined. However, the relative rate of straining in the three instruments used will be in inverse proportion to the total times before failure in Table 8.1, i.e. the rate of straining up to yield point of the Weissenberg Rheogoniometer will be nearly seventy times greater than that of the Fann-Viscometer although they were both nominally rotated at 6 r.p.m

It can therefore be seen from Table 8.1 that the stiffness of the instrument measuring system can alter the real (as opposed to instrument setting) strain rate to first flow by several orders of magnitude and it would be expected, therefore, that the stiffer systems such as the Weissenberg would give a higher value for the gel strength at the same instrument setting speed.

8.3.3: Effects of Shape of the Vane

The cylindrical vane is the most common vane shape, though other shapes such as the diamond have also been used in soil mechanics to
CHAPTER 8

study anisotropy. However, if the study of anisotropy is not needed, the use of non-cylindrical vanes is not recommended for two reasons. Firstly, they increase the effect of progressive failure, the error caused by the diamond shaped vane may reach 32%, Merrifeild (1980). Secondly, the variation of radius with non-cylindrical vanes produces a variation in linear speed at the outside edges of the vane which may affect the measured gel strength as described in section 8.2.2.

8.4: Experimental Techniques
8.4.1: Mixing

Approximately 600ml of material of standard proportion, were mixed according to the standard procedure given in Chapter 4.

8.4.2: Co-axial Cylinder Viscometer

Three different types of viscometer were used known as the Fann Viscometer, the Weissenberg Rheogoniometer and Contraves Rheomat 15. The same procedure was followed for tests using a bob or vane. Immediately after mixing, the cement slurry was transferred to the container of the viscometer and the bob/vane was lowered and immersed completely in the cement slurry 13mm below the surface. Depending on the instrument used the outer cylinder or the bob/vane was rotated at constant speed after a given standing time.

Various vanes and outer cylinders were used. These are described in Table 8.2.

8.5: Results and Discussion
8.5.1: Comparison Between the Bob and the Vane

The results obtained using the vane technique were compared with those obtained using the smooth bob (inner cylinder) on the co-axial cylinder viscometer in order to demonstrate the large effect which slip layers have on the results from the bob. In this set of tests, the co-axial cylinder arrangement on the Weissenberg Rheogoniometer was used with the cup (outer cylinder) C2 (Table 8.2). For the vane test the bob was replaced by a vane V4 (Table 8.2). The dimensions of the vane and bob, and the rotation speed of the cup were exactly the
same in both tests. The results are presented in Table 8.3 which shows that the value of the measured gel strength obtained using the vane was much higher than that obtained using the bob. The large

Table 8.2: Vanes and Cups Details

<table>
<thead>
<tr>
<th>Vane</th>
<th>Height (mm)</th>
<th>Diameter (mm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>V1</td>
<td>15</td>
<td>15</td>
<td>Six blades</td>
</tr>
<tr>
<td>V2</td>
<td>25</td>
<td>15</td>
<td>..</td>
</tr>
<tr>
<td>V3</td>
<td>35</td>
<td>15</td>
<td>..</td>
</tr>
<tr>
<td>V4</td>
<td>38.4</td>
<td>20.8</td>
<td>..</td>
</tr>
</tbody>
</table>

Cup

<table>
<thead>
<tr>
<th>Cup</th>
<th>Height (mm)</th>
<th>Diameter (mm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>58</td>
<td>36.8</td>
<td>Serrated</td>
</tr>
<tr>
<td>C2</td>
<td>110</td>
<td>96</td>
<td>Smooth</td>
</tr>
</tbody>
</table>

Table 8.3 Gel Strength Obtained with the Co-axial Cylinder Arrangement of Weissenberg Rheogoniometer

- Standing time = 15 minutes
- Nominal Rotation Speed = 0.1 r.p.m.

<table>
<thead>
<tr>
<th>Attachment</th>
<th>Vane</th>
<th>Bob</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of results</td>
<td>7</td>
<td>7</td>
</tr>
<tr>
<td>Arithmetic mean (Pa)</td>
<td>154.5 ± 13 *</td>
<td>65.6 ± 7.1 *</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>14.06</td>
<td>7.68</td>
</tr>
</tbody>
</table>

* 95% confidence limits.
difference between the two sets of results may be explained by the slippage which probably occurs on the smooth surface of the bob. This is supported by the breakdown curve of cement paste obtained by both techniques, as shown in Figure 8.2. With the bob, the material had a small elastic deformation followed by an exponential form of breakdown curve. However, with the vane, the material continued to deform without the torque reducing until relatively large rotation. The peak elastic stress obtained using the bob was much less than that obtained using the vane, which indicates that slip occurred before the full elastic deformation of the material had taken place.

8.5.2: Effect of Vane Size and Slurry Air Content on Progressive Failure

In section 8.3.1 two types of progressive failure were discussed. These were: Progressive failure due to the progressive breakdown of the material in front of the blades of the vane and progressive failure due to the differential straining between the horizontal and vertical edges of the vane. The cement slurry used in this work contained approximately 5% of air which caused the mix to compress when subjected to a relatively small pressure as shown in Chapter 7. Hence, since the shear strain required to break the structure of cement slurry was small, probably less than $10^{-4}$ radians, Hannant and Keating (1985), the effect of having air in the mix may have caused the material to compress, and hence to fail progressively, in front of the vane blades. This effect would reduce the maximum torque on the vane shaft. The results, Figure 8.6, show that de-airing the sample increased the measured gel strength by approximately 20% in these particular conditions. This supports the idea of progressive failure in the case of retained air. Figure 8.6 also shows that the average measured gel strength with three different vanes of the same diameter and different H/D ratio are not statistically different within their own groups of normal and de-aired material. This implies that the effect of progressive failure, due to the differential straining at the horizontal and vertical vane edges is small in each case. Thus supporting the hypothesis of rectangular stress distribution described in section 8.3.1.
Standing time = 30 minutes

Obtained with the Fann Viscosimeter at rotation speed of 3 r.p.m.

Figure 9.6: Mean gel strength against height/diameter ratio of the vane.
8.5.3: Effect of Nominal Rotation Speed

The effect of nominal instrument rotation speed on the measured gel strength of cement slurry was investigated using the V4/C2, vane/cup-combination (Table 8.2), with the Weissenberg Rheogoniometer. The results are shown in Figure 8.7. It can be seen from this figure that the measured gel strength is affected to a large extent by the nominal rotation speed in agreement with the data obtained with soils as described in section 8.3.2.

8.5.4: Effect of Different Test Instruments with Different Measuring System Stiffness

Three rotational viscometers (Fann viscometer, Weissenberg Rheogoniometer and Rheomat 15) with the V4/C2 combination (Table 8.2) were used to investigate whether or not the operational differences between them, as outlined in section 8.3.2, had an effect on the measured gel strength, even although the nominal rotation speed was 6 r.p.m for all instruments. The results obtained by these instruments are shown in Table 8.4. These results indicate that in accordance

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Rheomat 15</th>
<th>Fann Viscometer</th>
<th>Weissenberg Rheogoniometer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of results</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>Mean gel strength (Pa)</td>
<td>58.5 ± 10.5 *</td>
<td>87.2 ± 9.5 *</td>
<td>111.05 ± 10.2 *</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>16.52</td>
<td>14.95</td>
<td>16.05</td>
</tr>
</tbody>
</table>

* 95% confidence limits
Standing time = 15 minutes

Figure 8.7: Effect of Vane Rotation Speed on the Measured Gel Strength

Bars indicate 95% confidence limits.

Each point represents the mean of eight results.
CHAPTER 8

with the prediction in section 8.3.2, the means of the measured gel strength obtained by the three instruments are statistically different at the 95% confidence level with the Weissenberg Rheogoniometer giving the highest results. However, the time to break the material down in the Rheomat was probably greater than shown in Table 8.1 because, the driving motor on the Rheomat, as opposed to a rigidly supported motor on the other instruments, was suspended on a soft torsional spring which had to be rotated to produce the opposing torque generated at the vane shaft. The effect of this was to increase the time to failure and hence reduce the shear rate below that described in section 8.3.2. Therefore the true total time to failure with the Rheomat is considerably less than that given in Table 8.1. An additional problem which may have resulted in rather a low value for gel strength when measured on the Rheomat was that when the motor was switched on, a rapid flick of the vane was observed which may have broken the weak gel structure before the normal rotation was initiated. The torque associated with this instantaneous flick could not be measured with the soft spring and hence the apparent maximum torque may have been related to a partly broken system.

8.5.5: Concluding Remarks

The above discussion shows that if the vane tests are to be used to measure gel strength, great care has to be taken to define the actual time to maximum torque rather than to define the nominal instrument rotation speed. If this is not done, widely different results will be obtained from different instruments.

The results of other workers with more plastic systems requiring rotation of several degrees before reaching the yield stress are not directly applicable to cement paste systems because the cement based systems breakdown at such small strain values.

8.6: Prediction of Minimum Pressure Required to start up Flow in Complex Geometries.

8.6.1: Introduction

In the oil drilling industry the prediction of flow of cement slurries in complex geometries such as eccentric annuli, fissures and cavities
is of great practical importance. A complete mathematical solution of flow in these geometries is difficult even for the relatively simple Newtonian and Bingham fluids. However, numerical solutions are possible for fluids with a well defined flow model, (see for example, Theodoron et al (1984), Gallagher et al (1975), Davies et al (1984), Kim-e et al (1983) and Josse and Finlayson (1984). For the material used in this work where the material possesses a variety of complex rheological characteristics (see Chapter 5), a comprehensive solution which includes inertia effects viscous effects, elastic effects and build up and breakdown of structure has proved impossible to achieve. Nevertheless, an approximate solution may be obtained if the material behaviour is simplified. Below, a simple method to calculate the minimum pressure to cause flow in complex geometries will be discussed. The method is based on the assumption that the material behaviour can be described by a flow equation of an ideal plastic solid, the yield stress of which is equal to the gel strength measured by the shear vane test described above.

8.6.2: Extrusion Through a Conical Die

To examine the validity of the method a solution for the problem of extrusion through an axisymmetric die will be considered, because it resembles the flow through the complex practical geometries mentioned above and requires shear within the material rather than at surfaces. Furthermore, a mathematical solution of quasi-static extrusion through this geometry is possible (Slater (1977)), if the assumptions stated below are valid. Other methods can be used to solve the same problem such as the upper-bound theorem (Calladine (1969)) and a numerical analysis method (Zienkiewicz (1977)). However, these methods will not be considered in this thesis.

8.6.2.1: Assumptions

i) The material has a well defined yield point and yield stress which is equal to the gel strength measured by the vane test.
ii) The material is isotropic, rigid- perfectly plastic and obeys the Tresca yield criterion and associated flow rules.
iii) The effects of inertia, viscous resistance and build up or breakdown of structure are negligible.

None of these assumptions is actually correct for cement paste.
Figure 8.8 shows the stresses acting on an elemental frustum extruded through a straight conical die of dimensions given in the figure.

P and \( \tau \) are the normal die pressure and the shear stress at the die surface respectively.

From the geometry of the section

\[
\begin{align*}
\text{dr} &= ds \sin \alpha \\
\text{dz} &= ds \cos \alpha \\
\text{dz} &= \text{dr} \cot \alpha
\end{align*}
\]  

...(8.2) ...(8.3) ...(8.4)

Resolving forces exerted on the elemental frustum in the axial direction

\[
(\sigma_z + d\sigma_z) \pi (r + dr)^2 - \sigma_z \pi r^2 - P2\pi r \ ds \sin \alpha - \tau 2\pi r \ ds \cos \alpha = 0
\]  

...(8.5)
Neglecting differential quantities of the second order and using Equation 8.2 - 8.4, Equation 8.5 reduces to:

$$2(\sigma_Z - P - \tau \cot \alpha) \left(\frac{dr}{r}\right) + d\sigma_Z = 0 \quad \ldots(8.6)$$

Similarly, resolving forces in the radial direction

$$\sigma_r 2\pi r \ dz - P 2\pi r \ ds \cos \alpha + \tau 2\pi r \ ds \sin \alpha = 0 \quad \ldots(8.7)$$

Substituting for $dz$ from Equation 8.3
Equation 8.7 reduces to:

$$\sigma_r - P + \tau \tan \alpha = 0 \quad \ldots(8.8)$$

Assuming $\sigma_z$ and $\sigma_r$ are the principal stresses the Tresca yield criterion gives

$$- \sigma_z - (-\sigma_r) = 2 \tau_y$$

or

$$- \sigma_z + \sigma_r = 2 \tau_y \quad \ldots(8.9)$$

where $\tau_y$ is the yield stress.

Combining Equation 8.6, 8.8 and 8.9 yields:

$$-2(2 \tau_y + \tau \tan \alpha + \tau \cot \alpha)(dr/r) + d\sigma_z = 0 \quad \ldots(8.10)$$

Let

$$B = 2 \tau_y + \tau \tan \alpha + \tau \cot \alpha \quad \ldots(8.11)$$

Then

$$-2 B \left(\frac{dr}{r}\right) + d\sigma_z = 0 \quad \ldots(8.12)$$

Integrating Equation 8.12 produces

$$\sigma_z = 2 B \ln r + C \quad \ldots(8.13)$$

where $C$ is constant of integration and can be found from the boundary
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conditions. Assuming that the material is discharged into the atmosphere and residual stresses are absent so that \( \sigma_z = 0 \) at \( r = R_2 \)

Therefore \( 0 = 2B \ln R_2 + C \)

or \( C = -2B \ln R_2 \) \( \ldots (8.14) \)

Substituting for \( C \) in Equation 8.13

\[ \sigma_z = 2B \ln (r/R_2) \] \( \ldots (8.15) \)

Let \( P_e \) be the extrusion pressure then at entrance to the die where

\[ r = R_1, \quad \sigma_z = P_e \]

Hence \( P_e = 2B \ln (R_1/R_2) \) \( \ldots (8.16) \)

Equation 8.16 estimates the extrusion pressure for open die extrusion with \( B \) obtained from Equation 8.11. If the surface of the die is perfectly rough so that slip will not occur, the shear stress at the surface of the die \((\tau)\) will be equal to the yield stress \(\tau_y\). Hence Equation 8.11 will become

\[ B = \tau_y (2 + \tan \alpha + \cot \alpha) \] \( \ldots (8.17) \)

This will be an upper bound solution.

Further, if the die surface is smooth then \( \tau \) may be equated to the shear stress \((\tau_s)\) measured with a smooth surface such as a bob of co-axial cylinder viscometer, hence Equation 8.11 produces

\[ B = 2\tau_y + \tau_s (\tan \alpha + \cot \alpha) \] \( \ldots (8.18) \)

This will be a lower bound solution.

8.6.3: Axisymmetric Extrusion Through Square Die

In the case of square (i.e. \( \alpha = 45^\circ \) in Figure 8.8) die, flow may occur with the material sliding over the face of the die or the formation of
a dead material zone as shown in Figure 8.9a. The latter case has been shown (Calladine (1969)) to be the most likely case over almost all the practical range of reductions of diameter. This was (as will be seen later) the case in our experiments. Consequently, with a dead material zone, $B$ in Equation 8.17, and hence $P_e$ in Equation 8.16, are minimum when $\alpha = 45^\circ$

Hence the minimum value of $P$ when a dead material zone occurs is

$$P_e = 8 \gamma y \ln(R_1/R_2) \ldots (8.19)$$

8.7: Experimental Techniques
8.7.1: Mono Pump with Sharp Orifice
8.7.1.1: Experimental Procedure

Approximately 4 litres of the standard proportions, Table 4.1, were mixed as given in Chapter 4. The vertical tube with a sharp orifice at the top end (Figure 8.10) was filled from the bottom leaving an air gap for 300 mm from the orifice. The tube was then stored vertically for a given time after which it was attached to the Mono pump and a bentonite suspension was pumped behind the cement plug until the bentonite started to flow through the orifice. Meanwhile the pressure was recorded continuously using the pressure transducer shown in Figure 8.10. A typical pressure time curve is shown in Figure 8.11.

The pump was switched off after the bentonite suspensions started to flow through the orifice. The top 300 mm of the test section including the orifice was then detached from the rest of the test section and the bentonite inside it was allowed to flow out under its own weight. Thereafter, this 300 mm of the tube was filled with hot wax which was then allowed to cool. After the wax solidified the orifice plate was detached from the tube and the wax-cement plug inside the tube was then pushed out carefully to keep the plug intact. Material from the same mix was transferred to the Fann viscometer cup, using the cup C1 and the vane V2 or V3 (see Table 8.2), the vane was submerged 13 mm below the surface. The material was allowed to stand and then tested simultaneously with the Mono pump test.
FIGURE 8.9: EXTRUSION THROUGH SQUARE DIE

(a) Assumed profile

(b) Measured profile

FIGURE 8.10: SHARP ORIFICE ARRANGEMENT USED WITH THE MONO PUMP
Examination of the wax-cement plug produced as described in section 8.7.1.1 has shown that a dead material zone occurred with the test section used in this investigation. This result agrees with the hypothesis used to derive Equation 8.19. Further, it was assumed when deriving Equation 8.19 that the material would flow in a conical frustum with a shown in Figure 8.9a equal to 45°. However, a careful study of the plug revealed that this is not exactly true and the dead material zone formed a surface of conical frustum with a curvature at both ends (Figure 8.9b) rather than a straight surface of conical frustum as was assumed (Figure 8.9a). However, the exact shape of the dead material zone varied from one test to another and, from side to side of the tube which made it impossible to draw a consistent picture of the dead material zone.

8.7.1.2.2: Extrusion Pressure

Table 8.5 gives the theoretical (Equation 8.15) and measured extrusion pressures through the sharp orifice shown in Figure 8.10, the pressures being obtained using the measured gel strengths from the shear vane test for standing times of 15, 40 and 70 minutes. For the 15 minutes standing time the theoretically calculated extrusion pressure is only approximately half of the measured one. For the 40 and 70 minutes standing time the correlation between the calculated and measured extrusion pressures is much improved, although all the
calculated pressures are still smaller than the measured ones. The reasons for this difference could be:-

a) The extrusion speed is fluctuating or/and is not sufficiently small for inertia and viscous effects to be ignored.
b) The air content of the mix (approximately 5%) may influence the shear vane results and the extrusion pressure.
c) The effects of the curvature at both ends of the extrusion surface are large compared with the extrusion pressure with the sharp orifice geometry.

These factors are investigated below.

Table 8.5: Theoretical and Experimental Extrusion Pressures for the 10 mm Sharp Orifice

<table>
<thead>
<tr>
<th>Standing time (min)</th>
<th>Test Number</th>
<th>Gel Strength from Shear Vane (Pa)</th>
<th>Theoretical Pressure (k Pa)</th>
<th>Experimental Pressure (k Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>S.D. Mean</td>
<td>S.D. Mean</td>
</tr>
<tr>
<td>-14</td>
<td>1</td>
<td>211</td>
<td>2.11</td>
<td>4.23</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>219</td>
<td>2.19 0.04 2.15</td>
<td>4.43 0.11 4.36</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>215</td>
<td>2.15</td>
<td>4.42</td>
</tr>
<tr>
<td>40</td>
<td>4</td>
<td>1169</td>
<td>11.69</td>
<td>12.28</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>908</td>
<td>9.08 1.66 11.36</td>
<td>17.71 2.55 15.45</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>1305</td>
<td>13.05</td>
<td>17.31</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>1163</td>
<td>11.63</td>
<td>14.49</td>
</tr>
<tr>
<td>70</td>
<td>8</td>
<td>2968</td>
<td>29.68</td>
<td>36.23</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>3193</td>
<td>31.93</td>
<td>43.08</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>2763</td>
<td>27.63 4.33 27.77</td>
<td>42.87 6.26 37.64</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>2183</td>
<td>21.83</td>
<td>29.39</td>
</tr>
</tbody>
</table>
8.7.2: Instron Pump with Tapered Tube or Sharp Orifice

8.7.2.1: Experimental Procedure

Approximately 2 litres of material with the standard proportions (Table 4.1) were mixed as described in Chapter 4.

After mixing the material was de-aired in a vacuum flask (-700 mm of mercury) for 5 minutes with the flask continually tapped to encourage the air bubbles to move to the surface. Then the material was agitated for 10 minutes using the Crypto Peerless mixer. The material was de-aired for 8 minutes for the second time to ensure effective removal of the air bubbles. The structure formed during the latter de-airing process was broken by material re-agitation for further 2 minutes. Thereafter the material was transferred to the Weissenberg Rheogoniometer cup (C2, Table 8.2) and either the tapered tube section or the sharp orifice section shown in Figure 8.12. The vane (V4, Table 8.2) was then immersed 13mm below the surface of the material in the cup. After 15 minutes standing time the Instron head (see Chapter 4) was used to push down the plunger shown in Figure 8.12 at a specified constant speed. The output from the pressure transducer shown in Figure 8.12 was recorded using the Bryans xy/t recorder. Almost simultaneously a shear vane test was carried out by setting the cup of the Weissenberg Rheogoniometer to rotate at a specified constant speed.

8.7.2.2: Results and Discussion

8.7.2.2.1: Shear Vane Test

The results from 16 tests conducted with the Weissenberg Rheogoniometer at a cup rotation speed varying from 0.00475 to 11.9 r.p.m are shown in Figure 8.13. It can be seen from this figure that over a wide range of low rotation speeds, less than 0.6 r.p.m, the measured gel strength is almost constant. At higher rotation speeds (greater than 0.6 r.p.m), however, the measured gel strength increase rapidly with the increase in rotation speed, probably because at these higher speeds, forces such as inertia in the vane and in the recording system have a larger effect on the results. Therefore, one could assume that the gel strength measured at low rotation speed is more identifiable with the true gel strength of the material. It is
FIGURE 8.12: EXTRUSION TEST SECTIONS
Figure 8.13: Gel strength against log rotation speed of the cup obtained with the Weissenberg rheogoniometer.
important to note that at extremely low rotation speed (lower than 0.001 r.p.m) the torque measured on the vane shaft continued to increase for a very long time without any tendency to reach a maximum value. Perhaps because at very low rotation speeds, the rate of bond formation, due to thixotropic recovery and/or chemical reaction, is higher than that of bond destruction caused by the rotating vane. Consequently, since the gel strength is calculated from the maximum torque, gel strength cannot be found using these low rotation speeds. This is one of the inevitable problems when a reacting system is used.

A check on the capability of the xy/t recorder to cope with the rapid increase in torque on the vane shaft was carried out with Gould Digital Storage Oscilloscope, Type 4035. Four tests were performed with the Weissenberg Rheogoniometer at rotation speed of 1.9 r.p.m with the xy/t recorder and the oscilloscope recording the torque generated. The torque recorded with both systems were virtually identical.

8.7.2.2.2: Results for the Instron Plunger Plus Sharp Orifice

The gel strength measured with the vane at low rotation speeds as described in section 8.7.2.2.1 was used to calculate the theoretical pressure required to extrude the material through the orifice described in section 8.7.2.1. The resulting extrusion pressure is shown in Figure 8.14 together with measured extrusion pressure at various flow speed. Figure 8.14 displays little or no variation in the measured pressure with the increase in flow speed up to 500 mm/minute. However, as before (section 8.7.1.2.2) the calculated pressure is still smaller than the measured one. Hence, since the material was de-aired, one could conclude that the air content of the mix and the flow speed are not responsible for the difference between the experimentally measured extrusion pressure and the theoretically calculated one.

The deviation of the dead material zone from the assumed profile, as described in section 8.7.1.2.1, may explain the difference between the experimental and the theoretical extrusion pressures. To investigate this further, the tapered tube described earlier in section 8.7.2.1 was employed in the subsequent experiments.
FIGURE 8.14: EXTRUSION PRESSURE AGAINST PISTON SPEED FOR THE SHARP ORIFICE

Piston Speed (mm/minute)

Calculated extrusion pressure

Extrusion Pressure (kPa)
CHAPTER 8

8.7.2.2.3: Results for Instron Plunger Plus Tapered Tube

The tapered tube was employed based on the hypothesis that the effects of the deviation of the flow zone from the assumed profile will be smaller in magnitude and more importantly, in comparison with the extrusion pressure than those which exist with the sharp orifice geometry. Gel strength at low rotation speeds as given in section 8.7.2.2.1 was utilised to calculate the extrusion pressure as given by Equation 8.17. The resulting pressure is shown in Figure 8.15 together with the measured extrusion pressure for flow speed varying from 10 to 1000 mm/minute. Figure 8.15 shows that, as the case with the sharp orifice, there exists little or no dependence of the measured extrusion pressure on the flow speed up to flow speed of 1000 mm/minute. The correlation between the measured and calculated pressure is much better than the case for the sharp orifice, with the calculated pressure providing a slight overestimation. It must be noted that the calculated pressure was determined based on the assumption that the internal surface of the tapered tube is perfectly rough so that slip will not occur. This would be not entirely true and, hence, some slip may have taken place. If the shear stress at the surface of the tapered tube was assumed to be equal to the gel strength measured with a smooth steel bob, i.e. 41 Pa at 0.75 r.p.m, as determined in Chapter 7 (Appendix 7.9) then, Equation 8.18 would give the extrusion pressure as 5.5 kPa. This pressure provides a considerable underestimate of the measured extrusion pressure displayed in Figure 8.15. This is not entirely unexpected since the internal surface of the tube was designed to be rough in order to minimise slippage. Furthermore, as demonstrated in Chapter 5, section 5.4, slip effects become insignificant if shearing takes place throughout the material which is the case with the tapered tube. Therefore one may deduce that the no slip assumption is more identifiable with the true material behaviour.

The correlation between the theoretically calculated pressure, based on the gel strength measured with the shear vane technique, and the experimentally determined ones displayed above is less than perfect. Nonetheless, considering the several assumptions and simplifications involved in the derivations of the theory, these results indicate that this gel strength is a parameter which can be related to the material
Figure B.45: Extrusion pressure against piston flow (flow) speed for the tapered extruder.

Piston Speed (mm/minute)

Extrusion Pressure (kPa)

Calculated extrusion pressure
flow behaviour in complex geometries. Hence, one may conclude that
the shear vane technique appears to give a true material parameter.

8.8: Conclusions

1) The shear vane technique appears to offer a method by which the gel
strength of cement slurries may be measured and which is superior
to many existing techniques.

2) The effective rotation speed of the vane is dependent on the
instrument nominal rotation speed and the stiffness of the torque
measuring system.

3) Gel strength measured with the vane generally increases with the
effective rotation speed of the vane. However, it is virtually
constant over a wide range of low effective rotation speeds.

4) The gel strength measured with the vane together with the Tresca
failure criterion gives extrusion pressures which are identifiable
with the experimentally measured ones.
CHAPTER 9

CONCLUSIONS

For completeness, the conclusions drawn in previous chapters are collected here.

9.1: Concentrated Suspensions and Cement Slurries

Concentrated suspensions in general and cement slurries in particular still present a problem for scientists and engineers. The rheological properties of these systems, in most cases cannot be determined from the basic constituents. Consequently the engineer or scientist has to rely on experimental methods to obtain the rheological parameters suitable for making engineering predictions or for understanding the materials science aspects of slurries.

The rheological properties of cement slurries are shown to be dependent on a variety of factors including the experimental techniques and conditions. Hence, theoretical predictions, based on experimentally determined parameters, are valid only if the practical conditions are very similar to the experimental ones.

9.2: Co-axial Cylinder Viscometer

The co-axial cylinder viscometer is a very useful tool for measuring the rheological properties of cement slurries. However, the results obtained in this research clearly show that this instrument suffers from inherent problems, mainly plug flow and slippage at the surfaces of the cylinders, and unless these are accounted for, inaccurate results may be obtained. It is recommended that a minimum of two gap widths are used in the viscometer and the results accepted only if the results correlate with each other.

9.3: Material Used in This Investigation

The cement slurry used in this investigation possesses a complex rheological behaviour for which no general equation to describe the flow behaviour has been found. The material's resistance to flow was measured to increase with time under shearing or when at rest. This
increase was found to be dependent on many factors such as shear rate, time, slurry age and experimental procedure.

The method recommended by the API (1982 and 1986) for the determination of the rheological properties of cement slurries was found to be too simplistic, and may lead to wrong conclusions if used with materials which exhibit complex rheological behaviour similar to that possessed by the slurry used in this investigation.

9.4: Pipe Flow

A new method is proposed to predict the pressure drop for plug flow in pipelines. The technique involves establishing a similar flow in the co-axial cylinder viscometer to that in the pipe and is based on the assumption that the sheared layers in both systems are similar. The experimental and analytical work involved is simple and the accuracy of the prediction is adequate for most engineering applications. The method is also applicable for the prediction of flow start-up in pipes if inertia effects in the pipe and in the co-axial cylinder viscometer are negligible, and the material does not fail progressively in the pipe.

9.5: Shear Vane Test

In order to overcome the limitation of many of the existing techniques for measuring gel strength, the shear vane test was suggested and thoroughly investigated at various rotation speeds. The techniques appeared to be superior to standard methods in many respects although the gel strength was shown to increase when the time to reach maximum torque became less than 0.2 seconds. The rotation speed at which this occurs will depend on the stiffness of the torque measuring system and will vary between different instruments.

The gel strength measured with the vane when used with the Tresca failure criterion gives extrusion pressures which are of the same order as the experimentally measured ones.
Recom-endations for Further Work

The work described here has by no means completely solved the problems associated with highly thixotropic slurries, and much further work is needed for a full understanding of such systems. The work required is not restricted solely to the fields touched on in this thesis. However, there are a number of points raised in the course of this investigation which require further attention:

i) The method proposed for the prediction of pumping pressure in pipes was shown to be effective for materials with a relatively high yield stress. For materials with low yield stress, some of the assumptions involved are not appropriate. Experimental and analytical study should therefore be made to establish the applicability of the method to materials which possess a low yield stress.

ii) Experimental studies should be made to examine the applicability of the method proposed for pipe flow to the case of annuli.

iii) Flow start-up in pipes may occur by progressive failure of the material in the pipe. However, this phenomenon is not fully understood and the pressure to cause this type of failure has not been investigated. Work in this area is therefore required.

iv) The shear vane test and the factors influencing the results it produces have not been fully investigated, particularly if the material possesses a low yield stress. The whole range of high water content oil well cement slurries should therefore be examined with the shear vane.
REFERENCES


Budge, C.J., Cement and Concrete Association publication 47.012, 1981.

Cadling, L. and Odenstad, S., The Vane Borer, Royal Swedish Geotechnical Institute, Proceedings No. 2, 1950.


Casson, N., Paper Presented to Joint Meeting of the British, Italian and Netherlands Societies of Rheology, Amsterdam, 18 - 20, April 1979.


Harris, J., Rheol. Acta, Band 6, Heft 1, 6 - 12, 1967.


Appendix 7.1

Inertia Effects on the Pressure Required to Start Up Flow in Tubes

The case of Transducer 3 with the 10.55 millimetre diameter tube, Figure 7.24, will be used as an example to calculate the inertia effect involved in the problem of flow start-up in tubes. For this case:

Initial flow velocity = 0.0 m/sec.
Final flow velocity = 100 mm/min. = 0.0017 m/sec.
Tube length = 3.63 m
Material measured density = 1.75 x 10^3 Kg/m^3
Time to achieve steady flow (Figure 7.24) = 1.0 sec
Pressure to flow (Figure 7.24) = 68000 Pa

Now, inertia force \( F \) = rate of change of momentum

Assuming that the material in the tube moves as one solid body at constant acceleration gives:

Rate of change of momentum = \( \text{mass} \times \frac{\text{final velocity} - \text{initial velocity}}{\text{time}} \)

or \( F = \pi r^2 \rho \times \frac{(V_F - V_i)}{t} \) ....(1)

where \( r \) and \( l \) are tube radius and length respectively, and \( \rho, t, V_F \) and \( V_i \) are material density, time, final flow velocity and initial flow velocity respectively.

Inertia pressure \( P_i \) = \( F/\text{Area} \)

or \( P_i = \frac{F}{\pi r^2} \) ....(2)

Substituting equation 1 in 2 yields:

\( P_i = \frac{(\pi r^2 \rho \times (V_F - V_i))}{(\pi r^2 t)} / (\pi r^2 t) \)

\( = \frac{\rho \times (V_F - V_i)}{t} \)
\[ Pi = \frac{(3.63 \times 1.75 \times 10^3 \times (0.0017 - 0.0))}{1.0} \]

\[ = 11.9 \text{ Pa}. \]

Comparing this with the 67,000 Pa needed to start flow, it may be concluded that inertia effects are negligible.

**Appendix 7.2: Gun Rheometer Results: Gel Strength (Calculated from the Maximum Pressure Needed to Start Flow); 30 Minutes Standing Time.**

<table>
<thead>
<tr>
<th>Tube Diameter (mm)</th>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>53.3</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>70.7</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>93.9</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>63.7</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>83.4</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>71.8</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>97.4</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>76.3</td>
</tr>
<tr>
<td>S.D.</td>
<td></td>
<td>16.0</td>
</tr>
</tbody>
</table>

S.D. = Standard Deviation.
Appendix 7.3: Gun Rheometer Results: Gel Strength (Calculated from the Maximum Pressure Needed to Start Flow); 54 Minutes Standing Time

<table>
<thead>
<tr>
<th>Tube Diameter (mm.)</th>
<th>3.25</th>
<th>4.9</th>
<th>7.82</th>
<th>10.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test Number</td>
<td>Gel Strength (Pa)</td>
<td>105.5</td>
<td>127.8</td>
<td>153.6</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>127.4</td>
<td>161.0</td>
<td>198.1</td>
</tr>
<tr>
<td>3</td>
<td>102.0</td>
<td>143.5</td>
<td>156.4</td>
<td>129.9</td>
</tr>
<tr>
<td>4</td>
<td>92.7</td>
<td>113.7</td>
<td>159.2</td>
<td>198.4</td>
</tr>
<tr>
<td>5</td>
<td>135.6</td>
<td>129.4</td>
<td>164.7</td>
<td>220.0</td>
</tr>
<tr>
<td>6</td>
<td>82.2</td>
<td>151.9</td>
<td>162.0</td>
<td>205.6</td>
</tr>
<tr>
<td>7</td>
<td>124.0</td>
<td>159.2</td>
<td>139.6</td>
<td>183.9</td>
</tr>
<tr>
<td>8</td>
<td>86.9</td>
<td></td>
<td></td>
<td>216.4</td>
</tr>
<tr>
<td>9</td>
<td>90.4</td>
<td></td>
<td></td>
<td>181.8</td>
</tr>
<tr>
<td>10</td>
<td>129.8</td>
<td></td>
<td></td>
<td>181.8</td>
</tr>
<tr>
<td>11</td>
<td>121.1</td>
<td></td>
<td></td>
<td>160.6</td>
</tr>
<tr>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td>160.6</td>
</tr>
<tr>
<td>Mean</td>
<td>108.9</td>
<td>140.9</td>
<td>161.9</td>
<td>176.4</td>
</tr>
<tr>
<td>S.D.</td>
<td>19.3</td>
<td>17.8</td>
<td>17.9</td>
<td>31.0</td>
</tr>
</tbody>
</table>
Appendix 7.4: Gun Rheometer Results with Internally Threaded Tube: Gel Strength (Calculated from the Maximum Pressure Needed to Start Flow); 30 Minutes Standing Time

Tube Diameter = 5.2 mm

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>194.2</td>
</tr>
<tr>
<td>2</td>
<td>194.2</td>
</tr>
<tr>
<td>3</td>
<td>213.1</td>
</tr>
<tr>
<td>4</td>
<td>265.2</td>
</tr>
<tr>
<td>5</td>
<td>260.5</td>
</tr>
</tbody>
</table>

Mean = 225.4
S.D. = 35.1

Appendix 7.5: Piston Pump Results: Gel Strength (Calculated from the Maximum Pressure Needed to Start Flow) for Non-De-Aired Material in the 10.55 mm Tube.

Pumping Speed = 100 mm/minute.

<table>
<thead>
<tr>
<th>Tube Length (m)</th>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.515</td>
<td>1</td>
<td>79.9 58.3 59.6 43.3</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>193.9 133.5 57.2 38.8</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>177.2 112.6 67.3 58.3</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>119.5 77.0 58.3 38.0</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>206.2 131.3 64.4 43.3</td>
</tr>
<tr>
<td>1.33</td>
<td>6</td>
<td>115.5 81.2</td>
</tr>
<tr>
<td>1.785</td>
<td>7</td>
<td>148.5 89.7</td>
</tr>
<tr>
<td>3.785</td>
<td>Mean</td>
<td>148.7 97.7 61.4 44.3</td>
</tr>
<tr>
<td></td>
<td>S.D.</td>
<td>46.3 28.7 4.3 8.2</td>
</tr>
</tbody>
</table>
Appendix 7.6: Piston Pump Results: Gel Strength for Non-De Aired Material (Calculated from the Maximum Pressure Needed to Start Flow) In the 19.54 mm Tube.

Pumping Speed = 100 mm /minute.

<table>
<thead>
<tr>
<th>Tube Length (m.)</th>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.785</td>
<td>1</td>
<td>182.9</td>
</tr>
<tr>
<td>1.785</td>
<td>2</td>
<td>129.5</td>
</tr>
<tr>
<td>3.785</td>
<td>3</td>
<td>101.4</td>
</tr>
<tr>
<td>3.785</td>
<td>4</td>
<td>120.7</td>
</tr>
<tr>
<td>3.785</td>
<td>5</td>
<td>114.6</td>
</tr>
<tr>
<td>3.785</td>
<td>6</td>
<td>115.3</td>
</tr>
<tr>
<td>3.785</td>
<td>7</td>
<td>91.4</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>127.8</td>
</tr>
<tr>
<td></td>
<td>S.D.</td>
<td>137.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>104.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>207.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>172.2</td>
</tr>
</tbody>
</table>

Mean: 137.6
S.D.: 42.7
Appendix 7.7: Piston Pump Results: Gel Strength (Calculated from the Maximum Pressure Needed to Start Flow) for De-Aired Material.

Tube Diameter = 10.55 mm.
Pumping Speed = 100 mm/minute.

<table>
<thead>
<tr>
<th>Tube Length (m.)</th>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.515</td>
<td>1</td>
<td>45.4</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>47.4</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>61.9</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>55.7</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>28.9</td>
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<tr>
<td></td>
<td>6</td>
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<tr>
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<td>7</td>
<td>39.2</td>
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<td>45.3</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>47.4</td>
</tr>
<tr>
<td>S.D.</td>
<td></td>
<td>10.5</td>
</tr>
</tbody>
</table>

Mean Gel Strength (Pa): 50.7
S.D.: 2.8
Appendix 7.8: Co-axial Cylinder Viscometer Results: Gel Strength
(Calculated from the Maximum Torque Recorded on the Bob Shaft)
for Non-De Aired Material.

Rotation Speed = 0.75 r.p.m.
Equivalent Linear Speed = 100 mm /minute.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>137.3</td>
</tr>
<tr>
<td>2</td>
<td>128.3</td>
</tr>
<tr>
<td>3</td>
<td>79.0</td>
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<tr>
<td>4</td>
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<tr>
<td>5</td>
<td>184.0</td>
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<tr>
<td>6</td>
<td>130.1</td>
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<tr>
<td>7</td>
<td>152.6</td>
</tr>
<tr>
<td>8</td>
<td>72.7</td>
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<tr>
<td>9</td>
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<tr>
<td>10</td>
<td>121.2</td>
</tr>
<tr>
<td>11</td>
<td>139.1</td>
</tr>
<tr>
<td>12</td>
<td>194.8</td>
</tr>
</tbody>
</table>

Mean 132.8
S.D. 35.6
Appendix 7.9: Co-axial Cylinder Viscometer Results: Gel Strength
(Calculated from the Maximum Torque Recorded on the Bob Shaft for
De-Aired Material.

Rotation Speed = 0.75 r.p.m.
Equivalent Linear Speed = 100 mm./minute

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Gel Strength (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>33.2</td>
</tr>
<tr>
<td>2</td>
<td>37.7</td>
</tr>
<tr>
<td>3</td>
<td>31.9</td>
</tr>
<tr>
<td>4</td>
<td>37.1</td>
</tr>
<tr>
<td>5</td>
<td>50.3</td>
</tr>
<tr>
<td>6</td>
<td>56.3</td>
</tr>
<tr>
<td>7</td>
<td>33.2</td>
</tr>
<tr>
<td>Mean</td>
<td>41.4</td>
</tr>
<tr>
<td>S.D.</td>
<td>9.8</td>
</tr>
</tbody>
</table>
Figure 4.13: Instron piston pump