SUSPENSION DEWATERING:
CHARACTERISATION AND OPTIMISATION

by

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PREFACE
I hereby declare that, except where due reference is made in the text, this thesis is solely my own original work, and is less than 100,000 words in length.
ACKNOWLEDGMENTS

The project was undertaken within the Particulate Fluids Processing Centre, a Special Research Centre of the Australian Research Council, formerly known as the Advanced Minerals Products Special Research Centre at the University of Melbourne. The centre is a unique interdisciplinary group of engineers and scientists from the departments of chemical engineering, chemistry and mathematics. This project would not have been possible without the help of a great number of people from the Particulate Fluids Processing Centre and the AMIRA Bayer Process Flocculants (P527) project sponsor companies. I would like to thank all of you for your assistance and support.

Particulate Fluids Processing Centre

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Industrial Project Sponsors

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I would like to acknowledge the assistance of all of the on-site staff at each Alumina Refinery sponsor site, ALCOA Pinjarra, Worsley Alumina and Queensland Alumina during the nine site visits over the course of the project. This included coordination of safety inductions, orientations and use of laboratory facilities in addition to assistance with collecting samples and process data and providing other in kind support of food, accommodation and transport for a total of 20 weeks of site work. To name just a few of the many people involved, the coordination efforts of Mike Shaw, Evan Jamieson, Geoff Bauer, Steve Rosendeg, Ron Kahane, Brad Hogan Alan Kane and Dale Hosking are truly appreciated.

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Australian Research Council

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Australian Mineral Industries Research Association

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ABSTRACT

The alumina industry produces a significant quantity of bauxite residue suspension (red mud) that must be washed and dewatered in trains of thickeners and residue disposal areas to recover valuable alumina and sodium hydroxide. The Australian Alumina Industry have come together to sponsor a project to address waste minimisation and environmental impact issues collectively through the optimisation of dewatering in their washer trains and residue disposal areas. The project aims to maximise thickener underflow and tailings dam solids concentrations.

The gel point, compressibility and permeability have been established as the fundamental physical properties that determine suspension dewaterability. In this project, experimental techniques have been developed to allow rapid and comprehensive dewaterability characterisation over a wide range of solids concentrations using batch settling, gravity permeation and pressure filtration tests. Newly developed techniques include a transient batch settling test, a gravity permeation test and a stepped pressure filtration test.

On-site testing was performed at the ALCOA, Pinjarra Alumina Refinery, the Worsley Alumina Refinery and the Queensland Alumina (QAL) Refinery. Dewaterability characterisation down an entire washer train was performed at each sponsor site. Subsequently comprehensive dewaterability characterisation was performed on selected underflow suspensions at each refinery. Flocculation testing and subsequent dewaterability characterisation were also performed on simulated thickener feed suspensions to test the influence of flocculant chemical type on dewaterability. Characterisation of the shear yield stress of red mud suspensions identified that the shear yield stress increases with increasing temperature. Thixotropy in red mud suspensions was observed to be relatively insignificant over the short time scale of dewatering processes. It is proposed that thixotropy in red mud suspensions is predominantly due to precipitation products from supersaturated liquor.

Fundamental theoretical models have been applied to predict the operation of individual pieces of process equipment including thickeners and pressure filters and also model settlement in tailings dams. Thickener dimensions were used in a steady state thickener model to predict how the solids flux varies with the underflow solids concentration based on the characterised dewaterability of both the flocculated and underflow suspensions. The underflow solids concentration predictions highlight that most thickener operation is limited by suspension permeability. These predictions also demonstrate that flocculation effectively doubles this permeability. At the actual underflow solids concentration, comparison of the predicted solids flux with the actual value has suggested that there is something in the thickener operation that effectively improves permeability by a factor ranging from 2 to 50 for various thickeners. This
permeability enhancement has been attributed to raking and other shear processes. The knowledge gained from this modelling is presented as an optimisation tool to maximise liquor recovery in washer trains through variations in process conditions. Red mud dewaterability was used for the prediction of rotary drum filtration performance, highlighting the effect of changes in process variables and potential directions for process optimisation. End of washer train red mud dewaterability was used in prediction of residue disposal area consolidation to give an indication of the time scale of consolidation.

The methods developed and the results of this body of work have implications, beyond the alumina industry, for a wide range of process and mining industries where suspensions are dewatered. The techniques developed and knowledge gained have already been applied by numerous other Australian and international companies and research institutions seeking to better understand and improve dewatering operations.
KEYWORDS

Compressive yield stress, hindered settling function, stepped pressure filtration, flocculated suspension, dewaterability characterisation, filtration modelling, thickener modelling, permeability enhancement, red mud.
PUBLICATIONS

The following papers and reports have been published as a result of and in support of this work.


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\(a\) empirical fitting parameter
\(A\) dissolved solids weight fraction of the liquor defined in Equation 4.3 (-)
\(A\) specific volume filter cake resistance
\(A_1\) specific volume filter cake resistance corresponding to the application of pressure \(P_1\)
\(A_2\) specific volume filter cake resistance corresponding to the application of pressure \(P_2\)
\(A_{\text{cloth}}\) filter cloth area (m²)
\(A_{\text{cloth washed}}\) area of filter cloth subjected to washing (m²)
\(b\) empirical fitting parameter
\(B\) dry weight fraction of the suspension with dissolved solids defined in Equation 4.4 (-)
\(B\) \(\phi_1\) filter cake height (m)
\(B’\) \(\phi_2\) filter cake height during \(\phi_1\) filter cake compaction (m)
\(B”\) \(\phi_2\) filter cake height during filter cake growth (m)
\(B_1\) \(\phi_1\) filter cake height at time \(t_1\) (m)
\(B_2\) \(\phi_2\) filter cake height at time \(t_1 + t_2\) (m)
\(d\) internal cylinder diameter (m)
\(d(z)\) thickener diameter (m)
\(d_{\text{max}}\) maximum thickener diameter (m)
\(d_p\) particle diameter (m)
\(D(\phi)\) solids diffusivity (m² s⁻¹)
\(D_c\) sample container diameter (m)
\(D_v\) vane diameter (m)
\(E_1\) fitting parameter
\(E_2\) fitting parameter
\(E_3\) fitting parameter
\(f_{\text{lr}}\) fractional liquor recovery (-)
\(f_{\text{submerged}}\) fraction of submerged filter cloth (-)
\(f_{\text{washing}}\) fraction of filter cloth subjected to washing (-)
\(F_0\) first Fourier series term defined in Equation 7.29
\(F_n\) Fourier series term defined in Equation 7.26
\(g\) magnitude of the gravitational acceleration (9.8 m s⁻²)
\(gpl\) grams of solids per litre of suspension (g L⁻¹)
\(h\) suspension height (m)
\(h_0\) initial suspension height (m)
\( hb \)  thickener bed height (m)
\( h_c \)  critical suspension height (m)
\( h_{\text{cake}} \)  filter cake height (-)
\( h_f \)  final suspension height (m)
\( h_f,\text{pred} \)  predicted final or equilibrium suspension height (m)
\( h_{\text{liq}} \)  liquor height (m)
\( h_{\infty} \)  equilibrium suspension height (m)
\( h_{\infty, f} \)  final equilibrium solids volume fraction (-)
\( H \)  initial suspension height before slumping in a slump test (m)
\( H_p \)  packed bed height (m)
\( H_0 \)  initial packed bed suspension height (m)
\( H_v \)  vane height (m)
\( k \)  empirical fitting parameter
\( k(\phi) \)  traditional Darcian permeability (m²)
\( K_{\text{cf}} \)  calibration constant for Cannon-Fenske viscometer (m²)
\( K_v \)  vane constant (m³)
\( L \)  rheometer bob length (m)
\( m \)  empirical fitting parameter
\( m_{\text{dry}} \)  combined minimum weight of dried suspension and sample container with lid (g)
\( m_{\text{empty}} \)  weight of empty sample drying container with the lid if applicable (g)
\( m_{\text{liq, dry}} \)  combined minimum weight of dried liquor and sample container with lid (g)
\( m_{\text{liq, empty}} \)  weight of empty sample drying container with the lid if applicable (g)
\( m_{\text{liq, wet}} \)  combined weight of the wet liquor and sample container with the lid if applicable (g)
\( m_s \)  mass of suspension (kg)
\( m_{\text{wet}} \)  combined weight of the wet suspension and sample container with the lid (g)
\( n \)  empirical fitting parameter in Equation 6.3
\( p \)  solids network pressure (Pa)
\( p_a \)  empirical fitting parameter
\( p_b \)  empirical fitting parameter
\( p_m \)  empirical fitting parameter
\( p_n \)  empirical fitting parameter
\( P \)  applied piston pressure (Pa)
\( P_1 \)  applied piston pressure (Pa)
\( P_2 \)  applied piston pressure (Pa)
\( P_{\text{base}} \)  equilibrium solids network pressure at the base of a suspension (Pa)
\( PE \)  effective permeability enhancement factor

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\[ P_y(\phi) \quad \text{compressive yield stress (Pa)} \]
\[ P_y(\phi)_{\text{pred}} \quad \text{predicted compressive yield stress (Pa)} \]
\[ q \quad \text{volumetric solids flux (m s}^{-1}\text{)} \]
\[ q_u \quad \text{underflow volumetric solids flux (m s}^{-1}\text{)} \]
\[ r \quad \text{membrane resistance} \]
\[ r(\phi) \quad \text{hindered settling factor (-)} \]
\[ r_a \quad \text{empirical fitting parameter} \]
\[ r_b \quad \text{empirical fitting parameter} \]
\[ r_g \quad \text{empirical fitting parameter} \]
\[ r_n \quad \text{empirical fitting parameter} \]
\[ R \quad \text{rheometer bob radius (m)} \]
\[ R(\phi) \quad \text{hindered settling function (Pa s m}^{-2}\text{)} \quad (R(\phi) = (\lambda/V_p)r(\phi)) \]
\[ s \quad \text{slump height (m)} \]
\[ s' \quad \text{dimensionless slump height defined in Equation 5.8 (-)} \]
\[ S \quad \text{rheometry parameter given Equation 5.3} \]
\[ t \quad \text{time, time of filtration (s)} \]
\[ t_0 \quad \text{initial filtration time (s)} \]
\[ t_1 \quad \text{time when pressure is stepped (s)} \]
\[ t_2 \quad \text{time for filter cake compaction (s)} \]
\[ t_c \quad \text{critical time of filtration (s)} \]
\[ t_{\text{cycle}} \quad \text{total cycle time (s)} \]
\[ t_{\text{fall}} \quad \text{measured fall time in a Cannon-Fenske viscometer (s)} \]
\[ t_p \quad \text{previous time (s)} \]
\[ t_{\text{res}} \quad \text{solids residence time (s)} \]
\[ t_{\text{submerged}} \quad \text{Submerged filtration time (s)} \]
\[ t_{\text{washing}} \quad \text{washing filtration time (s)} \]
\[ T \quad \text{temperature (°C)} \]
\[ T \quad \text{torque (N m)} \]
\[ T_c \quad \text{scaled critical time of filtration (s)} \]
\[ T_f \quad \text{scaled filtration time (-)} \]
\[ T_m \quad \text{modelling temperature (°C)} \]
\[ T_{\text{max}} \quad \text{maximum vane torque (N m)} \]
\[ T_p \quad \text{process temperature (°C)} \]
\[ T_t \quad \text{test temperature (°C)} \]
\[ u \quad \text{downward solids settling rate (m s}^{-1}\text{)} \]
\[ u_0 \quad \text{initial settling rate (m s}^{-1}\text{)} \]
\( u_0(\phi) \) free settling rate \((\text{m s}^{-1})\)

\( u_{St} \) magnitude of the Stokes settling rate given by Stokes’ Law in Equation 3.4 \((\text{m s}^{-1})\)

\( v \) upward liquor velocity \((\text{m s}^{-1})\)

\( V \) specific filtrate volume, volume of filtrate divided by membrane area \((\text{m})\)

\( V_0 \) initial specific filtrate volume \((\text{m})\)

\( V_1 \) specific filtrate volume when pressure is stepped \((\text{m})\)

\( V_2 \) specific filtrate volume required to compact filter cake from \(\phi_1\) to \(\phi_2\) \((\text{m})\)

\( V_p \) average particle or floc volume \((\text{m}^3)\)

\( w \) empirical fitting parameter

\( w \) weight of cake solids per unit volume of filtrate \((\text{kg m}^{-3})\)

\( x \) solids weight fraction of the suspension (-)

\( z \) vertical displacement with up signified as positive \((\text{m})\)

\( z_{\text{above}} \) distance from the top of the vane to the suspension surface \((\text{m})\)

\( z_{\text{below}} \) distance from the base of the vane to the base of the sample container \((\text{m})\)

\( z_c \) critical height \((\text{m})\)

\( \alpha(z) \) thickener shape factor (-)

\( \rho_{\text{water}} \) density of water \((\text{kg m}^{-3})\)

**Greek**

\( \alpha \) filtration parameter

\( \alpha \) membrane resistance \((\text{m}^{-1})\)

\( \alpha_m \) specific mass filter cake resistance

\( \alpha_v \) specific volume cake resistance \((\text{m}^2)\)

\( \beta^2 \) filtration parameter \((\text{m}^2 \text{ s}^{-1})\)

\( \chi \) filtration parameter given by Equation 7.10

\( \Delta P \) applied compressive stress or pressure drop \((\text{Pa})\)

\( \Delta t \) time step \((\text{s})\)

\( \Delta \rho \) solid-liquid density difference \((\text{kg m}^{-3})\)

\( \varepsilon \) settling parameter defined by Equation 6.24 (-)

\( \phi \) solids volume fraction (-)

\( \phi^* \) filtration parameter given by Equation 7.11

\( \phi_0 \) initial solids volume fraction (-)

\( \phi_1 \) average filter cake solids volume fraction corresponding to piston pressure \(P_1\) (-)

\( \phi_2 \) average filter cake solids volume fraction corresponding to piston pressure \(P_2\) (-)
\(\phi_{av}\) average final solids volume fraction (-)

\(\phi_{base}\) solids volume fraction at suspension base (-)

\(\phi_{cake}\) average solids volume fraction in filter cake (-)

\(\phi_r\) final solids volume fraction (-)

\(\phi_g\) suspension gel point as a solids volume fraction (-)

\(\phi_p\) previous solids volume fraction (-)

\(\phi_u\) underflow solids volume fraction (-)

\(\phi_{equ}\) equilibrium solids volume fraction (-)

\(\phi_{equ,f}\) final equilibrium solids volume fraction (-)

\(\dot{\gamma}\) shear rate (s\(^{-1}\))

\(\eta\) fluid or liquor viscosity (Pa s)

\(\eta_{liq}\) liquor viscosity (Pa s)

\(\eta_m\) liquor viscosity at modelling temperature (mPa s)

\(\eta_r\) liquor viscosity at test temperature (mPa s)

\(\eta_{equ}\) empirical parameter in the Casson model equation (Equation 3.2)

\(k^{-1}\) debye length

\(\lambda\) Stokes drag coefficient (Pa s m)

\(\nu_{liq}\) kinematic viscosity of the liquor (m\(^2\) s\(^{-1}\))

\(\rho_{liq}\) liquor density (kg m\(^{-3}\))

\(\rho_{sol}\) solid density (kg m\(^{-3}\))

\(\rho_{susp}\) suspension density (kg m\(^{-3}\))

\(\tau\) shear stress (Pa)

\(\tau_y\) shear yield stress (Pa)

\(\tau_y'\) dimensionless shear yield stress defined in Equation 5.8 (-)

\(\Omega\) angular velocity in radians per second (s\(^{-1}\))

\(\zeta\) zeta potential
SECTION A
INTRODUCTION AND THEORY
Chapter 1

INTRODUCTION

1.1 BACKGROUND

The minerals industry produces large quantities of waste tailings. Indeed, many ores produce more tailings than product. A notable example is gold which can be extracted from ore bodies with less than three grams of gold per tonne of ore (Wills 1976; Boger 1998). An aqueous dissolution process typically removes the valuable product. The insoluble residue tailings end up in a tailings pond, often in the form of an aqueous particulate suspension at low solids concentration. These suspensions settle over a period of years and pose numerous environmental hazards. Because of the dilute nature of these suspensions, the ponds cover large areas and require extensive dam wall construction. Over the last two decades, the trend has been towards disposing at increasingly higher solids concentrations to reduce the land area requirements and allow quicker land reclamation. High solids disposal is achieved through the use of settling aids called flocculants in large scale gravity settling equipment. Contaminated liquors can also cause degradation by leaching into the water table and poisoning sensitive ecosystems. Many ponds are clay and even plastic lined to prevent leakage, but to minimise the risk, liquors need to be cleaned to remove chemicals such as cyanide, sulphuric acid, sodium hydroxide and poisonous organic chemicals. This decontamination can be achieved through repeated washing and dewatering of suspensions in washer trains. Washing generally involves a series of gravity settlers, but often includes alternative separation methods such as filtration and liquor removal from sand layers in tailings ponds.

For example, the Bayer process produces alumina from bauxite ore and in Australia generates over two tonnes of fluid tailings for each tonne of bauxite mined. The process involves grinding bauxite ore and dissolving soluble alumina compounds in a concentrated caustic solution at an elevated temperature. The insoluble residue, containing mainly iron and silica compounds is separated through gravity settling aided by flocculant addition. This phase of mud washing is focused on clarification of the thick aluminate rich liquor at over 100°C. The underflow residue suspension is then repeatedly washed and dewatered in a train of washers to recover caustic for repeated use in the process. The final stage dewateres to a high solids suspension that is pumped to landfill, where caustic liquor is recovered. The alkaline nature of the resulting suspensions makes revegetation difficult and is a key environmental concern. Maximising the caustic recovery and the final solids concentration requires improvement in the dewaterability of these
suspensions and more efficient operation of process equipment. Unfortunately, most flocculant selection and dosing is based on settling tests that optimise liquor clarification but ignore the dewaterability of the residue suspension.

A challenge for all minerals industries is to optimise process conditions for the recovery of liquor contaminants from residue suspensions. This thesis describes techniques to characterise suspension dewaterability and tools to predict the extent of dewatering in compression.

Compressional dewatering is a method of removing liquid from particulate suspensions through the application of a net compressive force. A mathematical theory of compressional dewatering of flocculated suspensions was developed by Buscall and White (Buscall and White 1987). They established compressibility and permeability as physical properties that determine the dewaterability of flocculated suspensions. The theory assumes that when the solids volume fraction, $\phi$, is above the gel point, $\phi_g$, the suspension forms a continuous network structure from the aggregation of flocs (Landman et al. 1991). The gel point, $\phi_g$, is defined as the minimum concentration at which a suspension forms a continuous network structure which transmits its weight to the suspension below. The compressibility determines the concentration to which a suspension can be compressed under an applied pressure. Compressibility is defined as a compressive yield stress, $P_y(\phi)$, which is the minimum compressive stress required for the network structure to compress irreversibly and is a function of solids volume fraction. Permeability, which influences the rate of dewatering is quantified with a hindered settling function, $R(\phi)$, which accounts for the hydrodynamic interaction between particles and is also a function of solids volume fraction. Using experimental $P_y(\phi)$ and $R(\phi)$ data, the dewatering behaviour for a range of solid-liquid separation equipment can be predicted from the theory of Buscall and White (Buscall and White 1987). Examples of process equipment that have been modelled using this theory include continuous flow gravity settlers (Landman et al. 1988), pressure filters (Landman et al. 1991; Landman and White 1994; Landman et al. 1995; Landman and White 1997) and batch settling processes (Howells et al. 1990; Landman and White 1994).

This thesis presents techniques that can be used to characterise dewaterability and predict dewatering performance in washer trains and tailings dams for use as optimisation tools. The techniques build on the PhD works of Nguyen (Nguyen 1983a) and Pashias (Pashias 1997) where the shear and compressional rheology of red mud suspensions were characterised. The samples characterised were underflows from the final stage of washing before being pumped to tailings dams. The work in this project extends analysis of shear and dewatering properties to entire washer trains. This thesis also builds on the work of Green (Green 1997) where
techniques were developed for the characterisation of suspension dewaterability and attempts were made to predict the operation of process equipment. These techniques have been developed further to allow characterisation at elevated temperatures and in reduced time. Experimental results have been applied to predict the operation of process equipment.

1.2 RESEARCH OBJECTIVES

The research objectives were to validate the fundamental mathematical dewatering model proposed by Buscall and White (Buscall and White 1987) through application to a number of dewatering operations including pressure filters, thickeners and consolidation in tailings dams and to subsequently enable improvements in the dewatering performance of industrial processes. The process under investigation in this project is the washing and dewatering of red mud suspensions in countercurrent washer trains at three Australian alumina refineries. The refineries include the Pinjarra Alumina Refinery (ALCOA, Western Australia), the Worsley Alumina Refinery (Worsley, Western Australia) and the Queensland Alumina Refinery (QAL, Queensland, Australia). In this project, achieving the research objectives required that a number of sub-tasks be completed:

Develop suspension characterisation and modelling tools

- Develop suspension dewaterability characterisation techniques that are quick and reproducible.
- As a model system, use zirconia suspensions to validate the experimental techniques developed.
- Model the effect of processing changes on dewaterability and dewatering performance in red mud thickeners, washer trains, rotary drum filters and residue disposal areas.
- Establish dewaterability characterisation methods and dewatering modelling tools as standard industry measures of process performance.

Application of tools to red mud dewatering

- Characterise red mud dewaterability over the entire washer train at each sponsor site.
- Identify the physicochemical reasons for thixotropy in red mud suspensions.
- Optimise the washing and dewatering of red mud suspensions.
- Model the operation of thickeners/washers and other dewatering equipment in red mud washer trains.

Improve knowledge of how dewatering is influenced by processing conditions

- Identify the factors that influence suspension dewaterability.
• Quantify the effect of flocculant chemical type and dose on the extent of dewatering.
• Predict the relative importance of process variables such as flocculation and shear processes on dewatering performance.
• Provide a tool for predicting the effect of a range of processing conditions such as flocculant dose on dewatering performance.
• Develop methods of optimising dewatering performance through exploiting potential improvements that are identified through characterisation and modelling.
• Identify research directions that will produce further improvements in knowledge and process performance.

The long term objectives involve developing and marketing the technical tools in this body of work to the stage where they are accepted as dewatering industry standards and incremental improvements in knowledge to the eventual extent that these tools form the basis of dewatering equipment design.

1.3 THESIS OUTLINE

This thesis is directed towards gaining an understanding of the flow and dewatering behaviour of particulate suspensions. Particular attention is given to process variables which influence the flow and dewatering behaviour of red mud suspensions.

This thesis is broken into four key sections. In Section A, the purpose of the research is introduced along with a background to tailings issues in the minerals industries and an overview of the basic scientific concepts on which the project builds. In Section B, an array of suspension characterisation and equipment modelling tools are introduced. In Section C, the results of red mud characterisation and dewatering equipment modelling is presented. Finally, in Section D, the results are summarised and interpreted leading to discussion of directions for optimisation, conclusions and further work.

1.3.1 SECTION A: INTRODUCTION AND THEORY

In Chapter 1, the motivation for the research is overviewed, followed by a statement of the research objectives and an outline of the thesis structure.

Chapter 2 provides a background to mineral tailings disposal with reference to the specific issues faced in the alumina industry and how they are addressed in red mud washer trains and tailings disposal.
Chapter 3 provides an introduction to the scientific concepts that are used to tackle flow and dewatering issues. Initially the concept of a particulate suspension is introduced. Then, the way in which particulate suspensions exhibit non-Newtonian shear rheology is described. The dewatering model of Buscall and White (Buscall and White 1987) is presented with the physical properties compressibility, permeability and diffusivity defined in terms of the model. The effects of flocculation and shearing processes such as raking are also overviewed along with current practices in terms of characterisation and modelling techniques.

1.3.2 SECTION B: CHARACTERISATION TECHNIQUES AND MODELLING TOOLS

Chapter 4 details the analytical techniques used to characterise properties of red mud suspension liquors including density and viscosity. In addition, methods of determining the solids concentration are also reviewed.

Chapter 5 reviews the experimental methods used to characterise the shear rheology of particulate suspensions in terms of the shear yield stress, shear stress versus shear rate behaviour and thixotropy.

Chapter 6 describes experimental methods used to characterise particulate suspension dewaterability in terms of compressibility, permeability and diffusivity.

Many of the techniques described in Chapters 4, 5 and 6 are considered relatively generic. However, the methods have been presented with the specific requirements of characterising red mud suspensions at process temperatures in mind.

Chapter 7 describes the characterisation techniques used to comprehensively characterise red mud suspensions. Methods of curve fitting compressibility and permeability data are described for subsequent use in modelling dewatering performance. The mathematical models and algorithms used to predict the operation of pressure filtration, steady state thickening and tailings dam consolidation are also presented.

1.3.3 SECTION C: EXPERIMENTAL CHARACTERISATION AND MODELLING RESULTS

Chapter 8 introduces the red mud characterisation and process modelling work program conducted.
Chapters 9, 10 and 11 contain the detailed results of red mud washer train suspension characterisation and dewatering equipment modelling for the ALCOA Pinjarra, Worsley and QAL refineries respectively.

1.3.4 SECTION D: SUMMARY OF RESULTS, CONCLUSIONS AND FUTURE DIRECTIONS

Chapter 12 overviews the generic properties of red mud suspensions.

In Chapter 13, the key observations resulting from comparisons of thickener dewatering predictions with operational reality are summarised. Then, thickener performance influences are overviewed with a discussion of future directions for optimisation of process performance.

Chapter 14 provides an overview of the conclusions and major outcomes of the work measured against the research objectives, followed by a summary of potential further work.
2.1 TAILINGS AND THE AUSTRALIAN MINERALS INDUSTRY

Mineral Processing

The mining industry is involved in the business of digging up an ore body, crushing the ore and extracting the valuable product through an extensive hydrometallurgical and/or pyrometallurgical processing operation before disposing of the waste tailings. After crushing, particles containing the highest mineral value are concentrated and then, in hydrometallurgical operations, an aqueous dissolution process is typically employed to extract the valuable product in strongly acidic or basic conditions. The unwanted insoluble residue tailings are sometimes partially decontaminated through a washing and dewatering process before being disposed of, typically in a tailings dam. In some circumstances, an end use is found for this waste product.

As already noted in Chapter 1, many ores produce more tailings than product, with gold being a notable example (Wills 1976; Boger 1998). Historically, as the high yielding ore bodies have been exhausted, developments have enabled many ore bodies to be viably mined with increasingly lower yields (CRNET 1972). The continuing trend of processing increasingly larger volumes of lower grade ore will inevitably produce greater volumes of waste (Pashias 1997).

Australian Tailings Production

On a world scale, Australia is a major player in the minerals industry, and a major producer of tailings. Table 2.1 shows the Australian production and world market share for the major minerals mined and processed in Australia. Australia is the world’s largest producer of alumina, diamonds, ilmenite, rutile and zirconia and also a major player in the mining and processing of many other metals (Boger 1998). As a result, the largest producer of waste in Australia is the mining industry (Boger 1998). Mining and mineral processing accounts for about 80% of the solid waste produced in Australia (Boger 1998).
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina</td>
<td>13.3 million tonnes</td>
<td>30 %</td>
</tr>
<tr>
<td>Copper</td>
<td>186 000 tonnes</td>
<td>1.8 %</td>
</tr>
<tr>
<td>Diamonds</td>
<td>42 million carats</td>
<td>35 %</td>
</tr>
<tr>
<td>Gold</td>
<td>243 tonnes</td>
<td>10 %</td>
</tr>
<tr>
<td>Lead (refined)</td>
<td>228 300 tonnes</td>
<td>3.9 %</td>
</tr>
<tr>
<td>Nickel (refined)</td>
<td>73 000 tonnes</td>
<td>7.8 %</td>
</tr>
<tr>
<td>Silver</td>
<td>1020 tonnes</td>
<td>6.9 %</td>
</tr>
<tr>
<td>Titanium (concentrate)</td>
<td>2.2 million tonnes (50 % TiO$_2$)</td>
<td>24 %</td>
</tr>
<tr>
<td>Zinc (ore)</td>
<td>1.008 million tonnes</td>
<td>13.8 %</td>
</tr>
<tr>
<td>Zirconium (concentrate)</td>
<td>462 000 tonnes</td>
<td>46 %</td>
</tr>
</tbody>
</table>

Table 2.1: Australian minerals production (BGS 1998).

13.3 million tonnes of alumina was produced from bauxite in Australia in 1996, accounting for 30 % of world production (BGS 1998). Since then, Australian capacity has expanded to 16 million tonnes of alumina per annum, increasing market share to 33 % in 2000-2001 (Alcoa 2001c; Nabalco 2001; QAL 2001; Worsley 2001). In the future, with planned expansions to a number of Australian refineries and with Comalco to build a new refinery with an initial capacity of 1.4 million tonnes per annum by 2005 (Comalco 2001), Australian alumina production will soon exceed 20 million tonnes per annum. This will produce over 40 million tonnes of red mud waste tailings (dry solids basis) per annum. These tailings have the potential to fill 3.3 square kilometres of tailings dams 10 metres deep, at a solids concentration of 1200 g/L, every year. Relative to the total landmass of the country, this lost area is not very significant, but ongoing management of these areas is expensive.

Australia was the 3rd biggest producer of gold in 1996, producing 243 tonnes which is over 10 % of world production (BGS 1998). Since gold extraction produces very low yields, the process also produces a massive amount of discarded tailings. The massive quantity of tailings produced is mirrored in the production of the other minerals as well, creating a very significant management issue.

**Decontamination & Disposal**

The major environmental impact from mineral processing is due to solid waste disposal, through severely affecting air, water and land resources (Norman 1972). However, these effects can be minimised if the problems are anticipated, impact minimisation strategies are adopted and follow up efforts are employed (Norman 1972). The challenge is to balance the economic objectives of mining and mineral processing with the social and environmental consequences of the operation. At the outset, it needs to be recognised that the tailings produced by many mineral extraction processes are highly toxic, requiring decontamination and potentially long term containment. Continual improvements in processing technology is enabling finer ground ores to be treated. Mine cycle analysis suggests that stabilisation and rehabilitation of finer tailings is increasingly expensive and exacerbates problematic leaching of unwanted materials.
from the solids in tailings dams (Sutherland and Richards 1997; Sutherland 1998). One of the major shifts in recent decades is the trend towards high solids disposal, including paste backfill. Achieving higher solids concentrations where fine particles are involved has required improved understanding of how to manipulate and exploit the complex rheological and dewatering properties of tailings (Pashias 1997). This is achieved through deliberate process modifications (de Kretser 1995) and through additives such as pH modifiers, dispersants and flocculants (Scales et al. 1998a). Improved material properties and understanding of the process allows solid-liquid separation and pumping equipment optimisation.

2.2 DEWATERING AND DISPOSAL METHODS

All mineral processing operations use different ore-bodies as raw materials which are processed under different physical and chemical conditions with a wide variety of processing equipment. Understanding and exploiting these differences requires an appreciation of the process unit operations employed. Some methods utilise thermal drying processes, but the energy costs are very high and generally not applicable to the massive quantities involved in minerals processing. The majority of dewatering operations utilise one of two physical phenomenon. The first is a pressure gradient driven by either a vacuum or fluid pressure in filtration operations. The second is solid-liquid density differences exploited through gravitational or centrifugal acceleration to be utilised in settling, compression and centrifugation. These methods have been overviewed in the Kirk-Othmer Encyclopedia of Chemical Technology (Nagaraj 1995) and Perry’s Chemical Engineer’s Handbook (Dahlstrohm et al. 1997b) provides a higher level of detail. What follows in this section is a brief overview of pressure filtration, centrifugation, gravity thickening and tailings disposal operations employed in the minerals industry for the washing, dewatering, disposal and rehabilitation of tailings suspensions.

2.2.1 PRESSURE FILTRATION

An extensive review of pressure filtration equipment is presented in the Kirk-Othmer Encyclopedia of Chemical Technology (Svarovsky 1993). In general, filtration operations involve either surface filters, where a membrane or cloth barrier prevents the passage of particles, or deep bed filters where for instance sand layers provide the solids barrier. The purposes of filtration processes include solids removal from liquor, washing of suspension liquors and also suspension dewatering to form a high solids cake. Suspensions are often pre-treated to improve filtration properties through the addition of chemical additives such as coagulants, pH modifiers, flocculants and permeable solids that act as filter aids and reduce membrane fouling. Physical methods of enhancing dewatering include mechanical squeezing of
filter cakes and the application electric and magnetic fields. The filtration pressure can be provided through a hydraulic liquid pressure applied with a pump, through forced displacement, such as with a piston, and under vacuum from the other side of the membrane or filter cloth. Filtration processes can be either batch or continuous. Batch pressure filters include plate and frame filter presses, pressure vessel filters, cartridge filters, mechanical batch compression filters and presses and fixed bed centrifuge filters. Continuous pressure filters include disk filters, drum filters, horizontal belt pressure filters, continuous compression filters, thickening pressure filters and moving bed centrifuge filters. Examples of vacuum filters include the Nutsche filter, which is an industrial scale equivalent of the Buchner funnel, the vacuum leaf filter, the tipping Pan Filter, the horizontal rotating pan filter, the horizontal belt vacuum filter, the rotary drum vacuum filter and the rotary vacuum disk filter. The most popular vacuum filter is the rotary drum filter.

2.2.2 CENTRIFUGATION

Centrifugation is a common method of exploiting solid-liquor density differences in dewatering processes. It is most commonly applied to small scale batch processes in the laboratory as a quick method of attaining suspensions at high solids concentrations and/or removing solids from liquors. On a large scale, the equipment can be very complex, expensive to operate and thus inappropriate for the high throughputs involved in the minerals industry. Nevertheless, numerous niche applications have been developed and an extensive review is presented in the Encyclopedia of Separation Technology (Letki et al. 1997). Letki et al. describe a number of batch and continuous systems and suggest that the factors influencing centrifugal separation include viscosity, solid-liquid density difference and particle size, shape and size distribution in addition to sedimentation velocity.

2.2.3 GRAVITY THICKENING

Gravity thickening operations are the most common methods of dewatering employed in the minerals industry for processing substantial suspension volumes, because of the relatively low costs involved. The purposes of the dewatering include either or both of liquor clarification and dewatering to high solids concentrations and often form part of a large counter-current washing and dewatering process known as a washer train. As such, the vessels involved are often called clarifiers, washers, settlers and thickeners, but the equipment involved is basically the same and it is just the position in the process or the purpose of the equipment that often determines its name. In both clarification and dewatering, other methods such as filtration can achieve better performance, but at significant financial cost. Like centrifugation, gravity sedimentation equipment exploits the solid-liquid density difference in particulate suspensions to achieve
separation. For some applications, additional external forces are employed to assist with material flow and dewatering through rakes, shearing rods, sloped vessel walls, ultrasound, electric fields and magnetic fields.

Reviews of sedimentation equipment are presented in the Chemical Engineer’s Condensed Encyclopedia of Process Equipment (Cheremisinoff 2000), the Kirk-Othmer Encyclopedia of Chemical Technology (Svarovsky 1997) and Perry’s Chemical Engineer’s Handbook (Dahlstrohm et al. 1997b). The most common form of sedimentation device involves a large tank where a dilute suspension feed is mixed with chemical additives (most commonly flocculants) to aggregate particles and improve the settling rate in a central feed well. The particles settle and form a bed. The suspension bed flows and is directed towards an underflow outlet at the base of the thickener through rakes and sometimes the converging base of the thickener. The underflow is generally pumped away using a centrifugal pump. Near the top of the thickener, a virtually solids free liquor zone develops. The liquor at the top is often designed to overflow the top of the thickener and be collected in a trap and pumped away for further processing. In some processes, a layer of undesirable scum forms on the top of the liquor and the clarified liquor is collected through an outlet port slightly below the top of the liquor.

The influences on thickener performance include suspension material properties and many operational variables. The relevant material properties are not consistently defined in the literature because fundamental knowledge is incomplete and research is ongoing. In general, key influences include particle size, shape and size distribution, solid-liquid density differences, liquor and suspension viscosities, surface properties such as zeta potential and the influence of chemical additives in influencing floc structure. The influence of these factors on settling rate/permeability, compressibility and shear rheology is of critical importance. The operational variables include, but are not limited to, feed solids concentration, solids throughput, suspension bed height, process temperature, tank geometry, rake design and rake speed.

2.2.4 TAILINGS DISPOSAL, CONSOLIDATION AND REHABILITATION

Ideally, waste tailings suspensions from mineral processing operations are decontaminated and then dewatered to a high solids concentration allowing safe disposal, further consolidation and then rehabilitation. In some cases, the tailings may even be put to practical use as a feedstock for another industry or process. Unfortunately, this is often not easily achieved, if at all possible. Complete decontamination is often unattainable due to slow leaching of toxic chemicals from the porous tailings over many years. In addition, it is often very difficult and
expensive to dewater fine tailings to high solids concentrations and then transport the material to disposal areas, though continual progress is being made.

**Disposal & Containment Issues**

The majority of tailings disposal involves long term containment in tailings ponds, otherwise termed residue disposal areas (Nagaraj 1995). There have been at least 69 tailing dam failures since 1960, where hundreds of people have died, hundreds of homes have been destroyed, thousands of kilometres of rivers have suffered severe environmental damage and the financial costs have run into many millions of dollars (WISE 2001). These failures included wall breaches, wall collapses, overflows and other containment issues caused by earthquakes, excessive rain, stormy weather, foundation failure, leaky seals, erosion and mismanagement. The sad fact is that the issue of long term management of storage facilities is often overlooked (Pashias 1997) and many of these disasters are preventable (Sofra’ 2000; WISE 2001).

**Disposal Methods**

Today, most tailings are thickened before discharge into residue disposal areas to recover valuable process water and reduce the volume of tailings (Nagaraj 1995). This lower volume of tailings requires a smaller disposal area, lowers management costs and reduces the environmental hazard. To ensure containment, the coarse tailings fraction is often used for pond dam wall construction. In addition, clay and plastic lining of the dam are often used to prevent seepage of contaminated water and potentially hazardous chemicals into the surrounding natural environment through the underground water system, rivers and streams (Nagaraj 1995).

The extent to which tailings are dewatered before disposal, varies significantly throughout the world. In the past, the environmental consequences and long term costs of tailings disposal methods were not a major consideration (Kealy and Busch 1979; Pashias 1997; Boger 1998). However, through increasingly higher community expectations and more stringent environmental legislation, tailings disposal has become an important component of process design (Wood and McDonald 1986; Pashias 1997). Pashias (Pashias 1997) has conducted a review of tailings disposal operations where suspension solids concentrations range from the historically very dilute suspensions to current goals of maximum practically achievable solids concentrations in thickened tailings disposal and dry stacking. This review is summarised below. As an addition to these disposal methods, paste backfill is increasing in use because of the small associated footprint.
**Wet Disposal (Pashias 1997)**

Historically, tailings were typically produced at low solids concentrations below 30 % by weight. Wet disposal involves the discharge of these dilute tailings into surface impoundments with constructed embankments or dams to complete the containment. The coarser fractions of the tailings themselves are used to construct the embankments (Cooling 1989; Cooling and Glenister 1992; Hutchison and Ellison 1992). The tailings slurry is transported by pipeline and deposited by either point or line discharge. The tailings are allowed to consolidate and the subsequent clarification of the effluent allows its reuse. The high volume of tailings with low structural stability involved in wet disposal leads to a high risk of dam failure. Wet disposal also increases the threat of tailings seepage into the surrounding environment and groundwater. Additionally after decommissioning the tailings ponds remain difficult to cap due to the low mechanical stability of the dilute tailing (Cooling 1989).

**Semi-Dry Disposal (Pashias 1997)**

Thickening of suspensions prior to disposal produces deposited tailings at intermediate solids concentrations of 40-60 % by weight (Cooling 1989; Cooling and Glenister 1992). Semi-dry disposal of tailings usually involves depositing the tailings over an impoundment area that is underlain by a drainage and seepage collection system. By controlling the discharge, consecutive batch areas can be covered. Once an area has been covered, the discharge pipe is moved and the area allowed to dry. As layers of fresh tailings are deposited, the water is absorbed by the dried layers immediately below. One advantage of this method of disposal is better utilisation of the available storage area. Furthermore, the dried nature of the tailings offers benefits including, increased resistance to flow, the option for more immediate capping and rehabilitation as well as reducing the potential for seepage (Cooling 1989). Disadvantages include the high labour costs of using amphibious dozers to move the mud around to ensure even drying and to break the surface crust which can inhibit drying of the suspension below (Cooling and Glenister 1992). Alternatively, in cases of excessive drying, erosion can be a problem (Cooling and Glenister 1992).

**Thickened Tailings Disposal (Pashias 1997)**

Thickened tailings disposal starts with a tailings slurry thickened to as high a solids concentration as is practically pumpable and involves discharge from an elevated position in the centre of the impoundment to create a conical stack (Robinsky 1975; Shields 1975; Robinsky 1986; Wood and McDonald 1986; Yeomans 1986). The higher solids concentration gives the suspension increased mechanical stability, which is exploited by the cone formation (Poulos et al. 1985). The advantages of the cone include reduced dyke perimeter height and construction
costs or alternatively reduced land area requirements. In addition to improved recovery of valuable process water, product and hazardous chemical contaminants, thickened tailings disposal provides better impoundment stability and quicker land reclamation. The disadvantages include increased thickener construction costs and issues associated with transportation of high solids tailings. Also, this method requires a consistent feed solids concentration to maintain the integrity of the cone and ensure that the dykes don’t overflow (Sofra 2000).

**Tailings Consolidation**

Once in the residue disposal areas, the tailings tend to consolidate further. Depending on the permeability/settling rate of the suspensions, this process can be virtually complete in a number of months, or can last many decades. Settling and consolidation of solids in tailings ponds can be problematic in situations where fine particles and clay suspensions are involved. In these slow settling cases, careful planning is required to ensure that consolidation can be achieved. Failure to consolidate to a mechanically stable solids concentration can result in a permanent environmental hazard that can not be rehabilitated. During consolidation, liquor, lost through surface evaporation, can be decanted from the pond surface and can also removed from strategically placed sand layers underneath and within the consolidating bed. In this way, toxic liquor can be recovered from contaminated tailings for as long as is required to hasten site rehabilitation. The rate of liquor recovery and evaporation is hindered by the permeability of the mud and the development of a hard thin crust on the surface of the drying area (Cooling and Glenister 1992). Ploughing the surface of the drying area can destroy any crust that may form. Often, when significant drying has been achieved at the surface, dust and erosion can become a problem (Cooling and Glenister 1992). This has been addressed at Worsley by ploughing to create a rough surface that buffers the effects of wind and keeps enough moisture at the surface in an attempt to maintain mechanical strength and structural integrity. However, this ploughing method also has the potential to increase the surface area and drying rate, thus increasing the erosion problem in hot weather with high winds, where dampening of the surface with irrigation sprays can be more effective (Cooling and Glenister 1992). Finally, when the tailings surface is decontaminated, it is time to cap the consolidated tailings, add topsoil and revegetate the surface. Regardless of surface rehabilitation, contaminated liquor recovery from bore holes into the consolidated tailings is often required for many more years.
2.3 ALUMINA AND THE BAYER PROCESS

2.3.1 INTRODUCTION

In the production of aluminium from bauxite, the intermediate alumina (aluminium oxide) is produced through the Bayer process. Austrian chemist Karl Bayer patented this process in 1888 (Jarrett 1987). It is still the most economic method of alumina production and is used to produce virtually all alumina produced in the world today. The process utilises aqueous caustic soda to dissolve and extract the alumina present in the bauxite ore in the form of gibbsite, boehmite and diaspore. The Bayer process involves four basic steps: digestion, clarification, precipitation and calcination. Every alumina refinery process is different, but the basic process is still universal. Each of the four steps is overviewed in the sections that follow, which are collated from a number of references (Hudson 1987; Jarrett 1987; Alcoa 2001b; QAL 2001; Worsley 2001). More detailed site specific information for all Australian refineries can be found in Australasian Mining and Metallurgy (Woodcock and Hamilton 1993) and the respective company websites (Alcoa 2001c; Nabalco 2001; QAL 2001; Worsley 2001).

Terminology

Before describing the Bayer process, it is important to recognise that there are many terms that are peculiar to this industry. For example, the process solution, called liquor, is referred to as green or pregnant liquor when it has a high alumina content and it is called spent liquor after precipitation of alumina from solution. The remaining insoluble residue after the digestion of bauxite is interchangeably referred to as both red mud and bauxite residue. The different forms of alumina are given industry specific names. Gibbsite (Al(OH)$_3$) is referred to as trihydrate or just hydrate and given the chemical formula Al$_2$O$_3$.3H$_2$O, while boehmite (AlOOH) and diaspore (Al$_2$O$_3$.H$_2$O) are both referred to as monohydrate and given the same chemical formula, Al$_2$O$_3$.H$_2$O. However, gibbsite and boehmite are not strictly hydrates at all (Hudson 1987).

2.3.2 DIGESTION OF BAUXITE

Grinding & Desilication

Bauxite is finely ground in rod and ball mills to allow better solid liquid contact during digestion. Some recycled caustic soda solution, known as spent liquor, is added to produce a pumpable slurry. Lime is introduced for phosphate control and to regenerate sodium hydroxide, allowing the insoluble calcium carbonate to be removed with the waste mud according to the following reaction (Hudson 1987; QAL 2001).
\[ \text{Ca(OH)}_2 + \text{Na}_2\text{CO}_3 \rightarrow \text{CaCO}_3 + 2\text{NaOH} \quad (2.1) \]

The reactive silica component of the bauxite is dissolved in spent liquor containing concentrated sodium hydroxide. In solution, this dissolved silica then reacts with dissolved alumina and sodium hydroxide to precipitate as insoluble desilication products (DSP) (QAL 2001). The reaction results in a largely non-recoverable loss of alumina and caustic from the process (Hudson 1987). The slurry is held for several hours in agitated tanks to react most of the reactive silica to DSP prior to digestion and minimise scaling in downstream equipment and contamination in the final alumina product (Hudson 1987).

**Digestion**

The bauxite slurry is then digested in hot caustic soda solution to dissolve the alumina. Digestion involves the sodium hydroxide preferentially dissolving alumina under pressure at elevated temperatures (Jarrett 1987). The sodium hydroxide concentration, temperature and pressure required depend on the least reactive form of alumina present in the ore, which can include gibbsite, boehmite and diaspore.

Digestion of gibbsite (trihydrate), the most reactive form of alumina, is achieved at relatively low temperatures (under 200°C) and pressures (Worsley 2001). When there is less reactive boehmite (monohydrate) present, reaction conditions required to extract the monohydrate alumina are more extreme, being about 250°C and 3500 kPa at QAL (QAL 2001). Extraction of diaspore requires even more extreme conditions (Hudson 1987). Under the appropriate conditions, the following dissolution reactions are rapid:

\[ 2\text{NaOH} + \text{Al}_2\text{O}_3.3\text{H}_2\text{O} \rightarrow 2\text{NaAlO}_2 + 4\text{H}_2\text{O} \quad (2.2) \]

\[ 2\text{NaOH} + \text{Al}_2\text{O}_3.\text{H}_2\text{O} \rightarrow 2\text{NaAlO}_2 + 2\text{H}_2\text{O} \quad (2.3) \]

The digestion product is a concentrated sodium aluminate solution with undissolved impurities including principally inert iron and titanium oxides and silica compounds (QAL 2001). The slurry leaving digestion is flash-cooled to atmospheric boiling point by flowing through a series of flash vessels. Condensate from the flash process is used for boiler feed water and washing waste mud (QAL 2001).

### 2.3.3 CLARIFICATION OF THE LIQUOR STREAM

The alumina rich liquor is separated from the insoluble red mud residue, which is disposed of as waste in most cases. Separation is achieved by settling in a thickener, and then filtration to remove the remaining fine particles. The thickener underflow suspension is subjected to further
processing via a counter current washer train to recover process liquor components and allow residue disposal at low moisture content, thus minimising the level of pollutants accompanying the output residue (Worsley 2001). In many processes, large sand particles are removed via a hydrocyclone prior to the thickener or the first washer to improve handleability.

**Thickeners/Settlers & Washers**

Most red mud waste solids are settled from the liquor stream in large-scale gravity thickeners/settlers at 100-110°C. Flocculants are added to the settler feed stream to aggregate solid particles into flocs that improve mud settling rate and improve clarity in the overflow liquor (Hudson 1987; QAL 2001). The suspension feed enters through the feed well in the centre of the vessel. The liquor moves to the overflow launder at the perimeter, from where it is pumped to filtration, while the solid particles consolidate in a suspension bed at the base and are raked to an outlet and pumped to a washer train.

The thickener underflow suspension is washed with water in counter-current washer trains to recover the soda and alumina content in the mud before being pumped to large disposal impoundments, termed residue disposal areas (QAL 2001). A washer train usually involves four to seven washers operated in series, as shown in Figure 2.1. The inputs to the washer train include the underflow from the thickener which is fed to the 1st washer and water (most often process condensate) which is fed to the last washer. The outputs from the washer train include the concentrated 1st washer overflow liquor and the washed high solids concentration final washer underflow. The feed to any washer is composed of the underflow from the previous washer (or thickener if 1st washer) and the overflow from the next washer (or condensate if final washer) and process additives such as flocculants and lime.

Measuring the settling rate as a function of solids concentration enables equipment performance to be estimated, but because of fragile flocs and laboratory limitations, the equipment usually out performs these predictions (Hudson 1987).

In some plants alternative process equipment, such as vacuum filters (Worsley 2001), high rate/deep bed thickeners or very large diameter thickeners (Cooling 1989) now known as superthickeners are used for the final stage of thickening. The purpose of this final stage is to ensure high solids concentrations in the underflows that maximise the recovery of valuable process liquor prior to residue disposal.
Figure 2.1: Washer train flowsheet.

**Filters**

Thickener/settler overflow liquor containing traces of fine mud is filtered in Kelly-filters, most often using polypropylene filter cloth (Hudson 1987; QAL 2001). Slaked lime slurry or desilication product, DSP, is used as a filter aid to produce a permeable filter cake (Hudson 1987; QAL 2001). Solid particles are held on the filter leaves for removal when filters are taken off line, and added to red mud washer feeds (QAL 2001).

### 2.3.4 PRECIPITATION OF ALUMINA HYDRATE

The clarified and filtered pregnant liquor is cooled, seeded and cooled again to a final temperature of 60-70°C, in several flashing stages to maximise supersaturation and to precipitate alumina as gibbsite (hydrate) (Hudson 1987). The steam produced in flash evaporation is used to heat spent liquor returning to digestion (QAL 2001). The hydrate suspension is thickened, separated by filtration and then washed to remove sodium hydroxide and other impurities (Worsley 2001).

**Crystallisation**

In 20-30 m tall agitated vessels known as precipitators, with a holding time of about 3 hours, dissolved alumina is recovered from the liquor by precipitation of gibbsite crystals in a reaction which is the reverse of digestion (Hudson 1987; QAL 2001):

\[
2\text{NaAlO}_2 + 4\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3.3\text{H}_2\text{O} + 2 \text{NaOH}
\]  

Precipitation occurs in series of 6-13 precipitators which are seeded with crystalline gibbsite, with an intermediate or fine particle size, to promote crystal growth (Hudson 1987; QAL 2001). A wide crystal particle size distribution is produced and since correct particle size is important to smelter operations (QAL 2001), classification is necessary.
Classification

The precipitated suspension is settled from the liquor and separated into 3 size ranges, fine, intermediate and coarse (QAL 2001). The coarse fraction becomes the product hydrate, while the intermediate and fine crystals from the secondary and tertiary classifiers are returned to the precipitation tanks as seed (QAL 2001).

Spent Liquor

Spent caustic liquor is re-concentrated through an evaporation stage, heated by heat exchange with incoming liquor and returned to digestion with fresh sodium hydroxide added to make up for process losses (QAL 2001).

2.3.5 CALCINATION OF ALUMINA

Classified coarse hydrate (+45 µm) is washed, filtered and then heated to a temperature of around 900-1100°C (QAL 2001; Worsley 2001). This heating process is called calcination or roasting and occurs in rotary kilns or fluidised beds of hot gas from the combustion of natural gas or oil (Hudson 1987; Worsley 2001). Fluidised bed calciners are more energy efficient than the older rotary kilns (QAL 2001). The purpose of calcination is to remove both free moisture and chemically-combined water from the hydrate (QAL 2001), for conversion to anhydrous alumina (aluminium oxide), a fine, white powder with a purity in excess of 99% alumina (Worsley 2001). Calcined alumina then is cooled to less than 90°C, in a number of stages, before it is discharged onto conveyor belts and transported to stockpiles ready for shipment (QAL 2001).

2.3.6 WASTE PRODUCTION AND MANAGEMENT

The Nature of Red Mud

Bauxite residue suspension (red mud), the waste suspension remaining after alumina extraction in the refinery, always contains some unrecovered dissolved liquor contaminants. One of these contaminants is sodium hydroxide where the two principal reasons for the incomplete recovery are the high porosity of the red mud solids (Sarmiento 1978; Geppert 1981) and DSP in the residue exchanging Na⁺ for H⁺ in contact with water (Hudson 1987). Both of these factors cause the slow release of dissolved liquor contaminants over a number of years. Thus red mud has a persisting alkaline nature, cannot be allowed to enter the ground water and must be properly managed (Hudson 1987; Alcoa 2001b). The other liquor contaminant components include dissolved alumina and toxic organics such as oxalates. Tailings management provides a significant environmental challenge to the alumina industry, that coupled with the cost reduction
of improved efficiency, has been a driving force behind improvements in residue storage practices (Cooling and Glenister 1992). The improvements have generally involved disposal at higher solids concentrations, which increases handling complexity. At high solids concentrations, red mud suspensions exhibit a shear yield stress in addition to displaying time and shear history dependant rheological behaviour in the form of thixotropy (Nguyen 1983a; Nguyen and Uhlherr 1983; Nguyen and Boger 1985b; Pashias 1997). Rheological properties in shear and compression form the basis for the design of pipeline transport and dewatering operations and thus high solids suspensions require different processing regimes to low solids suspensions.

Disposal & Containment of Red Mud Solids

The red mud disposal methods available include pumping to a marine environment, forming an impoundment by damming a valley or building dykes on level land. Pumping to a marine environment has been practiced in the past, but environmental concerns have eliminated this option (Hudson 1987). As a result, most of the red mud produced today is contained in impoundments. These impoundments vary in the feed solids concentration, use a variety of methods to contain the suspensions and liquors and have varying levels of sophistication in strategies for liquor drainage.

The traditional method of disposing at low solids concentrations is disappearing. This is due to the obvious benefits of disposing at higher solids concentrations including reduced soda and alumina losses and a smaller initial volume of waste leading to reduced land area requirements. Because of the thixotropic nature of red mud, agitation can reduce viscosity by two orders of magnitude enabling pumping to disposal areas at high solids concentrations (Hudson 1987). Concentrated disposal strategies are now the preferred method of red mud disposal, where the waste suspension is concentrated in thickeners or pressure filters, to over 50 % w/w prior to disposal at Alcoa refineries and 62 % w/w at the Worsley refinery (Alcoa 2001b; Worsley 2001). Some of the sand fraction of the red mud is used in dyke construction. Semi-dry disposal and thickened tailings disposal, introduced in section 2.2.4, are the two most common disposal methods employed in the alumina industry. In short, semi-dry disposal involves disposal of a layer of red mud up to 5 m high and allowing to dry before depositing subsequent layers. Thickened tailings disposal involves disposal of suspension, at a slightly higher solids concentration, from an elevated position at the centre of the disposal area leading to a cone shaped deposit with good mechanical stability that dries quickly and requires minimal dyke construction. Despite higher initial capital costs, disposing at higher solids concentrations allows quicker consolidation and drying to high solids concentrations providing long term
economic benefit from reduced impoundment construction costs, and a reduced land requirement for storage areas (Alcoa 2001b).

Liquor Containment & Recovery

In older dam structures, liquor was prevented from entering ground water by the constructed dykes and assumed to be contained underneath because consolidated red mud is less permeable than clay (Hudson 1987). However construction errors and geological problems have resulted in ground water contamination (Hudson 1987). Options commonly adopted for enhanced containment include lining the impoundment with compacted clay and then with a polymeric membrane (Hudson 1987; Alcoa 2001b; Worsley 2001). The liquor in the residue disposal areas contains valuable process chemicals that are well worth recovering, especially when process water is scarce or when no discharges into the natural environment are allowed. Liquor can be recovered from surface water and from a sand bed and/or a pipe network which can be placed over the lining prior to the disposal of red mud. The surface water comes from rainfall onto the disposal area and liquor from within the consolidating residue suspension.

Recovery of liquor that drains into the sand bed and/or pipe network underneath the residue enhances caustic recovery rates and reduces the possibility of leakage (Hudson 1987; Alcoa 2001b; Worsley 2001). The recovered liquor is usually pumped to a holding pond ready for reuse as necessary in the Bayer process. The holding time in the pond allows biodegradation and photodegradation of some of the organic species that build up in Bayer liquors.

An alternative method to recovery and reuse of red mud liquor is partial neutralisation of red mud with seawater, enabling liquor to be discharged into a river, as is done at QAL, after filtration to remove fine solids (QAL 2001).

Rehabilitation of Residue Disposal Areas

Once the disposal areas are full of consolidated dried residue that is significantly decontaminated, it is time for rehabilitation. As described in section 2.2.4, red mud disposal area rehabilitation involves capping the consolidated tailings, adding permeable topsoil and revegetating the surface with native plants. Even after surface rehabilitation is complete, alkaline liquor recovery from the sand bed and/or pipe network underneath the residue is still required for many years.

Potential uses for red mud as alternatives to disposal

Considering the large quantities of red mud generated in the Bayer process, there is the desire to find practical uses for this product that is generally considered waste. Potential alternatives to disposal are very orebody specific and depend mainly on the composition of impurities. For
this reason, each unique application must be developed for the specific orebody in question, requiring extensive research and development and in many cases is not economically viable. Irrespective of these difficulties, one application includes use as a fertiliser (Alcoa 2001b). Research has shown that red mud can be used to improve agricultural productivity of sandy soils (Alcoa 2001b). Because of its high porosity, bauxite residue has the ability to absorb nutrients such as phosphorous and nitrogen, and can substantially decrease the rate at which these nutrients leach to groundwater (Alcoa 2001b). Pasture improvements of 20-50 percent have been noted in farm trials (Alcoa 2001b). Though the use of what was formerly a waste product as a tool for improving agricultural activity is environmentally advantageous, Alcoa derives no profit from this use (Alcoa 2001b). There is potential for use as a feedstock for the production of iron and titanium dioxide (Mishra et al. 2001) and for the production of a ceramic building material, but these applications still require development.
Chapter 3

THEORY OF SUSPENSION RHEOLOGY AND DEWATERING

3.1 PARTICULATE SUSPENSIONS

An explanation of flow and dewatering phenomenon in addition to numerous concepts and definitions associated with particulate suspensions are introduced in this chapter. Particulate suspensions basically consist of solid particles suspended in a liquid medium. They are everywhere in our everyday world and in the minerals industries and must be handled in countless process operations. In situations where particle sizes are less than 50 µm, the flow and dewatering behaviour becomes very complex, especially at high solids concentrations. Factors that influence this behaviour include solids concentration, particle size distribution, liquor density, liquor viscosity, liquor chemistry, the surface charge on the solid particles, interparticle forces, chemical additives such as flocculants and coagulants and a host of other influences. Solids concentration is an important parameter in describing particulate suspensions and can be described in many ways. Throughout this thesis, the standard method of describing solids concentration is as a solids volume fraction, \( \phi \). In the sections that follow, a brief overview of surface chemistry, flocculation, suspension shear rheology and dewatering theory are presented.

3.2 SURFACE CHEMISTRY

3.2.1 INTRODUCTION

In depth description of colloid and surface chemistry is beyond the scope of this thesis. The theory of colloid and surface chemistry as it applies to high solids particulate suspensions has been reviewed by Pashias (Pashias 1997). As an introduction, the key concepts have been summarised here to help us better understand the implications of particulate suspension surface chemistry on shear and compressive rheology. For more comprehensive surface chemistry information, the reader is referred to texts by Hunter and Israelachvili (Israelachvili 1991; Hunter 2001).

Suspension rheology is influenced by three important phenomenon including Brownian motion, hydrodynamic interactions and colloidal forces. These phenomenon are influenced by
interparticle interactions, particle geometry, species adsorbed onto the particle surface and the nature of the liquid medium. Pashias (Pashias 1997) discussed two limits of idealised suspension behaviour including hard sphere dispersions and soft sphere dispersions. These concepts along with flocs and zeta potential are discussed below.

**Flocs**

A floc is a loose structure of particles that may be deformed or destroyed by the application of stress. Flocs may result from the process of flocculation, which involves particle bridging through the addition of polymer flocculant. Flocs may also form from the coagulation of particles which involves particles attraction due to the electrolytic environment in the suspending fluid medium.

### 3.2.2 HARD SPHERE DISPERSIONS

Hard sphere dispersions involve rigid force free dispersions where only Brownian motion and hydrodynamic interactions are important. This idealised state is approached when the electrical double layer thickness is much smaller than the particle radius and repulsive forces are large enough to prevent the formation of flocs (Hunter 1989).

**Brownian Motion**

Brownian motion determines the rate at which particles rearrange themselves through both translational and rotational movement driven by a statistical distribution of particle thermal energies in the viscous liquid medium. The relative influence of Brownian motion is enhanced as particle size decreases and leads to a range of significant particle alignment effects from low to high shear rates.

**Hydrodynamic Interactions**

Hydrodynamic interactions which manifest themselves as an increase in viscosity are influenced by the fluid velocity distribution around the particles causing an increase in energy dissipation. The key variables include solids volume fraction, particle shape and particle size distribution. The strength of interaction and thus viscosity increases with solids volume fraction and particle asymmetry, but increased polydispersity allows small particles to be interposed between larger ones to give a lower viscosity at the same solids volume fraction.

### 3.2.3 SOFT SPHERE DISPERSIONS

In flow processes, at low solids concentrations, particulate suspension material properties are largely determined by the viscosity of the liquor. However, at higher solids concentrations, the
interaction forces between particles are the key determinants of particulate suspension material properties. These forces are strongly influenced by the chemical environment on the surface of the particles and in the liquor. When repulsive colloidal forces are present, dispersions are considered soft sphere dispersions. The repulsive force can be due to either polymer adsorbed or electrical charges on the particle surfaces.

**Attractive Colloidal Interactions**

Attractive forces between particles are generally dominated by van der Waals forces, which are always present. These forces are due to electrical dipole orientation, induction and dispersion. In dipole orientation, two molecules with permanent dipoles orient themselves to give a net attraction. Induction involves dipolar molecules inducing dipoles on other molecules such that a net attraction results. London dispersion forces, which act between non-polar molecules and account for most of the attraction between particles, are the result of localised molecular charge fluctuations associated with the motion of electrons causing polarisation in another molecule and vice versa. London forces are inversely related to a power of the inter-particle distances.

**Repulsive Colloidal Interactions**

Stabilising repulsive colloidal interactions include both electrostatic and steric repulsion. Electrostatic repulsion between particles results from their like charge. Metal oxide particles have an overall surface charge that can be net positive or negative depending on pH and the presence of various ions that chemically absorb. The magnitude of the potential depends on the pH, a measure of the concentration of hydronium and hydroxyl ions in the suspending fluid. The repulsive forces cannot be calculated from Coulomb's law because charged particles are surrounded by a diffuse double layer of counter-ions resulting in overall neutrality. In the double layer, counter-ions are attracted to the particle surface while ions of like charge are repelled. Some of the counter-ions occur in an immobilised tightly bound layer around the particle, known as the Stern layer, while the rest move freely in the fluid by Brownian motion. The outer edge of this bound Stern layer is known as the Stern plane. The shear plane, the surface at which shear occurs when the particles are subject to flow processes, is slightly further out than the Stern plane due to the strength of molecular interactions. The repulsive surface potential decays linearly within the Stern layer and then decays exponentially at larger separations. The surface potential at the shear plane is termed the zeta potential, $\zeta$. The pH at which the zeta potential, $\zeta$, is zero is called the isoelectric point (IEP). The debye length, $\kappa^{-1}$, interpreted as the thickness of the electrical double layer, represents the particle separation over which the surface potential decreases by one exponential factor. The double layer thickness is inversely related to the ionic strength of the suspending fluid medium. This implies that the double layer is compressed as the ionic strength increases.
Total Colloidal Interactions

Deryaguin and Landau, and Verwey and Overbeek (Verway and Overbeek 1948) independently developed a quantitative theory of inter-particle forces that enable understanding of coagulation and flocculation phenomena. The theory assumes that the total interaction potential between two particles is the sum of the attractive van der Waals potential and the repulsive electrostatic potential. The shape of the curve representing the total interaction potential between two particles as a function of distance depends on particle size, surface potential, ionic strength and other factors.

For the case of low surface potential and/or high ionic strength, the repulsive component is not significant and the total potential follows the van der Waals attractive potential. The insufficient shielding inevitably leads to particle aggregation at close separation in the primary minimum, that is very stable and difficult to reverse.

For significant repulsive potential, as a result of high surface potential and/or low ionic strength, the total potential exhibits an energy barrier. If the energy barrier is large compared to the thermal energy of the particles, the system is stable, otherwise aggregation into the primary minimum will result.

Intermediate between the two extremes already mentioned is the case of a secondary minimum where particles are weakly stable at intermediate separations allowing flocs to form. The origin of the secondary minimum relates to the rate of decay of the attractive potential being slower than that for the repulsive potential at intermediate separations. These flocs can be broken down under shear leading to shear thinning and sometimes thixotropy.

Polymers in Stabilisation and Flocculation

Only a brief overview of the processes of polymeric stabilisation and flocculation is given here, but detailed discussions are provided by Vincent and Dobias (Vincent 1974; Dobias 1993).

The repulsive forces already mentioned are due to electrostatic repulsion, but can also be due to the presence of a polymer layer at the surface of the particles. This repulsion is due to an osmotic effect caused by the high concentration of overlapped polymer elements between the particles and a volume restriction effect on polymer conformations. This steric stabilisation results in the small separations where attractive van der Waals forces would dominate being unattainable.

Polymers can enable flocculation through a depletion mechanism of destabilising colloidal suspensions. The method exploits the osmotic pressure gradient between the polymer depleted
inter-particle gaps and the surrounding solution to create an effective attractive force between the particles. In this way, the polymer concentration controls the interaction strength and thus, the extent of flocculation.

Polymer bridging is an important method of flocculation where flocs are held together by bridges of individual polymer from the surface of one particle to another. These polymer flocculants have high molecular weight chains with functional groups designed to attach to the particle surfaces through electrostatic or other bonding methods. The mechanism of flocculation requires particle collision for the bridges to be formed to create relatively stable flocs. The strength of these flocs, the reversibility of the floc formation and the effect on rheological and dewatering properties can vary greatly from system to system.

### 3.3 SHEAR RHEOLOGY

#### 3.3.1 INTRODUCTION & PREVIOUS WORK

The simplest measure of a liquid flow property is the concept of a viscosity developed by Newton. Fluid viscosity, \( \eta \), is defined as the shear stress, \( \tau \), divided by the shear rate, \( \gamma \), where the shear rate is the velocity gradient. The simplest type of rheological behaviour, called Newtonian, occurs when the viscosity is constant over a wide range of shear rates. Non-Newtonian fluids have viscosities that can vary with shear rate, but can also vary with time and the sample’s shear and thermal history, and can also exhibit a shear yield stress at zero shear rate. Since Newton, many shear rheology theories have been developed to enable description of fluid flow behaviour for the wide range of complex fluids found in nature. In the middle of the 20th century, extensive research was conducted into non-Newtonian polymer rheology and these theories have been applied to particulate suspensions since they are just as applicable (Nguyen and Boger 1992). The main material properties of relevance in particulate suspension fluid flow are the shear stress versus shear rate behaviour. This behaviour has been shown to vary as a function of solids concentration, chemical environment, temperature and sometimes shear history. For particulate suspensions at high enough solids concentrations, the flow behaviour is intermediate between that of a solid and a liquid (Nguyen and Boger 1992). The behaviour is such that when the applied shear stress is less than a critical stress known as the shear yield stress, the suspension does not flow, but deforms plastically like a solid (Nguyen and Boger 1992). Also, when the shear yield stress is exceeded, the material flows like a viscous fluid (Nguyen and Boger 1992), with a viscosity that varies with shear rate. Throughout the 20th century, numerous researchers have proposed that the shear yield stress is not a truly real phenomenon (Barnes and Walters 1985), suggesting that the shear yield stress disappears at
very low shear rates. There is still contention in the literature (Pashias 1997) and some researchers still thrive off the debate. Regardless of the results of this debate, from a practical perspective over the time scales of industrial interest, the shear yield stress is real. The rheological behaviour of time independent and time dependent yield stress fluids are overviewed in the sections that follow. For a more detailed review of particulate suspension rheology, beyond the scope of this thesis, the reader is directed to the work of Pashias (Pashias 1997) and the numerous texts and reviews recommended by him (Metzner 1956; Wilkinson 1960; Schowalter 1978; Cheremisinoff 1986; Barnes et al. 1989; Boger and Yeow 1990; Macosko 1994).

3.3.2 TIME INDEPENDENT BEHAVIOUR

3.3.2.1 Newtonian, Pseudoplastic and Dilatant Behaviour

The rheological behaviour of particulate suspensions is considered time independent when rheological properties are a reproducible function of shear rate and do not depend on the material’s shear history (Pashias 1997). The rheological behaviour of time independent particulate suspensions can be categorised according to the characteristics of its shear stress versus shear rate curve. When there is no shear yield stress, the behaviour is Newtonian for constant viscosity, pseudoplastic (shear thinning) where the viscosity decreases with increasing shear rate and dilatant (shear thickening) where the viscosity increases with increasing shear rate. These three types of behaviour are shown in Figure 3.1.
3.3.2.2 Bingham Plastic, Yield-Pseudoplastic and Yield-Dilatant Behaviour

When the solids concentration is high enough, particulate suspensions exhibit a shear yield stress, which is the minimum shear stress required for a material to flow. The magnitude of the shear yield stress is related to the strength of interparticle interactions and the suspension microstructure (Green 1997). When a suspension exhibits a shear yield stress, the behaviour can again be categorised according to the shear stress versus shear rate behaviour. The suspension is termed a Bingham plastic when the shear stress versus shear rate gradient is constant, yield-pseudoplastic when the shear stress versus shear rate gradient decreases with increasing shear rate and yield-dilatant when the shear stress versus shear rate gradient increases with increasing shear rate. These three types of behaviour are shown in Figure 3.2.

Figure 3.1: Depiction of pseudoplastic, Newtonian and dilatant shear stress versus shear rate behaviour.
Figure 3.2: Depiction of yield-pseudoplastic, Bingham plastic and yield-dilatant shear stress versus shear rate behaviour.

3.3.2.3 Overview of Flow Models

There are two commonly used empirical rheological models that generically describe shear stress versus shear rate behaviour of yield stress materials. The first is the Hershel-Bulkley model and the second is the Casson model.

Hershel-Bulkley Model

The Hershel-Bulkley model equation, given below in Equation 3.1, describes the shear stress, $\tau$, as a function of shear rate, $\dot{\gamma}$, where $\tau_y$ is the shear yield stress with $k$ and $m$ being empirical parameters.

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

(3.1)

For rheological behaviour with no yield stress ($\tau_y = 0$), the value of $m$ determines whether the suspension rheology is Newtonian. The rheology of such suspensions is Newtonian if $m = 1$, pseudoplastic if $m < 1$, and dilatant if $m > 1$. For rheological behaviour with the presence of a shear yield stress ($\tau_y > 0$), the suspension is a Bingham plastic if $m = 1$, yield-pseudoplastic if $m < 1$, and yield-dilatant if $m > 1$. This model has applicability to a wide range of shear rheology behaviour.


Casson Model

In some situations, shear yield stress behaviour is not well described by the Hershel-Bulkley equation and a more complicated alternative is required. A commonly adopted alternative is the Casson equation shown below in Equation 3.2, which describes the shear stress, \( \tau \), as a function of shear rate, \( \dot{\gamma} \), where \( \tau_y \) is the shear yield stress and \( \eta_\infty \) is an empirical parameter related to the viscosity.

\[
\tau^{1/2} = \tau_y^{1/2} + (\eta_\infty \dot{\gamma})^{1/2}
\]

(3.2)

3.3.3 TIME DEPENDENT BEHAVIOUR

Time dependent flow behaviour is usually associated with structural changes that occur during and after cessation of shearing processes (Pashias 1997). This can involve changes in observed viscosity or shear yield stress as a function of time and shear history. The two extremes of time dependent behaviour are thixotropy and rheopexy.

3.3.3.1 Thixotropic Suspensions

Thixotropy is a continuously decreasing resistance to flow with time of shear, until some structural equilibrium is reached (Nguyen 1983a). This shear rate dependent equilibrium limit is explained as the rate of structural breakdown equalling the rate of structural recovery (Nguyen 1983a). On cessation of shear, Brownian motion enables a gradual recovery of structure (Nguyen 1983a). Thixotropic behaviour is associated with concentrated particulate suspensions, but is not common (Pashias 1997). The origins of thixotropy lie in the nature of particle interactions, described in section 3.2, and the influence of shear processes on these interactions and is thus complex. The primary difference between thixotropy and pseudoplasticity is the relative rate of structural change, which is finite and measurable for thixotropic suspensions, but undetectably rapid for pseudoplastic suspensions (Nguyen 1983a; Pashias 1997). The rate of structural regrowth is often slower than the breakdown because structural regrowth is particle diffusion limited and rough or non-spherical particles have reduced mobility (Nguyen 1983a; Nguyen and Boger 1985b; Pashias 1997).

3.3.3.2 Rheopectic Suspensions

Rheopectic suspensions exhibit anti-thixotropic behaviour in that an increasing resistance to flow is observed with time of shear, with this increased structure sometimes dissipating with time after cessation of shear. The phenomenon is proposed to be due to collision induced structural build up (Cheng 1973), analagous to dilatancy, which decays slowly due to Brownian
motion after cessation of shear, providing that interacting particles are not in a primary minimum.

### 3.3.4 SHEAR RHEOLOGY CHARACTERISATION TECHNIQUES

(OVERVIEW)

#### 3.3.4.1 Steady Shear Stress Measurement

There is a wide range of techniques for the measurement of shear stress versus shear rate behaviour. These techniques include the standard range of capillary and rotational rheometers. Capillary rheometers range from lab scale Cannon-Fenske viscometers for the measurement of Newtonian liquid viscosities to large scale tubing for characterising the shear rheology of particulate suspensions. Rotational rheometers include geometries such as “cone and plate”, “parallel plate” and “concentric cylinders”. These methods are overviewed below.

**Capillary Rheometry**

Capillary rheometry is generally considered a reliable and accurate method of characterising shear stress versus shear rate behaviour for particulate suspensions. However, the technique, described by Nguyen and Boger (Nguyen and Boger 1992), uses relatively large scale, expensive and non-portable equipment. A capillary rheometer basically consists of a straight tube of known length and internal diameter through which a fluid flows at a measured rate under a known pressure drop (Nguyen and Boger 1992). Major limitations of this method for particulate suspensions are the labour intensive nature of the measurements and the occurrence of wall slip (Nguyen and Boger 1992). The method can use many litres of suspension, and thus can only be applied when there is an adequate quantity of material available. In addition, it is generally only dedicated rheological research laboratories that possess this equipment. For materials with time dependant properties, the implication is that this method will not produce representative results after the extended time required to transport the sample to the dedicated laboratory.

**Cone and Plate Rheometry**

In situations where only small quantities of sample are available, cone and plate rheometry is preferred to capillary rheometry. The technique, described by Nguyen and Boger (Nguyen and Boger 1992), involves sandwiching the fluid between a flat plate and a cone. Either the cone or the plate is rotated with the other stationary and the torque measured at a number of rotational rates. Particulate suspensions require a truncated cone to prevent grinding, where the minimum spacing exceeds the largest particle size by a factor of at least ten (Nguyen and Boger 1992).
Such a large gap leads to non-uniform shear rate distributions and thus calculation errors (Nguyen and Boger 1992). Experimentally, it can be difficult to ensure that suspension particles do not settle over the timescale of the experiment and slip is difficult to eliminate, leading to more errors. As for capillary rheometry, this method requires expensive, sophisticated and non-portable equipment that is generally not available on site at industrial laboratories, and thus the sample must be transported to a laboratory dedicated to rheological characterisations.

**Concentric Cylinder Rheometry**

Concentric cylinder rheometry, sometimes referred to as cup and bob rheometry, is a relatively simple and low cost method of characterising shear rheology. The technique, described by Nguyen and Boger (Nguyen and Boger 1992), involves rotation of a cylinder in a cup of fluid, with the torque measured at a number of rotational speeds. Traditionally, the gap between the cup and the bob is very small and laminar, non-slip flow regimes are assumed. When measuring the shear rheology of particulate suspensions, a small gap is impractical due to coarse sand like particles that get stuck in the gap and compromise the test. Therefore, a relatively wide gap is recommended for suspensions with coarse particles. For a suspension that exhibits a shear yield stress, one method that eliminates the gap problem altogether is to use a very large “infinite” gap. This large gap method was found to be useful in on site testing and is detailed in Chapter 5.

### 3.3.4.2 Shear Yield Stress Measurement

Measurement of the shear yield stress provides valuable information regarding the handleability of a thickener output as a function of the solids concentration. This information, when combined with modelling data can be very useful in isolating an operating window of acceptable and achievable solids concentration output, with the shear rheology indicating a practical upper limit to the solids concentration that can be handled. There are many commonly used techniques for the determination of the shear yield stress including extrapolation of shear stress versus shear rate data, rotational rheometer methods, the vane technique, the cone penetration method and the slump test. The advantages and disadvantages of each method is discussed below.

**Extrapolation of Shear Stress versus Shear Rate Data**

The traditional method of determining the shear yield stress of a particulate suspension is through the extrapolation of shear stress versus shear rate data, to zero shear rate. The method can involve fitting data with a straight line or fitting to a rheological model such as the Hershel-
Bulkley or Casson model. The value of the shear yield stress determined often depends on the accuracy of low shear rate data and the curve fit method used. In addition, as has already been noted, it is very difficult to eliminate slip at low shear rates in steady shear rheological testing (Nguyen and Boger 1992) and thus there is always the potential for significant error in the data even before yield stress extrapolation.

**Rotational Rheometer Methods**

Rotational rheometer methods of determining the shear yield stress such as stress relaxation, creep recovery and stress growth tests are described by Nguyen and Boger (Nguyen and Boger 1992). Each of these techniques can be applied to the conceptually simple concentric cylinder rheometry apparatus, but each requires a complicated experimental method and is also subject to significant slip.

**The Vane Technique**

The vane technique, developed by Nguyen (Nguyen 1983a; Nguyen and Boger 1985a) overcomes the slip problem. The technique uses a vane consisting of four thin blades arranged at equal angles around a small cylindrical shaft. The method involves rotating the vane immersed in the suspension at a low rate and measuring the maximum torque. It is such a simple and reliable technique that it has found widespread application and become a standard characterisation tool for particulate suspensions. Indeed, this technique has been useful in on site testing and is detailed in Chapter 5.

**The Cone Penetration Method**

The cone penetration method, described by Nguyen and Boger (Nguyen and Boger 1992), is a standard method for soil and foundation testing (ASTM D217-82; AS 1289.6.5.1-1999) and has been applied in the dairy industry (Dixon 1981). The method involves calculation of the shear yield stress from the gravitational penetration of a cone of known weight into a yield stress material. The time required for equilibrium penetration to be achieved can depend heavily on the strength of the suspension and it is often not clear whether equilibrium has been achieved. This method works reasonably well for paste like suspensions, but sedimentation causes significant error for low yield stress materials.

**The Slump Test**

The slump test, adapted from soil consistency tests by Pashias (Pashias et al. 1996), is an extremely simple test that can be applied on site without any electronic equipment. It requires only an open-ended PVC cylinder, a ruler and a flat surface. The method involves filling the
cylinder with suspension, lifting the cylinder and measuring the final slump height. This simple test has been useful for spot characterisations and is also detailed in Chapter 5.

3.3.4.3 Thixotropic Determination

The use of the shear yield stress as a method for the characterisation of thixotropy has many advantages over conventional rheological techniques. Most thixotropic suspensions are extremely shear sensitive and the time scale of recovery is such that conventional shear measurements are not appropriate since the act of placing a sample in the rheometer causes structural breakdown. It is easier on this basis to monitor the recovery process after shearing the sample to an equilibrium state, as has been proposed by numerous workers (Nguyen and Boger 1985b; Pashias 1997). The vane yield stress measurement technique is ideal in this context since a shear yield stress can be monitored in a series of samples as a function of recovery time for a number of days.

3.4 DEWATERING

3.4.1 INTRODUCTION

The mineral, pigment, water and waste water industries produce millions of tonnes of wet particulate suspensions as process intermediates, wastes and final products. It is usual to increase the solids concentration of these suspensions at some point in the process through methods including gravity settling, vacuum filtration, centrifugation and pressure filtration. Flocculants may be added to encourage floc formation and thus enhance settling and compression rates. Flocculant selection and dose is often based on measurement of settling rates, while neglecting to characterise the dewaterability of the sediment.

A mathematical theory of the dewatering of flocculated suspensions has been developed by Buscall & White (Buscall and White 1987) using the concept of a flocculated particle network structure. They established the gel point, compressibility and permeability as the fundamental physical properties that determine the dewaterability of flocculated suspensions. Compressibility determines the maximum possible extent of dewatering for a given applied compressive stress while permeability determines the rate at which this extent of dewatering can be achieved.

The gel point, $\phi_g$, is the critical solids concentration at which discrete flocs or particles come into contact to form a continuous network structure. Thus, the gel point is the maximum solids concentration at which the suspensions are dewaterable.
concentration that a suspension can attain through free settling alone with no compression or mechanical enhancement (eg. rakes).

In a flocculated suspension, at a solids concentration above the gel point \( \phi > \phi_g \), the interconnected particle network has a physically measurable strength or compressibility, which depends on the local solids volume fraction, \( \phi \). The magnitude of the network strength is quantified in terms of the compressive yield stress, \( P_y(\phi) \), which is the minimum compressive stress required for the network structure to yield and compress irreversibly at a given solids volume fraction, \( \phi \). For all solids concentrations below the gel point \( \phi < \phi_g \), the particle concentration is too small to allow a particle network to develop and thus the compressive yield stress is zero. For all solids concentrations above the gel point, the network will remain in its original form until an applied stress, \( \Delta P \), on the network exceeds the compressive yield stress. At this stage, the structure of the network collapses, irreversible particle consolidation occurs and the local volume fraction increases. Clearly, \( P_y(\phi) \) increases with \( \phi \), as the number of inter-particle linkages increases.

In suspension dewatering processes, the fluid flows past and interacts with the solid particles, which exert a drag force and cause a hydrodynamic resistance to flow through the suspension. This resistance to flow is quantified in terms of a property called the hindered settling function, \( R(\phi) = \left( \lambda / \langle V_p \rangle \right) r(\phi) \), with units Pa s m\(^{-2}\), which is inversely related to the permeability and free settling velocity of the suspension. \( \lambda / \langle V_p \rangle \) represents the Stokes drag coefficient, with units Pa s m, divided by the average particle or floc volume. The hindered settling factor, \( r(\phi) \), is a dimensionless quantity which accounts for the influence of hydrodynamic interactions between particles on the rate of dewatering relative to the Stokes settling velocity for a single settling particle or floc. As the solids concentration increases, the interactions between particles increase and \( R(\phi) \) increases in a non-linear fashion.

### 3.4.2 DEWATERING MODEL FORCE BALANCE

The particulate suspension force balance proposed by Buscall and White (Buscall and White 1987) has been modified by a factor of \((1 - \phi)\) to maintain consistency with current practices (Green 1997). Therefore, the force balance over a volume element of particulate suspension includes the sum of a hydrodynamic drag force, a network pressure gradient and the effective gravitational force exerted upon the particles, as shown below,

\[
\frac{\Delta \rho \phi}{u_{se}} \left( \frac{\phi}{1 - \phi} \right) \left( u - v \right) + \frac{\partial P}{\partial z} + \Delta \rho g \phi = 0
\]  

(3.3)
where $\Delta \rho$ is the solid-liquid density difference, $g$ represents the magnitude of the gravitational acceleration, $u_{St}$ is the magnitude of the Stokes settling rate, $r(\phi)$ is the hindered settling factor, $u$ is the downward solids settling rate, $v$ is the upward liquor velocity, $p$ is the solids network pressure and $z$ is vertical displacement with up signified as positive.

The magnitude of the Stokes settling velocity, $u_{St}$, is given by Stokes’ Law which predicts the terminal settling velocity for an isolated spherical particle in creeping flow according to the following relation,

$$u_{St} = \frac{\Delta \rho g d_p^2}{18\eta} \quad (3.4)$$

where $d_p$ is the particle diameter and $\eta$ represents the fluid viscosity. The Stokes settling rate, $u_{St}$, can also be related to the particle volume, $V_p$, and the Stokes drag coefficient, $\lambda$, according to the following relation,

$$u_{St} = \Delta \rho g \frac{V_p}{\lambda}. \quad (3.5)$$

The hindered settling factor, $r(\phi)$, is defined as the ratio of the resistance to flow through the suspension network structure at solids volume fraction $\phi$ relative to that of an isolated particle. However, since knowledge of the floc density and size is very difficult to obtain experimentally and has little physical meaning at solids concentrations above the gel point, the physical properties mentioned are grouped with the hindered settling factor to give the hindered settling function, $R(\phi)$ which is a readily determined material property.

$$R(\phi) = \frac{\lambda}{V_p} r(\phi) = \frac{\Delta \rho gr(\phi)}{u_{St}} \quad (3.6)$$

Substitution of the hindered settling function, $R(\phi)$, into the force balance of Equation 3.3 gives,

$$R(\phi) \frac{\phi}{1-\phi} (u - v) + \frac{\partial p}{\partial z} + \Delta \rho g \phi = 0. \quad (3.7)$$

This equation forms the basis for much of the dewaterability characterisation and modelling tools that are presented in this thesis.

**Batch Settling**

In a batch settling situation, a material balance through any suspension plane requires that the solids flux and liquor flux sum to zero to maintain conservation of mass, as shown below,
\[ \phi u + (1 - \phi) v = 0. \]  

(3.8)

Transforming this equation to give the liquor velocity in terms of the solids velocity and substituting for the liquor velocity in the force balance gives,

\[ R(\phi) \frac{\phi}{(1 - \phi)^2} u + \frac{\partial p}{\partial z} + \Delta \rho g \phi = 0, \]

(3.9)

where transformation of the force balance in Equation 3.9, gives the linear solids velocity,

\[ u = -\frac{\Delta \rho g (1 - \phi)^2}{R(\phi)} \left( 1 + \frac{\partial p}{\partial z} \frac{1}{\Delta \rho g \phi} \right). \]

(3.10)

For free settling particles at a concentration below the gel point, there is no network pressure gradient, and thus the downward free settling rate reduces to the following;

\[ u_f(\phi) = \frac{\Delta \rho g (1 - \phi)^2}{R(\phi)}. \]

(3.11)

**Continuous Thickening**

In a continuous thickening situation, a material balance through any suspension plane requires that the solids flux and liquor flux sum to the operating flux to maintain conservation of mass, as shown below,

\[ \phi u + (1 - \phi) v = \frac{q_u}{\phi_u}, \]

(3.12)

where \( q_u \) is the underflow solids flux and \( \phi_u \) is the underflow solids volume fraction, with volumetric solids flux, \( q \), given by,

\[ q = -u \phi. \]

(3.13)

Equations 3.12 and 3.13 above can be combined to give the relative fluid solids settling velocity, \( u - v \), as

\[ (u - v) = -\frac{1}{(1 - \phi)} \left( q - \frac{q_u}{\phi_u} \right), \]

(3.14)

where substitution into Equation 3.7 gives
\[ \frac{\partial P_s(\phi)}{\partial z} = \frac{R(\phi)}{(1-\phi)^2} \left( q - q_u \frac{\phi}{\phi_u} \right) - \Delta P \cdot g \cdot \phi . \] (3.15)

Equation 3.15 above can be transformed to the differential equation,

\[ \frac{\partial \phi}{\partial z} = \frac{\frac{R(\phi)}{(1-\phi)^2} \left( q - q_u \frac{\phi}{\phi_u} \right) - \Delta P \cdot g \cdot \phi}{\frac{dP_s(\phi)}{d\phi}} , \] (3.16)

which when combined with the solids continuity equation,

\[ \frac{\partial q}{\partial z} = \frac{\partial \phi}{\partial t} , \] (3.17)

forms a set of coupled partial differential equations defining the consolidation dynamics of a suspension bed in a continuous thickening operation.

At steady state, the solids flux is equal to the underflow solids flux, \( q = q_u \), which allows the solids continuity equation to be ignored and Equation 3.16 to be simplified to

\[ \frac{\partial \phi}{\partial z} = \frac{\frac{R(\phi)}{(1-\phi)^2} q_u \left( 1 - \frac{\phi}{\phi_u} \right) - \Delta P \cdot g \cdot \phi}{\frac{dP_s(\phi)}{d\phi}} . \] (3.18)

Alternatively, when the underflow solids flux equals zero, \( q_u = 0 \), such as in a batch settling situation, Equation 3.16 simplifies to become,

\[ \frac{\partial \phi}{\partial z} = \frac{\frac{R(\phi)}{(1-\phi)^2} q - \Delta P \cdot g \cdot \phi}{\frac{dP_s(\phi)}{d\phi}} . \] (3.19)

### 3.4.3 COMRESSIBILITY

Compressibility determines the solids concentration, above the gel point, \( \phi_g \), that a suspension can compress to for a given applied solids network pressure, and is quantified by the compressive yield stress, \( P_s(\phi) \).
3.4.3.1 Gel Point

The gel point, $\phi_g$, of a suspension is a useful parameter for gaining information regarding the floc structure in particulate suspensions. The gel point is defined as the lowest solids volume fraction at which particles or flocs form a self-supporting network. Within a suspension network structure, the weight of each individual floc or particle is transmitted down through the network and, as a result, particles or flocs within a suspension are subject to compressive forces due to the self-weight of the overlying material. Thus material at the top of a bed will experience no compressive forces and moving down through the bed, as the distance from the top of the bed is increased, the compressive force correspondingly increases. At solids concentrations above the gel point the network has an integral strength, which can resist an applied load, i.e. a compressive yield stress. Thus the gel point may alternatively be defined as the solids concentration at which the compressive yield stress becomes zero.

3.4.3.2 Compressive Yield Stress $P_y(\phi)$

The compressive yield stress is the applied compressive pressure that must be exceeded to make a networked suspension, at a given solids volume fraction, $\phi$, yield and concentrate to a higher solids concentration. Conversely, $\phi$ may be viewed as the solids concentration to which a material will concentrate at equilibrium when subject to an applied compressive pressure. As shown in Figure 3.3, the compressive yield stress increases from 0 at the gel point and exhibits exponential behaviour at higher solids concentrations. As the solids volume fraction rises even higher than 0.4, towards a close packing fraction, the compressive yield stress would be expected to asymptote towards infinity.

![Figure 3.3](image-url)

**Figure 3.3**: Typical compressive yield stress, $P_y(\phi)$, behaviour as a function of solids volume fraction, $\phi$. (A) In linear coordinates and (B) in semi-logarithmic coordinates.
3.4.4 PERMEABILITY

There are numerous methods of defining suspension permeability and resistance to flow though suspensions as a physical quantity. This list of quantities includes, but is not limited to, the hindered settling function, $R(\phi)$, a free settling velocity, $u_{fs}(\phi)$, a traditional Darcian permeability, $k(\phi)$, and a specific cake resistance. The concept of permeability is generally well understood for suspension filter cakes. However we do, at times, refer to “permeability at low solids concentrations” when quantifying settling rates. What is meant here is that high settling rates imply high permeabilities and vice versa. The important message is that all of these measures of permeability are continuous functions of solids concentrations from dilute suspensions all the way up to pastes and filter cakes. Each of the quantities mentioned is physically related and is introduced below.

3.4.4.1 Hindered Settling Function, $R(\phi)$

The hindered settling function, $R(\phi)$, is defined as the hydrodynamic resistance to flow of liquor through a particulate suspension as a function of solids volume fraction, $\phi$. This flow can be interpreted as the movement of either a particle through a liquid or liquid past a collection of particles and is thus applicable to the full range of solids concentrations from very dilute to very concentrated suspensions. As a result, the hindered settling function is a valid quantity for all solids volume fractions from the case where a single particle is moving through liquid to liquid moving through a concentrated bed of many particles. As the solids volume fraction increases, the hindered settling function also increases. This factor quantifies the rate at which a suspension can dewater as a function of solids concentration. For flocculated suspensions, typical variation of the hindered settling function, $R(\phi)$, as a function of solids volume fraction, $\phi$, is shown in Figure 3.4. Theory suggests that the behaviour would produce relatively straight lines on semi-logarithmic coordinates, but the typical behaviour shown in Figure 3.4 (B), indicates more complex behaviour for flocculated suspensions.

3.4.4.2 Free Settling Rate, $u_{fs}(\phi)$

The free settling rate of a particulate suspension is directly related to the hindered settling function according to the following relation given by Equation 3.11;

$$u_{fs}(\phi) = \frac{\Delta \rho g (1 - \phi)^2}{R(\phi)}.$$

(3.11)

Free settling rate is defined as the settling rate at which a suspension settles when there is no compressibility influence to retard gravitational settling. This is always the case at solids
concentrations less than the gel point, but at solids concentrations greater than the gel point an asymptotic limit, equal to the free settling rate, is approached as suspension height increases. A typical example of free settling rate behaviour as a function of solids concentration, \( u_f(\phi) \), is given in Figure 3.4 (C), where the permeability decreases with increasing solids concentration.

### 3.4.4.3 Traditional Darcian Permeability, \( k(\phi) \)

According to Green (Green 1997), many workers have determined permeability, \( k(\phi) \), by constant pressure filtration through a packed bed of known height and concentration. According to Darcy’s law, for a filtration system with a pressure drop \( \Delta P \) across a packed bed of height, \( H \), solids volume fraction, \( \phi \) and liquor viscosity, \( \eta \), the volumetric rate of filtration per filter cross sectional area, \( dV/dt \) is shown below with the addition of a membrane resistance \( \alpha \).

\[
\frac{dV}{dt} = \frac{\Delta P}{\eta \left( \frac{H}{k(\phi)} + \alpha \right)}
\]

The traditional Darcian permeability, \( k(\phi) \), with units m\(^2\), is related to the hindered settling function, \( R(\phi) \), according to the following equation (Green 1997).

\[
k(\phi) = \frac{\eta}{R(\phi)} \frac{1-\phi}{\phi}
\]

A typical example of Darcian permeability, \( k(\phi) \), is given in Figure 3.4 (D), where the permeability decreases with increasing solids concentration.

### 3.4.4.4 Specific Cake Resistance

Darcy’s Law can also be expressed in term of a specific volume cake resistance, \( \alpha_v \), with units m\(^{-2}\) according to the equation

\[
\frac{dV}{dt} = \frac{\Delta P}{\eta \left( \frac{H}{k(\phi)} + \alpha_v \right)}
\]

where, \( \alpha_v \) is related to \( R(\phi) \) by

\[
\alpha_v = \frac{R(\phi)}{\eta (1-\phi)}
\]
for an incompressible suspension. The relationship is more complex for compressible suspensions, since \( \alpha_r \) depends on resistance over the range of solids concentrations in the filter cake, and is thus only a filtration property, rather than strictly being a material property.

**Figure 3.4:** Typical permeability behaviour as a function of solids volume fraction, \( \phi \). (A) Hindered settling function, \( R(\phi) \), in linear coordinates, (B) hindered settling function, \( R(\phi) \), in semi-logarithmic coordinates, (C) free settling rate, \( u_{fs}(\phi) \), in semi-logarithmic coordinates and (D) traditional Darcian permeability, \( k(\phi) \) in semi-logarithmic coordinates.

### 3.4.5 SOLIDS DIFFUSIVITY, \( D(\phi) \)

Solids diffusivity, \( D(\phi) \), combines the competing effects of compressibility and permeability into a single term. This term can be used to fully describe a filtration process when gravitational forces are insignificant, given only the feed solids concentration and the final solids concentration for the applied pressure. \( D(\phi) \) is directly related to the compressive yield stress, \( P_y(\phi) \), and the hindered settling function, \( R(\phi) \), according to the following relation,
The solids diffusivity is often considered a measure of the overall dewaterability of a suspension. Because $D(\phi)$ is inversely related to $R(\phi)$, a suspension with a higher $D(\phi)$ is often considered more dewaterable, however this is not necessarily the case because of the $P_y(\phi)$ dependence. Therefore, extreme caution should be used when comparing suspension dewaterability on the basis of $D(\phi)$. It is suggested that comparisons of $D(\phi)$ can be given improved physical meaning when solids diffusivity is plotted against pressure rather than solids concentration. A typical example of $D(\phi)$, behaviour for a flocculated suspension is given in Figure 3.5, showing $D(\phi)$ to be zero at solids concentrations below the gel point, to have a discontinuity at the gel point and to increase at an exponential rate at solids concentrations above the gel point. Of course, if the solids concentrations becomes high enough, $D(\phi)$ is predicted to reach a maximum and then decrease, but the solids concentration at which this would occur is material specific.

![Figure 3.5](image)  
*Figure 3.5*: Typical solids diffusivity, $D(\phi)$, behaviour as a function of solids volume fraction, $\phi$. (A) In linear coordinates and (B) in semi-logarithmic coordinates.

### 3.4.6 OVERVIEW OF DEWATERABILITY CHARACTERISATION TECHNIQUES AND DEWATERING PREDICTION TOOLS

The compressibility, permeability and solids diffusivity behaviour demonstrated in Figures 3.3, 3.4 and 3.5 are typical examples for a flocculated suspension. The dewaterability curves of different suspensions can vary dramatically in shape and by numerous orders of magnitude. Thus, there is no universal correlation that can describe all dewatering behaviour. This necessitates sound reproducible techniques for the characterisation of suspension dewatering material properties. The application and limitations of traditional relatively empirical
dewaterability characterisation methods and dewatering prediction tools are overviewed below, followed by a brief overview of fundamentally based characterisation methods and prediction tools.

3.4.6.1 Traditional Characterisation

Filtration Design

Filtration design is often achieved using empirical knowledge of process operation combined with permeabilities measured for a given applied pressure in filtration tests that assume an incompressible suspension (Green 1997). This kind of semi-empirical methodology can enable optimisation of processes within a narrow range of operation parameters, but lacks the potential to confidently predict the effect of operating outside these parameters.

Thickener Design

In thickener design, equipment sizing uses empirical settling rate methods (Coe and Clevenger 1916; Kynch 1952; Talmage and Fitch 1955; Fitch 1966) that don’t properly account for suspension compressibility. Batch settling tests giving an initial settling rate and compacted solids concentrations are the norm for thickener sizing and flocculant dosing in industry. The simplest method involves flocculation with a plunger in a one litre measuring cylinder, measuring the initial settling rate and then waiting a preset period of time (1-24 hours) before determining the compacted solids concentration. The initial settling rate is often quoted as a measure of the effectiveness of flocculation for a range of flocculant chemical types and doses. This method has limitations in predicting the most effective conditions for dewatering, especially since this settling rate is only at one solids concentration. In addition, the qualitative compacted solids trends can vary with the amount of time over which the suspension is allowed to consolidate.

Tailings Dam Consolidation

Consolidation in tailings dams is a very complex field of study that has been approached diligently by a small number of civil and geotechnical engineers, such as Vick who published a book on tailings dams (Vick 1983). Vick (Vick 1983) introduced some rules of thumb and industrial observations for consolidation rates and final solids concentrations, but failed to introduce a predictive consolidation model. Some consulting engineers utilise concepts analogous to compressibility and permeability determined by application of a modified Darcy’s law to filtration behaviour in a number of filtration tests. The results are then applied to computer models to predict consolidation and drainage behaviour. These methods are expected
to be applicable for relatively incompressible suspensions, but not for flocculated suspensions with colloidal sized particles.

**Standardisation of Material Properties**

A large degree of empiricism exists in the design and optimisation methods for pressure filter, thickener and residue consolidation process operations. The methods are based on measured quantities that often have a loose relationship with physical material properties. The methods are limited by the lack of standardisation in the nomenclature of the properties that are measured to enable performance prediction. With the development of the dewatering theory of Buscall and White (Buscall and White 1987), all of these processes can be predicted from the compressibility and permeability. If these fundamental material properties can be determined from standard sets of experimental tests, then a wide range of dewatering phenomenon can be predicted under the banner of a single theory.

**3.4.6.2 Fundamental Material Property Characterisation**

The compressive yield stress, \( P_y(\phi) \), can be determined from batch settling, centrifugation and pressure filtration tests. The hindered settling function, \( R(\phi) \), can be determined from batch settling, gravity permeation, centrifugation and pressure filtration tests. The solids diffusivity, \( D(\phi) \), can be calculated from \( P_y(\phi) \) and \( R(\phi) \), but can also be determined directly from pressure filtration tests. These techniques and their potential applications are described below.

**Settling Tests**

An equilibrium batch settling test has been developed to provide an estimate of the gel point, \( \phi_g \). The test can also be used to characterise \( P_y(\phi) \) at solids concentrations near the gel point. The test involves allowing suspension in a number of cylinders to settle to equilibrium. The gel point, \( \phi_g \), and some \( P_y(\phi) \), values are calculated from the initial solids volume fraction, the initial suspension heights and final suspension heights. This test procedure is simple to apply and is detailed in Chapter 6.

A transient batch settling test has been developed to characterise \( R(\phi) \) at solids concentrations ranging from equipment feed solids concentrations to slightly above the gel point. The test involves measuring the initial settling rate of a homogeneous suspension. An \( R(\phi) \) value is calculated for the initial solids concentration from the initial settling rate. This test procedure is quick, simple to apply and also detailed in Chapter 6.
Tomography of the solids concentration profile of consolidating and consolidated suspensions, using gamma rays (Rui et al. 2002) and microelectrical resistance tomography (Tapp and Williams 2000), can be applied to characterise suspension compressibility and also gain information about permeability. At present, the key limitation of these methods is insufficient spatial resolution for accurate reproducible results. However, the concept has significant potential for future applications as this limitation is resolved. The more expensive, but potentially very accurate method of nuclear magnetic resonance computerised tomography (NMR-CT) has been successfully applied to monitor the solids concentration profile of consolidating suspensions (Chang et al. 1997).

Permeation Tests

Landman and White and Green (Landman and White 1992; Landman and White 1994; Green 1997) have demonstrated how \( R(\phi) \) can be determined from the rate of permeation of liquor through a packed suspension bed subject to a high applied pressure. The theory applies Darcy’s Law assuming that gravity is insignificant relative to the applied pressure. The techniques are very tedious and time consuming with limited practical application, especially in light of recent advances in piston driven pressure filtration tests.

Nonetheless, a novel application of the permeation phenomenon has been developed to characterise \( R(\phi) \) at an applied pressure of about 1 kPa, using a gravitational liquor head for the driving force. The advantage of this technique is that it characterises \( R(\phi) \) at a solids concentration that is intermediate between those which can be characterised by settling tests and piston driven pressure filtration, while using a very small volume of sample. The gravity permeation technique is detailed in Chapter 6.

Centrifugation Tests

Centrifugation techniques for the characterisation of \( P_y(\phi) \) have been described and investigated in detail by Green (Green 1997). Tests include the multiple speed equilibrium sediment height technique (Green 1997) and the concentration profile technique (Green 1997). The multiple speed technique is very simple to apply, but can take a number of weeks to perform, leading to significant sample degradation. The concentration profile technique can be performed in a number of days, but is very labour intensive. Both techniques are subject to significant systematic error.

Attempts have been made to characterise \( R(\phi) \) (Rui et al. 2002) from the solids concentration profile over time with a gamma ray detector in centrifugation tests. Unfortunately the requirement of accelerating and decelerating the centrifuge between each profiling of the sample
rendered the method very labour intensive and also compromised accuracy. In addition, these tests took weeks to perform.

**Pressure Filtration**

A piston driven pressure filtration rig can be used to determine both $P_y(\phi)$ and $R(\phi)$ at solids concentrations above the gel point (Landman and White 1994; Landman *et al.* 1995; Green 1997; Landman *et al.* 1999). The advantage of piston driven filtration is that piston displacement can be accurately logged using a linear encoder. Older test methods used a single filtration test for each pressure that needed to be tested and thus characterisation was very labour intensive. A new stepped pressure filtration technique (de Kretser *et al.* 2001b; Usher *et al.* 2001) has been developed that cuts down sample characterisation time dramatically relative to the previous single pressure technique. Stepped pressure filtration is detailed and validated in Chapter 6.

Circumstances often arise when it is impractical to wait until a compressibility filtration test runs to completion, where filtration has ceased completely. Landman and White (Landman and White 1997) have developed a functional relation to predict the pressure filtration during the final stages of compression. This functional relation can be simplified to a generic functional form, which relates filtration time to the specific volume of filtrate. Using this functional form, the final solids concentration and the solids diffusivity at this solids concentration can be predicted by an iterative curve fitting optimisation procedure using data from an incomplete compressibility filtration test (de Kretser *et al.* 2001a). The method involved in such a predictive “$\phi_{\infty}$ estimation” technique is detailed and validated in Chapter 6.

The limitation of piston driven filtration is that it is difficult to reliably apply a pressure lower than 5 kPa. An automated air driven filtration test rig developed by de Kretser (Scales *et al.* 2001) enables pressure filtration for pressures down as low as 0.5 kPa. The drawback of air driven filtration is a loss of accuracy in measuring the extent of filtration due to the linear encoder being replaced by a less accurate analytical balance that logs the mass change after each drop of filtrate. Nonetheless, both stepped pressure filtration and $\phi_{\infty}$ estimation tests can be applied to air driven filtration.

**Comprehensive Characterisation**

In Chapter 6, numerous practically applicable dewaterability characterisation techniques are described. The techniques include equilibrium batch settling, transient batch settling, gravity permeation, stepped pressure filtration and $\phi_{\infty}$ estimation filtration tests. There are many other tests proposed in the literature, but those listed above were found to be sufficient to reliably
characterise permeability over almost the full range of solids concentrations from dilute particulate suspensions to concentrated networked suspensions. The one important area that is missing is settling rates of isolated particles from floc size and density analysis, ie. $\phi = 0$. This type of work has been performed by numerous workers (Farrow and Warren 1993; Glover et al. 2000), but the process involves very labour intensive, time consuming work making it difficult to find volunteers. Work on automating floc size and density analysis methods is under development at The University of Melbourne (Scales et al. 2001).

### 3.4.6.3 Fundamental Dewatering Predictions

The same fundamental material properties including $P_\lambda(\phi)$, $R(\phi)$, and $D(\phi)$ can be used to predict dewatering behaviour in a wide range of dewatering processes based on the same fundamental dewatering theory (Buscall and White 1987). Landman and co-workers and Bürger and Concha have developed numerical integration algorithms for the prediction of batch consolidation processes (Howells et al. 1990; Landman and White 1994), continuous flow gravity thickening (Landman et al. 1988; Green 1997; Bürger and Concha 1998) and pressure filtration (Landman et al. 1991; Landman and White 1994; Landman et al. 1995). In addition, Landman and White (Landman and White 1997) have developed an analytical solution to pressure filtration for situations where the influence of gravity is insignificant.

These methods generally involve solution of a dewatering problem in only one dimension, sometimes generalised to two dimensions through the use of a shape factor for cross sectional area variations. This simplification has generally been necessary to ensure that computer modelling simulations can be completed within an acceptable time. The primary limitation of modelling in one dimension is that the influence of shear and flow processes in dewatering cannot be properly accounted for. Nevertheless, these simplistic models still provide useful information about the influence of variations in process variables on dewatering performance.

Fundamental theories of multiphase fluid mechanics incorporating compressional dewatering phenomena in three dimensions have been proposed (Drew 1983; Soo 1989; Ungarish 1993; Bürger 2000), however the practical application of these fundamentally based models to predict the operation of industrial dewatering systems is still to be realised. The principal obstacles are the development of robust numerical computation algorithms that can be performed in reasonable time for these very complex models.
3.4.7 VARIABLES INFLUENCING DEWATERABILITY

As has been noted, there are many variables that influence suspension dewaterability. The influence of solids concentration, solids and liquor properties, flocculation and shear processes during consolidation are overviewed below.

**Solids Concentration**

The most significant contribution to suspension dewaterability is due to the solids concentration as demonstrated in Figures 3.3, 3.4 and 3.5. Methods for determining the solids concentration are described in Chapter 4.

**Solid and Liquor Properties**

Liquor density, viscosity and pH all influence suspension dewaterability and are all influenced by the dissolved solids content of the liquor and the process temperature. In addition, the nature of the undissolved solid particles also influence dewaterability due to solids density, particle size and size distribution, surface chemical properties and inter-particle forces. Methods for the determination of liquor density and viscosity are also described in Chapter 4.

**Flocculation**

The primary purpose of flocculation is to improve the settling rate and permeability of a particulate suspension, without significantly compromising compressibility. This enables either a higher output solids concentration or increased throughput to be achieved in a dewatering operation such as filtration or thickening. The mechanism of flocculation combines small particles to form larger agglomerates called flocs that have enhanced settling rates at low solids concentrations and a more permeable suspension network structure at higher solids concentrations. Flocculation is also employed to reduce particle size segregation that can cause fouling on filter membranes and rake bogging in thickeners. The chemical nature of the flocculant, the molecular weight of the flocculant, the dose of the flocculant and the shear regime during flocculation all have significant influence on floc structure. This floc structure can influence supernatant turbidity, suspension shear rheology as well as suspension dewaterability and have significant process implications.

There are several methods of flocculating suspensions in the laboratory. They can involve continuous flocculation equipment such as a shear vessel and a pipe reactor and batch process equipment such as a plunger in a measuring cylinder and a baffle reactor. Because the shear vessel and pipe reactor flocculate continuously they are more likely to represent flocculation conditions in the plant. However, these units require large quantities of sample, (which also needs to be disposed of) significant laboratory space and appropriate equipment for sample
preparation, hence making them less suitable for many laboratory based experiments. Flocculant suppliers commonly utilise the plunger test to compare the effectiveness of various flocculants and determine optimum flocculant dosages. The technique is simple and fast to carry out, making it especially suitable for on-site work. The baffle reactor, based on the standard tank configuration described by Holland and Chapman (Holland and Chapman 1966), consists of a flat-based glass vessel fitted with four baffles where agitation is provided through a centrally located, six bladed Rushton impeller that is fitted to a variable speed drive (Scales et al. 2001). The plunger and baffle reactor flocculation testing procedures are described in the Pinjarra and QAL site visit testing reports in section 9.3.3.1 and section 11.3.3.1 respectively.

Shear Processes During Consolidation

In a thickening process, there are numerous shear related phenomena that contribute to reducing and enhancing the extent of dewatering relative to what could be predicted by application of the fundamental dewatering theory introduced here. This includes an observed permeability enhancement due to channelling and shear processes involved with raking and converging thickener bases, but can also include permeability reduction due to the destruction of the permeable floc structure. The influence of raking can be investigated in the laboratory through the use of flat forks rotating in suspension.
SECTION B

CHARACTERISATION TECHNIQUES AND MODELLING TOOLS
Chapter 4

SOLID AND LIQUOR PROPERTY MEASUREMENT

4.1 INTRODUCTION

Liquor Density and Viscosity

Liquor density is a necessary piece of information for the determination of solids concentration, characterisation of particulate suspension dewaterability and process modelling. Likewise, liquor viscosity plays an important role in determining suspension viscosities and is also necessary for characterisation of particulate suspension dewaterability and for process equipment modelling. For many process operations, liquors closely resemble water and thus no measurement is necessary. In many industries however, dissolved solids concentrations are in excess of 300 g/L leading to significant increases in liquor density and viscosity.

Many industrial laboratories have equipment for the determination of liquor densities including hydrometers, which work on the principle of displaced volume buoyancy, online gamma radiation density gauges and many others. Where these methods are not available, use of a density bottle provides a quick and simple characterisation method that is presented in this chapter. It is not always feasible to measure liquor densities at the applicable temperature and thus corrections sometimes need to be applied. Variations of liquor density with temperature can be of the order of 1% for every 20°C change in temperature.

For Newtonian fluids, the Cannon-Fenske viscometer provides a simple method of determining process liquor viscosities. The experimental method is presented in this chapter. Understanding the variation of liquor viscosity with temperature enables prediction of dewatering behaviour over a range of operating temperatures. Additionally, if the liquor viscosity is known, then hindered settling function test results can be normalised to a standard viscosity (e.g. the viscosity of water at 20°C) or a traditional Darcian permeability, enabling comparison against results from other washers, where the liquor viscosities are different. In such a way, the influence of the flocculated network on the permeability alone can be gauged.

Solids Density

Solids density can be determined from a measured suspension density using a density bottle, but the method is not discussed here. The solids densities presented in this work are values
accepted as representative of red mud solids at each respective site. Metal oxide solids density values, such as red mud, measured at one temperature can generally be applied over a very wide temperature range with minimal error. This is because the thermal expansion coefficient of metal oxide solids is very small, generally less than 1 % for an 80°C change in temperature (Israelachvili 1991).

**Solids Concentration**

Traditional methods of determining solids concentration in the alumina industry include:

- in process radiation detectors,
- measurement of suspension density and
- a process that involves weighing or measuring the volume of a suspension sample, washing the suspension with hot water to remove dissolved solids from the entrained liquor, oven drying and then weighing the dried sample to determine the solids weight fraction or grams of solid per litre of suspension.

The method of determining particulate suspension solids concentrations employed in this work involves measuring the weight loss on drying of a suspension sample and a liquor sample in order to calculate the solids volume fraction. This technique differs from those that are traditionally used in the alumina industry in that there is the absence of a solids washing stage and also because the result expresses solids concentration in non-traditional units. Therefore, this non-traditional method is detailed in this chapter, as are conversion equations for the conversion of solids volume fraction to grams of solid per litre of suspension, solids mass fraction and suspension density. Use of a non-traditional method is employed because hot washing invariably leaves some dissolved solids in the suspension.

### 4.2 LIQUOR DENSITY MEASUREMENT

#### 4.2.1 PRINCIPLE OF MEASUREMENT USING DENSITY BOTTLES

Most process sites have the capability to measure the density of a Bayer process liquor, however a common problem on site is the inability, or impracticability of changing the measurement temperatures. Thus the density of the liquors can be measured using density bottles held in a thermostated water bath at a number of temperatures. The density bottle, generally of a standard volume of around 25 or 50 mL, is filled with the test liquor to enable the total liquor mass to be recorded and the density then calculated.
4.2.2 MEASUREMENT PROCEDURE

Part 1: Calibrate the Density Bottle Volume at the Test Temperature:

(i) Clean and dry the density bottle and lid.
(ii) Measure the mass of the clean, dry bottle and lid.
(iii) Fill the density bottle to the top with water and then insert the lid, displacing any excess liquid.
(iv) If the test temperature is above room temperature, place the bottle in a controlled temperature bath at the test temperature. Note that the initial filling of the bottle should always be at the lowest temperature that the liquor is to be characterised at.
(v) Wait at least 20 minutes for thermal equilibrium to be attained in the density bottle.
(vi) Once at thermal equilibrium, if the testing temperature is higher than the fill temperature excess liquor will have been expelled from the top of the capillary in the lid due to the expansion of the bottle contents. Remove the density bottle from the water bath and dry the outside with a tissue. (Liquid will be present at the end of the capillary on the lid – this should be dabbed away with a tissue, ensuring not to wick any excess liquid out of the capillary. At the end of this process, the liquid meniscus should be close to or at the top of the capillary in the lid.)
(vii) Quickly weigh the full density bottle and return it to the water bath.
(viii) Calculate the mass of water present and using the known density of water at the measurement temperature, calculate the volume of the density bottle.
(ix) Increase the water bath temperature set point to the next highest characterisation temperature and wait until the water bath temperature has stabilised at the new set point before repeating the procedure from step (v).

This process is repeated for each bottle, ensuring always to keep the same lid-bottle combination.

Part 2: Determine the Liquor Sample Density at the Test Temperature:

(i) Clean and dry the calibrated density bottle and lid.
(ii) Filter the solids out of the required amount of liquor for density determination using a syringe filter.
(iii) Fill the calibrated, clean and dry density bottle to the top with liquor and then insert the lid, displacing any excess liquid and dry the bottle with a tissue.
(iv) If the test temperature is above room temperature, place the bottle in a controlled temperature bath at the test temperature.
(v) Wait at least 20 minutes for thermal equilibrium to be attained in the density bottle.
Once at thermal equilibrium, if the testing temperature is higher than the fill temperature excess liquor will have been expelled from the top of the capillary in the lid due to the expansion of the bottle contents. Remove the density bottle from the water bath and dry the outside with a tissue.

Quickly weigh the bottle and return it to the water bath.

Calculate the density of the liquor from the calibrated density bottle volume and the measured mass of the liquor.

Increase the water bath temperature set point to the next highest characterisation temperature and wait until the water bath temperature has stabilised at the new set point before repeating the procedure from step (v).

### 4.3 LIQUOR VISCOSITY MEASUREMENT

#### 4.3.1 MEASUREMENT PRINCIPLE USING A CANNON-FENSKE VISCOMETER

The measurement of the liquor viscosities was performed using capillary viscometry in a Canon-Fenske viscometer (Figure 4.1). The principle of use of the Canon-Fenske viscometer is simple; a standard amount of the liquor to be tested is filled into the viscometer and allowed to drain into the lower chamber. Using a suction bulb, the liquor is drawn up into the higher of the upper two chambers and above the highest line on the tube (Line A). The suction is released and then, as the level of the liquid falls, the time taken for the liquid level to fall from Lines A to B, $t_{fall}$, is measured. The kinematic viscosity of the liquor, $\nu_{liq}$, is calculated by multiplication of this time by a simple calibration constant, $K_{cf}$, supplied by the viscometer manufacturer and checked using a viscosity standard.

$$\nu_{liq} = K_{cf} t_{fall} \quad (4.1)$$

The regular dynamic liquor viscosity, $\eta$, is calculated by multiplication of this kinematic viscosity, $\nu_{liq}$, by the liquor density, $\rho_{liq}$.

$$\eta = \rho_{liq} \nu_{liq} \quad (4.2)$$

Viscometers with different calibration constants are available for the testing of materials with a wide variety of viscosities. For the work presented in this thesis it was found that the best viscometers to use, based on typical liquor viscosities down the washer train were #25 and #75.
4.3.2 MEASUREMENT PROCEDURE

The following is a step-by-step procedure for the measurement of the viscosity of red mud washer overflow liquors. Generally the liquor viscosity was measured over a range of temperatures according to the procedure detailed below:

(i) Before commencement of filling of the viscometer, filter the liquor to be tested using a standard Millipore syringe filter to remove any suspended solids.

(ii) Place approximately 50 ml of the liquor to test in a container with low edges such as a petri dish.

(iii) Place the suction bulb over the end of Arm D of the viscometer. Invert the viscometer and place the end of Arm C in the liquor and draw the liquor up into the viscometer. When the level of the liquor reaches Line B, stop drawing the liquor up, remove the end of the tube from the liquor and invert the viscometer to prevent liquor draining out.

(iv) Place the filled viscometer in a water bath at the desired testing temperature. (Note that if viscosity testing is to be combined with density testing over a range of temperatures it is best to commence testing at the lowest temperature due to the testing method employed for the density).

(v) Allow at least 15 minutes after the temperature set point is reached in the bath for the liquid to thermally equilbrate in the viscometer.

**Figure 4.1:** Cannon-Fenske viscometer
Once at thermal equilibrium, place the suction bulb over the end of Arm C and suck the liquid above Line A. Release the suction, by removing the bulb and then measure the fall time between Lines A and B. Repeat this test at least three times or until a consistent fall time is obtained.

Take the average of the fall times and calculate the viscosity using the calibration constant for the viscometer.

Adjust the temperature of the water bath to the next highest desired temperature and wait for the bath temperature to stabilise. Return to Step (v).

4.4 SOLIDS CONCENTRATION MEASUREMENT

4.4.1 MEASUREMENT PRINCIPLE OF WEIGHT LOSS ON DRYING

The drying method employed for the determination of solids concentration involved oven drying of the suspension and a liquor sample representative of the liquor within the suspension. This method is the only practical method that gives accurate results for small sample analyses and accounts for the dissolved solids component of the liquor. The drying was performed under vacuum where available to accelerate the drying process.

Generally the drying containers employed for the measurement of solids concentration were made of polypropylene, had a mass of 6.8 g, a diameter of around 4 cm, a height of 3 cm and had a removable lid. If a sample had a very high level of dissolved solids, then a wider dish was employed to maximise the surface area available for drying. It was found that concentrated liquors had the effect of forming an impermeable “skin” which massively reduced the rates of drying in the oven and to avoid the drying problems a squat drying dish was required along with only a small amount of material.

4.4.2 WEIGHT LOSS ON DRYING MEASUREMENT PROCEDURE

For any washer stage, the first thing to do after sampling was to determine the suspension solids concentration. This involved measuring the dissolved solids concentration the overflow liquor and the weight loss on drying of a homogenised suspension sample. The solids concentration measurement procedure is detailed below:

Measurement of Total Dissolved Solids Concentration in Liquor

(i) Collect a liquor sample that is representative of the liquor within the particulate suspension of interest.
(ii) Select and weigh an appropriate sample drying container including the weight of the lid if applicable and note the weight as $m_{\text{liq, empty}}$. A wider, low dish is often necessary for viscous liquor samples.

(iii) Transfer 10-20 g of the liquor to the sample drying container and, if available, immediately place the lid on the drying container to prevent any evaporation over the period between sampling and weighing the wet sample.

(iv) Record the combined weight of the wet sample container with the lid on as $m_{\text{liq, wet}}$.

(v) Remove the lid, place it on the underside of the sample container and then place the sample in an oven at around 105°C to dry by evaporation. The drying of the liquor may take a significant amount of time due to the “skin-forming” nature of the liquor. If available, a vacuum oven can enhance the drying rate, but the vacuum must be applied slowly to prevent excessive bubbling that can make the sample overflow and render the test results invalid.

(vi) Periodically remove the sample from the oven and record the combined weight of the container, lid and dried liquor, before replacing it back in the oven. Repeat this process until the weight is constant and then record the minimum weight as $m_{\text{liq, dry}}$.

**Measurement of Particulate Suspension Weight Loss on Drying**

(i) Collect the particulate suspension at the solids concentration of interest.

(ii) Homogenise the particulate suspension to ensure that a sample with a representative solids concentration can be obtained. Depending on the thickness and volume of sample, this can involve shaking the sample container or mixing with a spatula.

(iii) Select and weigh an appropriate sample drying container including the weight of the lid if applicable and note the weight as $m_{\text{empty}}$.

(iv) Transfer 10-20 g of the homogenised suspension to the sample drying container and immediately place the lid on the drying container to prevent any evaporation over the period between sampling and weighing the wet sample. This proves to be critical when trying to obtain accurate determination of the solids concentration when hot suspensions at high solids concentrations are involved.

(v) Record the combined weight of the wet sample container with the lid on as $m_{\text{wet}}$.

(vi) Remove the lid, place it on the underside of the sample container and then place the sample in an oven at around 105°C to dry by evaporation. If available, a vacuum oven can enhance the drying rate, but be wary of excessive bubbling that can lead to the sample overflowing when solids concentrations are low.

(vii) Periodically remove the sample from the oven and record the combined weight of the container, lid and dried sample, before replacing it back in the oven. Repeat this process until the weight is constant or no longer decreases with time and then record the minimum
weight as \( m_{\text{dry}} \). It should be noted that, due to the nature of the solids, prolonged periods in the drying oven will actually lead to the weight increasing after some time. Thus “constant weight” should be taken as the minimum weight measured before the onset of weight increase.

**Calculations**

(i) Calculate the dissolved solids weight fraction of the liquor, \( A \), using

\[
A = \frac{m_{\text{liq,dry}} - m_{\text{liq,empty}}}{m_{\text{liq,wet}} - m_{\text{liq,empty}}}.
\]  

(ii) Calculate the dry weight fraction of the suspension including dissolved solids, \( B \), using

\[
B = \frac{m_{\text{dry}} - m_{\text{empty}}}{m_{\text{wet}} - m_{\text{empty}}}.
\]  

(iii) Calculate the solids weight fraction of the suspension, \( x \), corrected for dissolved solids, using Equation 4.5 or alternatively, calculate the solids volume fraction of the suspension, \( \phi \), corrected for dissolved solids using Equation 4.6.

\[
x = \frac{B - A}{1 - A}
\]  

\[
\phi = \frac{1}{1 + \left( \frac{\rho_{\text{sol},}}{\rho_{\text{liq}}} \left( \frac{1 - B}{B - A} \right) \right)}.
\]

Conversions between solids mass fraction, solids volume fraction, grams per litre and suspension density are detailed in the next section.

**4.4.3 SOLIDS CONCENTRATION CONVERSION EQUATIONS**

Though the standard method of expressing solids concentration in this body of work is as a solids volume fraction, \( \phi \), there are many other ways of expressing the solids concentration of particulate suspensions. These include grams of solids per litre of suspension, \( gpl \), solids weight fraction, \( x \), and suspension density, \( \rho_{\text{susp}} \). The conversion equations required to convert solids volume fractions to these alternatives are given in Equations 4.7 to 4.9 below, where solid, liquid and suspension densities, \( \rho_{\text{sol}} \), \( \rho_{\text{liq}} \) and \( \rho_{\text{susp}} \) respectively, are expressed in kg m\(^{-3}\).

\[
gpl = \phi \rho_{\text{sol}}
\]
The equations for conversion back to solids volume fractions are given by Equations 4.10 to 4.12 below.

\[ x = \frac{1}{1 + \frac{\rho_{liq}}{\rho_{sol}} \left( \frac{1}{\phi} - 1 \right)} \]  
\[ \rho_{susp} = \phi \rho_{sol} + (1 - \phi) \rho_{liq} \]  
\[ \phi = \frac{\rho_{pl}}{\rho_{sol}} \]  
\[ \phi = \frac{1}{1 + \frac{\rho_{sol}}{\rho_{liq}} \left( \frac{1}{x} - 1 \right)} \]  
\[ \phi = \frac{\rho_{susp} - \rho_{liq}}{\rho_{sol} - \rho_{liq}} \]
Chapter 5

SUSPENSION RHEOLOGY CHARACTERISATION TECHNIQUES

5.1 INTRODUCTION

Characterisation of shear rheology can be achieved using a wide range of experimental techniques. The range of techniques has been overviewed in Chapter 3. Unfortunately, many of the techniques available are not applicable to particulate suspensions or require non-portable equipment that is impractical for on site testing. The relevant techniques described here are those which determine steady shear stress measurements in the form of shear stress versus shear rate behaviour, those which determine the shear yield stress and those which readily enable thixotropy to be probed. As a result, the three most practical techniques include concentric cylinder rheometry with the bob rotating in an infinite media suspension or very large gap, the vane technique and the slump test. These techniques were used in the characterisation of the shear rheology data presented in this thesis.

5.2 CONCENTRIC CYLINDER RHEOMETRY (WITH A VERY LARGE GAP)

5.2.1 INTRODUCTION

A simple characterisation technique for determining the shear stress versus shear rate behaviour of particulate suspensions involves a bob rotating in an infinite media solution, described by Nguyen and Boger (Nguyen and Boger 1992). This method has the advantage of requiring only a portable rheometer for the characterisation of suspension shear rheology. Also, this technique overcomes the limitations of small gaps posed by traditional concentric cylinder rheometry for coarse particulate suspensions. The experimental technique involves measurement of the torque exerted on a bob at a number of rotational rates when rotating in a sample vessel of radius much larger than the radius of the bob. The calculation method exploits the fact that when a bob is rotated in a yield stress suspension, there will be a radius beyond which the suspension will not flow, and thus, if the bob is large enough, the torque will be independent of the vessel radius and calculations will be simplified (Nguyen and Boger 1987; Nguyen and Boger 1992).
5.2.2 THEORY

The shear stress at the bob surface, \( \tau \), equals the torque, \( T \), divided by the sheared surface area of the bob which is given by

\[
\tau = \frac{T}{2\pi R^2 L},
\]

where \( R \) is the radius of the bob and \( L \) is the length of the bob. The shear rate at the surface of the bob, \( \dot{\gamma} \), is given by

\[
\dot{\gamma} = \frac{2\Omega}{S},
\]

where \( \Omega \) is the angular velocity in radians s\(^{-1}\). With \( S \) being given by the following equation:

\[
S = \frac{d(\log T)}{d(\log \Omega)},
\]

5.2.3 EXPERIMENTAL PROCEDURE

Presented here is the standard experimental procedure employed at the University of Melbourne (de Kretser 1998), that was used to obtain the shear stress versus shear rate data presented in this thesis:

(i) Zero the rheometer.
(ii) Immerse the bob in the fluid such that the top of the bob is at the same level as the level of the suspension.
(iii) Rotate the bob at the desired rates from the lowest to the highest rates and back again. Wait for the reading to stabilise at each rate and then note the torque reading, \( T \).
(iv) Convert the rotation rates to angular velocities, \( \Omega \), using the conversion 1 rotation = \( 2\pi \) radians.
(v) Plot \( \log(T) \) versus \( \log(\Omega) \) and fit the data by a regression method to determine the slope of the plot, \( S \), at each of the data points obtained according to Equation 5.3.
(vi) Calculate the shear stresses and shear rates using Equations 5.1 and 5.2.
(vii) Calculate the viscosity from the following equation

\[
\eta = \frac{\tau}{\dot{\gamma}}.
\]

(viii) Plot viscosity versus shear rate (\( \eta \) vs. \( \dot{\gamma} \)).
It is important to note that particulate suspensions with low shear yield stresses tend to settle over time, especially the coarse particles, so it was sometimes necessary to re-homogenise the suspension during the testing process.

5.3 THE VANE TECHNIQUE

5.3.1 INTRODUCTION

A simple method of determining the shear yield stress of a suspension is the vane technique, developed by Nguyen (Nguyen 1983a; Nguyen and Boger 1985a). The advantage of this technique is that it provides a very simple and quick single point measurement of shear rheology. This technique can be applied to suspensions with shear yield stresses as low as 5 Pa and as high as 1000 Pa.

The vane technique, adapted from soil and cement characterisation techniques, involves rotating a vane, attached to a rheometer spring, in the suspension until the material yields. The principle behind the technique is that the application of a torque to a four (or more) bladed vane in a suspension will cause yielding. As the driving motor stretches the spring attached to the vane, the rotational torque rises and the suspension yields elastically until the torque exceeds a critical value. At this critical torque, the stress generated on the surface of the cylindrical yielding element (See Figure 5.1) is greater than the shear yield stress of the sample and the suspension yields irreversibly and the vane rotates. The yield stress is then calculated from the cylindrical yielding surface area determined from the vane dimensions and the maximum torque interpreted as the point of irreversible yielding. Thus the torque needed for yielding of a given material will be dependent on the dimensions of the vane used.

5.3.2 THEORY

The shear yield stress can then be calculated from the maximum torque obtained during testing, \( T_{\text{max}} \), divided by a vane constant, \( K_v \),

\[
\tau_y = \frac{T_{\text{max}}}{K_v} \quad (5.5)
\]

Nguyen and Boger (Nguyen 1983a; Nguyen and Boger 1985a) showed that the vane constant is dependent on the vane dimensions according to the following equation,

\[
K_v = \frac{\pi D_v^3}{2} \left( \frac{H_v}{D_v} + \frac{1}{3} \right), \quad (5.6)
\]
where \( D_v \) is the vane diameter and \( H_v \) is the vane height.

**Figure 5.1:** Schematic of the vane used for shear yield stress measurement, highlighting the height, \( H_v \), and the diameter, \( D_v \).

### 5.3.3 APPARATUS

The viscometers employed to obtain the shear yield stress data presented in this thesis were a Brookfield DVIII and a Haake VT550 Viscometer. In practice it does not matter what viscometer is employed for yield stress testing as long as the torque range is satisfactory for the range of yield stresses it is desired to test. The main stipulation is that a rotational speed as low as 0.2 rpm is achievable and that the maximum torque values are within an accurate range for the rheometer spring. This generally requires selection of an appropriately sized vane. That is, a small vane for weak springs and a large vane for strong springs. The Brookfield had a weak spring, and even with a small vane, the maximum yield stress that could be measured was around 200 Pa, while the Haake had a strong spring and lacked accuracy for shear yield stresses below 5 Pa, even for larger vanes.

### 5.3.4 EXPERIMENTAL PROCEDURE

*Prepare the viscometer and vane*

(i) Zero the viscometer.

(ii) Select the desired vane, connect it to the rheometer spindle and note its vane constant, \( K_v \). If the vane constant is not known, then measure the vane diameter, \( D_v \) and vane height \( H_v \).
(iii) Either set the viscometer to display torque readings or, if set up, initialise a yield stress measuring protocol and set up test parameters, one of which is a speed of rotation 0.2 rpm.

*Prepare the suspension and insert the vane into the suspension*

(iv) Equilibrate the suspension sample at the test temperature and homogenise the suspension to eliminate the solids concentration distribution that sometimes occurs due to particle settling.

(v) Place the sample to be tested on a stand under the rheometer (a scissor stand works well) and raise the stand slowly so that the vane gently penetrates the surface of the material. Insert the vane to a depth such that it is completely covered and immersed up to the ridge on the shaft.

*Perform the measurement*

(vi) Start the motor rotating and watch the build up of torque. The torque will rise until a maximum reading is reached, followed by stabilisation to a slightly lower value over time. Note that vibrations on the bench can affect the results, so attempts should be made to prevent vibrations.

(vii) Note the maximum torque, $T_{\text{max}}$.

(viii) Lower the sample stand and clean the vane. It is advisable to repeat the measurements up to three times to ensure accuracy.

*Calculate the shear yield stress*

(ix) If necessary, calculate the vane constant, $K_v$, using Equation 5.5.

(x) Calculate the shear yield stress, $\tau_y$, from $T_{\text{max}}$ and $K_v$ using Equation 5.6.

**5.3.4.1 Notes on Shear Yield Stress Measurement**

In order to obtain accurate results when taking measurements, the following considerations were observed.

*Vane Issues*

Due to the wide range of vanes available for use, materials with a wide range of yield stresses can be measured. Sometimes, the size of the vane selected was dictated by the amount of sample available for testing or the size of the sample vessel. To minimise errors, some guidelines for the required relationship between vane dimensions and sample vessel size provided by Nguyen (Nguyen 1983b) are given below in Equations 5.7,
\[
\begin{align*}
D_v &\leq 0.5D_c, \\
&\left\{z_{\text{above}} \geq D_v, \\
&z_{\text{below}} \geq 0.5D_v\right\},
\end{align*}
\]

(5.7)

where \(D_c\) is the sample container diameter, \(D_v\) is the vane diameter, \(z_{\text{above}}\) is the distance from the top of the vane to the suspension surface and \(z_{\text{below}}\) is the distance from the base of the vane to the base of the sample container. As a result, the vane diameter dictated the minimum vane insertion depth. Vanes produced at The University of Melbourne have a ridge on the shaft that indicates this depth. If the vane was not immersed in the sample up to the ridge on the vane shaft then the measured shear yield stress was potentially under-estimated. Nguyen (Nguyen 1983b), described the optimum rotational speed for the application of torque to the vane. Too fast and the true \(\tau\) will be overshot. Too slow and creeping of the suspension structure will occur. A speed of 0.2 rpm was adopted as a standard.

**Process Materials Issues**

Testing of process materials was usually at elevated temperatures and this led to further considerations.

- The temperature dependence of the shear yield stress (See Chapter 12) dictated that measurements be conducted at process temperatures where practical. Thus process samples were contained in a temperature bath and adequately covered and sealed from the environment to prevent evaporation and thermal losses.
- Many process samples were prone to settling and required re-suspension in between measurements. This also served the purpose of homogenising the thermal distribution in the sample.
- Air was easily entrained in yield stress suspensions during stirring. The presence of air bubbles in samples during testing was a common source of error that manifested as poor reproducibility. Tapping of the sample container on the bench or floor prior to testing assisted in the removal of entrained air and improved the accuracy of the results.
- The net effects of temperature, settling, thixotropy and evaporation on the shear yield stress of individual samples were difficult to isolate from each other. Therefore obtaining a reliable measure of the shear yield stress of a process sample required repeated measurements until the measured yield stress was consistent, with replicate values within about 10%.
5.4 THE SLUMP TEST

5.4.1 INTRODUCTION

The slump test, adapted from soil consistency tests by Pashias (Pashias et al. 1996) is the simplest technique available for the determination of the shear yield stress of paste-like materials. The test has a number of advantages, the most significant of which are the simplicity of the test and the in-expensive equipment requirement of a piece of PVC pipe, a ruler and a smooth flat surface. The test can be performed in the field or at the thickener underflow with minimal sample preparation. Unfortunately, the test is limited to the range of solids concentrations where the material has a paste-like consistency. As such, it is not possible to use the test to measure low yield stresses associated with dilute, runny suspensions.

The test uses the principle that if an open-ended cylinder is placed vertically on a flat surface and filled with suspension, that on lifting the cylinder, the material will slump under its own weight. By measuring the amount of slump for a given height of material, the yield stress may be calculated. Thermal control is indirectly possible by quickly testing materials that have been immersed in a high temperature environment and only just removed, prior to the onset of appreciable cooling. This method was applied for some of the shear yield stress measurements presented in this thesis for the Worsley and QAL site work in 2000.

5.4.2 THEORY

The theoretical validation of the extraction of a shear yield stress from slump measurements was first presented by Murata (Murata 1984) and corrected by Christensen (Christensen 1991). An overview of this theory, as presented by Pashias (Pashias et al. 1996), is given here. The basis of the slump test is that in an unsupported vertical column of suspension, the shear stress generated via the compressive weight of the material itself can be calculated as a function of the distance from the top of the column. For yield stress materials, solid behaviour is exhibited down to a critical point some distance below the top of the column where the self-weight stress equals the shear yield stress of the material. Below this point, the shear yield stress is exceeded and the material will yield and flow resulting in slumping of the column. On slumping, the material flows and spreads out, resulting in an increase in the area over which the self-weight force acts, causing a reduction in the shear stresses in the system. Thus there will be a point at which the spreading of the material reduces the shear stresses in the system to the point that at every level in the slumped column of material, the self-weight shear stresses are in equilibrium with the shear yield stress. At this time the slumping will stop and an equilibrium slump height is attained. In practice, slumping is over in a matter of seconds. The equilibrium height will be
composed of an undeformed region where the shear yield stress was not exceeded and the slumped region, where spreading occurred to reduce the internal stresses in the material. Thus the final height of the slumped material will be related to its shear yield stress. A schematic diagram of the slump test is presented in Figure 5.2, displaying the important variables in slumping. The diagram also indicates the stress distributions involved in slumping.

Figure 5.2: Schematic diagram of the cylinder slump test, showing initial and final stress distributions (from Pashias et al. (1996)).

Pashias et al. (Pashias et al. 1996) related the slump height, $s$, to the shear yield stress, $\tau_y$, using dimensionless variables. Calculation of the shear yield stress is performed using the dimensionless shear yield stress, $\tau'_y$, and the dimensionless slump height, $s'$, defined below,

$$
\begin{align*}
\tau'_y &= \frac{\tau_y}{\rho_{susp} g H} \\
\frac{s'}{H} &= \frac{s}{H}
\end{align*}
$$

where $\rho_{susp}$ is the suspension density and $H$ is the initial suspension height before slumping. From a slump test measurement, the dimensionless shear yield stress, $\tau'_y$, is related to the dimensionless slump height, $s'$, by the following equation;

$$s' = 1 - 2\tau'_y [1 - \ln(2\tau'_y)].$$

(5.9)

As slump height is the measurable variable, it would be desirable to re-express Equation 5.9 as a dimensionless yield stress in terms of dimensionless slump height. However, this is not mathematically possible. An approximate solution to Equation 5.9, shown below in Equation 5.10, was developed by Pashias et al. (Pashias et al. 1996) using a series expansion of the logarithmic term.

$$\tau'_y \approx 0.5 - 0.5\sqrt{s'}$$

(5.10)

Caution should be employed when using the approximation in Equation 5.10 because of significant inaccuracies for large dimensionless slump heights. A more accurate determination
requires an iterative procedure. A rearrangement of Equation 5.9 gives the following implicit equation:

$$
\tau'_y = \frac{1 - s'}{2(1 - \ln(2\tau'_y))}
$$

A self-focussing iterative method enables an accurate solution by first applying Equation 5.10 for an initial guess that is then applied in Equation 5.11. Iterative calculations using Equation 5.11 were repeated until a stable solution was obtained. The actual shear yield stress was then calculated by multiplying the dimensionless yield stress by $\rho_{susp}gH$.

In the development of the slump test, the slump model was observed to accurately predict the relationship between the cylinder slump height and the shear yield stress determined by the vane technique. The results showed the technique most accurately measures the shear yield stress for dimensionless yield stresses ranging from 0 to 0.15 (or dimensionless slump height values greater than 0.4). In simplified terms, the slump test is accurate when the yield stress is relatively low and the slump height is large. Consequently, increasing the cylinder height should maintain a dimensionless yield stress value and dimensionless slump height within the accurate region.

### 5.4.3 APPARATUS

It was mentioned in the previous section that to measure the yield stress of materials of a range of consistencies, cylinders of a range of different dimensions were required. The shear yield stress data presented in this thesis was derived from the slump tests that utilised two PVC cylinders, with an internal diameter of 70 mm and heights of 64 mm and 100 mm.

### 5.4.4 EXPERIMENTAL PROCEDURE

The slump test was applied to the measurement of the shear yield stress of thickener underflow suspensions in this thesis. The standard slump test methodology that was used is given below:

(i) Locate a clean, smooth, flat and level surface to perform the slump test on. If a smooth surface cannot be located, then a flat perspex board will usually suffice. Use of a board often makes cleaning up simple.

(ii) Place the cylinder on the testing surface and fill it with sample while holding the cylinder firmly in place. Using a thin spatula, remove any trapped air bubbles and smooth the suspension level at the top of the cylinder.
(iii) Then, lift the cylinder slowly and evenly. The material will begin to slump and flow of the material will occur as soon as the removal of the cylinder commences.

(iv) Measure the slump height, \( s \), with a ruler. The slump height is the change in height between the cylinder and deformed material. On occasion, the top surface of the slumped material is not level. In this case, the mid-point of the top level of the slumped material is taken as the representative height.

(v) Determine the suspension density using a standard technique.

(vi) Convert the slump height, \( s \), to a dimensionless slump height, \( s' \), using Equation 5.8.

(vii) Determine an approximate guess of the dimensionless shear yield stress, \( \tau_y' \), using Equation 5.10.

(viii) Calculate the dimensionless shear yield stress by iterative solution of Equation 5.11. If however, an on the spot result is required, apply the approximation in Equation 5.10, but beware of potential inaccuracies.

(ix) Convert the dimensionless shear yield stress, \( \tau_y' \), to the standard shear yield stress, \( \tau_y \), using Equation 5.8.
Chapter 6

DEWATERABILITY CHARACTERISATION TECHNIQUES

6.1 INTRODUCTION

There are many methods of measuring suspension dewaterability in terms of $P_f(\phi)$, $R(\phi)$ and $D(\phi)$, many of which have been overviewed in section 3.4.6.2, with each method having notable advantages and disadvantages. This project has involved the refinement of current techniques and the development of a number of new techniques. Some of those with practical application and potential are introduced and reviewed in this chapter. In brief, the techniques include gravitational batch settling tests for low solids concentration characterisation, gravity permeation for intermediate solids concentration characterisation as well as piston driven filtration for high solids concentration characterisation. It should be noted that the suspension dewaterability characterisation techniques described in this chapter assume familiarity with the measurement of material properties including liquid densities, liquid viscosities and solids volume fraction. Summaries of these techniques have already been presented in Chapter 4.

6.2 EQUILIBRIUM BATCH SETTLING

<table>
<thead>
<tr>
<th>Parameters Measured:</th>
<th>$\phi_g$, $P_f(\phi)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Concentration Range:</td>
<td>$\phi_g &lt; \phi &lt; \phi_g + 0.05$</td>
</tr>
<tr>
<td>Pressure Range:</td>
<td>$0 &lt; P &lt; 500$ Pa</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>10-95°C*</td>
</tr>
<tr>
<td>Time Required:</td>
<td>2 hours – 7 days</td>
</tr>
</tbody>
</table>

Table 6.1: Equilibrium batch settling test method overview.

6.2.1 INTRODUCTION

Equilibrium batch settling provides the only simple technique available for determining the gel point, $\phi_g$, and the compressive yield stress, $P_f(\phi)$, at solids volume fractions near the gel point. The gel point provides useful information regarding the structure of flocs and nature of inter-particle interactions in the system in addition to being an important modelling parameter. The test, overviewed in Table 6.1 is a long term test that requires at least 2 hours, with 1 or more

* Temperatures of up to 95°C are achievable using a thermostated bath, however there are problems with excessive vibration.
days being recommended for most samples. The test is quantitatively useful for coagulated and
well sheared flocculated suspensions, but is of questionable accuracy for freshly flocculated
material. The long test time makes vibration free temperature control difficult to obtain.
Limitations regarding the long-term stability of samples and particle size segregation provide
the major disadvantages of this test procedure, however, the advantages far outweigh the
shortcomings.

In short, the experimental technique involves allowing a number of suspension samples at low
solids concentrations, with varying initial suspension heights or initial solids concentrations, to
settle to equilibrium.

6.2.2 THEORY

A flocculated or coagulated particulate suspension with an initial solids volume fraction, \( \phi_0 \), and
initial suspension height, \( h_0 \), is settled under gravity in a batch settling experiment until
consolidation stops and the settling rate, \( u = 0 \). According to Equation 3.9, this implies that the
suspension network pressure, at all heights, is equal to the compressive yield stress of the
suspension. Therefore, the pressure gradient in such a suspension is a function of the fluid-solid
density difference, \( \Delta \rho \), the local solids volume fraction, \( \phi \), and gravitational acceleration, \( g \), and
is given by

\[
\frac{dp}{dz} = -\Delta \rho g \phi
\]  

(6.1)

This equation can be transformed to give

\[
\int dz = \frac{1}{\Delta \rho g} \int \frac{-1}{\phi} dp
\]  

(6.2)

According to Howells (Howells et al. 1990), the compressive yield stress, \( P_f(\phi) \), can be
modelled using an equation of the form

\[
P_f(\phi) = k \left( \frac{\phi}{\phi_g} \right)^n - 1
\]  

(6.3)

where \( \phi_g \), \( k \) and \( n \) are fitting parameters, with \( \phi_g \) representing the gel point. This equation can be
rearranged to give
Curve fitting experimental compressive yield stress data has shown that the Howells equation shape is not applicable for suspensions over a wide solids volume fraction range, but is still applicable for fitting low solids volume fraction data from equilibrium batch settling tests alone.

**Initial Solids Concentration Below the Gel Point**

Consider a suspension with an initial solids concentration that is below the gel point, $\phi_0 < \phi_g$. Once the suspension has completely settled to equilibrium, at the top of the settled suspension bed, $z = h_f$, the solids network pressure, $p = 0$, and at the base of the suspension, $z = 0$, the solids network pressure, $P_{\text{base}}$, is determined from the amount of suspension above it, given by

$$P_{\text{base}} = \Delta \rho g \phi_0 h_0.$$  

Insertion of these boundary conditions into Equation 6.2 gives

$$\int_0^{h_f} dz = \frac{1}{\Delta \rho g} \int_0^{\phi} -\frac{1}{\phi} dp.$$  \hspace{1cm} (6.6)

At equilibrium the suspension has settled to satisfy the following condition at all sediment heights.

$$p = p_y(\phi)$$  \hspace{1cm} (6.7)

Thus substitution of Equation 6.4 for $\phi$ and Equation 6.7 for $P_y(\phi)$ into Equation 6.6 and integrating gives the predicted equilibrium suspension height

$$h_{f,\text{pred}} = \frac{1}{\Delta \rho g \phi_g (1 - (1/\eta))} \left( \frac{\Delta \rho g h_0 \phi_0}{k} + 1 \right)^{-(1/(1/\eta))}.$$  \hspace{1cm} (6.8)

**Initial Solids Concentration Above the Gel Point**

For a suspension with an initial solids concentration that is greater than the gel point, $\phi_0 > \phi_g$, that has settled to equilibrium, there is a critical height, $z = h_c$, where the pressure is equal to the compressive yield stress at the initial solids volume fraction, $p = p_y(\phi_0)$. As for the previous case, the equilibrium pressure at the base of the suspension, where $z = 0$, is given by Equation 6.5. Insertion of these boundary conditions into Equation 6.2 gives
\[ \int_{0}^{h_{c}} dz = \int \frac{P_{s}(\phi_{0})}{\Delta \rho g} \frac{-1}{\phi} dp \quad (6.9) \]

At equilibrium, over this height range, \( 0 < z < h_{c} \), the suspension has again settled to satisfy Equation 6.7. Thus substitution of Equation 6.4 for \( \phi \) and Equation 6.3 for \( P_{s}(\phi) \) into Equation 6.9 and integrating gives the critical height

\[ h_{c} = \frac{k}{\Delta \rho g \phi_{g}} \left( \frac{\Delta \rho g h_{0}(\phi_{0})}{k} + 1 \right)^{(l-(1/n))} \left( \frac{\phi_{0}}{\phi_{g}} \right)^{n-1} \]  

(6.10)

Above this critical height, for a suspension with initial solids volume fraction greater than the gel point, the pressure is below the compressive yield stress such that, \( p < P_{s}(\phi_{0}) \), and as a result, this suspension has not compressed and remains at the initial solids volume fraction, \( \phi_{0} \). Equating the compressive yield stress of a suspension at the initial solids volume fraction, \( P_{s}(\phi_{0}) \), with the pressure due to suspension above the critical height, \( z > h_{c} \), gives

\[ P_{s}(\phi_{0}) = \Delta \rho g (h_{f,\text{pred}} - h_{c}) \phi_{0} \]  

(6.11)

Which rearranges to give the predicted equilibrium suspension height

\[ h_{f,\text{pred}} = \frac{P_{s}(\phi_{0})}{\Delta \rho g \phi_{0}} + h_{c} \]  

(6.12)

Substitution of Equation 6.3 for \( P_{s}(\phi_{0}) \) and Equation 6.10 for \( h_{c} \) into Equation 6.12 gives

\[ h_{f,\text{pred}} = \frac{k}{\Delta \rho g \phi_{0}} \left( \frac{\phi_{0}}{\phi_{g}} \right)^{n} - 1 \right] + \frac{k}{\Delta \rho g \phi_{g}} \left( \frac{\phi_{0}}{\phi_{g}} \right)^{n} - \left( \frac{\phi_{0}}{\phi_{g}} \right)^{n-1} \]  

(6.13)

**Gel Point Estimation**

Estimation of the gel point requires appreciation of the fact that as soon as particles or flocs form a network, the solids at the base of the network will experience a compressive force due to the weight of the overlying material. As a flocculated network is compressible, the compressive force acts to squeeze water out of the flocs, thereby concentrating the solids to a concentration higher than \( \phi_{g} \). Thus in settling experiments the sediment interface will not stop moving as soon as the gel point is reached and the sediment will continue to concentrate due to compression. However, at higher solids concentrations the network will be stronger, so settling continues until the network strength matches the compressive force supplied by the solids network above. As the compressive force increases with depth in the bed, the strength of the network required to
balance the compressive forces also increases with depth. Thus the solids concentration in the settled bed will change with depth from $\phi_g$ at the top of the sediment to a higher, maximum value at the base of the sediment. Hence the average solids concentration in a sediment will always be higher than the gel point as some compression of the network will always occur due to the weight of solids present.

Equation 6.5 shows that as $\phi_o$ and $h_o$ are changed, the maximum compressive force in the settled suspension, $P_{base}$, also varies and logically, so does the average suspension solids concentration, $\phi_{av}$. The key to estimating the gel point is determining the suspension solids concentration as the base pressure approaches zero ($P_{base} = 0$). If settling tests were set up with a range of different base pressures, then a corresponding range of $\phi_{av}$ would result. The experimental set up would involve small equilibrium batch settling tests where suspensions with varying initial heights, $h_0$, but a constant initial solids volume fraction, $\phi_0$, (or vice versa) settle to average equilibrium solids volume fractions, $\phi_{av}$. The gel point, $\phi_g$, could then be estimated from the extrapolation of $\phi_{av}$ to $P_{base} = 0$ on a plot of $\phi_{av}$ versus $P_{base}$. Some compressive yield stress, $P_y(\phi)$, data can also be determined from these equilibrium batch settling tests.

6.2.3 EXPERIMENTAL METHOD

Described below is the experimental characterisation methodology that was adopted for equilibrium batch settling tests conducted in this thesis.

6.2.3.1 Apparatus

The only equipment that was required included a range of settling cylinders, the dimensions of which depended on the volume of material available for testing, a ruler and the means to determine solids concentration. The cylinders had flat bottoms, a diameter of at least 40 mm and heights of up to 100 mm. Three to four cylinders were generally used in each test to produce reasonably accurate results, however when material was limited, one cylinder tests were used to provide valuable qualitative trends. When testing more readily available material such as red mud thickener underflow samples, more cylinders ranging up to much larger heights could have been employed to generate extra $P_y(\phi)$ data at higher solids concentrations. Unfortunately, it was evident that precipitation and thixotropy issues would have compromised those extra results.
6.2.3.2 Sample Preparation

Process Underflow Samples

Samples taken from thickener underflows generally had a solids concentration that was slightly above the gel point. The optimum solids concentration for starting the test was below the gel point, and as such, a 30-50% dilution from the as-received solids concentration, using thickener overflow liquor, was deemed sufficient.

Freshly Flocculated Samples

Generally, flocculated samples were produced via either a baffle reactor or a settling cylinder plunger test. The resulting flocculated suspension was generally collected at a solids concentration near the gel point and a dilution to 30-50% of this solids concentration using supernatant from the initial flocculation test gave an appropriate solids concentration for testing. Flocculated sample treatment for equilibrium batch settling tests required a significant degree of consistency because the freshly flocculated suspensions were very shear sensitive. The key to accurately measuring the gel point of the as-flocculated network was to consistently minimise the amount of shear imparted to the sample in the dilution process and thus the constant initial height method was deemed impractical. In cases where it was impractical to even transfer the suspensions to multiple cylinders, the final suspension height in the plunger flocculation test itself was used as single cylinder equilibrium batch settling data.

6.2.3.3 Test Procedures

There are numerous methods of preparing suspensions for equilibrium batch settling tests. These methods include using the final suspension heights of;

- a single suspension,
- multiple initial suspension heights with a constant initial solids concentration and
- multiple initial solids concentrations with a constant initial suspension height.

The choice of whether to use a single suspension or multiple suspensions in equilibrium batch settling tests generally depended on the amount of sample available. The choice of whether to use the constant initial solids concentration or constant initial suspension height method generally depended on which was logistically simpler. For this reason, the constant initial solids concentration method was used for all red mud multiple cylinder equilibrium batch settling results presented in this thesis. The test procedure for each method is outlined below.
Single Height Technique

i) Prepare the suspension at a solids concentration that is known to be below the gel point, $\phi_g$.

ii) Determine the initial solids concentration of the suspension, $\phi_0$.

iii) Add a volume of the suspension sample to the settling cylinder such that the final suspension height will be about 25 mm.

iv) Record the initial suspension height, $h_0$.

v) Allow the suspension to settle in the cylinder until an equilibrium sediment height is reached. This may take days, so leave sample undisturbed, minimising bumps and vibrations that can compromise the final height.

vi) Record the final suspension height, $h_f$.

vii) Calculate an estimate of the gel point, $\phi_g$, and/or $P_y(\phi)$ values at a low $\phi$ using an approximation method or an iteration method.

Constant Initial Solids Concentration Technique

(i) Prepare the suspension at a solids concentration that is 35-50% of the solids volume fraction at the gel point, $\phi_g$, by dilution with process liquor.

(ii) Determine the initial solids concentration of the suspension, $\phi_0$.

(iii) Add the sample to 3 or more settling cylinders at initial suspension heights, $h_{0,i}$, ranging from 25 to 100 mm.

(iv) Record the initial suspension height, $h_{0,i}$, for each cylinder.

(v) Allow the suspension in each cylinder to settle until an equilibrium sediment height is reached. This may take days, so leave samples undisturbed, minimising bumps and vibrations that can compromise the final height.

(vi) Record the final suspension height, $h_{f,i}$, for each cylinder.

(vii) Calculate an estimate of the gel point, $\phi_g$, and/or $P_y(\phi)$ values at low $\phi$ using an extrapolation and/or iteration method.

Constant Initial Height Technique

(i) Prepare the suspension at a range of initial solids concentrations that go as low as 30-50% of the solids volume fraction at the gel point, $\phi_g$ by dilution with process liquor. If taking a process underflow suspension, a number of dilutions should be made down to a lowest dilution that is around 30% of the original suspension solids concentration.

(ii) Determine the initial solids concentration of the suspensions, $\phi_{0,i}$.

(iii) Add a constant volume of each suspension to individual settling cylinders. Aim for a suspension height in the range 50-100 mm.
(iv) Record the initial suspension height, \( h_{0,i} \), for each cylinder.

(v) Allow the suspension in each cylinder to settle until an equilibrium sediment height is reached. This may take days, so leave samples undisturbed, minimising bumps and vibrations that can compromise the final height.

(vi) Record the final suspension height, \( h_f,i \), for each cylinder.

(vii) Calculate an estimate of the gel point, \( \phi_g \), and/or \( P_y(\phi) \) values at low \( \phi \) using an extrapolation and/or iteration method.

6.2.4 CALCULATIONS

The gel point is an important physical property of particulate suspensions that can be estimated by simply approximating the final average solids concentration as the gel point. This method generally overestimates the gel point. Using an extrapolation method can reduce this inaccuracy. In addition, an iterative method has been developed to determine a \( P_y(\phi) \) value for each cylinder in a test. These methods are detailed below.

6.2.4.1 Approximation of the Gel Point

When an estimate of the gel point must be determined from the equilibrium batch settling of a single cylinder test, the best approximation is the average solids volume fraction in the settled suspension calculated from the following equation.

\[
\phi_g = \phi_{av} = \frac{\phi_{0,i} h_0}{h_f}
\]  

(6.14)

This same approximation is also useful for equilibrium batch settling involving multiple cylinders when the quality of the data is low. In this case, the gel point can be quoted as the average of the estimates for each of the \( n \) cylinders.

\[
\phi_g = \frac{\sum_{i=1}^{n} \phi_{av,i}}{n}
\]  

(6.15)

6.2.4.2 Extrapolation of the Gel Point

Extrapolation is a simple, approximate method of estimating the gel point from equilibrium batch settling tests involving multiple cylinders whether the constant initial solids concentration or the constant initial height method is used. The solids pressure at the base of each test cylinder, \( i \), is determined using the following equation based on Equation 6.5:
The average solids concentration in the suspension bed for each test cylinder, $i$, may be determined from

$$P_{\text{base},i} = \Delta \rho g \phi_{0,i} h_{0,i}$$  \hspace{1cm} (6.16)$$

where $h_{f,i}$ is the final suspension height in each cylinder. The resulting values of $\phi_{av,i}$ are plotted against $P_{\text{base},i}$ and fitted to a curve. The type of curve used to fit to the data is arbitrary. A linear fit generally describes the data well, however in other cases a quadratic may be required. Alternatively, as it is common to get some curvature in data obtained for higher base pressures, this data may be ignored and a straight line fitted to the low base pressure data. The key is to get a good fit in the low base pressure region of the data. The curve is then extrapolated to zero base pressure and the intercept on the $\phi_{av}$ axis gives the gel point. An example of the results for a constant initial solids volume fraction test and a constant initial height test are presented in Figure 6.1. As can be seen in Figure 6.1, the base pressure that is used in testing should be as low as is practical in order to improve the accuracy of the extrapolation. This requires consideration of the implications of suspension height reading errors that are introduced when final suspension heights are less than 10 mm.

**Figure 6.1:** Determination of the gel point of a coagulated zirconia suspension by extrapolation of data from equilibrium batch settling tests with i) a constant initial suspension height of 0.05 m and ii) a constant initial solids volume fraction of 0.025.
The constant initial solids concentration technique gives a reasonably accurate measure of the gel point as long as the initial solids concentration is below the gel point. Obviously this is a somewhat circular argument as at the outset, the gel point is not known. Hence the importance of allowing a large safety zone in the dilution of material to a suitable solids concentration for testing. The constant initial height technique has only recently been trialed and results here suggest that it gives more indicative gel points, however this work is ongoing. Its major limitations are that it can require more sample than the other method and is impractical for characterising freshly flocculated suspensions.

6.2.4.3 Approximation of Compressive Yield Stress Data

In addition to the gel point, low $P_y(\theta)$ values can be obtained from equilibrium batch settling data. A simple approximation method involves assuming that the final average solids volume fraction for each settling test, $\phi_{av,i}$, is determined by a compressive force equal to the average applied pressure over the suspension, $P_{base,i}/2$. This gives a compressive yield stress datum point $(\phi_{av,i}, P_{base,i}/2)$ for each settling test using Equations 6.16 and 6.17. This approximation is of limited accuracy. However, the ease of calculation makes it useful for obtaining on the spot data.

6.2.4.4 Iterative Determination of Compressive Yield Stress Data

A more accurate iterative method of determining compressive yield stress data from batch settling test data involves optimisation of the fitting parameters in a $P_y(\theta)$ functional form. In this method, iterations continue until an error function comparing the predicted and actual final suspension heights is minimised. The calculation method is explained below.

A multiple cylinder batch settling test is performed using $n$ cylinders with initial solids volume fractions, $\phi_{0,i}$, below the gel point and with initial suspension heights, $h_{0,i}$. The suspensions settle over time to equilibrium heights, $h_{f,i}$. The value of the gel point is estimated using one of the methods already detailed.

If a pressure filtration test rig is available, compressive yield stress values, $P_y(\theta)$, are determined at $p$ pressures for this same flocculated suspension using pressure filtration. The pressure filtration data is then fitted to a functional form, such as Equation 6.3, to give initial guesses for the other equation parameters (eg. $k$ and $n$ for Equation 6.3) by minimising the sum of the squares of the fractional errors according to the following equation
\[
\text{Error} = \sum_{j=1}^{n} \left( \frac{P_j(\phi_j)^\text{pred} - P_j(\phi_j)}{P_y(\phi_j)^\text{pred}} \right)^2,
\]  

(6.18)

In principle, any compressive yield stress functional form could be used, provided the equilibrium suspension height can be predicted. For each equilibrium height, \(h_{f,i}\), a predicted steady state height is determined (eg. using Equation 6.8 if the compressive yield stress functional form given by Equation 6.3 is used). A fit error is calculated using the following equation

\[
\text{Error} = \sum_{j=1}^{n} \left( \frac{h_{f,\text{pred},j} - h_{f,j}}{h_{f,j}} \right)^2 + \sum_{j=1}^{n} \left( \frac{P_j(\phi_j)^\text{pred} - P_j(\phi_j)}{P_y(\phi_j)^\text{pred}} \right)^2,
\]  

(6.19)

where \(h_{f,\text{pred},i}\) and \(P_j(\phi_j)^\text{pred}\) are the batch settling equilibrium heights and pressure filtration compressive yield stresses as predicted by the parameter guesses. Optimisation of the fitting parameter guesses to minimise the error in Equation 6.19 generally provides a good estimate of the parameter values.

For the red mud results presented in this thesis, the functional form given by Equation 6.3 was applied in the optimisation of Equation 6.19 above. However, because Equation 6.3 does not represent red mud compressibility well over a wide range of solids concentrations, only the lowest pressure filtration datum point was used in the optimisation. The shortcoming of this was that the curve fits were only valid for the lower solids concentration range. The optimised fitting parameter values were then used to calculate the solids concentration at the base of each test cylinder using Equation 6.4. Subsequently, the optimised fitting parameter values were used to determine the equilibrium solids concentration and pressure at the base of each cylinder to provide a compressive yield stress datum point for each settling test cylinder using Equation 6.4 and Equation 6.5.

As a further extension, if the initial gel point estimate was not considered reliable, the gel point could also have been included as a variable in optimising the error given by Equation 6.19, but this was not attempted.

6.2.5 APPLICATION SUMMARY

Red mud equilibrium batch settling results are presented for each site in Chapters 9, 10 and 11. The constant initial height method was generally employed using three to four cylinders for each sample. For some flocculated samples, the final height in a single plunger flocculation test cylinder was used so as to minimise disruption to the sample. For multiple cylinder tests, the
gel point was determined by extrapolation, but the final average solids concentration was used as a qualitative approximation when only one cylinder was used. $P_y(\phi)$ data points were determined through the iterative optimisation method.

### 6.3 TRANSIENT BATCH SETTLING

<table>
<thead>
<tr>
<th>Parameters Measured:</th>
<th>$R(\phi)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Concentration Range:</td>
<td>$0.01 &lt; \phi &lt; 0.25$ for red mud (approximately 30-750 gpl).</td>
</tr>
<tr>
<td>Pressure Range:</td>
<td>$0 &lt; P_y(\phi_o) &lt; 200$ Pa</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>$10-95^\circ$C</td>
</tr>
<tr>
<td>Time Required:</td>
<td>10 minutes – 1 day</td>
</tr>
<tr>
<td>Advantages:</td>
<td>Quick and simple test, enables characterisation at low solids concentrations, simple analysis, short times relative to precipitation.</td>
</tr>
<tr>
<td>Limitations:</td>
<td>Average accuracy, only one datum point produced per test, relatively large volumes of material required.</td>
</tr>
</tbody>
</table>

**Table 6.2:** Transient batch settling test method overview.

### 6.3.1 INTRODUCTION

Transient batch settling tests, overviewed in Table 6.2, provide a method of determining the hindered settling function, $R(\phi)$, over a wide range of solids concentrations. These solids concentrations cover the full range found in thickener operations, varying from the low solids concentrations associated with feed suspensions to solids concentrations slightly above the gel point. The solids concentration upper limit for applicability of this test is determined by the suspension compressive yield stress, which should be less than about 200 Pa. The test can be conducted in one litre measuring cylinders at $10-95^\circ$C while kept in temperature controlled water baths. The time required for the test depends strongly on the initial suspension solids concentration and can vary from a number of minutes for dilute suspensions such as thickener feeds to a whole day for concentrated thickener underflows.

The advantages of the transient batch settling technique include its simplicity, its applicability to a wide range of solids concentrations, the relatively short experimental times required and its adaptability from traditional flocculation testing methods. Additionally the data treatment is relatively simple. Results have shown that some scatter exists in the data generated. However, considering that settling rates can vary by five or more orders of magnitude from low to high solids concentrations, the significance of this error is diminished.

In this test the initial settling rate of the suspension-supernatant interface is tracked in a number of settling cylinders with different initial solids concentrations above or below the gel point. The hindered settling function, $R(\phi)$, is calculated using the suspension’s initial solids volume
fraction, $\phi_0$, initial suspension height $h_0$, initial solids settling rate, $u$, the solid-liquid density difference, $\Delta \rho$, and the compressive yield stress at the initial solids concentration, $P_y(\phi_0)$.

### 6.3.2 THEORY

The dewatering model force balance (Equation 3.7) and the batch settling material balance (Equation 3.8) combine to form Equation 3.10. Application of this equation to a batch settling situation with an initial solids volume fraction $\phi_0$ gives the following:

$$u = \frac{\Delta \rho g (1 - \phi_0)^2}{R(\phi_0)} \left(1 + \frac{\partial p}{\partial z} \frac{1}{\Delta \rho g \phi_0}\right).$$  \hfill \text{(6.20)}$$

Batch settling is assumed to occur through consolidation that starts at the base of a suspension through the upward flow of liquor and downward flow of solids (Howells et al. 1990). It is inferred that as consolidation commences, the network pressure varies from zero at the top of the suspension to the suspension compressive yield stress at the base of the suspension. Therefore, in the initial stages of a batch settling test, the pressure gradient over the suspension is given by

$$\frac{\partial p}{\partial z} = \frac{0 - P_y(\phi_0)}{h_0 - 0} = -\frac{P_y(\phi_0)}{h_0}$$  \hfill \text{(6.21)}$$

where $h_0$ is the initial suspension height. Substitution of Equation 6.21 for the solids network pressure gradient into the force balance in Equation 6.20 gives the following for the initial settling rate $u_0$:

$$u_0 = \frac{\Delta \rho g (1 - \phi_0)^2}{R(\phi_0)} \left(1 - \frac{P_y(\phi_0)}{\Delta \rho g \phi_0 h_0}\right)$$  \hfill \text{(6.22)}$$

The behaviour represented in this equation is predicted by Howells et al (Howells et al. 1990). For small times based on the fundamental mathematical theory of Buscall and White (Buscall and White 1987). This equation can be simplified to the following

$$u_0 = \frac{\Delta \rho g (1 - \phi_0)^2}{R(\phi_0)} (1 - \varepsilon)$$  \hfill \text{(6.23)}$$

where

$$\varepsilon = \frac{P_y(\phi_0)}{\Delta \rho g \phi_0 h_0}$$  \hfill \text{(6.24)}$$
is the ratio of the compressive yield stress of the initial suspension to the equilibrium solids network pressure at the base of the suspension. The \( \epsilon \) term accounts for and gives a measure of the retarding effect of the suspension network on the settling rate. Therefore, the compressive yield stress can influence the initial settling rate. Of course, when the initial solids volume fraction is below the gel point \( (\phi_0 < \phi_g) \), the compressive yield stress is zero and so is \( \epsilon \), giving the following equation for the initial settling rate (which is equivalent to the free settling rate derived in Chapter 3, Equation 3.11):

\[
u_0 = \frac{\Delta p g (1 - \phi_0)^2}{R(\phi_0)}
\]  

(6.25)

Transformation of Equation 6.23 gives the hindered settling function in terms of the initial settling rate, \( u_0 \), and a minor compressibility dependence, \( \epsilon \).

\[
R(\phi_0) = \left(1 - \epsilon\right) \frac{\Delta p g (1 - \phi_0)^2}{u_0}
\]  

(6.26)

As a result, when the initial solids volume fraction is above the gel point, \( (\phi_0 > \phi_g) \), the hindered settling function is calculated using the initial settling rate and the compressive yield stress at the initial solids volume fraction (determined via equilibrium batch settling detailed in section 6.2). If the compressive yield stress is small, but not known, then an upper bound estimate of the hindered settling function can be calculated by ignoring the \( \epsilon \) term. When the initial solids volume fraction is below the gel point, \( (\phi_0 < \phi_g) \), the hindered settling function can be calculated from the initial settling rate alone.

6.3.3 EXPERIMENTAL METHOD

6.3.3.1 Apparatus

The only equipment that was required for this test was a number of settling cylinders and a stopwatch. 1 litre cylinders, typical of those employed in plunger settling tests, were employed in the collection of the transient batch settling test data presented in this work. Use of smaller cylinders led to increases in experimental error and were avoided where practical.

6.3.3.2 Sample Preparation

*Thickener Underflow Suspensions*

For the characterisation of thickener underflow suspensions, enough overflow liquor and underflow suspension was sampled to enable dilution and concentration to produce five or more
litres of suspension at a range of solids concentrations. Typical dilutions used in this work were 0.2, 0.4, 0.6, 0.8 and 1.0 times the as-sampled underflow solids concentration in addition to concentration of one sample to 1.1-1.2 times as-sampled underflow solids concentration. All dilutions were performed using thickener overflow liquor so that the effect of surface chemistry was minimised.

**Flocculated Suspensions**

For the testing of freshly flocculated suspensions, the solids concentration of flocculation was the lower solids concentration limit for the $R(\phi_0)$ data that could be generated. Traditional flocculation tests were used to measure the initial settling rate at the flocculation solids concentration. Obtaining data at higher solids concentrations required the products of multiple flocculation tests to be combined into a single test cylinder at a higher solids concentration. This process was labour intensive and therefore only a small number of transient batch settling tests conducted at higher solids concentrations, where flocculated suspensions were involved. When transferring freshly flocculated suspensions, it was important to be gentle on the flocs to prevent significant permeability loss. However, some degradation always resulted. The most important thing was to shear the suspensions consistently during sample transfer and homogenisation so that the influence of flocculation condition variations could be reliably observed.

### 6.3.3.3 Test procedure

**Thickener Underflow Suspensions**

(i) Measure the solids concentration of each sample to be tested.

(ii) Add the homogenised suspension samples to the settling cylinders so that the initial heights are close to the top of the cylinders.

(iii) If the tests are to be conducted at non-ambient temperatures, transfer the cylinders to a temperature controlled water bath with a viewing window and allow the suspension temperatures to equilibrate at the test temperature.

(iv) When ready, use a traditional flocculation test plunger to homogenise the suspensions.

(v) Record the initial suspension height, $h_{in}$, for each cylinder.

(vi) Allow the contents of each cylinder to settle. As settling occurs, record the height of the settling interface periodically until the interface settling rate slows. The rate of settling will vary depending on the material and the initial solids concentration but the constant rate period will generally be over within a number of hours.
For each settling rate test, calculate the value of $R(\phi_0)$ using Equation 6.26. This may require determination of the compressive yield stress at the initial solids concentrations, $P_y(\phi_0)$, using a method from section 6.2.

Flocculated Suspensions

(i) Determine the solids concentration of the suspension samples to be flocculated.

(ii) Flocculate each of a number (2-5) of suspension samples under the same conditions, determining the initial settling rate.

(iii) Decant the supernatant liquor from each flocculated suspension and transfer the remaining suspension samples to a single settling cylinder so that the initial heights are close to the top of the cylinders.

(iv) If the test is being conducted at a non-ambient temperature, transfer the cylinder to a temperature controlled water bath with a viewing window.

(v) Optionally, use a traditional flocculation test plunger to gently homogenise the suspension, but be aware that all shearing will degrade the permeability. The recommendation is to always be consistent, to allow a valid comparison of results.

(vi) Record the initial suspension height, $h_0$.

(vii) Allow the contents of the cylinder to settle. As settling occurs, record the height of the settling interface periodically until the interface settling rate slows. The rate of settling will vary depending on the material and the initial solids concentration but the constant rate period will generally be over within a number of hours.

(viii) For each settling rate test, calculate the value of $R(\phi_0)$ using Equation 6.26. Generally, $\varepsilon$ can be assumed as zero.

6.3.4 APPLICATION SUMMARY

Transient batch settling tests were applied to the characterisation of red mud suspensions at each sponsor site in 2000 and the results are presented in Chapters 9, 10 and 11. The tests were conducted over a wide range of solids concentrations from thickener feed solids concentrations to underflow solids concentrations for both underflow suspensions and laboratory flocculated suspensions. When the initial solids concentration was below the gel point, an estimate of $P_y(\phi_0)$ was obtained from equilibrium batch settling results.
6.4 GRAVITY PERMEATION

<table>
<thead>
<tr>
<th>Parameters Measured:</th>
<th>$R(\phi)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Concentration Range:</td>
<td>$0.15 &lt; \phi &lt; 0.30$ for red mud (approximately 600-900 gpl).</td>
</tr>
<tr>
<td>Pressure Range:</td>
<td>$500 &lt; P,(\phi_{av}) &lt; 1500$ Pa</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>$20-25^\circ$C (Temperature control up to perspex limit of 60°C is possible using a water jacket)</td>
</tr>
<tr>
<td>Time Required:</td>
<td>3 hours – 1 day</td>
</tr>
<tr>
<td>Advantages:</td>
<td>Operates at lower solids concentrations than pressure filtration, mechanically and computationally simple, only small samples required.</td>
</tr>
<tr>
<td>Limitations:</td>
<td>Average reproducibility, only single $R(\phi)$ point, relatively long test times.</td>
</tr>
</tbody>
</table>

Table 6.3: Gravity permeation test method overview.

6.4.1 INTRODUCTION

Gravity permeation tests, overviewed in Table 6.3, provide a method of determining $R(\phi)$ at intermediate solids concentrations, slightly above the gel point, corresponding to an applied pressure of about 1 kPa. Accurate modelling of dewatering processes requires complete dewaterability characterisation from dilute low solids concentrations that can be characterised in transient batch settling and paste/cake behaviour that can be characterised using pressure filtration. As a result, gravity permeation is a useful technique providing data that bridges the gap between dilute suspension behaviour and paste/cake behaviour. The test can be conducted at ambient temperatures, or at elevated temperatures up to 60°C using a water jacketed perspex cylinder. The test can take from 3 to 24 hours to complete. The principal advantage of this technique is that only small sample sizes are required. As a result, this technique has been used extensively in the laboratory to supply $R(\phi)$ data for freshly flocculated suspensions, where only limited suspension is available (Scales et al. 2001). The practical limitation of this test is that it can only characterise $R(\phi)$ at a single solids concentration.

The method involves steady state gravity permeation of liquor through a packed bed of suspension. The liquor above the suspension provides the pressure head, while the hindered settling function, $R(\phi)$ is calculated from the permeation rate, the pressure head, the suspension bed height and the average solids volume fraction of the suspension bed.

6.4.2 THEORY

According to Green (Green 1997), many workers have determined permeability, $k(\phi)$, by constant pressure filtration through a packed bed of known height and concentration. Green suggests that the hindered settling function can be derived from the same experiment using Darcy’s law with a pressure drop $\Delta P$ across a packed bed of height, $H$, solids volume fraction, $\phi$
and liquor viscosity, \( \eta \). The volumetric rate of filtration per filter cross sectional area, \( \frac{dV}{dt} \) is shown below with the addition of a membrane resistance \( \alpha \).

\[
\frac{dV}{dt} = \frac{\Delta P}{\eta \left( \frac{H}{k(\phi)} + \alpha \right)}
\]

(6.27)

The permeability, \( k(\phi) \), is related to the hindered settling function, \( R(\phi) \), by

\[
k(\phi) = \frac{\eta}{R(\phi)} \left( 1 - \frac{\phi}{f} \right)
\]

(6.28)

Substitution and transformation gives

\[
R(\phi) = \left( \frac{\Delta P}{dV/dt} - \eta \alpha \right) \frac{1}{H} \frac{1 - \phi}{\phi}
\]

(6.29)

In cases where the membrane resistance is insignificant, the hindered settling function is given by

\[
R(\phi) = \left( \frac{\Delta P}{dV/dt} \right) \frac{1}{H} \frac{1 - \phi}{\phi}
\]

(6.30)

Thus, by setting up a bed of known solids concentration, \( \phi \), and known bed thickness, \( H \), \( R(\phi) \) may be measured by applying a constant known head of fluid pressure across the bed and measuring the rate of liquid flow. The true value of the pressure drop, \( \Delta P \), is difficult to determine, but is expected to lie in the range,

\[
\rho_{liq}g h_{liq} < \Delta P < \rho_{liq}g (h_{liq} + (1 - \phi_{av})H),
\]

(6.31)

where \( h_{liq} \) is the height of liquor above the top of the suspension bed of average solids volume fraction, \( \phi_{av} \). For the experimental results that follow, the pressure drop has been assumed to be equal to the liquor pressure head at the top of the suspension according to Equation 6.32, but assuming it is equal this liquor head plus the liquor head in the suspension bed according to Equation 6.33 is also reasonably justifiable.

\[
\Delta P = \rho_{liq}g h_{liq}.
\]

(6.32)

\[
\Delta P = \rho_{liq}g (h_{liq} + (1 - \phi_{av})H)
\]

(6.33)

For a system with no suspension, the membrane resistance can be determined using
\[
\frac{dV}{dt} = \frac{\Delta P}{\eta \alpha}
\]  \hspace{1cm} (6.34)

which transforms to give

\[
\alpha = \frac{\Delta P}{\eta \frac{dV}{dt}}
\]  \hspace{1cm} (6.35)

### 6.4.3 EXPERIMENTAL METHOD

#### 6.4.3.1 Apparatus

The gravity permeation apparatus employed for transient batch settling tests had a transparent perspex cylinder as shown in Figure 6.2. The cylinder height was 110 mm with an internal diameter of 40 mm. There was a permeable steel sintered disc supporting the membrane. The membrane used was a Millipore-Durapore membrane type DVPP with 0.65 \(\mu\)m pore size. It was important to control temperature reasonably well in these experiments so all permeate was kept in a thermostated water bath. The liquid height was kept at a constant level using two peristaltic pumps to add and remove permeate from the top of the cylinder. By removing liquid at a faster rate than it was replenished the liquid level was always maintained just under the point of liquid removal. In this way, by adjusting the vertical location of the suction tube, the level of liquid, and hence the pressure drop, was held constant at any desired level. An electronic balance underneath the cylinder logged the permeate mass passing through the membrane into a beaker. The mass was logged as a function of time using a LabVIEW\textsuperscript{®} application and saved as a Microsoft Excel spreadsheet, from which an average rate of permeation, \(dV/dt\), was be determined.
6.4.3.2 Sample Preparation

The most critical aspect of the permeation test was to obtain an even sediment with minimal inhomogeneities such as air gaps. This would have been difficult to achieve if the loading solids concentration was such that the shear yield stress was too high. Essentially, the suspension needed to be able to flow into the cylinder and not leave any air gaps.

Conversely, a common problem with running tests on freshly flocculated material or a material that had a low solids concentration was that settling and densification of the sediment occurred over the time frame of the test. The simplest solution to this problem was to wait until the sediment level and permeation rate had stabilised before calculating the permeation rate. Obviously, the settled bed height and final average solids concentration was then used in the calculation of $R(\phi_{av})$.

There were no notable differences in the sample treatment required for freshly flocculated suspensions relative to that of unflocculated and process based suspensions, with the exception that flocculated suspension required more care to minimise shear degradation.

6.4.3.3 Test Procedure

i) Measure the initial solids concentration, $\phi_0$, of the suspension sample to be analysed.

ii) Ensure that the permeate collection beaker is on the measuring balance under the permeation test rig.
iii) Start the LabVIEW® gravity permeation control program and enter the relevant test information when prompted.

iv) When prompted, wet the membrane and load it onto the sintered disk before bolting the cylinder in place. It is important to avoid air bubbles under the membrane, as these may affect permeation rates, so it is a good idea to degass the sintered disk in advance of the test.

v) Load the suspension into the cylinder with a bed height of 1-3 cm, tapping the cylinder until the bed is flat and level.

vi) Calculate (by difference) and record the mass of suspension added to the cylinder, \( m_s \).

vii) Gently decant liquor onto the top of the sediment up to the desired level, which is usually about 1 cm below the top of the cylinder.

viii) Set the liquor supply and removal hoses in place, each with one end positioned inside the cylinder, about 1 cm below the top, then passing through peristaltic pumps and then into a beaker of liquor that is representative of that in the suspension. It is desirable that the liquor be held in a temperature controlled water bath. Adjust the pumping rates so that the liquor level is held constant.

ix) The program will then log the data and display transient \( dV/dt \) data as it is logged, until the rate of permeation is stable.

x) Once a stable rate of permeation has been obtained, record the liquid level in the permeation cylinder, \((h_{\text{liq}} + H)\), and final suspension bed height, \( H \).

xi) Calculate the final average bed solids volume fraction, \( \phi_{av} \) using

\[
\phi_{av} = \frac{\phi_0 H_0}{H}.
\]

where the initial suspension height, \( H_0 \) is calculated using

\[
H_0 = \frac{4 m_s}{\left(\rho_{\text{sol}} \phi_0 + \rho_{\text{liq}} (1 - \phi_0)\right) \pi d^2}.
\]

Where \( m_s \) is the initial mass of suspension, \( \rho_{\text{sol}} \) is the solid density, \( \rho_{\text{liq}} \) is the liquid density and \( d \) is the internal diameter of the cylinder.

xii) Calculate the pressure drop across the suspension bed using either Equation 6.32 or 6.33.

xiii) Calculate the hindered settling function using Equation 6.29, given a membrane resistance of \( 6.66 \times 10^9 \text{ m}^{-1} \).
6.4.4 RESULTS

Initially, the membrane resistance needed to be determined. The cylinder was loaded with milli-Q filtered water, the liquor height maintained by the pumps and the filtration rate measured over an hour or more. The experiment was repeated a number of times to test reproducibility. During some tests, the filtration rate decreased over time and inspection revealed air bubbles entrained underneath the membrane as shown in Figure 6.3. As a result, the steel sintered disk, which supported the membrane, was de-aerated before filtering for all subsequent tests. The membrane resistance for seven separate tests was calculated using Equation 6.35, giving a membrane resistance of \((6.66 \pm 0.46) \times 10^9\) m\(^{-1}\) with 95% confidence.

A flocculated haematite suspension in 1 M NaOH was tested in triplicate. Each suspension was loaded into the cylinder at an initial height of around 25 mm and an initial solids volume fraction of around 0.20. The flocculated suspensions generally always settled at the start of the test, but the bed height remained constant after about an hour as shown by the stabilisation in the liquor mass flowrate, \(\frac{dm}{dt}\) versus \(t\), data in Figure 6.4. Once the bed height had stabilised, the bed height was recorded and the permeation rate measured over a couple of hours. The hindered settling function could then be calculated from Equation 6.29 assuming Equation 6.32 represented the pressure drop and assuming a measured evaporation rate of \(6.7 \times 10^{-8}\) kg s\(^{-1}\). The results for the three tests are shown in Figure 6.5 with calculated uncertainties. The measured hindered settling function appears to be reasonably reproducible giving results within 7%. The only significant uncertainty in the calculation procedure involved the real value of the pressure drop over the bed.

![Figure 6.3: Wetted membranes (left) not degassed, with entrained air that can slow permeation when subjected to low applied pressures, (right) degassed, with no entrained air.](image-url)
Figure 6.4: Example of gravity permeation test mass flowrate $dm/dt$ data for a haematite suspension.

Figure 6.5: Hindered Settling function determined by gravity permeation tests, using Equation 6.40 in calculation of pressure drop, $\Delta P$. 
6.4.5 APPLICATION SUMMARY

Gravity permeation has been applied to the characterisation of flocculated haematite suspensions and red mud suspensions at intermediate solids concentrations by Hulston (Scales et al. 2001). Other workers at The University of Melbourne and Rio Tinto have also found this technique useful when there are sample volume limitations.

6.5 STEPPED PRESSURE FILTRATION

Parameters Measured: $P_y(\phi)$ & $R(\phi)$ (& $D(\phi)$)
Solids Concentration Range: $0.25 < \phi < 0.45$ for red mud.
Pressure Range: 5-300 kPa
Temperature Range: 10-90°C (Thermally jacketed filtration cylinder).
Time Required: 1-8 hours for red mud
Advantages: Robust, accurate, transportable, rapid characterisation, requires only small samples.
Limitations: Mechanically complex, no data for very low solids.

Table 6.4: Stepped pressure filtration test method overview.

6.5.1 INTRODUCTION

Using stepped pressure filtration, compressive yield stress, $P_y(\phi)$, and hindered settling function, $R(\phi)$, data are determined as a function of the solids volume fraction, $\phi$, for flocculated suspensions over a number of hours. Traditionally, characterisation of the compressibility and permeability of flocculated suspensions is a laborious time consuming experimental process that often takes days to perform. Standard constant pressure filtration requires five or more individual filtration tests to characterise a suspension across a range of solids concentrations. A new stepped pressure filtration technique (de Kretser et al. 2001b; Usher et al. 2001) has thus been developed. The new method uses only one stepped pressure compressibility filtration test and one truncated stepped pressure permeability filtration test to determine $P_y(\phi)$ and $R(\phi)$ at five or more solids volume fractions and substantially decreases the time required for sample characterisation. Alternatively, the data from these same filtration tests can be used to determine the solids diffusivity, $D(\phi)$, at these same solids concentrations.

The stepped pressure filtration test described here can characterise suspensions over a wide range of pressures from 5 kPa to 300 kPa, using only two filtration tests. The base plate and filtration cylinder are water jacketed allowing thermal control from 10-90°C. However, with different equipment, there is the potential to build a filtration test rig that operates at lower pressures, at higher pressures or at higher temperatures. Characterisation usually takes of the order of 1-8 hours depending on the permeability of the suspension. Computer control and semi-automation of the technique makes the method robust and accurate. The reduction in the
number of tests involved in stepped pressure filtration has the implication that only a small amount of sample is required. The other positive aspect of the filtration test rig is that it can be disassembled for transportation, enabling use on site. The limitation of this technique is that it is mechanically complex, the equipment has been expensive to develop and the results are only produced for high solids concentrations.

Traditional pressure filtration experiments involve the pressure filtration of a particulate suspension in which the time of filtration, \( t \), is monitored as a function of piston height from which the specific volume of filtrate, \( V \), is determined. \( P_y(\phi) \) is determined from the equilibrium solids volume fraction. The gradient \( d(t/V)/dV \) was previously used to determine \( R(\phi) \), using fundamental dewatering theory (Landman et al. 1999). To eliminate the influence of a stepped pressure on calculated \( R(\phi) \) values, a modification to the analysis of experimental results has been developed to use the gradient \( dt/dV^2 \) instead.

In the following sections, the assumption of a uniform cake porosity has been modelled to enable a simple physical understanding of why stepped pressure filtration is valid. Then, the stepped pressure filtration test method presented in detail and the technique is validated by presenting results for a zirconia suspension comparing both single and stepped pressure filtration.

### 6.5.2 THEORY

#### 6.5.2.1 Pressure Filtration Tests

For modelling purposes, filtration apparatus is considered to include a cylinder filled with a flocculated suspension of particles at an initial volume fraction \( \phi_0 \). A pressure is applied to the top of the suspension via a downward moving piston with a pressure transducer mounted flush with the piston face. The pressure at the piston face, \( P \), is used in a feedback control loop with a computer and pneumatic cylinder to maintain a constant applied pressure. A permeable membrane at the base of the cylinder allows liquid to pass through but retains the solids, gradually building up a filter cake. Filtration progress is measured over time, \( t \), as a function of specific filtrate volume, \( V \), which is calculated from measured piston displacement using a linear encoder.

*Single Pressure Filtration*

Standard pressure filtration involves the application of a single constant pressure and enables determination of one compressibility and one permeability value for each test. A combination of individual tests can be completed to establish a compressibility and permeability profile for a
sample. In a standard single pressure filtration test, a constant pressure, $P$, is applied at the piston face until the filter cake stops compressing. The compressive yield stress, $P_y(\phi)$, at the equilibrium solids volume fraction, $\phi$, is equal to the applied pressure, $P$. The gradient $\frac{d(t/V)}{dV}$ is relatively constant during the middle stages of a filtration test. A single value of the hindered settling function, $R(\phi)$, is calculated for each gradient as demonstrated by Green (Green et al. 1998). Landman and White (Landman and White 1997) have shown that this is sufficient to fully characterise, predict and optimise a filtration process. This single pressure method is adequate, but requires significant time with five or more individual filtration tests required to effectively characterise a sample.

Stepped Pressure Filtration

To reduce characterisation time, a new stepped pressure filtration technique has been developed. Stepped pressure filtration provides the option to fully characterise a sample from only one compressibility filtration test and one truncated permeability filtration test. The first test determines the compressibility at a range of pressures and another truncated permeability test determines the hindered settling function over the same range of pressures. The step technique makes efficient use of time in terms of equipment use and labour.

In the compressibility test, the lowest pressure is applied until the filter cake stops exuding liquid and then the pressure is stepped and so on. The data obtained give the compressive yield stress $P_y(\phi)$ for a number of solids volume fractions. Time is saved in that the cake formation time is avoided for all but the initial pressure.

In the permeability test, the first pressure is applied until a specified gradient is stable, then the pressure is incremented. The hindered settling function, $R(\phi)$, is traditionally calculated from the gradient of a $t/V$ versus $V$ plot for a single applied pressure. It will be shown that this method is not mathematically justified when a number of pressures are applied in the one test. Experimentally, the $t/V$ versus $V$ method produces erroneous gradients and subsequently incorrect $R(\phi)$ values when the pressure is stepped. It will be shown that the problem becomes progressively worse with the number of pressure steps applied. Thus, the $t/V$ versus $V$ method is not applicable to the stepped pressure technique and another calculation method using $t$ versus $V^2$ is proposed.

6.5.2.2 Mathematical Modelling

The experimental observation that a plot of $t/V$ versus $V$ is approximately linear, in the initial stages of filtration, suggests that the behaviour is similar in form to Darcy's law based on the Hagen-Poiseuille equation (Dahlstrohm et al. 1997a) given by
\[
\frac{dV}{dt} = \frac{P}{\eta(\alpha_m w V + r)}
\]  

(6.38)

where \( \eta \) is the filtrate viscosity, \( \alpha_m \) is the specific mass filter cake resistance, \( w \) is the weight of cake solids per unit volume of filtrate and \( r \) is the membrane resistance.

A modified differential equation has been developed to model the filtration behaviour of a compressible networked suspension. The specific mass filter cake resistance has been transformed to a specific volume filter cake resistance, \( A \), which is a function of the applied pressure, \( P \). The specific filtrate volume, \( V \), has been related to a physical filter cake height, \( B \), and membrane resistance redefined as \( \alpha \) to minimise confusion with the hindered settling factor. This gives

\[
\frac{dV}{dt} = \frac{P}{\eta(AB + \alpha)}
\]  

(6.39)

Neglecting membrane resistance, Equation 6.46 becomes

\[
\frac{dV}{dt} = \frac{P}{\eta AB}
\]  

(6.40)

The application of these concepts to the modelling of stepped pressure filtration involves three stages; initial filter cake formation, filter cake compaction and subsequent filter cake growth as depicted in Figure 6.6. Initial filter cake formation involves filter cake development upwards from the base of the suspension by compression of the initial suspension. After the pressure is stepped, filter cake compaction involves the development of a new filter cake at higher solids volume fraction developing upwards by compression of the filter cake already present. Once the previous filter cake has been completely compressed to the new filter cake solids volume fraction, filter cake growth involves the new filter cake developing upwards by compression of the initial suspension.

\emph{Initial Filter Cake Formation}

A pressure, \( P_1 \), is applied to a suspension at solids volume fraction \( \phi_0 \) and the suspension is assumed to compress to form a filter cake with average solids volume fraction \( \phi_1 \), from the base of the cylinder. This filter cake, with constant average solids volume fraction \( \phi_1 \), grows as the pressure \( P_1 \) is maintained and the filtrate passes through the membrane on the bottom plate of the cylinder. Given the filter cake resistance \( A_1 \), corresponding to the application of pressure \( P_1 \), Equation 6.40 becomes
\[
\frac{dV}{dt} = \frac{P_1}{\eta A B}
\]  
(6.41)

A solids mass balance relating the \( \phi_1 \) filter cake thickness, \( B \), to the specific filtrate volume, \( V \), gives

\[
B = \frac{\phi_0}{\phi_1 - \phi_0} V
\]  
(6.42)

Thus, substituting Equation 6.42 into 6.41 and then integrating from time \( t_0 \) to \( t \), and specific filtrate volume \( V_0 \) to \( V \), gives

\[
t - t_0 = \frac{\eta A_1}{2P_1} \phi_0 \left( \frac{V^2 - V_0^2}{\phi_1 - \phi_0} \right)
\]  
(6.43)

Assuming \((t_0, V_0) = (0,0)\) corresponds to the start of the filtration test gives

\[
t = \frac{\eta A_1}{2P_1} \phi_0 V^2
\]  
(6.44)

Consequently, it is predicted that during filter cake formation

\[
\frac{d(t/V)}{dV} = \frac{dt}{dV^2} = \frac{\eta A_1}{2P_1} \phi_0 \left( \frac{1}{\phi_1 - \phi_0} \right)
\]  
(6.45)

If however, \((t_0, V_0)\) does not correspond to the start of a filtration test, then \( dt/dV^2 \) remains unchanged but

\[
\frac{d(t/V)}{dV} = \frac{\eta A_1}{2P_1} \phi_0 \left( \frac{1}{\phi_1 - \phi_0} \right) \left( 1 + \frac{V_0^2}{V^2} \right) - \frac{t_0}{V^2}
\]  
(6.46)

**Filter Cake Compaction**

At time \( t = t_1 \) and specific filtrate volume \( V = V_1 \), the \( \phi_1 \) filter cake thickness \( B_1 \), derived from Equation 6.42, is given by

\[
B_1 = \frac{\phi_0}{\phi_1 - \phi_0} V_1
\]  
(6.47)

At this time, the pressure is stepped from \( P_1 \) to \( P_2 \) and filter cake compaction begins. A new filter cake with thickness \( B' \) and average solids volume fraction \( \phi_2 \) begins to form. The filter cake develops from the base of the cylinder by compression of the \( \phi_1 \) filter cake. With the
application of pressure $P_2$ and the corresponding filter cake resistance $A_2$, Equation 6.40 becomes

$$\frac{dV}{dt} = \frac{P_2}{\eta A_2 B'}$$  \hspace{1cm} (6.48)

A solids mass balance relating the $\phi_2$ filter cake thickness $B'$ to the specific filtrate volume $V$ gives

$$B' = \frac{\phi_1}{\phi_2 - \phi_1} (V - V_1)$$  \hspace{1cm} (6.49)

Thus, substituting Equation 6.49 into 6.48 and then integrating from time $t_1$ to $t$ and specific filtrate volume $V_1$ to $V$, gives

$$t = \frac{\eta A_2}{2P_2} \frac{\phi_1}{\phi_2 - \phi_1} (V - V_1)^2 + t_1$$  \hspace{1cm} (6.50)

Thus it is predicted that during filter cake compaction

$$\frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2} \frac{\phi_1}{\phi_2 - \phi_1} \left(1 - \frac{V_1^2}{V^2}\right) - \frac{t_1}{V^2}$$  \hspace{1cm} (6.51)

and

$$\frac{dt}{dV^2} = \frac{\eta A_2}{2P_2} \frac{\phi_1}{\phi_2 - \phi_1} \left(1 - \frac{V_1}{V}\right).$$  \hspace{1cm} (6.52)

**Filter Cake Growth**

Soon, at time $t = (t_1 + t_2)$ and specific filtrate volume $V = (V_1 + V_2)$, the $\phi_2$ filter cake reaches the top of the $\phi_1$ filter cake. At this point, Equation 6.50 simplifies to

$$t_2 = \frac{\eta A_2}{2P_2} \frac{\phi_1}{\phi_2 - \phi_1} V_2^2$$  \hspace{1cm} (6.53)

Also at this time, the $\phi_2$ filter cake thickness $B_2$, is given by

$$B_2 = \frac{\phi_1}{\phi_2 - \phi_1} V_2$$  \hspace{1cm} (6.54)

Equating the amount of solids in each filter cake at $V = V_1$ and $V = (V_1 + V_2)$ yields
\[ \phi_2 B_2 = \phi_1 B_1 \quad (6.55) \]

Substituting Equation 6.47 for \( B_1 \), Equation 6.54 for \( B_2 \) and then rearranging gives

\[ V_2 = \left( \frac{\phi_0}{\phi_2} \right) \left( \frac{\phi_2 - \phi_1}{\phi_1 - \phi_0} \right) V_1 \quad (6.56) \]

Equations 6.53-6.56 describe the material balance to calculate \( V_2 \) in terms of \( V_1 \) for use in the filter cake growth equations (Equations 6.57-6.61).

In the filter cake growth stage, the application of pressure \( P_2 \) continues and the \( \phi_2 \) filter cake height, \( B'' \) continues to grow by compression of the original suspension at solids volume fraction \( \phi_0 \). Thus, in the filter cake growth stage, Equation 6.40 becomes

\[ \frac{dV}{dt} = \frac{P_2}{\eta A_2 B''} \quad (6.57) \]

A solids mass balance relating the \( \phi_2 \) filter cake thickness, \( B'' \), to the specific filtrate volume, \( V \), during filter cake growth gives

\[ B'' = \frac{\phi_0}{\phi_2 - \phi_0} V \quad (6.58) \]

Thus, substituting Equation 6.58 into 6.57 and then integrating from time \( (t_1 + t_2) \) to \( t \) and specific filtrate volume \( (V_1 + V_2) \) to \( V \) gives

\[ t = \frac{\eta A_2}{2P_2} \frac{\phi_0}{\phi_2 - \phi_0} \left( V^2 - \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} V_1^2 \right) + t_1 \quad (6.59) \]

Consequently it is predicted that during filter cake growth

\[ \frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2} \frac{\phi_0}{\phi_2 - \phi_0} \left( 1 + \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} V^2 \right) - \frac{t_1^2}{V^2} \quad (6.60) \]

and

\[ \frac{dt}{dV^2} = \frac{\eta A_2}{2P_2} \frac{\phi_0}{\phi_2 - \phi_0} \quad (6.61) \]

The gradient \( dt/dV^2 \) in the filter cake growth stage given by Equation 6.61 is of the same form as that in the initial filter cake formation stage given by Equation 6.45. Therefore, the gradient
\( \frac{dt}{dV^2} \) in the filter cake growth stage could be used in the calculation of permeability in stepped pressure filtration.

**Membrane Resistance**

Membrane resistance contributes an error to the modelled filtration behaviour. The filtration behaviour with membrane resistance \( \alpha \), is modelled using Equation 6.39 with applied pressure \( P_1 \) and corresponding specific filter cake resistance \( A_1 \) to give

\[
\frac{dV}{dt} = \frac{P_1}{\eta(A,B + \alpha)} \tag{6.62}
\]

As calculated for the filter cake formation stage, integrating from time \( t_0 \) to \( t \), and specific filtrate volume \( V_0 \) to \( V \), predicts \( t \) versus \( V \) behaviour of the form

\[
t - t_0 = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} \left( V^2 - V_0^2 \right) + \frac{\eta \alpha}{P} \left( V - V_0 \right) \tag{6.63}
\]

The gradients are then calculated to be

\[
\frac{d(t/V)}{dV} = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} \left( 1 + \frac{V_0^2}{V^2} \right) + \frac{\eta \alpha}{2P_1} V - \frac{t_0}{V^2} \tag{6.64}
\]

and

\[
\frac{dt}{dV^2} = \frac{\eta A_1}{2P_1} \frac{\phi_0}{\phi_1 - \phi_0} + \frac{\eta \alpha}{2P_1} \frac{1}{V} \tag{6.65}
\]

The error term \((\eta \alpha/2P_1V)\) in Equation 6.65 diminishes with increasing extent of filtration due to the inverse \( V \) dependence and becomes insignificant at high extents of filtration.

**Modelling Summary**

The derived filtration equations (Equations 6.44, 6.50 and 6.59), are summarised in Table 6.5. Inspection of the filtration equations for the initial filter cake formation and new filter cake growth reveals that they are identical in form with the exception of a constant. This constant can be explained as a time correction. When the pressure is increased during a filtration test, the filter cake with average solids volume fraction \( \phi_1 \), that has already formed is gradually converted to a filter cake with average solids volume fraction \( \phi_2 \). This filter cake took time \( t \) to form, but would have taken a different amount of time if the second pressure had been applied from the beginning. Thus, modelling predicts that the normal \( t/V \) versus \( V \) analysis to determine
the hindered settling function would be in error for subsequent pressures due to the incorrect definition of zero time.

The predicted gradients $\frac{dt}{dV}$ for each filtration equation are also tabulated in Table 6.5. The predicted $1/V^2$ dependence of $\frac{dt}{dV}$ in the filter cake growth stage shows how the gradients in stepped pressure filtration differ from those in single pressure filtration. For single pressure filtration (i.e., the filter cake formation stage), Equation 6.45 predicts that $dt/dV^2$ is equal to $d(t/V)/dV$. Fortunately, in stepped pressure filtration, the predicted value for $dt/dV^2$ is also constant in the filter cake growth stage, after filter cake compaction. Use of the gradient, $dt/dV^2$, eliminates the influence of the time correction. This suggests that the gradient, $dt/dV^2$, be used in the calculation of $R(\phi)$ from stepped pressure filtration.

After the pressure is stepped in a permeability test, the $V$ at which compaction finishes, can be calculated from Equation 6.56. The calculation could use approximate values of $\phi_1$ and $\phi_2$ determined from the equilibrium solids volume fractions of compressibility filtration tests at pressures $P_1$ and $P_2$. It should be noted however, that the equilibrium solids volume fraction will be slightly higher than the average filter cake solids volume fractions, but not to the extent that it would cause too significant an error. Alternatively, this condition is easily satisfied without such a calculation by waiting until $dt/dV^2$ is stable. To detect stability, the variation in the gradient is monitored until the rate of change is below a certain level. In principle, this is the only control measure required because the gradient will not be stable during filter cake compaction.

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**Figure 6.6:** Schematic of solids concentration zones using a simplified model stepped pressure filtration showing a) initial conditions, b) initial filter cake formation, c) filter cake compaction and d) filter cake growth.
Initial Filter Cake Formation

\[ P = P_1; \ 0 \leq V \leq V_1 \]

\[ t = \frac{\eta A_1}{2P_1 \phi_1 - \phi_0} V^2 \]  
\[ \frac{d(t/V)}{dV} = \frac{dt}{dV^2} = \frac{\eta A_1}{2P_1 \phi_1 - \phi_0} V \]  
\[ \text{With membrane resistance:} \]

\[ \frac{dt}{dV^2} = \frac{\eta A_1}{2P_1 \phi_1 - \phi_0} + \frac{\eta \alpha}{2P_1 V} \]

Filter Cake Compaction

\[ P = P_2; \ V_1 < V \leq V_1 \frac{\phi_1(\phi_2 - \phi_0)}{\phi_1(\phi_1 - \phi_0)} \]

\[ t = \frac{\eta A_2}{2P_2 \phi_2 - \phi_1} (V - V_1)^2 + t_1 \]  
\[ \frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2 \phi_2 - \phi_1} \left(1 - \frac{V_1^2}{V^2}\right) - \frac{t_1}{V^2} \]  
\[ \frac{dt}{dV^2} = \frac{\eta A_2}{2P_2 \phi_2 - \phi_1} \left(1 - \frac{V_1}{V}\right) \]

Filter Cake Growth

\[ P = P_2; \ V_1 \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} < V \]

\[ t = \frac{\eta A_2}{2P_2 \phi_2 - \phi_0} \left(V^2 - \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} V_1^2\right) + t_1 \]  
\[ \frac{d(t/V)}{dV} = \frac{\eta A_2}{2P_2 \phi_2 - \phi_0} \left(1 + \frac{\phi_1(\phi_2 - \phi_0)}{\phi_2(\phi_1 - \phi_0)} V_1^2\right) - \frac{t_1^2}{V^2} \]  
\[ \frac{dt}{dV^2} = \frac{\eta A_2}{2P_2 \phi_2 - \phi_0} \]

Table 6.5: Key stepped pressure filtration equations derived from simplified modelling.

6.5.3 EXPERIMENTAL METHOD

6.5.3.1 Apparatus

The piston driven filtration test rig, shown in the schematic diagram and photograph in Figure 6.7, is a laboratory scale computer controlled filter press that was used for the rapid characterisation of the dewaterability of suspensions (de Kretser et al., 2001). The instrument differed from conventional filter presses in that times for full characterisation of \( R(\phi) \) and \( P_y(\phi) \) are reduced due to the employment of the Stepped Pressure Filtration Technique. The key components of the piston driven pressure filtration test rig included:

- a water jacketed filtration cylinder and base plate
• a permeable sintered disk
• a pneumatic cylinder
• a pressure controller
• a linear encoder
• a pressure transducer
• a 100 psi air supply
• an electronic data acquisition system
• a personal computer

The filtration cylinder was used to contain the suspension sample between an air driven piston above and a permeable membrane below. The filtration cylinder and base assembly was adapted to incorporate a water jacket that facilitated temperature controlled testing. The filtration rig employed a pneumatic cylinder to apply pressure to the suspension sample contained in the filtration cylinder, forcing liquid out through a filter membrane at the cylinder base, which was supported by a permeable sintered metal disk. The pressure of filtration was monitored using a pressure transducer mounted flush in the piston face and the rate of filtration was monitored through the rate of movement of the piston. Displacement in the piston was determined using a linear encoder with a spatial resolution of 10 $\mu$m. Computer control of the pressure stepping required for Stepped Pressure tests was facilitated by a Bronkhorst EL Press pressure controller, which controlled the pressure in the pneumatic cylinder. The pressure control system allowed pressures ranging from 5 kPa to 300 kPa to be tested. The pressure transducer was reliable up to temperatures of 90°C, but there is potential to increase this upper limit to 110°C with a different pressure transducer. Data from the pressure transducer and linear encoder was logged by the control program on the PC and analysed to provide run-time information on the progress of experiments and automatic control. A final data output file (in the form of an Excel spreadsheet) was generated at the conclusion of each test, providing the necessary information for use in the subsequent determination of the dewatering parameters.

When the cylinder diameter is small, the influence of wall friction can be significant, but the work of Green (Green 1997) suggests that a cylinder diameter of 26.5 mm should be sufficient to minimise this effect. The filtration test rig described here had a cylinder diameter of 40 mm and wall friction was ignored. The experimental set up also had a significant advantage relative to other systems in that the controlled pressure was the pressure measured at the piston face as measured by a pressure transducer in contact with the suspension, rather than an externally applied pressure assumed to be transmitted to the suspension.
Figure 6.7: Schematic and picture of the piston driven pressure filtration test rig.

6.5.3.2 Sample Preparation

*Underflow Suspensions*

Thickener underflow suspensions are generally subjected to significant amounts of shear within the thickener itself, thus it was not necessary to be too careful about how these samples were treated in the lab. For Bayer processes samples, the only requirements were that the samples be
kept at process temperatures and tests conducted within a reasonable time so that precipitation from the liquor did not compromise the validity of the results produced. When homogenisation was necessary, light shaking of the container and mixing with a spatula by hand was considered adequate.

**Flocculated Suspensions**

When testing freshly flocculated samples, produced either in a baffle reactor or a plunger type test, it was difficult to obtain a homogenous sample for measuring the initial solids concentration. The most important consideration was that the permeable floc structure was not destroyed during any mixing or homogenisation processes that were employed. As a result, mixing was very gentle, but some degree of shear degradation could not be avoided.

**Initial Solids Concentration**

Generally for characterisation of flocculated suspensions, samples needed to be prepared at a solids concentration that was in the region of the gel point. It was not advantageous to load suspensions at solids concentrations significantly lower than this because settling during the filtration test would have compromised the accuracy of calculated permeabilities. This would have also required more liquid to be filtered, thus increasing the total characterisation time. In addition, it was also not advantageous to load suspensions at solids concentrations that were significantly higher than the gel point. In this case the shear yield stress could have been too high, with the suspension exhibiting paste like behaviour that resisted flow, resulting in air gaps and compromising the test.

**Initial Suspension Height**

Determining the amount of sample to add, in a pressure filtration test, was a subjective art that was generally optimised through experience. The primary aims were to minimise characterisation time for the maximum number of pressures, while maintaining accuracy in the results.

For compressibility tests, the time of filtration is related to the square of the initial suspension height which therefore needed to be minimised as much as was practical. However, if the final suspension height was too low, then the accuracy of the test would have been compromised. Ideally, the initial suspension height was selected so that it produced a final suspension height of 6-12 mm. A rule of thumb for red mud underflow samples was an initial suspension height of 25-35 mm.

For permeability tests, the aim was to enable a large number of pressures to be tested before the filter cake touched the piston face. As a result, the amount of suspension added was generally
as much as the filtration cylinder could practically hold, while not creating too large a final filter cake that would have taken an excessively long time to filter at the highest test pressure. The filtration rig used in this work allowed initial suspension heights as high as 50-70 mm.

6.5.3.3 Test Procedure

The Stepped Pressure Filtration technique enabled dewaterability characterisation from a compressibility test at five or more pressures over about 3 hours and a permeability test at the same pressures over about 1 hour. Operation of the filtration test rig was via a semi-automated computer controlled LABVIEW application that guides the user through almost every step. A detailed procedure would be excessive, so a brief overview of the experimental test procedure is given below:

(i) Select the test temperature and set up the temperature control equipment.
(ii) Select the test pressures (For example use 5, 10, 20, 40, 80, 150, 230 and 300 kPa).

Compressibility Test

(iii) Allow the suspension sample to be tested, the filtration cylinder, base plate and pressure transducer to equilibrate at the test temperature.
(iv) Measure the initial solids concentration, \( \phi_0 \) (measured), of a suspension sample that is representative of the suspension to be characterised.
(v) Start the LABVIEW control program and start a “Compressibility Test”.
(vi) Enter all of the relevant test run details when prompted by the test control program, including the test pressures, estimates of the liquor density, solids density and the initial solids volume fraction of the sample to be tested. Note that the output results can be adjusted to account for the true values later.
(vii) When prompted, zero the linear encoder.
(viii) When prompted ensure that the pressure transducer is at thermal equilibrium at the test temperature and zero the pressure transducer. Note that the best indication of temperature equilibration can be observed as the reading from the pressure transducer not changing over a period of 5 minutes.
(ix) When prompted, load the filter membrane and bolt down the filtration cylinder.
(x) When prompted, load the filtration cell with an appropriate amount of sample and then position the piston in the cell, excluding as much air as is possible through the bleed line and then allow the program to record the initial suspension height, \( h_0 \).
(xi) Leave the sample for 5-15 minutes to reach thermal equilibrium.
(xii) When thermal equilibration is achieved, prompt the computer to commence the test. The program supplies air to the pneumatic cylinder to control the pressure logged by the
pressure transducer at the lowest set pressure. Filtration starts and as a filter cake builds up from the membrane, the rate of efflux of filtrate follows a $t$ versus $V^2$ relationship and thus $t$ will be linear in $V^2$. Eventually the filtration will slow as the piston impacts on the rising filter cake and $t$ is no longer linear in $V^2$. When an equilibrium criterion is met, the program determines that filtration has stopped, records the equilibrium suspension height for that pressure, $h_{\infty}$, and then steps to the next highest pressure and so on until filtration is complete at the final pressure. At the conclusion of the final pressure air is vented from the pneumatic cylinder, the test data is output to a spreadsheet file and the program stops. The spreadsheet file containing suspension height data as a function of pressure can subsequently be used with solids volume fraction data to calculate compressive yield stress data.

(xiii) When the test is complete, remove the compressed filter cake from the filtration cylinder as soon as possible and measure the solids concentration, $f_{\infty}$(measured). Speed is critical to minimise evaporation at high test temperatures. On withdrawal of the piston, immediately place the cake in a sealed container (25ml containers with a 40 mm internal diameter works well) and transfer to a balance for weighing – this prevents evaporation during weighing. After weighing, remove the lid and place it on the underside of the container, then transfer the container and cake to an oven for drying and later re-weighing.

Permeability Test

(xiv) Allow the suspension sample to be tested, the filtration cylinder, base plate and pressure transducer to equilibrate at the test temperature.

(xv) Measure the initial solids concentration, $\phi_0$(measured), of a suspension sample that is representative of the suspension to be characterised.

(xvi) Start the LABVIEW control program and start a “Permeability Test”.

(xvii) All sample preparation and loading considerations are basically the same as in Steps vi)-xi) for the compressibility test, with one exception. The pressures selected for the permeability test must be the same as for the compressibility test, but with the addition of an extra dummy pressure to allow compression of the final filter cake. This pressure should be set at the highest pressure used in the compressibility test (generally 300 kPa) to enable relevant comparison between the tests and verify that the tests are reliable.

(xviii) When thermal equilibration is achieved, prompt the computer to commence the test. The program supplies air to the pneumatic cylinder to control the pressure logged by the pressure transducer at the lowest set pressure. Filtration starts and as a filter cake builds up from the membrane, the program monitors the run data until the gradient $dt/dV^2$
reaches a stable value as determined by a linearity criterion (which can be adjusted with a tolerance, depending on the nature of the material or the pressure of operation). The gradient, \( \frac{dh}{dV^2} \), is then logged and the pressure stepped to the next highest pressure. A short non-linear region of filtration occurs initially and then a new linear region is attained with a new slope. When the linearity criterion is satisfied, stepping to the next pressure occurs and so on until the final dummy pressure is reached. For the final dummy pressure, the linearity criterion is switched off and replaced by the equilibrium criterion. When the equilibrium criterion is met, the program determines that filtration has stopped and records the final equilibrium suspension height, \( h_\infty \). Air is then vented from the pneumatic cylinder, the test data is output to a spreadsheet file and the program stops. The spreadsheet file containing suspension height and gradient data as a function of pressure can subsequently be used with solids volume fraction and compressive yield stress data to calculate hindered settling function data.

When the test is complete, remove the compressed filter cake from the filtration cylinder as soon as possible and measure the solids concentration, \( \phi_\infty \)(measured).

### 6.5.3.4 Important Considerations for Testing

#### Sample Loading Considerations

Generally, samples would have been loaded by pouring down the inside of the cylinder to minimise the presence of bubbles in the sample. However Bayer process muds generally contained a high proportion of grit or sand which, if poured down the side of the cylinder, would have adversely affected the smooth travel of the piston in the cylinder and compromised pressure control. To avoid such problems, in all tests on Bayer process muds, samples were poured into the centre of the cylinder, down the shaft of a spatula to minimise splashing.

#### Measure Both the Initial and Final Solids Concentrations

In principle, only the initial or final solids concentration needed to be directly measured for each filtration test performed, because the computer calculates the rest anyway. It was advantageous however, if both the initial and final solids concentrations were measured to enable the validity of the test to be evaluated. Thus, if one of the solids concentration measurements was in error, it provided instant evidence that a test result could have been invalid.

#### Dummy Pressure in Permeability Tests

In a permeability filtration test, the filter cake would not normally be fully compressed at the conclusion of the test, making reliable measurement of the final solids concentrations impossible. To combat this problem, an extra dummy pressure, 1 kPa higher than the highest
test pressure, was added to the test pressure list. When filtering at this pressure, the linearity criterion was switched off and the filtration tests went to completion with full compaction at the final solids concentration. This enabled the final solids concentration to be measured, allowing comparison with the result from the compressibility test and verifying that the tests were reliable.

6.5.4 CALCULATIONS

6.5.4.1 Compressive Yield Stress Calculation

Using the compressibility test output data and the measured initial solids volume fraction, a simple material balance enabled the equilibrium solids volume fraction, $\phi_\infty$, for each applied pressure to be calculated using

$$\phi_\infty = \frac{\phi_0 h_0}{h_\infty}, \quad (6.66)$$

where $\phi_0$ is the initial solids volume fraction, $h_0$ is the initial suspension height and $h_\infty$ is the final piston height at the end of each pressure step.

Comparison of the calculated final equilibrium solids volume fraction, $\phi_\infty$(calculated), calculated using Equation 6.66, with the measured value, $\phi_\infty$(measured), enabled evaluation of the validity of the measured initial solids volume fraction. Sometimes, there was a discrepancy and the initial solids volume fraction was deemed unreliable or was just a nominal value, but the final solids volume fraction was considered trustworthy. In this case, the calculated values of $\phi_\infty$, determined using Equation 6.66, were corrected using the ratio of the measured and calculated final filter cake solids volume fractions.

$$\phi_\infty(corrected) = \frac{\phi_{\infty,f(measured)}}{\phi_{\infty,f(calculated)}} \phi_\infty(calculated) \quad (6.67)$$

In addition, the measured or nominal initial solids volume fraction were corrected using this same ratio.

$$\phi_0(corrected) = \frac{\phi_{\infty,f(measured)}}{\phi_{\infty,f(calculated)}} \phi_0(measured) \quad (6.68)$$
At the conclusion of each pressure step with applied pressure, $P$, in the Compressibility Test, the resulting filter cake with solids volume fraction, $\phi_\infty$, had a compressive yield stress equivalent to the applied pressure such that:

$$P_y(\phi_\infty) = P$$

(6.69)

Therefore, each applied pressure in the compressibility test provided one compressive yield stress datum point $(\phi_\infty, P_y(\phi_\infty))$.

### 6.5.4.2 Hindered Settling Function Calculation

The hindered settling function, $R(\phi)$, was determined for each applied pressure using the following equation derived by Landman et al. (Landman et al. 1999),

$$R(\phi_\infty) = \frac{2}{d\beta^2/dP} \left( \frac{1}{\phi_0} - \frac{1}{\phi_\infty} \right) (1 - \phi_\infty)^2,$$

(6.70)

where $\beta^2$ is a filtration parameter that has traditionally been obtained from the inverse of the gradient of $t/V$ versus $V$ data,

$$\beta^2 = \frac{1}{\left( \frac{d(t/V)}{dV} \right)}$$

(6.71)

but in stepped pressure filtration, use of the gradient of $t$ versus $V^2$ data,

$$\beta^2 = \frac{1}{\left( \frac{dt}{dV^2} \right)}$$

(6.72)

has been experimentally demonstrated to be more appropriate (see section 6.5.5). Using the permeability test output data, the $\beta^2$ versus $P$ data for all applied pressures was fitted to a functional form, typically a power law such as,

$$\beta^2 = aP^b,$$

(6.73)

where $a$ and $b$ are empirical fitting parameters. For each applied pressure, $P$, the gradient $d\beta^2/d\Delta P$ was calculated from the derivative of Equation 6.73. Using these $d\beta^2/d\Delta P$ values, the $\phi_\infty$ values determined from the compressibility test data and the measured initial solids volume fraction from the permeability test, $\phi_0$ (corrected in the same way as compressibility test data...
using Equation 6.68 if necessary), the value of \( R(\phi_\infty) \) was calculated for each applied pressure using Equation 6.70.

### 6.5.4.3 Solids Diffusivity Calculation

Alternatively, the solids diffusivity, \( D(\phi) \), could have been directly determined from the pressure filtration output data. The method would use the output data for each applied pressure in the following equation derived by Landman et al. (Landman et al. 1999),

\[
D(\phi_\infty) = \frac{\left( \frac{d\beta^2}{d\phi_\infty} \right)}{2 \left( \frac{1}{\phi_0} - \frac{1}{\phi_\infty} \right)},
\]

(6.74)

where \( \beta^2 \) is defined by Equation 6.72 for stepped pressure filtration. Using the permeability test output data, the \( \beta^2 \) versus \( \phi_\infty \) data for all applied pressures would be fitted to a functional form, typically an exponential relation such as,

\[
\beta^2 = ae^{b_\phi_\infty},
\]

(6.75)

where \( a \) and \( b \) are empirical fitting parameters. The gradient \( d\beta^2/d\phi_\infty \) would then be calculated for each \( \phi_\infty \) value at each applied pressure. Using the \( \phi_\infty \) values determined from the compressibility test data, the \( d\beta^2/d\phi_\infty \) values and the measured initial solids volume fraction from the permeability test, \( \phi_0 \) (corrected in the same way as compressibility test data using Equation 6.68 if necessary), the value of \( D(\phi_\infty) \) would be calculated for each applied pressure using Equation 6.74. However, this method was not generally used for the work presented in this thesis which focussed more heavily on \( R(\phi) \) results rather than \( D(\phi) \) results.

### 6.5.5 VALIDATION OF STEPPED PRESSURE FILTRATION

#### 6.5.5.1 Introduction

To test the new method and modelling predictions, 600 mL of a model suspension was prepared. The model coagulated zirconia (ZrO2) suspension was prepared at a solids volume fraction of \((0.0991 \pm 0.0006)\). The zirconia was obtained from ICI Australia Operations Pty. Ltd., Rockingham, Western Australia. This zirconia (ELECTRO-FINE 0.5 zirconia) has been characterised in the work of Green (Green 1997; Green et al. 1998). The average particle size by volume was determined as 0.47 \( \mu \)m using a Leeds and Northrup Microtrac Particle Size
Analyser. The solids density was taken as 5720 kg m\(^{-3}\). A 0.01 M KNO\(_3\) solution, made up from distilled and Milli-Q filtered water, was used as the suspending fluid. The suspension was initially adjusted to pH < 3 with concentrated nitric acid (HNO\(_3\)) and samples were dispersed by shearing in an Ultra Turrax\textsuperscript{®} high shear mixer to break up any aggregates. The pH was then neutralised with concentrated potassium hydroxide solution (KOH) and allowed to stabilise. The experimental pH was (6.63±0.07). At the test pH of 6.63, the gel point, \(\phi_g\), was calculated to be 0.06 by extrapolation of equilibrium batch settling data.

The compressive yield stress and hindered settling function of the zirconia suspension were determined using single and stepped pressure filtration over a range of five pressures. The initial solids volume fraction, \(\phi_0\), was determined by weight loss on drying of a small sub sample.

**Single Pressure Filtration**

Five single pressure filtration tests, with initial suspension heights of around 25 mm, were performed at pressures ranging from 50 kPa to 200 kPa. The equilibrium solids volume fraction, \(\phi_{\infty}\), for the applied pressure in each filtration test was calculated from the initial solids volume fraction, \(\phi_0\), initial suspension height, \(h_0\), and equilibrium suspension height, \(h_{\infty}\), using Equation 6.66. The gradient \(d(t/V)/dV\) was determined using the middle 50\% of data points in an effort to eliminate errors. Errors are due to the influence of membrane resistance, the time for the set pressure to be reached at the beginning of the test and the compaction of the filter cake against the piston face at the end of the test. The filtration parameter \(\beta^2\) was determined from the traditional gradient \(d(t/V)/dV\) using Equation 6.71. The \(\beta^2\) versus \(P\) relationship was fitted to a power law curve and the hindered settling function \(R(\phi_{\infty})\) values were then determined for each measured \(\phi_{\infty}\) value using Equation 6.70.

**Stepped Pressure Filtration**

Stepped pressure filtration compressibility and permeability tests were performed over the same pressures as the single pressure filtration tests. The stepped pressure tests were conducted twice, both before and after the single pressure tests. The compressibility tests, also with initial suspension heights of around 25 mm, gave the equilibrium solids volume fraction for each pressure. The permeability tests, used an initial suspension height of about 50 mm. During the test, each datum point was detected by a pulse sent from the linear encoder and corresponded to an increment in \(V\) of 1.27 \times 10^{-5} m. The gradient, \(dt/dV^2\), was calculated from linear least squares fits of the previous 30 \((V,t)\) data points. At each pressure, the calculated gradient over the previous 120 data points was monitored until it was stable to within a 1.75\% tolerance. The
average calculated gradient over these 120 data points was recorded and the pressure stepped. Selection of these stability criteria are a balance between gaining accurate results and gaining results at enough pressures since waiting longer may give more accurate results, but reduce the number of pressures that can be tested. For each set pressure, $\beta^2$ was determined from $dt/dV^2$ using Equation 6.72. For each pressure, $R(\phi_c)$ was then calculated using Equation 6.70 as it was for single pressure filtration.

### 6.5.5.2 Results and Discussion

*Compressive Yield Stress Determination*

Data for a stepped pressure filtration compressibility test of a zirconia suspension at pH 6.67 are shown in Figure 6.8 using a plot of $t$ versus $V^2$. For the first pressure of 50 kPa, the data shows the initial filter cake formation region predicted by Equation 6.44, followed by filter cake compression until the whole suspension is at the equilibrium solids volume fraction for the applied pressure. The pressure is then stepped with the suspension compressed to equilibrium through the range of pressures up to 200 kPa.

The compressive yield stress, $P_y(\phi)$, measurements using both single and stepped pressure filtration have been collected and are shown in Figure 6.9. The measured solids volume fractions at each pressure vary by an average of 0.7%. These variations show no systematic difference between single and stepped pressure filtration, but are due to random and precision errors, in addition to variation of sample properties over time.
Figure 6.8: $t$ versus $V^2$ for a stepped pressure filtration compressibility test.

Figure 6.9: Comparison of the compressive yield stress, $P_y(\phi)$, determined from single and stepped pressure filtration.

**Hindered Settling Function Determination**

Experimental $t/V$ versus $V$ behaviour for single pressure filtration is shown in Figure 6.10, showing reasonably linear behaviour once the 50 kPa set pressure has been reached ($V = 0.035$ m), followed by compression at the end of the test. The experimental gradients $d(t/V)/dV$ and
\( \frac{dt}{dV^2} \) for the 50 kPa filtration test are shown in Figure 6.11. It is noted that all experimental and predicted gradients, \( \frac{d(t/V)}{dV} \) and \( \frac{dt}{dV^2} \), shown in Figures 6.11 and 6.12, were determined from least squares fits of ten consecutive data points to smooth out experimental fluctuations. Equation 6.45 predicts that \( \frac{d(t/V)}{dV} \) should be constant until the piston face reaches the filter cake, but Figure 6.11 shows that this gradient is not stable until approximately half way through the test. The problem is that, on start-up, the set pressure is not attained instantaneously. Initially, air pressure in the pneumatic cylinder must build up to overcome friction at the piston-cylinder contact. The pressure then slowly increases to the set point so as to minimise pressure overshoot. This leads to a miscalculation of the time when the experiment effectively starts. Equation 6.46 accounts for this time issue and predicts that \( \frac{d(t/V)}{dV} \) underestimates the desired quantity, even after the pressure set point has been reached. Fortunately, this time error is not introduced when \( \frac{dt}{dV^2} \) is used. The initially high experimental gradient, \( \frac{dt}{dV^2} \), is partially due to the lower applied pressure prior to reaching the pressure set point and the effect of membrane resistance. The influence of membrane resistance is predicted to dissipate with the extent of filtration, according to Equation 6.65, and Figure 6.11 shows that it is experimentally insignificant once the set pressure has been reached. Therefore, modelling predicts that \( \frac{dt}{dV^2} \) can produce results similar to those using \( \frac{d(t/V)}{dV} \) for a single applied piston pressure, but experimental results suggest an improvement in accuracy because \( \frac{dt}{dV^2} \) is unaffected by time errors associated with experimental start-up.

Experimental and predicted gradients for a stepped pressure permeability test are shown in Figure 6.12. The values of \( \frac{dt}{dV^2} \) obtained from single pressure filtration have been used to predict the results in stepped pressure filtration using Equations 6.45, 6.52 and 6.61. During initial filter cake formation at 50 kPa, the \( \frac{dt}{dV^2} \) prediction does not take membrane resistance into account and therefore underestimates the experimental gradient. This influence is diminished with time, as predicted by Equation 6.65. The gradient \( \frac{dt}{dV^2} \) is well predicted during filter cake growth at higher pressures in accordance with Equation 6.61. From Figure 6.12, it is evident that the gradient, \( \frac{d(t/V)}{dV} \), in a stepped pressure test is not constant during filter cake formation or growth. Equations 6.46 and 6.60 predict that the gradient will vary with the inverse of the specific volume of filtrate squared, \( (1/V^2) \). The predicted gradient \( \frac{d(t/V)}{dV} \) has been plotted against the experimental results and correlates well with the experimental data. During filter cake compaction, the dramatic changes in gradients predicted by Equations 6.51 and 6.52 are observed, but not as strongly as predicted due to the step in pressure not being instantaneous.

The observation that the gradients \( \frac{d(t/V)}{dV} \) and \( \frac{dt}{dV^2} \) are both well predicted using the modified Darcy’s law equations suggests that the model can predict stepped pressure filtration.
behaviour with reasonable accuracy. The shortcoming of the modified Darcy's Law modelling is that it assumes a uniform solids volume fraction in the filter cake. The true situation involves a filter cake with a solids volume fraction distribution varying from the final volume fraction at the base to the initial volume fraction at the top. Fortunately, for the modelling conclusions to be valid, it is only required that the same solids distribution profile be obtained from different pressure regimes. For example, the solids distribution profile of a suspension compressed 10 mm at constant pressure being equal to that obtained if it was compressed to the same extent using stepped pressure filtration. How this issue affects the applicability of the stepped pressure calculations using $\frac{dt}{dV^2}$ requires verification with more comprehensive modelling techniques such as those employed by Landman and Russel (Landman and Russel 1993). For now, experimental results are compared.

![Graph](image.png)

**Figure 6.10:** $t/V$ versus $V$ plot for single pressure filtration at 50 kPa.
Figure 6.11: Gradients $dt/dV^2$ and $d(t/V)/dV$ for single pressure filtration at 50 kPa.

Figure 6.12: Experimental and predicted gradients $dt/dV^2$ and $d(t/V)/dV$ for a stepped pressure filtration permeability test.

Figure 6.13 compares experimental gradients from single and stepped pressure filtration. As expected, all $d(t/V)/dV$ gradients calculated from single pressure filtration are significantly below the $dt/dV^2$ values (18% on average) due to the time error in Equation 6.46. The single and stepped pressure $dt/dV^2$ results are reliable with an average deviation of 3.6% for each
applied pressure. Figure 6.14 compares experimental hindered settling functions, $R(\phi)$, for single and stepped pressure filtration. Single pressure $d(t/V)/dV$ values under-predict $R(\phi)$ values (27% on average) and thus significantly over-predict the permeability of the suspension. $dt/dV^2$ values in both single and stepped pressure filtration predict $R(\phi)$ with an average deviation of only 4.3%. The data clearly indicates that $dt/dV^2$ should be used for $R(\phi)$ determination.

**Figure 6.13:** Comparison of gradients $dt/dV^2$ and $d(t/V)/dV$ determined from single and stepped pressure filtration.
Figure 6.14: Comparison of the hindered settling function, $R(\phi)$, determined from single and stepped pressure filtration using gradients $dt/dV^2$ and $d(t/V)/dV$.

Sample Characterisation Time

For the five single pressure filtration tests, the total characterisation time was around 7 hours, including a sample changeover time of 40 minutes per test. Figures 6.8 and 6.12 show filtration data for stepped pressure filtration compressibility and permeability tests. These two tests alone enable characterisation of compressibility and permeability of the zirconia suspension in a time that is halved to 3.4 hours. When results from more than 5 pressures are required, the time saving becomes even more significant. This quicker characterisation reduces the likelihood that suspension properties will change due to influences such as flocculation degradation, pH drift or chemical reaction. Thus characterisation via stepped pressure filtration is clearly advantageous.

6.5.5.3 Conclusions

Stepped pressure filtration allows characterisation of suspension compressibility and permeability using only one stepped pressure compressibility filtration test and one truncated stepped pressure permeability filtration test. The compressibility test determines the compressive yield stress, $P_c(\phi)$, for a range of solids volume fractions, $\phi$. The permeability test determines the hindered settling function, $R(\phi)$, for the same solids volume fractions. A new calculation method uses $dt/dV^2$ to accurately determine $R(\phi)$. The new $dt/dV^2$ method is more accurate than the previously used $d(t/V)/dV$ method because $d(t/V)/dV$ becomes a function of specific filtrate volume, $V$, with pressure variations.
6.5.5.4 Further Work

At low pressures, the influence of membrane resistance can become significant. In the proposed stepped pressure filtration technique, waiting for the membrane resistance to become insignificant in the calculated gradient increases filtration time. When sample availability is limited, this limits the number of pressure steps that can be performed. If this error is predicted and corrected for, then more pressures can be tested with the same quantity of sample.

Values of $P$ and $V$ are readily available during a filtration test, so only the filtrate viscosity, $\eta$, and membrane resistance, $\alpha$, need to be determined in advance to apply a correction to the gradient. The filtrate viscosity $\eta$, can be determined from a range of standard rheological techniques. The membrane resistance $\alpha$, can be determined from the filtration of a liquid with no suspended solids. Solids free filtration behaviour using Equation 6.46 with filter cake height $B = 0$, predicts constant velocity filtration

$$\frac{dV}{dt} = \frac{P}{\eta \alpha}$$  (6.76)

Therefore, the membrane resistance is calculated using

$$\alpha = \frac{P}{\eta \frac{dV}{dt}}$$  (6.77)

As a consequence, membrane resistance in stepped pressure filtration can be predicted and eliminated. Alternatively, if this influence cannot be quantified accurately, then the final results should just take longer, but not be affected in terms of accuracy.

6.5.6 APPLICATION SUMMARY

Stepped pressure filtration tests using $dt/dV^2$ rather than $d(t/V)/dV$ have become standard practice at The University of Melbourne. This method has been used to provide the backbone of the red mud dewaterability characterisation results presented in this thesis in Chapters 9, 10 and 11. In fact, all of the high solids concentration $P_s(\phi)$ and $R(\phi)$ red mud data presented in this thesis were obtained using stepped pressure filtration.
### 6.6 $\phi_\infty$ ESTIMATION

<table>
<thead>
<tr>
<th>Parameters Measured:</th>
<th>$\phi_\infty$ &amp; $D(\phi_\infty)$ for applied pressure $P = P_y(\phi_\infty)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Concentration Range:</td>
<td>$0.25 &lt; \phi &lt; 0.45$</td>
</tr>
<tr>
<td>Pressure Range:</td>
<td>5-300 kPa</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>10-90°C (Thermally jacketed filtration cylinder)</td>
</tr>
<tr>
<td>Time Required:</td>
<td>1-8 hours or more per test</td>
</tr>
<tr>
<td>Advantages:</td>
<td>Robust, accurate, transportable, rapid characterisation that doesn’t require a test to go to completion, requires only small samples</td>
</tr>
<tr>
<td>Limitations:</td>
<td>Mechanically complex, no data for very low solids and only works well for one pressure per test</td>
</tr>
</tbody>
</table>

**Table 6.6:** $\phi_\infty$ estimation filtration test method overview.

#### 6.6.1 INTRODUCTION

The method of $\phi_\infty$ estimation is a characterisation tool that can be applied to determine compressive yield stress, $P_y(\phi)$, and solids diffusivity, $D(\phi)$, data from an incomplete filtration test. This method has the potential to reduce characterisation times for impermeable suspensions, such as water treatment sludges, that would take many hours to characterise by standard pressure filtration techniques that have already been described in this chapter in section 6.5.

The test can characterise suspensions over the same wide range of temperatures and pressures applicable to stepped pressure filtration. Characterisation time depends strongly on the nature of the suspension being characterised and can vary from 1 to 8 hours or more depending on the rate and extent of filtration. For very slow filtration tests, the time required is a subjective compromise between filtration time constraints and accuracy. The significant limitation of using $\phi_\infty$ estimation relative to stepped pressure filtration is that each test produces only one $P_y(\phi)$ datum point and one $D(\phi)$ datum point.

As for traditional pressure filtration, experiments involve the pressure filtration of a particulate suspension in which the time of filtration, $t$, is monitored as a function of piston height from which the specific volume of filtrate, $V$, is determined. Providing that the test has progressed beyond the initial filter cake formation and has substantial filter cake compression data, the test can be terminated before filtration stops, thus giving a significant time saving. The equilibrium solids volume fraction, $\phi_\infty$, and the solids diffusivity at this solids concentration, $D(\phi_\infty)$, are predicted from the incomplete $(t, V)$ data set. This is achieved using an analytical solution to the single pressure filtration problem developed by Landman and White (Landman and White 1997) from the fundamental dewatering theory of Buscall and White (Buscall and White 1987). During the final stages of compression, this functional relation can be simplified and approximated as a generic functional form. This form predicts that filtration time $t$ is linearly related to $ln(E_3-V)$, with an appropriate choice of the equilibrium solids volume fraction, $\phi_\infty$. 

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where $E_3$ is a constant function of the initial suspension height, $h_0$, the initial solids volume fraction, $\phi_0$, and the equilibrium solids volume fraction, $\phi_\infty$. Iteration of $\phi_\infty$ guesses on the filtration data to optimise the linearity in the compression data for a plot of $t$ versus $\ln(E_3 - V)$ is used to determine the true value of $\phi_\infty$. This method predicts the equilibrium solids volume fraction, for an applied pressure, with greater accuracy than conventional methods in reduced time, allowing more accurate and timely determination of the compressive yield stress function. In addition, the gradient of this plot can be used to determine the solids diffusivity at the equilibrium solids volume fraction, $D(\phi_\infty)$.

In the following sections, the fundamental theory is introduced to demonstrate why $\phi_\infty$ estimation is valid, the experimental method is overviewed and the procedure is validated by presenting results for a zirconia suspension comparing pressure filtration methods described in section 6.5 with $\phi_\infty$ estimation.

### 6.6.2 THEORY

The linearised filtration theory predicts two stages of filtration behaviour including filter cake formation followed by filter cake compression. During cake formation, theory predicts linear $t$ versus $V^2$ behaviour according to the following relation

$$t = \frac{1}{\beta^2} V^2,$$

(6.78)

where $t$ is the filtration time, $V$ is the specific volume of filtrate and $\beta^2$ is a constant filtration parameter that is a complicated function of the initial solids volume fraction, $\phi_0$, the final solids volume fraction, $\phi_\infty$, and the solids diffusivity, $D(\phi)$, explained later in section 7.4.4.2.

Once the filter cake touches the piston, cake compression begins. During cake compression, the Landman and White solution predicts an explicit time-volume relationship for the cake compression region. Development of the solution required an approximation involving truncation of a Fourier series. The accuracy of the approximation is observed to improve with increasing extent of filtration and becomes an excellent approximation at high extents of filtration. The resulting cake compression equation, given by Equation 7.31 (see section 7.4.4.3), exhibits a logarithmic functional form given by;

$$t = E_1 - E_2 \ln(E_3 - V).$$

(6.79)

where $E_1$, $E_2$ and $E_3$ are all constant with respect to a given single pressure filtration test. $E_1$ is a complicated function of $h_0$, $\phi_0$, $\phi_\infty$ and $D(\phi)$ (see Equation 7.31). $E_2$ is given by...
\[ E_2 = \frac{4h_0^2\phi_0^2}{\pi^2\phi_d^2 D(\phi_\infty)} \]  

(6.80)

and \( E_3 \) is given by

\[ E_3 = h_0 \left( 1 - \frac{\phi_0}{\phi_\infty} \right). \]  

(6.81)

Equation 6.79 predicts that during filter cake compression, a linear plot of \( t \) versus \( \ln(E_3 - V) \) can be produced, assuming that \( \phi_0, \phi_\infty \) and \( h_0 \) are known. Since \( \phi_0 \) and \( h_0 \) are generally known, iterative optimisation of \( \phi_\infty \) guesses to produce the straightest line possible in a plot of \( t \) versus \( \ln(E_3 - V) \) for compression data would produce a very accurate determination of \( \phi_\infty \), providing that the theory is valid. As an added extra, the gradient \( E_2 \) could be used to determine \( D(\phi_\infty) \) using

\[ D(\phi_\infty) = \frac{4h_0^2\phi_0^2}{\pi^2\phi_d^2 E_2}. \]  

(6.82)

The nature of this functional behaviour enables exploitation in the determination of \( \phi_\infty \) and \( D(\phi_\infty) \) using \( (t,V) \) data from an incomplete filtration test.

### 6.6.3 EXPERIMENTAL METHOD

#### 6.6.3.1 Apparatus

This technique required a piston driven pressure filtration test rig as described in section 6.5.3.1.

#### 6.6.3.2 Sample Preparation

Sample preparation was as described for stepped pressure filtration in section 6.5.3.2. The initial solids concentration and initial suspension height were generally selected to minimise the required filtration time, while giving a final suspension height that was not so low that it caused accuracy errors.

#### 6.6.3.3 Test Procedure

The test procedure was the same as described in section 6.5.3.3, but for a single pressure compressibility test. Of course, because of the predictive nature of the analysis, the method has a flexibility that allows subjective judgement as to when the filtration test is close enough to completion, providing the tests were terminated during cake compression. This potential
subjectivity was not utilised when obtaining the test results presented here, but would be very useful if low permeability suspensions were involved. The filtration model predicts that all tests terminate before completion, such that the final solids volume fraction, $\phi_f$, is less than the equilibrium solids volume fraction, $\phi_{\infty}$. As a result, when measuring the final solids volume fraction, $\phi_f$, it was important to weigh the entire filter cake in the solids concentration measurement to eliminate the possible effects of a solids concentration profile.

### 6.6.3.4 Calculations

First, the solids concentration data needed to be calculated and corrected if necessary, according to the recommendations in section 6.5.4.1, to accurately determine the initial solids concentration, $\phi_0$, and the final solids concentration, $\phi_f$. $\phi_f$ was used as an initial guess of the equilibrium solids volume fraction, $\phi_{\infty}$. Then the initial suspension height, $h_0$, the initial solids volume fraction, $\phi_0$, and the equilibrium solids volume fraction guess, $\phi_{\infty}$, were used with the final 20-50 $(t,V)$ data points to produce a plot of $t$ versus $\ln(E_3 - V)$. A good rule of thumb was to only use $(t,V)$ data for times after the value of the gradient $\frac{dt}{dV^2}$ had risen to at least 30% higher than its filter cake formation value. Linear regression was used to provide a line of best fit through the selected compression data. The linearity of the plot was quantified using the value of the $r^2$ coefficient. A solver was used to maximise the value of the $r^2$ coefficient by varying $\phi_{\infty}$ guesses, to provide a more accurate prediction of $\phi_{\infty}$. Therefore, for the applied pressure, $P = P'_f(\phi_{\infty})$, one compressive yield stress datum point ($\phi_{\infty}, P'_f(\phi_{\infty}))$ was determined for each filtration test. In addition, one solids diffusivity datum point ($\phi_{\infty}, D(\phi_{\infty}))$ was provided using $h_0, \phi_0, \phi_{\infty}$, the optimised value of $\phi_{\infty}$ and the gradient of the final plot, $E_2$, in Equation 6.82.

### 6.6.4 VALIDATION OF $\phi_{\infty}$ ESTIMATION

#### 6.6.4.1 Materials and Methods

The material used in all tests was a zirconia supplied by Millennium Performance Chemicals, Rockingham, Western Australia, Grade HWA - Z 0.5. Preparation of the suspension was as described in section 6.5.5.1 according to the procedure described by Green (Green 1997; Green et al. 1998). The experimental pH was 7.0, just below the isoelectric point. At this pH the samples were strongly flocculated.

The solids diffusivity, $D(\phi)$, for this zirconia suspension was characterised by de Kretser (de Kretser et al. 2001a) from a number of single pressure filtration tests using the $\phi_{\infty}$ estimation method proposed in section 6.6.3. In addition, de Kretser (de Kretser et al. 2001a) produced
$D(\phi)$ values through stepped pressure filtration using Equation 6.74, according to the calculation method proposed in section 6.5.4.3. The results are compared in the following section.

### 6.6.4.2 Results and Discussion

Figure 6.15, below, presents a $t$ versus $V^2$ plot for a single pressure filtration test on a zirconia suspension at 300 kPa. The filter cake formation and filter cake compression regions are highlighted. Also shown in Figure 6.15 is a plot of the gradient $dt/dV^2$ versus $t$ data which more clearly demonstrates the transition from filter cake formation to filter cake compression through an increase in $dt/dV^2$ after $V = 0.021$ m. The selected data for $\phi_\infty$ estimation, when $dt/dV^2 > 3.47 \times 10^6$ s m$^{-2}$, is all at least 1.3 times the minimum value of the gradient $dt/dV^2$ during the filtration test.

Figure 6.16, below, presents two $t$ versus $\ln(E_3 - V)$ plots from this zirconia filtration test data. The first plot, (a), uses the final solids volume fraction, $\phi_f = 0.3399$, as a guess for the equilibrium solids volume fraction, $\phi_\infty$, in determining $E_3$. This value is traditionally considered accurate enough for most purposes. However, the data in the cake compression region is clearly not linear. Suggesting that the filtration test did not reach equilibrium and the true value of $\phi_\infty$ is indeed higher than $\phi_f$. If it is assumed that the control program had prematurely terminated the filtration and the $\phi_\infty$ input value is adjusted, it is revealed that the shape of the cake compression region of the data is highly sensitive to the input value of $\phi_\infty$. Performing iterations on $\phi_\infty$ to minimise the value of the $r^2$ coefficient of a linear regression through the selected cake compression data leads to the optimised result shown in Figure 6.16(b). Clearly, very good linearity is observed during cake compression with the optimised value of $\phi_\infty = 0.3419$. Such high sensitivity in linearity to small variations in $\phi_\infty$ suggests that this value can be accurately predicted from incomplete filtration tests. Currently the approach is being trialed on the modelling of the filtration behaviour of waste-water sludges, which are highly compressible and impermeable, leading to very long filtration times and the need predict equilibrium solids volume fractions from incomplete filtration data.
Figure 6.15: Plots of $t$ versus $V^2$ and $dt/dV^2$ versus $V$ data from a filtration test on zirconia suspension at 300 kPa, pH = 7.0, $\phi_0 = 0.0817$ and $h_0 = 0.03130$ m.

Figure 6.16: Plots of $t$ versus $\ln(E_3 - V)$ from a filtration test on zirconia suspension at 300 kPa, pH = 7.0, $\phi_0 = 0.0817$ and $h_0 = 0.03130$ m. (a) Using the final solids volume fraction, $\phi_f$, for the equilibrium solids volume fraction, $\phi_\infty$, in determining $E_3$ and (b) using the optimised value of $\phi_\infty$ in determining $E_3$.

Solids diffusivity, $D(\phi)$, characterisation results are presented in Figure 6.17, comparing results for the determined from single pressure $\phi_\infty$ estimation filtration tests with results from stepped pressure filtration tests. The stepped pressure filtration results show a consistent trend from low to high solids concentrations. The $\phi_\infty$ estimation method produced some scatter in the low solids concentration results obtained from low pressure filtration tests at 1 and 2 kPa. This scatter is due to a lack of pressure control at the low pressures involved that caused instabilities the filtration data. The agreement between the $\phi_\infty$ estimation and stepped pressure filtration
methods is very good at higher solids concentrations, supporting the validity of the \( \phi_\infty \) estimation method and validating the theory.

Obviously, it is necessary to have good pressure control for the \( \phi_\infty \) estimation method to produce valid results. As a result, \( \phi_\infty \) estimation is only recommended when good pressure control can be achieved, maintaining a constant pressure throughout the test. The implications of the results in Figure 6.17 are significant for the modelling of dewatering of wastewater sludge, where filtration is characterised by a very short cake formation region from which it is difficult to extract \( \beta^2 \). \( \phi_\infty \) estimation eliminates the need to isolate a linear filter cake formation region or even the exact location of the transition from cake formation to compression, since only the tail end of the cake compression data is required.

![Figure 6.17: Comparison of \( D(\phi) \) data determined from stepped pressure filtration tests and from single pressure \( \phi_\infty \) estimation filtration tests for zirconia suspension in 0.01 M KNO\(_3\) at pH 7.0.](image)

6.6.4.3 Conclusions

The utility of a functional form for \( t \) versus \( V \) data in the cake compression region for optimising filtration end point estimation and providing an alternate method of estimating the filtration diffusivity, \( D(\phi) \), has also been demonstrated. As a result, the equilibrium solids volume fraction can now be more accurately determined through an iterative \( \phi_\infty \) estimation method, which also allows accurate determination of the filtration diffusivity, \( D(\phi_\infty) \), at the applied pressure.
6.6.5 APPLICATION SUMMARY

The $\phi_m$ estimation filtration technique has significant potential for application in the characterisation of very low permeability suspensions where there is not enough time for complete filtration tests. This $\phi_m$ estimation filtration technique was not required for the red mud characterisations presented in this thesis, because the results from stepped pressure filtration were considered accurate enough and could also be produced much more quickly. However, there is potential for this $\phi_m$ estimation technique to be incorporated into the stepped pressure filtration methodology to improve the accuracy of results and also to reduce characterisation time further, but work is still ongoing.
Chapter 7

INDUSTRIAL PROCESS EQUIPMENT MODELLING TOOLS

7.1 INTRODUCTION

Utilising the dewatering theory of Buscall and White (Buscall and White 1987), this chapter describes methods that have been developed to model the operation of pressure filters, gravity thickeners and consolidation in tailings dams based on dewaterability data. Each of the methods described uses the compressive yield stress and the hindered settling function as the material properties that determine suspension dewaterability. Unfortunately there is no single technique that will measure these parameters quickly, accurately and over the entire range of solids concentrations of interest and a number of techniques have to be employed in combination. The next section of this chapter, section 7.2, describes the procedures used to fully characterise the process equipment output suspensions and the laboratory flocculated suspensions analysed in this thesis. The following section, section 7.3, describes how the dewaterability data was fitted to functions that accurately described the properties for all relevant solids concentrations. This was done in order to provide the necessary data for input to the computational models described in sections 7.4, 7.5 and 7.6 to predict the performance of industrial process equipment at the sponsor sites.

7.2 DEWATERABILITY CHARACTERISATION REQUIREMENTS

Whilst working on site during the course of this project, a minimum requirement for suspension dewaterability characterisation was developed with a view to providing the necessary input data for process modelling. To this end, the suspension characterisation data deemed to be required included:

a) Liquor viscosity and density data.

b) Suspension shear rheology data such as the shear yield stress, shear stress versus shear rate and shear history dependant behaviour such as thixotropy, where relevant.

c) Suspension dewaterability data, including gel point, compressive yield stress and hindered settling function, over the full range of solids concentrations of interest from the process equipment feed solids concentration to the output solids concentration.
Methods of determining liquor viscosity and density data are described in Chapter 4, while suspension shear rheology characterisation techniques appropriate for on site testing are described in Chapter 5. As for dewaterability characterisation, the relevant solids concentration range generally depends on the type of process equipment employed in the dewatering operation. Pressure filters have feed solids volume fractions ranging from 0.01-0.20, which is generally below the gel point and very high output solids volume fractions of 0.25-0.50, due to applied pressures of 30 kPa or higher. Thickeners generally have a feed solids volume fraction of 0.02-0.05 and an underflow solids volume fraction of 0.15-0.25, which is slightly above the gel point. Suspensions are generally fed to residue disposal areas at solids volume fractions of 0.15-0.25, which is slightly above the gel point, and consolidate to solids volume fractions of 0.25-0.50, which is similar to the concentrations achieved in pressure filtration due to the significant suspension network pressure head.

To enable full characterisation, over the solids concentration of interest, pressure filtration is generally combined with other dewaterability characterisation techniques that may include equilibrium batch settling tests, transient batch settling tests and gravity permeation tests. Each of these dewaterability tests and associated calculation methods has been described in Chapter 6. The most useful set of dewaterability characterisation tests depends on the nature and availability of the sample to be tested and the characterisation techniques available. In the absence of a pressure filtration test rig, batch settling tests alone can still provide valuable, material property data that will aid in optimising dewatering operations. The red mud dewaterability data presented in this thesis was derived from characterisations performed on thickener underflow suspensions and simulated thickener feed suspensions that were flocculated in the laboratory. As such, a different set of dewaterability tests was used for each of these samples as described below.

7.2.1 THICKENER UNDERFLOW CHARACTERISATION

On site, thickener underflow suspension samples are readily available in large quantities, enabling relatively comprehensive dewaterability characterisation over a wide range of solids concentrations. Equilibrium batch settling tests and transient batch settling tests provide methods of calculating $\phi_g$ in addition to $P_\eta(\phi)$ and $R(\phi)$ at solids concentrations applicable to thickener operations. Pressure filtration data at higher solids concentrations is still very useful for modelling purposes because it is relatively quick to obtain and removes the need for data extrapolation in process modelling. Since the $R(\phi)$ results of gravity permeation tests can generally be interpolated from the results of transient batch settling tests and stepped pressure filtration tests, gravity permeation tests were generally not considered necessary. Therefore, the
dewaterability characterisation tests used to determine the compressive yield stress, \( P_y(\phi) \), and hindered settling function, \( R(\phi) \), of thickener underflow suspensions included:

- Equilibrium batch settling test, \((\phi_g \text{ and } P < 1 \text{ kPa})\), for determination of \( \phi_g \) and \( P_y(\phi) \) data at low pressures at solids concentrations slightly above \( \phi_g \).
- Transient batch settling tests, \((\phi_0 < \phi < \phi_g + 0.02)\), for determination of \( R(\phi) \) data at solids concentrations ranging from the feed solids concentration to slightly above \( \phi_g \).
- Stepped pressure filtration tests, \((5 \text{ kPa} < P < 300 \text{ kPa})\), for determination of \( P_y(\phi) \) and \( R(\phi) \) data at high pressures.

### 7.2.2 FLOCCULATED SAMPLE CHARACTERISATION

Flocculation of suspensions in the laboratory is an involved process that can produce a wide range of results depending on the method used. Generally the aim is to reproduce the shear conditions in the process as closely as possible. In this work, flocculation of a simulated thickener feed suspension was achieved in batch tests using both the plunger method and the baffle reactor. Both of these methods are labour intensive and produce relatively small amounts of flocculated suspension. As a result, the comprehensiveness of the dewaterability characterisation was compromised. This required a balance between the number of tests performed on a sample and the amount of suspension available. Immediately after flocculation, the initial settling rate was measured, enabling determination of \( R(\phi) \) at the feed solids concentration, using calculations analogous to those used in transient batch settling. The number of actual transient batch settling tests that could be performed was especially limited and, for most flocculation conditions, only one other transient batch settling test was performed by combining suspension from a number of batch flocculation tests into a 1 litre cylinder. Equilibrium batch settling tests provided a method of calculating \( \phi_g \) in addition to \( P_y(\phi) \) at solids concentrations applicable to thickener operations. \( R(\phi) \) data, at solids concentrations slightly above the gel point, produced from gravity permeation tests would have been valuable, but the gravity permeation test rig was not available on site. Pressure filtration data at higher solids concentrations was very useful for modelling purposes because it produced results relatively quickly and removed the need for data extrapolation in process modelling. As a result, the dewaterability characterisation tests used to determine the compressive yield stress, \( P_y(\phi) \), and hindered settling function, \( R(\phi) \), of flocculated simulated thickener feed suspensions included:

- Measurement of initial settling rate after flocculation, \((\phi = \phi_0)\), for determination of \( R(\phi) \) at the feed solids concentration.
• Transient batch settling test, \((\phi = 3-5\phi_0)\), for determination of an \(R(\phi)\) value at a solids concentration that is 3-5 times the feed solids concentration.

• Equilibrium batch settling test, \((\phi_g\) and \(P < 1\ kPa)\), for determination of \(\phi_g\) and \(P_y(\phi)\) data at low pressures at solids concentrations slightly above \(\phi_g\).

• Stepped pressure filtration tests, \((5\ kPa < P < 300\ kPa)\), for determination of \(P_y(\phi)\) and \(R(\phi)\) data at high pressures.

### 7.3 DEWATERABILITY CURVE FITS

For use in process modelling, the dewaterability data determined according to section 7.2 must be accurately fitted to an appropriate functional form. The dewaterability data are fitted to empirical curves so that the compressive yield stress and hindered settling function can be predicted for any solids concentration. The curve fitting minimises the least squares of the proportional errors between the experimental data and the curve fits predictions.

There is an endless number of functional forms that could describe the compressive yield stress and hindered settling function, each with its own advantages and disadvantages. The key issues are the number of fitting parameters and accuracy. Some examples that have been previously proposed in the literature are presented below along with a discussion of their limitations. New functional forms that more accurately describe recently acquired dewaterability data over a wide range of solids concentrations are also presented and discussed.

Once dewaterability characterisation and curve fitting has been performed, the \(P_y(\phi)\) and \(R(\phi)\) curve fit equations can be used with fundamental dewatering theory to predict the separation performance of thickeners, centrifuges, filter presses and even consolidation in tailings dams.

#### 7.3.1 COMPRESSIVE YIELD STRESS

Numerous functional forms have been proposed to describe the behaviour of the compressive yield stress as a function of solids volume fraction. Commonly used functional forms, proposed by Landman and co-workers (Landman et al. 1988) are given by Equations 7.1 and 7.2 below,

\[
P_y(\phi) = k \left( \frac{\phi}{\phi_g} \right)^n, \quad (7.1)
\]

\[
P_y(\phi) = k \left( \frac{\phi}{\phi_g} - 1 \right)^n, \quad (7.2)
\]
where $k$ and $n$ are empirical fitting parameters. The limitation of these functional forms, and others like them, is that they cannot be accurately fitted to experimental data over the full range of solids concentrations from the gel point to the high solids concentrations found in filter cakes and at the base of consolidated tailings. To overcome this problem, a new more robust asymptote and exponential-power law functional form was developed. This functional form is given below in Equation 7.3,

$$P_y(\phi) = \left(1 - \left(\frac{\phi}{\phi_g}\right)^{p_n}\right) e^{(p_n \phi^{p_m} + p_0)},$$  \hspace{1cm} (7.3)

where $p_a, p_n, p_m$ and $p_0$ are empirical fitting parameters. Fitting of Equation 7.3 requires minimisation of the squares of the proportional difference between the fitted equation and the experimental data through varying the fitting parameters. A typical fit is displayed in Figure 7.1, where there is clearly good agreement between the curve fit and the experimental data.

Figure 7.1: Example of combined compressive yield stress data from an equilibrium batch settling test and a stepped pressure filtration test and a curve fit of this data using the asymptote and exponential-power law functional form given by Equation 7.3.

### 7.3.2 Hindered Settling Function

Numerous functional forms have also been proposed to describe the behaviour of the hindered settling function as a function of solids volume fraction. Commonly used functional forms, proposed by Eberl and co-workers (Eberl et al. 1995) are given by Equations 7.4 and 7.5 below,
\[ R(\phi) = w(1 - \phi)^m, \quad (7.4) \]

\[ R(\phi) = w(10)^{m0}, \quad (7.5) \]

where \( w \) and \( m \) are empirical fitting parameters. These functional forms work well for some coarse particle suspensions. However, the shape of the curve for flocculated suspensions such as red mud given in Figure 7.2 cannot be produced by these functional forms. Other functional forms that allow this shape to be fitted more adequately including a power law form, an exponential-power law form and a novel double exponential form are given below in Equations 7.6, 7.7 and 7.8,

\[ R(\phi) = r_a \phi^{-r_c} + r_b, \quad (7.6) \]

\[ R(\phi) = \text{Exp}\left(r_c \phi^{-r_c} + r_b\right), \quad (7.7) \]

\[ R(\phi) = \text{Exp}\left(\text{Exp}\left(r_a + r_b \left(-\ln\phi + r_c\right)^{r_c}\right)\right), \quad (7.8) \]

where \( r_a, r_b, r_c \) and \( r_n \) are empirical fitting parameters. Equations 7.6 and 7.7 tend to fit most experimental data reasonably well. However, even these functional forms are sometimes too rigid and only the more complex double exponential functional form given by Equation 7.8 seems to fit well. Equation 7.8 also has the advantage that only a small amount of experimental data is required to produce a realistic shape. Again, fitting of one of the equations requires minimisation of the squares of the proportional difference between the fitted equation and the experimental data through varying the fitting parameters. A typical fit is displayed in Figure 7.2, where there is good agreement between the curve fit and the experimental data. Clearly, the experimental data is scattered around the fit, however, when it is considered that the data are spread over five orders of magnitude, the significance of the scatter is diminished.
Figure 7.2: Example of hindered settling function, $R(\phi)$, data from transient batch settling tests and a stepped pressure filtration test and an associated curve fit using the functional form given by Equation 7.6.

7.4 PRESSURE FILTRATION MODELLING

7.4.1 INTRODUCTION

Landman and White (Landman and White 1997) have presented the results of a similarity solution to the problem of piston driven filtration where the influence of gravity and membrane resistance are negligible. The solution suggests that for a single applied pressure at the piston face, the filtration behaviour can be described as having two parts, firstly a filter cake formation stage and then a filter cake compression stage. The results suggest that the initial solids volume fraction, $f_0$, the equilibrium solids volume fraction at infinite time, $f_\infty$, the initial suspension height, $h_0$, and the solids diffusivity function, $D(\phi)$, can fully describe a filtration run. In the following sections, the assumptions of the model are presented, followed by a listing of the inputs required for prediction of the extent of filtration as a function of time. The derivations presented in the paper by Landman and White (Landman and White 1997) are not presented here, however the results are presented to a level of complexity that demonstrates the calculations involved in predicting filtration behaviour from material properties and operating conditions. Theory on the application of this model to rotary drum filtration is also presented. The model is validated by comparison of experimental zirconia suspension filtration data with model predictions. Finally, use of the model for filtration optimisation is discussed.
7.4.2 ASSUMPTIONS

As with all models, the solution to this dewatering model is subject to a number of limitations based on the assumptions in the dewatering model itself and the assumptions used in the derivations of the filtration solution. The most relevant assumption of the Buscall and White dewatering model is the assumption of line settling. This implies that settling rate and permeability are functions of solids volume fraction and all solid particles at the same height settle at the same rate in a one-dimensional model. This also implies that there is no particle size segregation and the model does not account for membrane fouling. The assumptions used in deriving the filtration solution are as follows:

- Filtration occurs in one direction (ie. a one-dimensional model).
- A single constant pressure is applied through piston driven filtration, but can be adapted to rotary drum filtration.
- No solids exit beyond the membrane.
- The effect of gravity is insignificant relative to the applied pressure.
- Membrane resistance is insignificant.

7.4.3 INPUTS

The inputs required to predict a filtration process include:

- Type of filtration process (eg. piston driven filtration or rotary drum filtration).
- Initial solids volume fraction, $\phi_0$.
- Initial suspension height, $h_0$.
- Filter area.
- Applied Pressure, $\Delta P$.
- Material properties including compressive yield stress, $P_y(\phi)$, and hindered settling function, $R(\phi)$ curve fits.

7.4.4 CALCULATIONS

From the data inputs, a few calculations provide all of the information required for filtration modelling. This section details the required preliminary calculations, calculations to predict filtration behaviour during the initial filter cake formation and then during filter cake compression. Application to rotary drum filtration predictions is also discussed.
7.4.4.1 Preliminary Calculations

Calculation of $D(\phi)$

If necessary, the solids diffusivity, $D(\phi)$, is calculated from the compressive yield stress, $P_y(\phi)$, and the hindered settling function, $R(\phi)$, using Equation 3.16.

Calculation of $\phi_\infty$

According to filtration theory, a suspension at solids volume fraction $\phi$ will yield and compress to a higher solids volume fraction when the applied pressure, $\Delta P$, exceeds the network strength. When filtering a suspension in a piston driven filtration system, a filter cake develops on the membrane. For cases where membrane resistance is negligible, the solids volume fraction at the base of the filter cake, $\phi_{\text{base}}$, is determined by the applied pressure, $\Delta P$, according to the following relation,

$$\Delta P = P_y(\phi_{\text{base}}). \quad (7.9)$$

This solids volume fraction, $\phi_{\text{base}}$, is the maximum solids volume fraction that the suspension will compress to, in the absence of gravity, even if compressed by the piston for an infinite time. Landman (Landman and White 1997) named this term $\phi_\infty$. If the compressive yield stress curve fit equation can be inverted, then $\phi_\infty$ can easily be calculated for any applied pressure, $\Delta P$, while an iterative search method is required when the equation can not be inverted.

Calculation of $\alpha$

Prediction of filtration behaviour requires the determination of the quantity $\alpha$, which is the solution to the following equation (Landman et al. 1999),

$$\chi = \left( \frac{1}{\phi^*} - \frac{1}{\phi_\infty} \right) = \pi^{0.5} \alpha e^{\alpha^2} Erf(\alpha), \quad (7.10)$$

such that $\phi^*$ is given by

$$\phi^* = \frac{\phi_\infty^2 D(\phi_\infty)}{\phi_\infty D(\phi_\infty) + \int_{\phi_0}^{\phi_\infty} D(\phi)d\phi} \quad (7.11)$$

Accurate determination of $\alpha$ can be difficult and involves an iterative numerical search. To aid in this search, Landman and White (Landman and White 1997) suggested the use of the
following approximate asymptotic solutions provided by Hill and Dewynne (Hill and Dewynne 1987) for use as initial guesses.

\[
\alpha = \left(\frac{2}{\chi + 2} + \frac{2}{3}\right)^{-0.5}; \chi < 3 \quad (7.12)
\]

\[
\alpha = \left(\ln\left(\frac{\chi + 1}{\sqrt{\pi}}\right) - \frac{1}{2} \ln \ln \left(\frac{\chi + 1}{\sqrt{\pi}}\right)\right)^{0.5}; \chi > 3 \quad (7.13)
\]

Where Equation 7.12 can be applied for \(\chi < 3\) and Equation 7.13 can be applied for \(\chi > 3\). Both equations produce reasonably significant prediction errors when \(\chi\) is between 2 and 4.

### 7.4.4.2 Filter Cake Formation

The time of filtration in the filter cake formation stage of filtration is determined by the equation,

\[
t = \frac{1}{\beta^2} V^2 \quad (7.14)
\]

assuming negligible membrane resistance, where \(t\) is the time of filtration and \(V\) is the specific volume of filtrate and \(\beta^2\) is a filtration parameter. Alternatively, Equation 7.14 can be rearranged to provide the extent of filtration using

\[
V = \beta \sqrt{t} \quad (7.15)
\]

The rate of filtration can be determined from the following relation

\[
\frac{dV}{dt} = \frac{\beta}{2\sqrt{t}} \quad (7.16)
\]

**Prediction of filtration parameter, \(\beta^2\)**

There are two methods of predicting \(\beta^2\) from the solids diffusivity of a particulate suspension. The first method uses an approximate integral, as shown below.

\[
\beta^2 = 2 \int_{\phi_0}^{\phi} \left(\frac{1}{\phi_0} - \frac{1}{\phi}\right) D(\phi) d\phi \quad (7.17)
\]
The second method is more accurate, but computationally difficult. First, \( \alpha \) must be calculated by solving Equation 7.10, and then applying Equation 7.18 below (Landman et al. 1999).

\[
\beta^2 = 4\Phi_0^2 D(\phi_\infty) \alpha^2 e^{-2\alpha^2} \left( \frac{1}{\phi_0} - \frac{1}{\phi^*} \right)^2
\]

*(7.18)*

**Prediction of average solids volume fraction in the filter cake, \( \phi_{\text{cake}} \)**

For a constant applied pressure, the average filter cake solids volume fraction, \( \phi_{\text{cake}} \), is predicted to be constant during filter cake formation, according to the following equation, derived from Equation B5 in Appendix B in Landman and White (Landman and White 1997),

\[
\phi_{\text{cake}} = \frac{1}{\frac{1}{\phi_0} - \left( \frac{1}{\phi_0} - \frac{1}{\phi^*} \right) e^{-2\alpha^2}}
\]

*(7.19)*

It should be noted that the solids volume fraction in the filter cake is not predicted to change with washing of the filter cake since the permeability is only expected to change by the ratio of the washed and unwashed liquor viscosities for all solids concentrations. However, filter cake washing does increase the solids weight fraction where liquor density changes are involved.

**Prediction of filter cake height, \( h_{\text{cake}} \)**

The height of the filter cake during filter cake formation is directly related to filtration parameters already derived according to the relation

\[
h_{\text{cake}} = \frac{\phi_0}{\phi_{\text{cake}} - \phi_0} V.
\]

*(7.20)*

### 7.4.4.3 Filter Cake Compression

**Scaled Filtration Time, \( T_f \)**

To enable simplification of some of the following equations, it is necessary to define a scaled time of filtration, \( T_f \), as a function of the time of filtration, \( t \), according to the following equation

\[
T_f = \frac{\phi_0^2 D(\phi_\infty)}{\phi^*_0 h_0^2} \cdot t.
\]

*(7.21)*

Conversely, the time of filtration, \( t \), is related to the scaled time of filtration, \( T_f \), according to the following equation
\[ t = \frac{\phi_0^2 h_0^2}{\phi^2 D(\phi_w)} T_f \]  

(7.22)

**Critical Filtration Time, \( t_c \)**

At a certain critical filtration time, \( t_c \), the filter cake reaches the piston and the average solids volume fraction of the suspension is equal to that of the filter cake during filter cake formation. This critical time, \( t_c \), can be calculated from the equation,

\[ t_c = \frac{\phi_0^2 h_0^2}{\phi^2 D(\phi_w)} T_c, \]  

(7.23)

where the scaled critical time of filtration, \( T_c \), is given by,

\[ T_c = \frac{1}{4\alpha^2}. \]  

(7.24)

**Determination of Extent of Filtration, \( V \)**

For the critical time onwards, an analytical solution to filter cake compression has been provided by Equation 102 in Landman and White (Landman and White, 1997). This equation can be transformed to give the extent of filtration, \( V \), as a function of the scaled time of filtration, \( T_f \), according to the following relation,

\[ V = h_0 \left(1 - \frac{\phi_0}{\phi_w}\right) \phi_0 h_0 \left(\frac{1}{\phi*} - \frac{1}{\phi_w}\right) \sum_{n=0}^{\infty} \frac{F_n}{(n+1/2)\pi} e^{-\frac{1}{4}(n+1/2)^2\pi^2(T_f - T_c)}, \]  

(7.25)

where, the \( F_n \) term is defined as,

\[ F_n = \frac{4}{\pi^{3/2}} \left(\frac{\alpha}{(n+1/2)\text{erf}(\alpha)}\right) \int_0^\alpha \cos \left(\pi z \right) e^{-z^2} dz. \]  

(7.26)

**Single Term Approximation**

Landman and White (Landman and White 1997) suggest that when,

\[ T_f - T_c >> \frac{4}{9\pi^2}, \]  

(7.27)

an approximation using only the first \( F_0 \) term in the infinite series yields sufficient accuracy. This gives the extent of filtration, \( V \), as a function of the scaled time of filtration, \( T_f \), according to the following equation,
\[ V = h_0 \left( 1 - \frac{\phi_0}{\phi_\infty} \right) - \phi_0 h_0 \left( \frac{1}{\phi^*} - \frac{1}{\phi_\infty} \right) \frac{2F_0}{\pi} e^{-\left(\frac{\pi}{2}\right) \left( r_f - r_i \right)}, \quad (7.28) \]

where \( F_0 \) is given by,

\[ F_0 = \frac{8}{\pi^{3/2}} \text{erf}(\alpha) \int_0^\alpha \cos \left( \frac{\pi z}{2\alpha} \right) e^{-z^2} dz \quad (7.29) \]

Equation 7.28 can be transformed to give the scaled filtration time, \( T_f \), as a function of extent of filtration, \( V \), according to the equation,

\[ T_f = T_c + \left( \frac{2}{\pi} \right)^2 \ln \left( \phi_0 h_0 \left( \frac{1}{\phi^*} - \frac{1}{\phi_\infty} \right) \frac{2F_0}{\pi} \right) - \left( \frac{2}{\pi} \right)^2 \ln \left( h_0 \left( 1 - \frac{\phi_0}{\phi_\infty} \right) - V \right). \quad (7.30) \]

This can be transformed to give the filtration time, \( t \), as a function of extent of filtration, \( V \), according to the equation,

\[ t = t_c + \frac{4\phi_0^2 h_0^2}{\pi^2 \phi_\infty^2 D(\phi_\infty)} \ln \left( \phi_0 h_0 \left( \frac{1}{\phi^*} - \frac{1}{\phi_\infty} \right) \frac{2F_0}{\pi} \right) - \frac{4\phi_0^2 h_0^2}{\pi^2 \phi_\infty^2 D(\phi_\infty)} \ln \left( h_0 \left( 1 - \frac{\phi_0}{\phi_\infty} \right) - V \right). \quad (7.31) \]

**Iterative Determination of Time of Filtration, \( t \)**

Sometimes, it may be necessary to calculate the time of filtration, \( t \), directly from the extent of filtration, \( V \), with high accuracy. However an explicit equation of this nature does not exist when many terms in the Fourier series are used. Instead, a self focusing iterative method is required. The method involves using Equation 7.30 as an initial guess for \( T_f \), followed by repeated iterative calculation of the time of filtration, \( t \), from the following equation,

\[ T_f = T_c - \frac{4}{\pi^2} \ln \left( \frac{\pi}{2F_0} \right) - \frac{4}{\pi^2} \ln \left( \frac{h_0 \left( 1 - \frac{\phi_0}{\phi_\infty} \right) - V}{\phi_0 h_0 \left( \frac{1}{\phi^*} - \frac{1}{\phi_\infty} \right)} \right) - \sum_{n=1}^{\infty} \frac{F_n}{(n+1/2)\pi} e^{-\left(\frac{n+1/2}{\pi}\right)^2 \left( r_f - r_i \right)}, \quad (7.32) \]

until the required accuracy is achieved.

**Discussion**

Filtration compression calculations can be computationally expensive if a large number of terms in the Fourier series are used. The optimum number of terms to use is a balance between computational time and accuracy. Generally a large number of terms, 10-100, is required for accuracy at the start of cake compression, but only one term is required towards the end of cake.
compression. Therefore, the single term approximations given by Equations 7.28 and 7.31 provide significant computational simplification. However, caution should be used when filtration is in the initial stages of compression, even if $T_f - T_c$ is greater than $4/(9\pi^2)$, because the required condition ($\gg$) implied in Equation 7.27 is rather subjective. The one term approximation has been found to produce predictions of $dV/dt$ and $dt/dV^2$ in error by up to 25% at the critical time, $t_c$, when bed formation ceases and bed compression commences. This error has been found to reduce to less than 0.3% in $dV/dt$ and $dt/dV^2$ predictions by using the first 101 terms in the infinite series. The first 30 terms are sufficient if only the time of filtration, $t$, is required to be accurately predicted.

### 7.4.4.4 Rotary Drum Filtration

Rotary drum filtration theory is directly related to the filter cake formation theory already described, with a few subtle variations relating to its nature as a continuous process rather than a batch process. The filtration parameter $\beta^2$ and average filter cake solids volume fraction are calculated as described in section 7.4.4.2. However, calculation of extent of filtration, solids throughput and extent of washing requires appreciation of the filtration cycle as discussed below.

**Cycle Time**

At any time in a rotary drum filter, fractions of the filter cloth area are in different modes of operation. These fractions include a fraction where development of the filter cake occurs through vacuum filtration of the feed, $f_{submerged}$, and a fraction where the filter cake is washed under vacuum with a clean liquor, $f_{washing}$. There is sometimes a mechanical squeezing mechanism at some stage in the cycle. In the other fractions of the cycle the filter cake may be drying under vacuum through air permeation. At the end of the cycle, there is always a cake removal mechanism using either positive pressure or a fixed blade.

Development of the filter cake occurs while the filter cloth is submerged in feed suspension and subject to a vacuum pressure. The total cycle time, $t_{cycle}$, is related to the submerged filtration time, $t_{submerged}$ and the fraction of filter cloth that is submerged, $f_{submerged}$, by the following,

$$ t_{cycle} = \frac{t_{submerged}}{f_{submerged}}. $$

(7.33)

The total cycle time, $t_{cycle}$, is also related to the washing filtration time, $t_{washing}$, and the fraction of filter cloth that is subjected to washing liquor at any time, $f_{washing}$, by the following,
Solids Throughput Predictions

Given the filter cloth area, \( A_\text{cloth} \), and the average filter cake solids volume fraction, \( f_\text{cake} \), rotary drum filter solids throughput (kg s\(^{-1}\)) is now easily calculated using,

\[
S\text{olids Throughput} = \frac{\rho_\text{solids} f_\text{cake} h_\text{cake} A_\text{cloth}}{t_\text{cycle}}.
\] (7.35)

Extent of Washing Predictions

Rigorous modelling of filter cake washing requires an estimate of the cloth area subjected to wash water under vacuum in addition to the temperature, density and viscosity of the wash water as well as properties of the original filter cake. Reliable prediction of the extent of washing is beyond the scope of this analysis. So instead, an approximate prediction is suggested, where the washing liquor is assumed to have the same physical properties (viscosity and density) as the feed suspension liquor. The following equation provides an initial guess of the washing rate using the rate of filtration at the end of the bed formation time period.

\[
W\text{ash Water Throughput} = \frac{\rho_\text{water} A_\text{cloth washed}}{2} \left( \frac{\sqrt{\beta}}{t_\text{submerged}} \right)
\] (7.36)

7.4.5 VALIDATION OF FILTRATION THEORY

A zirconia suspension in 0.01 M KNO\(_3\) at pH 6.8, with an initial solids volume fraction, \( \phi_0 = 0.0731 \), was prepared by de Kretser (de Kretser et al. 2001a; de Kretser et al. 2001b) according to the method described in section 6.5.5.1. de Kretser characterised the dewateribility of this suspension using stepped pressure filtration at pressures ranging from 5 to 300 kPa, fitted curves to the dewaterability data and then conducted a single pressure filtration experiment at 100 kPa, with an initial solids volume fraction, \( \phi_0 = 0.1316 \), and an initial suspension height, \( h_0 = 0.02122 \) m. To demonstrate the validity of the filtration equations described by Landman and White (Landman and White 1997), code written for the software application Mathematica was employed to simulate \((t, V)\) filtration behaviour based on the dewaterability curve fits with, \( \phi_0 = 0.1316 \), \( h_0 = 0.02122 \) m and \( P = 100 \) kPa. The simulation used Equation 7.18 to calculate \( \beta^2 \) for the filter cake formation stage and Equation 7.25 with 101 terms in the Fourier series for the filter cake compression stage. The \((t, V)\) filtration data from both the filtration experiment and filtration simulation are presented in Figure 7.3. There are differences between the simulation
and the experimental data at low times, but this is due to the time required for the applied pressure to reach the filtration set pressure of 100 kPa in the experimental filtration test. Apart from the early differences, comparison shows that very close agreement is obtained between the simulated and experimental filtration data. Another point of significance is that the accurate filtration simulation data was generated for a suspension with \( \phi_0 = 0.1316 \), but based on dewaterability data obtained from stepped pressure filtration tests conducted on a suspension with \( \phi_0 = 0.0731 \). This helps to validate the hypothesis that dewaterability data are indeed material properties and not just empirical parameters.

Figure 7.3: Comparison \( t \) versus \( V^2 \) data from a filtration simulation and a filtration experiment based on a zirconia suspension in 0.01 M KNO\(_3\) at pH = 6.8, with \( \phi_0 = 0.1316 \), \( h_0 = 0.02122 \) m and with an applied pressure of 100 kPa. Reproduced from de Kretser et al. (de Kretser et al. 2001a; de Kretser et al. 2001b).

7.4.6 OPTIMISATION

There are many ways in which filtration processes can be optimised, many of which have been developed through industry experience over many years. Some of these methods are clearly justifiable through improvements gained, but are quite empirical in their application. The fundamental basis of the filtration solution presented above can enable evaluation of the merits of given process conditions and determine if they actually do optimise process performance. Clearly, subject to the limitations of the model, there are some aspects of process operation that the model does not predict well and thus cannot be optimised with this theory. As noted in the
assumptions, the most significant limitations include an inability to predict variable pressure filtration, membrane fouling, membrane resistance and the influence of gravity. Caution should be used when applying the Landman and White (Landman and White 1997) filtration solution when these variables are significant. Process variables that can be examined include:

- Solids throughput
- Liquor recovery
- Final solids concentration
- Time of filtration
- Filtration surface area
- Filtration pressure
- Compressibility and permeability
- Type of flocculant and flocculation conditions.
- Operating temperature

The hope is that prediction of filtration behaviour as a function of process variables can help to demystify observations of complex filtration behaviour.

7.4.7 CONCLUSIONS

The practical application of the linearised filtration theory developed by Landman and White (Landman and White 1997) has been detailed for piston driven filtration and for rotary drum filtration assuming gravitational effects are insignificant. The theory has been converted into code via a computer software program called Mathematica, enabling timely simulation of pressure filtration from dewaterability data. The validity of the theory has been demonstrated by comparison of simulated filtration data with experimental filtration data and some directions for process optimisation have been proposed. Rotary drum filtration modelling has been applied to the operation of a red mud filter at the Worsley refinery in Chapter 10.

7.5 STEADY STATE THICKENER MODELLING

7.5.1 INTRODUCTION

A modelling tool has been developed to facilitate prediction of steady state thickener performance using curve fits of experimentally determined dewaterability data with the fundamentally based mathematical theory of Buscall and White (Buscall and White 1987). The technique predicts the steady state solids throughput for a given feed solids concentration and thickener dimensions as a function of suspension bed height and underflow solids concentration.
7.5.2 ASSUMPTIONS

As with all process models, there are a number of assumptions and these should be kept in mind when utilising the model output. The assumptions are as follows:

- The model used is one dimensional.
  It is converted to 2-dimensional model by the use of a shape factor, so the model does not account for short circuiting and mixing. This implies that only vertical dewatering is predicted and horizontal flow of liquor is ignored. As a result, non-isotropic permeability variations are not accounted for.

- The model assumes line settling.
  This implies that settling rate and permeability are functions of solids volume fraction and all solid particles at the same height settle at the same rate, with no particle size segregation.

- The model does not account for shearing.
  Shear forces in the thickener, caused by the action of rakes and shear rods, are expected to improve thickening. However wall friction which can slow consolidation is also not accounted for.

- The model assumes that no solids exit via the overflow.
- The model assumes steady state thickener operation.

7.5.3 INPUTS

The inputs required for the steady state thickener model include:

- A compressive yield stress, \( P_y(\phi) \), curve fit.
- A hindered settling function data, \( R(\phi) \), curve fit.
- The thickener dimensions, including diameter as a function of height, \( d(z) \), such as that shown in Figure 7.4 below.
- The feed solids concentration, \( \phi_0 \).
- Solid and liquor densities, \( \rho_{sol} \& \rho_{liq} \).
Figure 7.4: Schematic of a converging base thickener, with truncated cone, showing height in the thickener, \( z \), the suspension bed height, \( hb \), and the maximum thickener diameter, \( d_{\text{max}} \).

### 7.5.4 CALCULATIONS

Steady state thickener modelling involves prediction of the solids throughput as a function of underflow solids concentration and suspension bed height in two parts. The first part deals with free settling (generally called clarification) while the second part considers compression in the suspension bed (thickening). The free settling and compression predictions are combined to predict the steady state solids flux by taking the minimum predicted solids flux for each underflow solids concentration. It is important to note that in all of the following equations (unless otherwise specified), the solids flux, \( q \), is defined as a volume of solids per unit time per thickener cross sectional area, where the cross sectional area referred to is the maximum for the thickener in question. However, to adhere to industry conventions, all graphs of solids flux are presented in tonnes hour\(^{-1} \) m\(^{-2} \), while all graphs of solids throughput are presented in tonnes hour\(^{-1} \).

#### 7.5.4.1 Free Settling

Coe and Clevenger (Coe and Clevenger 1916) proposed that the settling rate is a function of solids concentration as long as no mechanical support is contributed from layers of suspension below (Fitch 1966). They called this condition free settling. Indeed, using the theory of Buscall and White (Buscall and White 1987), the free settling rate, \( u_{fs}(\phi) \), of a suspension in the absence of a compressive yield stress influence is predicted to be a function of the solids volume fraction, \( \phi \), according to Equation 3.11.
The traditional Coe and Clevenger method suggests that this settling rate be used in a material balance to determine the thickener steady state solids flux (for the maximum thickener cross sectional area), $q$, for suspension at any solids concentration, $\phi$, for a given underflow solids concentration, $\phi_u$ (Fitch 1966).

$$u_f(\phi) = \frac{\Delta p g (1 - \phi)^2}{R(\phi)}.$$ \hspace{1cm} (3.11)

$$q = \frac{u_f(\phi)}{\left( \frac{1}{\phi} - \frac{1}{\phi_u} \right)}$$ \hspace{1cm} (7.37)

A thickener shape factor, $\alpha(z)$, defined as,

$$\alpha(z) = \left( \frac{d(z)}{d_{\text{max}}} \right)^2,$$ \hspace{1cm} (7.38)

accounts for the cross sectional area variations with height in the thickener, $z$, such that $\alpha = 1$ when the thickener diameter, $d(z)$, is at its maximum, $d_{\text{max}}$, and the cross sectional area is also maximised. With the addition of $\alpha(z)$, the material balance equation becomes,

$$\frac{q}{\alpha(hb)} = \frac{u_f(\phi)}{\left( \frac{1}{\phi} - \frac{1}{\phi_u} \right)},$$ \hspace{1cm} (7.39)

where $hb$ is the suspension bed height for a converging base or flat based thickener.

For a given underflow solids concentration, $\phi_u$, and suspension bed height, $hb$, the maximum thickener capacity possible in free settling will be the minimum value of solids flux, $q$, obtained by applying the material balance for all solids concentrations, $\phi$, ranging from $\phi_0$ to $\phi_u$.

### 7.5.4.2 Compression

The underflow solids concentration predicted by free settling is often not achieved because suspension compressibility also limits the underflow solids concentration. Traditional methods of predicting compression in the suspension bed are fundamentally flawed in the way they empirically account for suspension compressibility. The technique used in this analysis involves integration of a one dimensional differential equation developed from fundamental dewaterability theory (Green 1997). The differential equation, shown below,
\[ \frac{d\phi(z)}{dz} = \frac{R(\phi(z))}{(1-\phi(z))^2} \frac{q}{\alpha(z)} \left( \frac{1-\phi(z)}{\phi_u} \right) - \Delta \rho g \phi(z) \], \tag{7.40} \]

relates the change in solids concentration with height in the thickener, \( d\phi(z)/dz \), for a given steady state solids flux, \( q \), and underflow solids concentration, \( \phi_u \). The change in thickening cross sectional area with height, \( z \), in the thickener is accounted for through the use of the shape factor, \( \alpha(z) \). At steady state, the solids concentration is equal to the gel point, \( \phi_g \), at the top of the suspension bed, \( z = hb \), and is equal to the given underflow solids concentration, \( \phi_u \), at the base of the thickener, \( z = 0 \).

To determine the solids flux, \( q \), required to produce a steady state suspension bed height, \( hb \), and underflow solids concentration, \( \phi_u \), the differential equation is integrated from the bottom to the top of the suspension bed, \( z = 0 \) to \( hb \), subject to the boundary condition,

\[ \phi(0) = \phi_u, \] \tag{7.41} \]

using an initial guess for, \( q \). The solids flux guess is iteratively adjusted through repeated integrations, until the solids concentration at the top of the bed equals the gel point,

\[ \phi(hb) = \phi_g. \] \tag{7.42} \]

The integration method is repeated for a number of underflow solids concentrations, \( \phi_u \), and suspension bed heights, \( hb \), to produce curves of \( q \) versus \( \phi_u \), for a number of \( hb \) values generally ranging from 10 cm to 20 m.

### 7.5.4.3 Combining Results

The free settling and compression predictions are combined to predict the limiting steady state solids flux by taking the minimum predicted solids flux for each underflow solids concentration as shown in Figure 7.5 below.
Figure 7.5: Combining free settling and compression predictions to predict the limiting steady state solids flux.

7.5.5 OUTPUTS

7.5.5.1 Solids Flux

An algorithm has been programmed in Mathematica code to take the input data and determine the steady state solids flux for a range of underflow solids concentrations and suspension bed heights. The resultant graph is presented in Figure 7.6. The data illustrate the expected underflow solids achievable for a given solids flux through the thickener (flux is calculated from the solids throughput per unit cross sectional area of the vessel at the top of the suspension bed). The effect of different suspension bed heights on the predicted underflow solids concentrations is illustrated. The gel point and feed solids concentration are also illustrated. The operation of a thickener may be broken into two distinct regions, each of which will be discussed in turn.
Figure 7.6: Typical steady state converging base thickener model prediction of the solids flux as a function of underflow solids volume fraction for a range of suspension bed heights.

**Permeability Limited Operation**

For moderate to high solids fluxes, > 0.007 t hr⁻¹ m⁻² in this example, thickener operation is described as permeability limited. This implies that the rate at which the solids are passed through the vessel is so fast that no transmission of compressive forces is achieved in the suspension bed. The limiting factor is the rate at which the liquid is able to escape from the solids network, which is dictated expressly by the permeability. Therefore, the model predicts that in permeability limited operation, the steady state underflow solids concentration is a function of solids flux alone, and not influenced by suspension bed height. Indirectly however, the suspension bed height may also influence the effectiveness of raking (not accounted for in the model) that also influences the underflow solids concentration.

**Compressibility Limited Operation**

At low to intermediate solids fluxes, < 0.007 t hr⁻¹ m⁻² in this example, the suspension bed height is observed to have an effect on the underflow solids concentration. In this region, the residence time of the solids in the suspension bed is long enough for compressive dewatering to occur and, as such, the amount of compressive force transmitted by the network structure of the suspension bed is the dominant effect that governs the underflow solids concentration.
In the limit of zero solids flux, the suspension will settle to equilibrium and the underflow solids concentration will be a function of steady state bed height only, dictated by the compressive force generated and the compressive yield stress behaviour for the material in question.

7.5.5.2 Solids Throughput

For a flat bottomed thickener, multiplying the solids flux by the cross sectional area of the thickener yields the solids throughput. Therefore, for a flat bottomed thickener, a plot of solids throughput (tonnes hr$^{-1}$) versus underflow solids concentration has the same shape as its solids flux counterpart (tonnes hr$^{-1}$ m$^{-2}$), with the same suspension bed height effects, just with the y-axis re-scaled. In the permeability-limited region of operation, for solids throughput and solids flux, the underflow solids concentration is predicted to be insensitive to the suspension bed height. In this region of operation, the model suggests that the only options available for changing the underflow solids concentration are either to reduce the solids flux (not practical) or to improve the permeability of the material in the low solids region, below the gel point.

For a converging base thickener, solids throughputs are determined by multiplying the solids fluxes by the cross sectional area of the thickener at the applicable suspension bed heights. The converging base thickener solids flux curves shown in Figure 7.6 have been replotted as solids throughput curves in Figure 7.7. The curves have separated to account for the variation in cross sectional area at the top of the suspension bed for the variation in suspension bed heights (up to 7 m in Figure 7.7). For a converging base thickener, the increasing cross sectional area as height in the suspension bed increases, has the implication that when the top of the suspension bed lies within the conical region, the solids throughput is a function of bed height even when the process is permeability limited. Therefore, for converging base thickeners, the solids throughput is a function of suspension bed height in situations where solids flux is not. However, for higher suspension bed heights, where thickener diameter does not vary with suspension bed height, the bed height effect is only observed for very low solids fluxes.
7.5.5.3 Solids residence Time

The steady state thickener model predictions may also be used to calculate the effective residence time of solids in the bed of a thickener, $t_{res}$, according to the following equation,

$$ t_{res} = \frac{1}{q_u} \int_0^{h_b} \alpha(z) \phi(z) dz . $$  \hspace{1cm} (7.43)

Figure 7.8 shows a typical bed solids residence time versus underflow solids concentration for a thickener based on model predictions. The type of data presented in Figure 7.8 gives insight into the interaction between underflow solids concentration and the existence of liquor stability issues and whether precipitation may be expected to be a problem in a given system. Note that the predicted residence times are impractically high, even for solids concentrations that are only just above the gel point. However, if solids concentrations up to 0.25 are achievable in practice, then the predicted residence times of 10-100 hours to reach these solids, suggested by Figure 7.8 seem unreasonable. This discrepancy will be discussed later in terms of comparison of the model predictions with real observations.
Figure 7.8: Typical steady state thickener model prediction of the solids residence time in the suspension bed as a function of underflow solids volume fraction for a range of suspension bed heights.

7.5.5.4 Fractional Liquor Recovery

A liquor material balance can be used to indicate the fractional liquor recovery, \( f_{lr} \), in a thickener (or any other dewatering operation for that matter) as a function of the underflow solids concentration. The fractional liquor recovery is defined as the liquor overflow flowrate divided by the total flowrate of liquor fed to the washer, assuming that all solids leave via the underflow and can be calculated from the following equation,

\[
f_{lr} = \frac{\phi_u - \phi_0}{\phi_u (1 - \phi_0)}.
\]  (7.44)

Figure 7.9 shows the fractional liquor recovery as a function of feed solids concentration, for the thickening example being used. For underflow solids volume fractions less than 0.09 (corresponding to the condition of high overall solids flux) the liquor recovery is low, below 50 \%. This indicates an impractical region of operation due to the free settling rate of the feed material. Alternatively, when underflow solids volume fractions greater than 0.30 are achieved, liquor recovery is over 90 \% and cannot be improved much, even with significantly higher underflow solids concentrations. As can be demonstrated, considering the fractional liquor recovery can sometimes put underflow solids concentrations into context by giving an alternative measure of dewatering efficiency to underflow solids volume fraction.
Figure 7.9: Typical steady state thickener model prediction of liquor recovery fraction as a function of the underflow solids volume fraction.

7.5.6 OPTIMISATION

It should be stressed that the model predictions are subject to limitations relating to the idealised nature of the model. The absence of the ability to account for shear, dead-zones and unstable operation mean that suggestions of process optimisation options available should be treated with caution. Also, considering that the compressibility and permeability data cover up to five orders of magnitude, the outputs of the model are very sensitive to measured and curve fitted permeability values. As a result, the model predictions are not applicable to extremely accurate prediction of thickener performance, however, they do improve understanding of why certain underflow concentrations are achieved, the trends involved and how performance can be improved by adjustment of process variables. Process variables that can be examined include:

- The influence of feed solids concentration, solids flux and thickener dimensions.
- Whether improvements in compressibility or permeability will be most beneficial.
- How sensitive the underflow solids concentration is to process variations.
- The influence of type of flocculant and flocculation conditions.
- The impact of upstream processing options such as coarse/fine separation or blending.
- The impact on downstream options such as management of tailings impoundments and water recovery.
• The response in terms of water recovery and underflow density to all of the suggestions above.

7.5.7 CONCLUSIONS
A useful steady state thickening model calculation algorithm has been developed by combining free settling rate theory (Fitch 1966) with suspension bed consolidation theory (Green 1997). The theory has been developed for both flat bottomed thickeners and converging base thickeners through the use of a shape factor (Green 1997). The theory has been converted into mathematical code via a computer software program called Mathematica, enabling timely simulation of steady state thickening from dewaterability data. Generic outputs of the model have been explained and interpreted and some directions for process optimisation have been proposed. The theory and software is applied through the prediction of dewatering in red mud thickeners and washers in Chapters 9, 10 and 11, where the predictions are compared with actual plant performance. The usefulness of the model and the influences not accounted for by the model are discussed in Chapter 13.

7.6 TRANSIENT CONSOLIDATION MODELLING

7.6.1 INTRODUCTION
A modelling tool has been developed to facilitate prediction of transient consolidation in batch processes using curve fits of experimentally determined dewaterability data in the fundamentally based mathematical theory of Buscall and White (Buscall and White 1987). The technique predicts the suspension height as a function of time for a given initial solids concentration and suspension height, for initial solids concentrations that are above the gel point. This modelling tool uses the method proposed by Howells et al. (Howells et al. 1990), but with a slightly different force balance (a factor of (1 - \(\phi\))) and modified boundary conditions.

7.6.2 ASSUMPTIONS
As with all process models, there are a number of assumptions and these should be kept in mind when utilising the model. The assumptions are as follows:

• The model used is one dimensional (see section 7.5.2).
• The model assumes line settling (see section 7.5.2).
• The model does not account for shearing (see section 7.5.2).
• The model assumes zero solid and liquor velocity at the base of the suspension.
This implies that nothing passes through the base of the suspension.

- The model assumes no time or shear history dependent rheological behaviour.
  It does not account for thixotropy, such as is observed for stagnant red mud suspensions.

### 7.6.3 INPUTS

The inputs required for the transient consolidation model include:

- A compressive yield stress data, \( P_y(\phi) \), curve fit.
- A hindered settling function data, \( R(\phi) \), curve fit.
- The initial suspension height, \( h_0 \).
- The initial solids volume fraction, \( \phi_0 \).
- Solid and liquor densities, \( \rho_{sol} \) & \( \rho_{liq} \).

### 7.6.4 CALCULATIONS

Transient consolidation modelling involves prediction of the suspension height as a function of time, by considering compression in the suspension bed enabled through the upward flow of liquor to the suspension surface. The initial settling rate, \( u_0 \), can be calculated from the initial suspension height, \( h_0 \), and the initial solids volume fraction, \( \phi_0 \), according to Equation 6.29,

\[
   u_0 = \frac{\Delta \rho g (1 - \phi_0)^2}{R(\phi_0)} \left( 1 - \frac{P_y(\phi_0)}{\Delta \rho g \phi_0 h_0} \right). \tag{6.29}
\]

The behaviour represented in this equation is predicted by Howells et al. (Howells et al., 1990) for small times based on the fundamental mathematical theory of Buscall and White (Buscall and White, 1987). Very soon however, the settling rate is slowed by suspension compressibility and numerical integration is required to predict consolidation. The technique proposed in this analysis involves integration of a set of coupled one dimensional differential equations developed from fundamental dewaterability theory (Howells et al. 1990), with a \((1 - \phi)\) correction. The first differential equation (from Equation 3.19), relates the change in solids concentration with height from the base, \( d\phi(z)/dz \), as a function of solids flux, \( q(z) \), as shown below,

\[
   \frac{d\phi(z)}{dz} = \frac{R(\phi(z))}{(1 - \phi(z))^2} q(z) - \Delta \rho g \phi(z) \frac{dP_y(\phi(z))}{d\phi(z)}. \tag{7.45}
\]
The second differential equation (from Equation 3.17), describes the solids flux continuity condition for discretised time steps, \( \Delta t \), as shown below,

\[
\frac{\partial q(z)}{\partial z} = \frac{\phi(z) - \phi_p(z)}{\Delta t},
\]

where \( \phi_p(z) \) is the solids volume fraction as a function of height from the base for the previous time, \( t_p = t - \Delta t \). Combined, this set of coupled partial differential equations defines the consolidation dynamics of a suspension bed in a batch settling operation.

To determine the solids volume fraction, \( \phi(z) \), and solids flux, \( q(z) \), at progressive times, \( t \), incremented by time steps, \( \Delta t \), the coupled differential equations are integrated from the base of the suspension, \( z = 0 \), to a critical height, \( z = z_c \), subject to two boundary conditions. Howells et al. (Howells et al. 1990) proposed that the integration be performed subject to the boundary conditions of an initial guess for the solids concentration at the base of the suspension and zero solids flux at the base of the suspension. However, iterative determination of the solids concentration at the base of the suspension for every time step proved to be unreliable. The problem was that the solids concentration needed to be determined to such an extreme accuracy that it became computationally impossible to complete most simulations. Using the same differential equations, but integrating from \( z_c \) down, with iterative guesses of \( z_c \) has proved to be more robust, subject to the boundary conditions that \( z_c \) is the lowest height at which,

\[
\phi(z_c) = \phi_0
\]

and

\[
q(z_c) = \frac{\Delta \rho g (1 - \phi_0)^2}{R(\phi_0)} \left( 1 - \frac{P_y(\phi_0)}{\Delta \rho g \phi_0 (h - z_c)} \right),
\]

using an initial guess for, \( z_c \). In Equation 7.48 above, the suspension height, \( h \), is estimated using,

\[
h = h_p - q_p(z_{cp}) \Delta t.
\]

where, \( h_p \) is the suspension height at the previous time step and \( q_p(z_{cp}) \) is the solids flux at the critical height at the previous time step. Guesses for \( z_c \) are iteratively adjusted until the condition of no flow at the base of the suspension,

\[
q(0) = 0,
\]
is satisfied. The true suspension height is then calculated more accurately using,

\[ h = h_0 + z_c - \frac{1}{\phi_0} \int_0^{z_c} \Phi(z)dz. \]  

(7.51)

The algorithm is then repeated for subsequent time steps, until the suspension height, \( h \), no longer changes with time.

### 7.6.5 OUTPUTS

An algorithm has been programmed in Mathematica code to take the input data and determine the transient consolidation of a suspension with an initial solids concentration above the gel point. The output data consists of a computer file that contains the suspension height and critical height for all consolidation times that were being modelled. The other output data file contains solids volume fraction as a function of height in the suspension for selected consolidation times. The outputs can be presented as plots of suspension height and critical height as functions of time, as shown for a red mud tailings suspension in Figure 11.25. An alternative method of presenting the output results is as a plot of suspension height versus solids volume fraction for selected consolidation times, as shown for a red mud tailings suspension in Figure 11.26. The predicted consolidation has three stages. There is an initial constant settling rate period where the permeability of the suspension at the initial solids concentration dominates. This is followed by a continuous slowing of the settling rate dictated by both compressibility and permeability considerations. Finally, as consolidation slows, the suspension height approaches an equilibrium suspension height, dictated by the compressibility of the suspension alone.

### 7.6.6 APPLICATIONS

#### 7.6.6.1 Tailings Dam Consolidation Modelling

As a demonstration, the consolidation of red mud suspension in a residue disposal area has been modelled in Chapter 11, section 11.5. The current limitation of the calculation algorithm is that it only works for initial solids concentrations above the gel point. If the calculation algorithm proves to be robust, small modifications to the coding may allow consolidation of suspensions with initial solids concentrations that are below the gel point. In addition permeation through sand layers in the base of the suspension could potentially be accounted for in the model through modified boundary conditions.
7.6.6.2 Transient Thickener Modelling
Modification of the calculation algorithm could allow prediction of transient thickener operation, enabling improved understanding of the time-scales involved with process variations and operational sensitivities. This will be achievable if the calculation algorithm proves robust enough to handle feed solids concentrations below the gel point in addition to the extra moving boundary condition.

7.6.6.3 Dewaterability Characterisation from a Single Batch Settling Test
One of the most valuable potential applications of transient consolidation modelling would be the ability to extract more low solids concentration dewaterability data from a single batch settling test. Achieving this would involve the following procedure:

- Measure the height versus time data for a batch settling test.
- Estimate the curve fits for \(P_y(f)\) and \(R(f)\), using only pressure filtration test data, the initial settling rate and the final suspension bed height as guides.
- Simulate the batch settling test.
- Compare the simulation results and provide a new guess for the \(P_y(f)\) and \(R(f)\) curve fits.
- Repeat the simulation of the batch settling test and adjustment of the \(P_y(f)\) and \(R(f)\) curve fits until the simulation matches the experimental data.

Initial implementation of this idea requires that the calculation algorithm allows feed solids concentrations that are below the gel point. After that, practical application of this tool would require that results could be produced in real time. The limitation to producing results in real time is that each settling simulation presently requires a number of hours of calculation time. Therefore, the number of \(P_y(f)\) and \(R(f)\) curve fit iterations might need to be limited, creating a compromise between accuracy and speed.

7.6.7 OPTIMISATION
Again it should be stressed that model predictions are subject to limitations. The absence of the ability to account for time dependent thixotropic behaviour, non-vertical liquor flows and drainage into sand layers below the suspension are significant limitations. However, predictions of the rate and extent of consolidation can still enable optimisation improvements in a wide range of scenarios. The model predictions are not applicable to extremely accurate prediction of consolidation; however, they do improve understanding of why certain levels of consolidation are achieved, the trends involved and how consolidation and liquor recovery can be improved by adjustment of process variables. Process variables that can be examined include:
• feed solids concentrations,
• suspension bed heights,
• the ratio of suspension disposal rates to tailings dam area (analogous to solids flux)
• compressibility and permeability changes due to variations in upstream flocculation conditions and processing conditions such as coarse/fine particle size separation or blending.

7.6.8 CONCLUSIONS

A useful transient consolidation model calculation algorithm has been developed by subtle modification of a method proposed by Howells et al. (Howells et al. 1990). The algorithm has been converted into Mathematica code, enabling simulation of transient consolidation from dewaterability data. Some potential real world applications of the model have been suggested, with most requiring some modifications to the algorithm. Potential areas where this tool could be used for process optimisation have been proposed. Finally, the theory and software is applied through the prediction of red mud consolidation in Chapter 11.
SECTION C

EXPERIMENTAL CHARACTERISATION AND MODELLING RESULTS
Chapter 8

RED MUD CHARACTERISATION WORK PROGRAM

8.1 INTRODUCTION

Red mud washer train suspensions from the ALCOA, Pinjarra Alumina Refinery, the Worsley Alumina Refinery and the Queensland Alumina (QAL) Refinery have been characterised. The primary purpose of the characterisations was to determine the differences in dewaterability down each washer train across all sites and to gain an understanding of the variables that influence dewaterability. The suspension samples were characterised in terms of their shear rheological properties and dewaterability. Modelling of the operation of separation process equipment was also performed. This chapter overviews the characterisation work program that was undertaken.

8.2 BAUXITE RESIDUE WASHER TRAINS

The initial stages of work included developing familiarisation with the entire process of washing and dewatering of red mud suspensions at each of the three sponsor refineries. This involved site visits and audits of how process equipment, process variables and material properties varied from the thickeners and settlers to the washers and subsequently to the residue disposal areas at each of the sponsor sites. An overview of the washing and dewatering process at each sponsor site is presented in the introductions of Chapters 9, 10 and 11.

During the first set of site visits, which took place in 1998, process equipment overflow liquors were collected from every processing stage and transported to Melbourne for analysis. Properties measured included the pH, electrical conductivity and density of the liquors. This work provides an overview of the effectiveness of washing at every process stage and results are presented in Chapter 12, section 12.3.

Chapters 9-12 provide significant amounts of process information and material properties from each sponsor refinery. However, in some instances key process information has been deemed commercially sensitive. Therefore, for confidentiality reasons, there are numerous instances where process information is referred to but can not actually be presented.
8.3 RED MUD STABILITY TESTING

At the first set of site visits, which took place in 1998, process equipment underflow suspensions were collected from every processing stage and transported to Melbourne for analysis. Properties measured included the pH, electrical conductivity and density of the liquors. The data gave an overview of the effectiveness of washing at every process stage. In addition, suspension dewaterability was characterised at ambient temperatures. The suspensions had changed significantly during transportation due to liquor instability and thus the results are considered unreliable. Later, in Chapter 12 section 12.5.3, the ambient dewaterability data produced in Melbourne in 1998 is compared with data produced on site at process temperatures in 1999.

8.4 SHEAR RHEOLOGY CHARACTERISATION

At the second set of site visits, which took place in 1999, process equipment underflow suspensions were collected from every processing stage. The shear rheology was characterised at process temperatures in terms of the shear yield stress and shear stress versus shear rate behaviour at underflow solids concentrations. These characterisations, presented for each site in sections 9.2.4, 10.2.4 and 11.2.4 enable an understanding of process equipment underflow rheology trends from the start to the end of each washing and dewatering process. The influence of temperature on shear rheology was also investigated in a number of ways. The knowledge gained about the shear rheology of red mud suspensions is summarised in Chapter 12, section 12.4.

8.5 DEWATERABILITY CHARACTERISATION

Using the process equipment underflow suspensions collected from every processing stage in 1999, suspension dewaterability was characterised at process temperatures in terms of the compressive yield stress (compressibility) and hindered settling function (permeability) using stepped pressure filtration and equilibrium batch settling tests. These characterisations, presented for each site in sections 9.2.3, 10.2.3 and 11.2.3 enable an understanding of process equipment underflow dewaterability trends from the start to the end of each washing and dewatering process. One of the limitations of the 1999 work was that it did not characterise suspension dewaterability over the solids concentration range of relevance to thickening operations. On site in 2000, the dewaterability of selected washer underflows at each site were characterised using transient batch settling to determine the shape of permeability curves over a wider range of solids concentrations, from feed concentrations to thickener underflow concentrations. The results of these comprehensive permeability characterisations are shown in
sections 9.3.2, 10.3.2 and 11.3.2. The effect of flocculation on dewaterability was also investigated for simulated feed suspensions on site in 2000 using a variety flocculants supplied by Ciba Specialty Chemicals and Cytec Australia. Flocculation test results are presented in sections 9.3.3 and 11.3.3.

8.6 PROCESS MODELLING

Using dewaterability characterisation data produced in 2000, curve fits to compressibilities and permeabilities of both underflow suspensions and flocculated simulated feed suspensions were produced. The curve fits were used along with thickener geometries and feed solids concentrations to predict thickener operation. The steady state thickener modelling predictions are presented and compared with process outputs in sections 9.4, 10.4 and 11.4. The Worsley Alumina refinery has a rotary drum filtration stage in its washing and dewatering process and thus some rotary drum filtration modelling is presented in section 10.4.6. The effect of washing red mud suspensions with seawater is shown to adversely affect dewaterability of red mud suspensions at QAL in sections 11.2.3, 11.3.2 and 11.3.3. So, the effect of washing red mud with seawater prior to consolidation in residue disposal areas is predicted with tailings dam consolidation modelling presented in section 11.5.

8.7 OVERVIEW OF RED MUD PROPERTIES

Finally in Chapter 12, the properties of red mud suspensions across all three sponsor sites are compared and combined to provide an insight into the generic properties of red mud suspensions at alumina refineries.
Chapter 9

ALCOA PINJARRA ALUMINA REFINERY

9.1 INTRODUCTION

The ALCOA (Pinjarra) Alumina Refinery located 100 km south of Perth in Western Australia processes bauxite from the local Darling range. The refinery is one of the world's biggest with an annual alumina production of 3.2 million tonnes (Alcoa 2001c).

The Refinery operates an extensive system of washing and dewatering of red mud suspensions. The Pinjarra red mud washing and dewatering process has 7-8 key solid-liquid separation stages. The first stage includes a number of thickeners running in parallel at over 100°C. The next stages include a number of counter-current washer trains also running in parallel and consisting of 4-5 washers in series for each train. In the washer trains, the temperature reduces at every process stage to about 50°C in the final washer. The second last stage involves a superthickener, so named because of its very large diameter, at about 35°C. The thickeners, washers and superthickener all have a converging base geometry. The final dewatering stage involves consolidation and drainage in a clay sealed residue disposal area.

In 1999, on site dewaterability and shear rheology characterisations were performed on underflow suspensions from a thickener, an entire washer train and the superthickener at as close to process temperatures as was practical. In 2000, more focussed underflow dewaterability characterisations were performed on 3rd, 4th and 5th washer underflow suspensions. Also in 2000, on site laboratory flocculation tests and dewaterability characterisations were performed on simulated 5th washer feed suspensions. Work included verification that settling rate is a strong function of solids concentration, reproducibility testing of plunger and stirred vessel flocculation methods and flocculation trials of copolymer (sodium polyacrylate / polyacrylamide) flocculants.

Dewaterability curve fits from characterisations in 1999 and 2000 were used in modelling of washer operation and evaluating the influence of rakes in increasing the effective suspension permeability. The experimental and modelling work is presented in the sections that follow and the implications of the results are discussed and overviewed at the end of the Chapter.
9.2 PINJARRA SITE VISIT 1999

9.2.1 WORK PROGRAM

With Ross de Kretser from the University of Melbourne, a temperature controlled pressure filtration rig and Brookfield rheometer were transported to the Alcoa Pinjarra Alumina Refinery on 12 July 1999. From 13-24 July 1999, the dewaterability and shear rheology of underflow samples from each stage of solid-liquid separation were characterised using batch settling tests, stepped pressure filtration, the vane technique and rotational viscometry.

9.2.2 EXPERIMENTAL METHOD

Sampling

Underflow suspensions and overflow liquors were collected one at a time, from each stage of the Pinjarra red mud washing and dewatering process. An important consideration in sampling was to ensure that suspensions were representative of what was in the process vessel. To best approximate this condition, the underflows were sampled before the pump and for overflows, effort was made to ensure that no dilution occurred.

Characterisation

For each piece of process equipment, the following tests were performed and data recorded:

1) Measurement of the underflow temperature.
2) Recording of process equipment temperature setpoint, mud bed height and flocculant dosage.
3) Measurement of underflow solids volume fraction by weight loss on drying of suspension and liquor.
4) Measurement of liquor viscosity with a Cannon-Fenske viscometer and liquor density using a densitometer.
5) Measurement of gel point using an equilibrium batch settling test.
6) Measurement of dewaterability (compressive yield stress and hindered settling function) using stepped pressure filtration. Characterisation temperatures were as close as was practical to process setpoint temperatures, subject to the constraint that the filtration test rig could not operate above 90°C.
7) Measurement of the shear yield stress using the vane technique.
8) Measurement of suspension viscosity using rotational viscometry of a bob in a very large gap.
9.2.3 DEWATERABILITY RESULTS

Properties of the vessels characterised are listed below in Table 9.1 including measured underflow temperatures, temperatures at which samples were characterised, liquor viscosities at filtration characterisation temperatures, approximate mud bed levels and an indication of flocculation regime used. Though the flocculant doses can not be presented here, each regime number 1-4 represents a different flocculant or combination of flocculants being used or a significantly different dose being applied.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Measured Underflow Temperature (°C)</th>
<th>Filtration Characterisation Temperature (°C)</th>
<th>Liquor Viscosity (mPa s)</th>
<th>Mud Bed Level (m)</th>
<th>Flocculation Regime</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickener</td>
<td>106</td>
<td>90</td>
<td>2.094</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1st Washer</td>
<td>101</td>
<td>90</td>
<td>1.113</td>
<td>0.005</td>
<td>2</td>
</tr>
<tr>
<td>2nd Washer</td>
<td>80</td>
<td>80</td>
<td>0.934</td>
<td>0.005</td>
<td>3</td>
</tr>
<tr>
<td>3rd Washer</td>
<td>68</td>
<td>70</td>
<td>0.847</td>
<td>1.553</td>
<td>3</td>
</tr>
<tr>
<td>4th Washer</td>
<td>58</td>
<td>60</td>
<td>0.724</td>
<td>2.0903</td>
<td>3</td>
</tr>
<tr>
<td>5th Washer</td>
<td>50</td>
<td>50</td>
<td>0.745</td>
<td>2.876</td>
<td>3</td>
</tr>
<tr>
<td>Superthickener</td>
<td>32</td>
<td>35</td>
<td>0.862</td>
<td></td>
<td>4</td>
</tr>
</tbody>
</table>

Table 9.1: Pinjarra alumina refinery washer train process temperatures

The underflow solids concentrations at all stages of the washing and dewatering process are compared with the gel points determined from batch settling experiments at ambient conditions in Figure 9.1. All of the solids volume fractions quoted assume a solids density of 3100 kg m⁻³, so multiplication by 3100 gives the concentration in grams per litre (g/L). Underflow solids concentrations become progressively higher from the thickener to the 5th washer and then decrease for the superthickener. The underflow concentrations in the thickener and 1st washer are substantially below the gel point such that compressional dewatering is not employed. Compressional dewatering occurs at solids concentrations above the gel point because it is then that the suspension flocs form a continuously networked structure that transmits weight to compress the suspension below. It is understood that this equipment is operated with low residence times and no bed height due to liquor stability issues. From the 2nd washer onwards, the underflow solids concentration is similar to or above the gel point, which occurs through compressional dewatering. The continually increasing underflow concentration down the train is attributed to progressively higher bed heights.

Using the pressure filtration test rig, the dewaterability of the underflow suspensions was determined from stepped pressure filtration tests. The filtration test rig was operated at the filtration characterisation temperatures listed in Table 9.1. These temperatures attempt to replicate typical process temperatures, subject to the constraint that the filtration test rig is limited to a maximum temperature of 90°C.
Compressibility defined as a compressive yield stress, \( P_y(\phi) \), is the compressive stress required to compress the suspension to a specified volume fraction, \( \phi \). The compressive yield stress data for each piece of process equipment is plotted in Figure 9.2. The compressibility values vary in a non-systematic manner with a range of 7% v/v over the entire washer train. However, the changes in compressibility show a strong correlation with changes in the flocculant used.

The permeability represented in terms of a hindered settling function, \( R(\phi) \), accounts for the hydrodynamic resistance to flow of liquor through a suspension. \( R(\phi) \) is inversely related to the permeability. The hindered settling function data measured at process temperatures for each piece of process equipment is plotted in Figure 9.3. The settler sample is considerably less permeable than all of the other samples, due to its significantly higher liquor viscosity. Down the washer train, permeabilities become progressively higher due to progressively lower liquor viscosities. The superthickener underflow is significantly less permeable than that of the final washer partly because it operates at a significantly lower temperature and thus has a higher liquor viscosity. Nothing can be inferred from Figure 9.3 about the influence of flocculation conditions on permeability because all samples have different liquor viscosities. However, the effect of liquor viscosity can be easily corrected for because permeability is proportional to liquor viscosity. The hindered settling functions corrected to a standard viscosity of 1 mPa s are shown in Figure 9.4. The trend in the results suggests that the flocculated network structure changes most significantly between the 1\textsuperscript{st} and 2\textsuperscript{nd} washers and between the 5\textsuperscript{th} washer and superthickener, which coincides with flocculant changes indicated in Table 9.1. Change in permeability due to change of flocculant is more easily seen in Figure 9.5 which shows the hindered settling function at a single solids volume fraction, \( \phi = 0.4 \).
Figure 9.1: Comparison of underflow solids volume fractions with gel point at Pinjarra refinery.

Figure 9.2: Compressive yield stress of Pinjarra refinery underflow suspensions at characterisation temperatures given in Table 9.1.
Figure 9.3: Hindered settling function of Pinjarra refinery underflow suspensions at characterisation temperatures given in Table 9.1.

Figure 9.4: Hindered settling function of Pinjarra refinery underflow suspensions corrected to a standard viscosity of 1 mPa s.
**Figure 9.5**: Hindered settling function of Pinjarra refinery underflow suspensions at $\phi = 0.4$ for characterisation temperatures given in Table 9.1 and corrected to a standard liquor viscosity of 1 mPa s.

### 9.2.4 SHEAR RHEOLOGY RESULTS

Results for the rheological characterisation of samples from the Pinjarra refinery are presented in Figures 9.6 (suspension viscosity versus shear rate) and 9.7 (shear yield stress versus solids concentration).

The viscosity results in Figure 9.6 are all for as-received samples taken from the plant and tested at a temperature close to the operating temperature. The broad trend is that the viscosity of the underflows tested increase at every processing stage. However, note that the temperature decreases down the train and the underflow solids concentrations increase down the train, both of which will contribute to a viscosity increase. Concentrations of dissolved solids in the liquor decreases at every processing stage, having the opposite effect on the viscosity. The net result of the factors influencing viscosity is a spread of viscosities over a range of about 1.5 orders of magnitude. The highest viscosities observed were for the 4th washer, 5th washer and superthickener underflows, all of which were tested at a low temperature and higher solids. Note that the 3rd washer underflow was the lowest viscosity sample, indicating that it lay at the optimum of the trade off between underflow solids and temperature and liquor concentration. All of the data are shear thinning and may be fitted with power law equations with a similar power law index in each case.
The shear yield stress results are presented in Figure 9.7 and in view of the different temperatures and chemical conditions of each sample, comparisons should be treated cautiously. However, comparing results for the 5<sup>th</sup> washer tested at both 50 and 70°C, an increase in the yield stress with temperature can be observed. Notably, most of the yield stresses look like they may be fitted with a single curve, with the exception of the yield stress of superthickener underflow. This difference reflects suspension processing differences that are partly temperature related and partly processing history and particle size distribution related.

![Figure 9.6: Viscosity versus shear rate behaviour of underflows of Pinjarra underflow suspensions at the temperatures and solids concentrations (volume %) indicated.](image)

**Figure 9.6:** Viscosity versus shear rate behaviour of underflows of Pinjarra underflow suspensions at the temperatures and solids concentrations (volume %) indicated.
Figure 9.7: Shear yield stress data as a function of solids volume fraction for Pinjarra underflows at temperatures indicated.

9.3 PINJARRA SITE VISIT 2000

9.3.1 WORK PROGRAM

The year 2000 Pinjarra site visit took place over four weeks from 8th August to 1st September 2000 in two parts. The major equipment requirements included transportation of a temperature controlled pressure filtration rig, a baffled flocculation vessel with overhead stirrer, numerous measuring cylinders, sample containers and a number of temperature controlled water baths.

The first part of the work program, performed solely by me, involved dewaterability characterisation of the red mud suspensions from the underflows of the 3rd-5th washers and from the bed of the 5th washer. The characterisation for each washer underflow included hindered settling function determinations over a wide range of solids concentrations using transient batch settling tests. Characterisation was completed in order to provide inputs to the steady state thickener model described in Chapter 7.

The second part of the work program involved comparison of CIBA’s plunger flocculation method and the University of Melbourne’s stirred vessel flocculation method. My collaborators included Kristen Bremmell and Janine Hulston from the University of Melbourne and Angela Beveridge from CIBA Specialty Chemicals. Settling rate reproducibility testing was performed for each flocculation method followed by flocculant trials with copolymer (NaAc/ACM)
flocculants with a range of anionic (NaAc) content. Dewaterability characterisation was completed using batch settling tests and pressure filtration.

9.3.2 WASHER UNDERFLOW AND BED DEWATERABILITY CHARACTERISATION

9.3.2.1 Experimental Method

Sampling

Underflow suspension samples were collected from 3rd to 5th washers in a washer train at the Alcoa, Pinjarra Alumina Refinery. 5 L of suspension was also collected from the top of the suspension bed in the 5th washer by repeatedly lowering a specially designed 300 mL collection device to the top of the bed. Overflow liquor was collected from overflow pump outlets.

Characterisation

A number of transient batch settling tests were conducted on each sample at 50°C to obtain permeabilities at solids concentrations relevant to thickener operation. The transient batch settling test procedure is detailed in Chapter 6.

9.3.2.2 Dewaterability Results

Dewaterability characterisation results from 1999 contained an inadequate amount of permeability data at solids concentrations of relevance to thickener operations. The pressure filtration data, though useful, is at concentrations well above that which is achieved in a thickener, so transient batch settling tests were planned to characterise permeabilities near the gel point. In addition, it was not known whether the permeability of the underflow suspensions was representative of the permeability in the thickener mud bed, so sampling directly from a mud bed was also a priority.

3rd – 5th washer underflow & 5th washer bed suspension permeabilities were characterised using the initial settling rates from transient batch settling tests at a range of solids concentrations at 50°C. The as-sampled material properties of each of these suspensions is listed in Table 9.2. For each sample, the hindered settling function corrected to the modelling process temperature is plotted in Figure 9.8, where the low volume fraction data (\(\phi < 0.25\)) comes from transient batch settling tests and the high volume fraction data comes from pressure filtration tests. The test procedures are detailed in Chapter 6. There are clearly some changes in suspension dewaterability between 1999 and 2000 as would be expected for any such industrial process with variable process conditions. The extra low volume fraction data produced in 2000
provides a more complete picture of how the hindered settling function varies at solids concentrations of interest to thickener operations. This new data enables more representative curve fitting and thus allows more appropriate modelling predictions. The hindered settling function of the 5th washer bed sample was found to be similar to that of the underflow sample. All suspensions sampled had a low shear yield stress that could not be measured using the slump test.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>3rd Washer U/F</th>
<th>4th Washer U/F</th>
<th>5th Washer U/F</th>
<th>5th Washer Bed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Volume Fraction</td>
<td>( \phi_u (v/v) )</td>
<td>0.226</td>
<td>0.209</td>
<td>0.217</td>
<td>0.137</td>
</tr>
<tr>
<td>Solids Weight Fraction</td>
<td>( \phi_u (w/w) )</td>
<td>0.448</td>
<td>0.434</td>
<td>0.453</td>
<td>0.321</td>
</tr>
<tr>
<td>Solids Concentration</td>
<td>( \phi_u (g/L) )</td>
<td>701</td>
<td>649</td>
<td>672</td>
<td>423</td>
</tr>
<tr>
<td>Shear Yield Stress</td>
<td>( \tau_y (Pa) )</td>
<td>&lt;20</td>
<td>&lt;20</td>
<td>&lt;20</td>
<td>&lt;20</td>
</tr>
<tr>
<td>Solids Density</td>
<td>( \rho_{sol} (kg , m^{-3}) )</td>
<td>3100</td>
<td>3100</td>
<td>3100</td>
<td>3100</td>
</tr>
<tr>
<td>Liquor Density (@ 25°C)</td>
<td>( \rho_{liq} (kg , m^{-3}) )</td>
<td>1116</td>
<td>1067</td>
<td>1037</td>
<td>1037</td>
</tr>
<tr>
<td>Process Temperature</td>
<td>( T_p (^\circ C) )</td>
<td>60</td>
<td>46, 53</td>
<td>42</td>
<td>42</td>
</tr>
<tr>
<td>Test Temperature</td>
<td>( T_t (^\circ C) )</td>
<td>50</td>
<td>50, 50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Liquor Viscosity at Test Temperature</td>
<td>( \eta_t (mPa , s) )</td>
<td>1.039</td>
<td>0.787</td>
<td>0.667</td>
<td>0.667</td>
</tr>
<tr>
<td>Modelling Temperature</td>
<td>( T_m (^\circ C) )</td>
<td>70</td>
<td>60</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Liquor Viscosity at Modelling Temperature</td>
<td>( \eta_m (mPa , s) )</td>
<td>0.753</td>
<td>0.670</td>
<td>0.667</td>
<td>0.667</td>
</tr>
</tbody>
</table>

Table 9.2: Summary of as-sampled material properties of Pinjarra washer suspensions characterised.

Figure 9.8: Hindered settling function, \( R(\phi) \), as a function of solids volume fraction, \( \phi \), for Pinjarra washers investigated at modelling temperatures given in Table 9.2. Data determined using transient batch settling and pressure filtration in 1999 and 2000.
9.3.3 FLOCCULATION TESTING

9.3.3.1 Experimental Methods

Materials

Suspension: 4th washer underflow suspension
Process Liquor: 5th washer overflow liquor
Lake Water: Approximately 0.4 M (16 g/L) NaOH with a visible presence of organics.

Flocculants: A number of copolymer (NaAc/ACM)† flocculants with a range of anionic (NaAc) content were used

<table>
<thead>
<tr>
<th>Name</th>
<th>Anionic content:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alclar W50</td>
<td>medium NaAc</td>
</tr>
<tr>
<td>Alclar 662</td>
<td>medium-high NaAc‡</td>
</tr>
<tr>
<td>Alclar HP22</td>
<td>medium-high NaAc‡</td>
</tr>
<tr>
<td>Alclar W23</td>
<td>medium-high NaAc‡</td>
</tr>
<tr>
<td>Alclar 661</td>
<td>high NaAc</td>
</tr>
<tr>
<td>Alclar 665</td>
<td>100% NaAc</td>
</tr>
</tbody>
</table>

Flocculant Makeup

A range of CIBA flocculants were prepared at 0.25% w/v by the following method:

1. weigh out 0.25 g of polymer
2. add 2 mL of methylated spirits (ethanol is more commonly used)
3. add 98 mL of lake water
4. mix by hand (about 2 hours) until dissolved
5. mix 10 mL of dissolved flocculant with 90 mL of lake water

Suspension Makeup (Simulated 5th Washer Feed)

1 part underflow suspension (4th washer) was mixed with 4 parts process liquor (5th washer overflow) and 1 part lake water in 4 by 20 litre drums to simulate a 5th washer suspension feed.

† NaAc is sodium polyacrylate poly(Na⁺(CH₂CHCOO⁻))
ACM is polyacrylamide poly(CH₂CHCONH₂)

‡ Polymers of similar anionic content, but with different average molecular weights and distributions.
The suspension was allowed to settle for 5 minutes and then decanted leaving coarse sand fraction behind to be discarded.

Flocculation Test Methods

CIBA’s plunger flocculation method and the University of Melbourne’s stirred vessel flocculation methods were compared in a number of settling rate reproducibility tests for each flocculation method and flocculant trials. These methods are briefly detailed below.

Plunger Flocculation Method:

In a thermally controlled water bath at 50°C, 1 L measuring cylinders were filled with suspension. A suspension was plunged strongly a few times to homogenise the sample, made up flocculant was added and the suspension was plunged 3 times over about 12 seconds using a metronome to time the plunge strokes. The settling time for the bed height to fall from 900 mL to 700 mL was recorded. This method can be used to measure relatively high settling rates because of the 10 cm distance from the 900 to 700 mL marks, but the method can lead to errors when settling slows before reaching the 700 mL mark.

Stirred Vessel Flocculation Methods:

The stirred vessel flocculation method has been developed by Hulston (Hulston and Scales 2001). In a thermally controlled water bath at 50°C, a baffled flocculation vessel was filled with 2.65 litres of made up suspension. The impeller was rotated at a high rate (700 rpm) for 2 minutes to mix the suspension before the flocculant was added. One minute before flocculant addition commenced, the impeller rate was lowered to the predefined rate that was used in the flocculation. Two variations on the stirred flocculation method were tested.

- Method 1: Flocculant was added simultaneously while stirring over 1 minute and the impeller was then stopped immediately when all flocculant was added.
- Method 2: Flocculant was added over 4 seconds while stirring with stirring continued for another 12 seconds before the impeller was stopped.

The initial settling rate was calculated from a line of best fit of height versus time of settlement data. The data used covered settlement from a height of 145 mm to 125 mm over 5 data points, where the initial height is just above 150 mm. Because settlement is measured over only 2 cm relative to 10 cm in the plunger method, using the 5 data points is necessary to maintain accuracy and is also useful in verifying whether settlement was slowing. It should be noted that the inclusion or exclusion of just one datum point can alter the measured settling rate by 20%.
Dewaterability Characterisation

Dewaterability characterisation involved measurement of the initial settling rate and gel point tests. For selected samples, the tests also included transient batch settling for permeabilities near the gel point and stepped pressure filtration for compressibilities and permeabilities at high solids concentrations.

9.3.3.2 Variation of Free Settling Rate with Solids Concentration

Many flocculant performance tests are based on determining the free settling rate as a function of flocculant dose for freshly flocculated suspensions. The tests are generally conducted at a single solids concentration somewhere near the thickener or washer feed solids concentration. Solids concentrations commonly used in testing range from 1.0-2.6% v/v which is about 30-80 g/L. In this experiment, plunger flocculation tests were performed in 1 litre measuring cylinders on suspensions with initial solids concentrations ranging from 0.29 to 4.6 % v/v (8.9 to 142 g/L). All suspensions were flocculated with a dose of 31.6 g of Alclar 662 per tonne of solids. As shown in Figure 9.9, before flocculation, the settling rate varied by a factor of 20 from solids concentrations of 0.29 % v/v to 4.6 % v/v. The free settling rate of the same suspensions when freshly flocculated is a very strong function of solids concentration, varying nearly three orders of magnitude from 73 m h\(^{-1}\) at 0.29 % v/v to 0.15 m h\(^{-1}\) at 4.6 % v/v. Therefore, the initial solids concentration should always be considered when comparing flocculants on the basis of settling rate.
Figure 9.9: Free settling rate, \( u_{fs}(\phi) \), as a function of solids volume fraction, \( \phi \), for simulated Pinjarra 5\textsuperscript{th} washer feed suspension before flocculation and when flocculated with 31.6 g/t Alclar 662.

### 9.3.3.3 Flocculation Reproducibility Testing

With the simulated 5\textsuperscript{th} washer feed suspension prepared at 69 g/L on 23\textsuperscript{rd} August, the primary task was to compare the settling rates produced by CIBA’s plunger flocculation method and the University of Melbourne’s stirred vessel flocculation method and also to verify the reproducibility of each method. Using the copolymer Alclar 662 as the standard flocculant, tests were performed at 50\textdegree{}C. As shown in Figure 9.10, the samples flocculated by the plunger method, all within a few minutes of each other, gave a clear trend in settling rate. However, the samples flocculated by the stirred vessel method 1 (adding polymer over 1 minute) gave significant variability in results with a stirrer speed of 450 rpm. The suspected reason for variability is because large flocs only formed during the last few seconds of flocculant addition. Hence, if addition of flocculant was stopped too early, only small flocs were formed and there was a decrease in settling rate, however if flocculant addition was stopped on time, large flocs were formed, leading to an increased settling rate.
Figure 9.10: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated Pinjarra 5th washer feed at 69 g/L, flocculated with Alclar 662 by plunger method and stirring method 1 on 23/8 and 24/8.

On 25th August, using a suspension at 64.5 g/L, a more controlled settling rate flocculation test compared the plunger method with the stirred vessel method 1 at 530 rpm by testing each method with the same frequency. As shown in Figure 9.11, there were no time dependant trends and the stirred vessel method 1 still had a high degree of variability.
It was noted that the plunger method usually gave higher settling rates due to quicker floculant addition and lower times of shear. The stirred vessel method 2, adding floculant over 4 seconds and then shearing at a low rate for a further 12 seconds, was developed to mimic the shear experienced in the plunger method. On 27th August, using a suspension at 73.1 g/L, the stirred vessel method 2 was tested at 350 and 450 rpm, with the results shown in Figure 9.12. The free settling rates from all reproducibility testing of the floculation methods are summarised in Table 9.3. The significant result from comparing different shear rates suggests that increased shear reduces the free settling rate. 350 rpm was selected as the standard impeller speed because, as shown in Figure 9.12, the resultant floc settling rate more closely mimicked the plunger method results while the impeller speed also provided enough turbulence to suspend most of the solid particles.

**Figure 9.11:** Free settling rate, $u_{fs}$, as a function of floculant dose for simulated Pinjarra 5th washer feed at 64.5 g/L, floculated with Alclar 662 by plunger method and stirring method 1 on 25/8.
**Figure 9.12:** Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated Pinjarra 5th washer feed at 73.1 g/L, flocculated with Alclar 662 by plunger method and stirring method 2 on 27/8.

<table>
<thead>
<tr>
<th>Alclar 662 Dose (g/t solid)</th>
<th>Plunger 450 rpm</th>
<th>Stirring Method 1 530 rpm</th>
<th>Stirring Method 2 350 rpm</th>
<th>Stirring Method 2 450 rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Settling rates (m/hr)</td>
<td>7.7</td>
<td>3.0</td>
<td>6.2</td>
<td>0.7</td>
</tr>
<tr>
<td></td>
<td>6.1</td>
<td>4.5</td>
<td>6.6</td>
<td>1.7</td>
</tr>
<tr>
<td></td>
<td>7.3</td>
<td>6.6</td>
<td>2.0</td>
<td>1.4</td>
</tr>
<tr>
<td></td>
<td>6.4</td>
<td>6.7</td>
<td>1.8</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>5.7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average (m/hr)</td>
<td>6.8</td>
<td>5.9</td>
<td>2.0</td>
<td>1.5</td>
</tr>
<tr>
<td>Standard Deviation (m/hr)</td>
<td>0.7</td>
<td>1.7</td>
<td>0.5</td>
<td>0.3</td>
</tr>
</tbody>
</table>

**Table 9.3:** Summary of free settling rates for flocculation reproducibility testing of plunger method, baffled vessel stirring method 1 and stirring method 2 on a simulated Pinjarra 5th washer sample at 50°C.

Finally on 29th and 30th August, using a suspension at 73.1 g/L, the plunger method was compared with stirred vessel method 2 at 350 rpm over a range of Alclar 662 doses. The settling rates are compared in Figure 9.13 and each method shows a trend of increasing settling rate with increasing flocculant dose. It is noted that on the second day, the plunger settling rates increased, while the stirred vessel settling rates decreased. No explanation is offered for this anomaly.
Figure 9.13: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated Pinjarra 5\textsuperscript{th} washer feed at 71.2 g/L, flocculated with Alclar 662 by plunger method and stirring method 2 at 350 rpm on 29/8 and 30/8.

9.3.3.4 5\textsuperscript{th} Washer Flocculant Test Results

Free Settling Rate & Permeability

On 23\textsuperscript{rd}-24\textsuperscript{th} and 29\textsuperscript{th}-30\textsuperscript{th} August, flocculant trials were conducted using the plunger method and the stirred vessel method 2 with a simulated 5\textsuperscript{th} washer feed suspension. The flocculants tested were copolymer (sodium polyacrylate / polyacrylamide) flocculants as listed in the “Flocculation Methods” section, ranging from the medium strength anionic Alclar W50 to the 100 % anionic Alclar 665. As shown in Figure 9.14 and Figure 9.15, settling rates generally increase with flocculant dose for all flocculants tested. The settling rates of the 100 % anionic Alclar 665 samples are amongst the lowest and the Alclar 662 samples give consistently high settling rates. The other samples have settling rates that do not show a trend in terms of anionic content and thus there must be other factors, such as molecular weight, that play just as significant a role.
Figure 9.14: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated Pinjarra 5th washer feed at 69 g/L, flocculated with a range of flocculants by plunger method on 23/8 and 24/8.

Figure 9.15: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated Pinjarra 5th washer feed at 71.2 g/L, flocculated with a range of flocculants by plunger method at 350 rpm on 29/8 and 30/8.

Some samples flocculated on 29th and 30th August were concentrated to 5 times their original concentration and the initial settling rate was determined from transient batch settling tests to
determine relative permeabilities near the gel point. Selected samples were also characterised by pressure filtration tests. The free settling rate results near the gel point, shown in Figure 9.16, give trends in accordance with those of the free settling rates at the solids concentration of flocculation with the exception of the Alclar W50 sample which unexpectedly is much more permeable. This anomaly seems reproducible, but further tests would be required. The hindered settling function results at high solids concentration from pressure filtration, shown in Figure 9.17, are relatively insensitive to the flocculant type and dose. However, the underflow sample appears to be more permeable than the synthetically flocculated samples, but it should be recognised that the removal of the sand fraction may alter the measured permeability at high solids concentrations.

Figure 9.16: Free settling rate, $u_{fs}(\phi)$, ($\phi = 0.115, 355$ g/L) as a function of flocculant dose for simulated Pinjarra 5th washer feed flocculated with a range of flocculants by plunger method and stirring method 2 at 350 rpm on 30/8.
**Figure 9.17:** Hindered settling function, $R(\phi)$, obtained by pressure filtration of Pinjarra 5th washer underflow suspension and a selection of flocculated simulated Pinjarra 5th washer feed samples.

*Gel Point & Compressibility*

For samples flocculated on 29th August at 50°C, the gel points were determined from equilibrium batch settling tests at ambient temperatures. The results from equilibrium settling tests at ambient temperatures are preferred over tests at 50°C because the vibration of the water bath caused extra consolidation that made analysis difficult. The gel point of the simulated 5th washer sample before flocculation was 0.155. Flocculated gel point results, shown in Figure 9.18, reveal the non-intuitive feature of this copolymer system that, for doses above 20 g/t, the gel point increases with increasing flocculant dose. This feature was exaggerated for the samples flocculated with Alclar 662 by the stirred vessel method 2. Overall, it is difficult to discern trends on the basis of anionic strength because there is a molecular weight dependence. The data from the stirred vessel indicate that floc rearrangement is occurring and that comparison of settling rate alone is not adequate.
The compressibilities of selected flocculated samples at high solids concentrations were determined from pressure filtration tests, as shown in Figure 9.19. The underflow sample is more compressible than the synthetically flocculated samples, but the removal of the sand fraction is responsible for most of this variation, which may also be due in part to shear in the washer mud bed. The compressibilities of the flocculated samples all seem very similar with the exception of a higher dosed Alclar 662 sample, which is less compressible. The reduction in compressibility is presumably due to the influence of extra flocculant (46.2 versus 27.8 g/t) on the related floe size and rearrangement.
9.4 STEADY STATE THICKENER MODELLING

9.4.1 WORK PROGRAM

The steady state thickening performance of a 5th washer at the Pinjarra Alumina Refinery has been modelled using material properties and relevant geometric information. The material properties include the dewaterability curve fits of a typical flocculated simulated feed suspension and an underflow suspension at process temperatures. Two types of modelling output have been presented:

i) Thickener underflow solids concentration as a function of feed solids flux calculated for a range of suspension bed heights.

ii) Solids residence time in the bed as a function of underflow solids concentration (linked to solids flux) for a range of different bed heights.

Comparison of the predicted output with the actual process output enables estimation of the permeability enhancement factor.
9.4.2 CURVE FITTING & PROCESS DATA

Steady state thickener modelling requires thickener dimensions, the feed solids concentration and suspension dewaterability in terms of the compressive yield stress and the hindered settling function. The thickener modelled in this study is a 5th washer with a truncated converging base as shown in the schematic in Figure 7.4. The feed solids concentration used in modelling work was a solids volume fraction of 0.08 (248 g/L) for the 5th washer. The solids flux has an average value of about 0.066 t hr$^{-1}$ m$^{-2}$.

Figure 9.20 presents the full characterisation of $P_y(\phi)$ over the range of solids from close to the gel point to high solids data for a 5th washer underflow sample and for a flocculated simulated 5th washer sample. The low solids data were obtained from sedimentation tests, while the high solids data were obtained from pressure filtration. The flocculated sample is a simulated 5th washer feed flocculated by the plunger method with 31.6 g/t Alclar 662 copolymer flocculant. Clearly, the freshly flocculated simulated feed sample is less compressible than the underflow sample. The difference is believed to be due to the underflow having experienced moderate shear in the washer bed, but is also due to some coarse sand particles being removed in the preparation of the simulated feed sample. The compressive data were fitted to an exponential-power law functional form, given by Equation 7.3, where $P_y(\phi)$ fitting parameter data are listed for the two washers in Table 9.4 below.

Figure 9.21 presents the hindered settling function over the complete range of solids concentrations from well below the gel point to high solids concentrations for the 5th washer underflow sample and for the flocculated simulated 5th washer sample. The underflow pressure filtration data has been used for both samples because suspension at the base of a thickener at high solids concentration will have properties resembling the underflow. The $R(\phi)$ data is fitted to a power law functional form given by Equation 7.6, where $R(\phi)$ fitting parameter data for the two samples are presented in Table 9.4 below.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\phi_g$</th>
<th>$P_a$</th>
<th>$P_b$</th>
<th>$P_m$</th>
<th>$P_n$</th>
<th>$r_a$</th>
<th>$r_b$</th>
<th>$r_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Underflow</td>
<td>0.154</td>
<td>33.752</td>
<td>-1.353</td>
<td>34.510</td>
<td>1.092</td>
<td>1.99E14</td>
<td>2.032</td>
<td>0.001</td>
</tr>
<tr>
<td>Flocculated</td>
<td>0.105</td>
<td>37.987</td>
<td>-12.299</td>
<td>110.96</td>
<td>0.453</td>
<td>9.43E12</td>
<td>3.373</td>
<td>1.19E6</td>
</tr>
</tbody>
</table>

Table 9.4: Summary of $P_y(\phi)$ and $R(\phi)$ fitting parameter data for Pinjarra 5th washer underflow and flocculated simulated Pinjarra 5th washer sample both at 50°C.
Figure 9.20: Compressive yield stress in normal and logarithmic co-ordinates as a function of solids volume fraction for Pinjarra 5\textsuperscript{th} washer underflow and flocculated simulated 5\textsuperscript{th} washer sample.
9.4.3 **5TH WASHER MODELLING RESULTS**

Figure 9.22 shows the results of modelling the steady state underflow solids concentration of the 5th washer. The model inputs included the 5th washer dimensions, a feed solids volume fraction of 0.08 (248 g/L) and the dewaterability curve fits of the underflow sample. The results show the predicted solids flux fed to the thickener as a function of the underflow solids concentration for a range of suspension bed heights. The flux is in units of tonnes per hour per m$^2$ of thickener surface area where the area used is the cross sectional area of thickener at the top of the bed. The feed solids concentration and gel point are also indicated.

Figure 9.22 suggests that the output solids from the 5th washer should be around a volume fraction of 0.09. Clearly this is substantially different to the measured underflow solids concentration of 0.217 listed earlier in Table 9.2. It noted from Figure 9.22 that for an underflow solids of around 0.22 to be reached the flux should be reduced to around 0.0017 t h$^{-1}$ m$^{-2}$. Thus, assuming that the calculations are correct, there is something that effectively increases the permeability of the suspension, by a factor of 39.

There are two factors that may have contributed to this discrepancy. The first lies in the assumption that the material that is taken from the underflow of the thickener is representative of the flocculated material that feeds into the top of the bed. Currently, little information is
available in this regard; however, observations of the effect of shear on freshly flocculated suspensions suggest that, once sheared, flocculated networks exhibit lower permeabilities. This could certainly contribute to the location of the flux-solids curve in the region between the feed solids concentration and the gel point. An increased permeability would shift the curve to higher underflow solids for the same flux. The second factor contributing to the discrepancy is the effect of shear on the flocculated solids due to raking. The simple 1-dimensional model for thickening employed here has no facility for the incorporation of shear. Previous research has suggested that the effect of shear on a flocculated network is to liberate floc-bound water, effectively increasing the permeability by a large extent. Whilst the model results do not incorporate shear, a measure of the permeability enhancement provided by shear can be found by the ratio of the predicted versus the actual solids flux for the measured underflow solids concentration, in this case a factor of 39.

The thickener model outputs may be converted to a bed solids residence time, indicating the average time a solid particle would spend in the bed. Figure 9.22 also shows the bed solids residence time versus underflow solids concentration for a range of suspension bed heights. Clearly the solids volume fraction of 0.09 corresponding to the true solids flux of 0.066 t hr$^{-1}$ m$^{-2}$ is outside the range of the data in Figure 9.22. However, for the actual underflow solids concentration of 0.22 (including the effect of raking), a bed solids residence time of around 243-660 hours is indicated for a bed height between 2 and 5 m. Scaling this residence time by the effective increase in permeability obtained through raking, a factor of 39, indicates that the real residence times of solids in the bed are of the order of 6.2-16.5 hours.

Figure 9.21 showed, that at low solids concentrations, the $R(\theta)$ data (which dictate washer output in this regime) for the underflow sample was higher than for the freshly flocculated sample. This implies that at low solids concentrations, the freshly flocculated sample is more permeable than the underflow sample and this would increase the predicted underflow solids concentration for a given solids flux.
Figure 9.22: Steady state thickener modelling predictions based on Pinjarra 5th washer underflow sample dewaterability, showing solids flux and solids residence time in suspension bed as functions of solids volume fraction for various bed heights.
Figure 9.23 shows the predicted solids flux – underflow solids concentration relationship for the same 5\textsuperscript{th} washer, with the same initial solids volume fraction of 0.08, but with the flocculated sample dewaterability curve fits.

As for the underflow sample modelling, a significant discrepancy is observed between the model output predictions and the observed underflow solids concentration. Reasons for the discrepancy have been discussed. Note that the flocculated sample is probably more representative of the suspension in the washer, so the same calculations are repeated here.

From Figure 9.23, an underflow solids of around 0.22 is predicted for a solids flux of 0.0035 t hr\textsuperscript{-1} m\textsuperscript{-2} for a bed height of 2 m and 0.0048 t hr\textsuperscript{-1} m\textsuperscript{-2} for a bed height of 5 m. The observed underflow solids concentration is actually moving into the regime of compressibility limited operation for small bed heights less than 5 m. The permeability enhancement factor is calculated to be 18.9 for 2 m of bed height and 13.8 for 5 m of bed height, which is the typical range of operation. This gives an average enhancement factor of 16.3.

Comparison of the permeability enhancement factors for the flocculated versus the underflow sample indicate that the effect of flocculation is to enhance permeability by a factor of 2.4 while shear influences such as raking enhance permeability by a factor of 16.3. Whilst these figures are qualitative, in that the permeability of the solids in the feed well may not be represented exactly, the numbers do correlate with other samples measured on similar thickening devices.

Figure 9.23 also shows the predicted bed solids residence time versus underflow solids concentration for the flocculated sample for a range of suspension bed heights. Again, for the actual underflow solids concentration of 0.22, a bed solids residence time of around 139-296 hours is indicated for a bed height between 2 m and 5 m. Scaling this residence time by the effective increase in permeability obtained through raking, a factor of 16.3, indicates that the real residence times of solids in the bed are of the order of 8.5-18.2 hours which compares well with the 6.2 to 16.5 hours calculated from the underflow sample analysis.
Figure 9.23: Steady state thickener modelling predictions based on laboratory flocculated Pinjarra 5th washer sample dewaterability, showing solids flux and solids residence time in suspension bed as functions of solids volume fraction for various bed heights.
9.5 GENERAL DISCUSSION AND OBSERVATIONS

Overall, Pinjarra suspensions appear to be more compressible than suspensions at other sites, with the least compressible superthickener underflow being as compressible as anything at other sites. Because of the high compressibility of the suspensions, Pinjarra has the potential to achieve higher extents of dewatering than other sites, possibly to even exceed the 22% v/v already achieved in the 5th washer. This potential improvement also requires that the shear yield stress stays below about 100 Pa for ease of material handling, which currently seems feasible for washer suspensions, but not superthickener suspensions due to processing differences.

Conversely, the suspensions are the least permeable of all, requiring numerous stages of clarification before compressional dewatering is ‘introduced’ from the 3rd washer onwards. Overall washing could be significantly improved if higher suspension bed heights were employed in the lead washers. However, to reach the higher solids concentrations requires operational changes. These changes include improved settling rate and better liquor stability to allow bed heights to be increased and/or solids throughputs to be reduced without solids residence times causing excessive precipitation and loss of product. Increasing process temperatures to improve permeability is considered impractical because process temperatures are already high. Alternatively, flocculant dosing and floc formation can be altered to give higher settling rates and permeabilities. In the longer term, compressional dewatering should be achievable when liquor stability issues are improved to allow substantial bed heights at the front end of the washer train without the risk of excessive precipitation and loss of product.

In converging base thickening, high underflow concentrations are controlled by suspension bed height, but the lead washers have minimal bed height. Achievable underflow concentrations are limited by the maximum practical bed height, which is limited by the influence of the suspension shear yield stress on rake torque and suspension pumpability. The shear yield stress of a suspension increases with increasing solids concentration and as such an excessively high shear yield stress can cause rakes to stop and also overstress underdesigned pumps and piping. In existing process operations, the maximum torque that the rakes are allowed to withstand and the maximum pumpable solids concentration determines the maximum practically achievable suspension bed heights and underflow solids concentrations. It is suggested that optimising suspension bed heights be seriously considered to improve underflow solids concentrations through compressional dewatering. Improved knowledge of the effects of suspension bed heights can only be achieved through fundamentally based process modelling and plant testing.

A significant issue arising from this analysis is the loss of dewaterability and pumpability between the 5th washer and the superthickener. This cannot be rationalised in terms of
temperature and liquor viscosity alone. The change arises partly from the remixing, pumping and separation of the sand fraction, resulting in the suspension settled in the superthickener being altered from that in the washer train. In addition, a different flocculant is used under significantly lower process temperature conditions. The data suggests that processing options including but not limited to the choice or utilisation of the flocculant are not optimal.

Flocculant testing has shown that both CIBA’s plunger flocculation method and the University of Melbourne’s stirred vessel flocculation method provide valuable information about the flocculation process. Each method produces different numbers in terms of settling rates and structure of flocs produced due to different shear regimes. The plunger method is subject to more systematic and random inconsistencies due to its manual nature, but copes reasonably well with the presence of large particles. The stirred vessel method provides the potential to control flocculation conditions over a wide range of shear rates and flocculant addition regimes, however the results are less reproducible due to the way that the flocs develop and especially in the presence of large sand particles.

Flocculated final washer suspensions have been shown to be less compressible at high solids concentrations than their sheared underflow counterparts. However, the trend is reversed at low solids concentrations with the gel point increasing with increasing flocculant dose. This behaviour is believed to be due to the chemical properties of the copolymer (NaAc/ACM) flocculants used. At low solids concentrations, permeability was found to be improved dramatically by increased flocculant doses, but this influence decreased as solids concentrations increased to the extent that no flocculant dose trends could be identified at high solids concentrations.

Modelling the operation of a 5th washer using the dewaterabilities from both flocculated and underflow suspensions predicts significantly different outputs. However, neither of these accurately predict the actual performance. Permeability enhancement factors of 39 for the underflow sample and 16.3 for the flocculated sample describe the magnitude of the discrepancy. The discrepancy is partly due to measured underflow permeabilities being lower than those of freshly flocculated suspensions because of the extreme shear sensitivity of flocculated suspensions. In addition, shear processes involving raking and the sloped

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† NaAc is sodium polyacrylate poly(Na+(CH₂CHCOO⁻))
ACM is polyacrylamide poly(CH₂CHCONH₂)
converging base of the thickener also contribute to the measured permeability enhancement factor.

9.6 CONCLUSIONS

Experimental dewaterability characterisation and solid-liquid separation equipment process modelling for the Alcoa (Pinjarra) Alumina Refinery included the following:

- Auditing of the process employed to wash and dewater red mud suspensions.
- Shear rheology and dewaterability characterisation of underflows from each solid-liquid separation stage.
- More complete permeability characterisation of 3rd-5th washer underflows near the gel point enabling more appropriate hindered settling function curve fitting.
- Verification of strong influence of solids concentration on free settling rate, even at very low solids concentrations.
- Flocculation reproducibility testing and comparison of the CIBA plunger flocculation method with the University of Melbourne’s stirred vessel flocculation methods.
- Flocculant trials comparing settling rate and other dewaterability parameters for a range of copolymer (sodium polyacrylate / polyacrylamide) flocculants with a range of anionic (sodium polyacrylate) content over a range of doses with a simulated 5th washer feed suspension. Results suggest that gel point increases for this copolymer system.
- Steady state thickener modelling of the 5th washer, with evaluation of the permeability enhancement of flocculation and raking and evaluation of solids residence time in the suspension bed.

Characterisation and modelling at Pinjarra revealed how red mud suspensions vary from having thick liquors at over 100°C in the thickeners to having dilute liquors in the superthickener at just over 30°C. As a result, trends in suspension shear rheology and dewaterability were observed down the washer train. Effects due to temperature and liquor viscosity were largely scalable, however there were some effects that were attributed to other process variations. More complete permeability characterisation demonstrated how permeability varies by more than five orders of magnitude from low to high solids concentrations. Flocculation testing demonstrated significant improvements in permeability and the extreme sensitivity of floc structures to shear processes. Shear can influence the permeability of floc structures produced, it can destroy permeability and, in compression, can also enable effective permeability enhancement. A permeability enhancement factor of 16.3 was attributed to shear processes such as raking. Modelling results show that improvements in extent of dewatering can potentially be achieved
with higher bed heights and lower solids fluxes, subject to rake torque limits not being exceeded. Higher solids can also be achieved through improved choice of flocculant type and dose to give higher permeabilities.
Chapter 10

WORSLEY ALUMINA REFINERY

10.1 INTRODUCTION

The Worsley Alumina Refinery, located 200 km south of Perth and 50 km inland from the port city of Bunbury in Western Australia, processes low quality bauxite by world standards from near Boddington in the local Darling range and now has an annual capacity of 3.1 million tonnes per annum of alumina as a result of a major plant expansion project completed in September 2000 (Worsley 2001).

Before the plant expansion, the refinery operated a system of washing and dewatering red mud suspensions that involved 7 solid-liquid separation stages. The first stage involved a number of flat bottomed, outward raking settlers running in parallel at over 100°C. The next 4 stages included a number of counter-current washer trains also running in parallel and utilising flat bottomed, outward raking thickeners. In the washer trains, the temperature reduced at every process stage to about 50°C in the 4th washer. The next stage involved rotary drum filtration under vacuum and washing with hot condensate. The filter cake was then collected, diluted slightly and pumped to sealed bauxite residue disposal areas (BRDA) where the final stage of consolidation and drainage occurred.

During the process expansion, a number of new high rate thickeners were built. These thickeners had converging bases like many others in the industry, but were taller and had smaller diameters than those used at Pinjarra and QAL for example. These thickeners replaced the settlers, which have become 1st washers and also took up the expansion load that the drum filters could not handle. As such, the new process configuration involves the same number of solid-liquid separation stages, but an altered configuration. The settlers use high rate thickeners, 1st-4th washers use flat bottomed, outward raking thickeners and the next stage is shared between high rate thickening and rotary drum filtration before pumping to the BRDAs.

In 1999, on site dewaterability and shear rheology characterisations were performed on underflow suspensions from a flat bottomed settler, and an entire washer train of flat bottomed washers at as close to process temperatures as was practical. In 2000, more focussed underflow dewaterability characterisations were performed on settler and 5th washer underflow suspensions.
from high rate thickeners. In addition, the influence of temperature on shear yield stress was also investigated.

Dewaterability curve fits from characterisations in 1999 and 2000 were used in modelling of settler and washer operation and evaluating the influence of rakes in increasing the effective suspension permeability. The experimental and modelling work is presented in the sections that follow and the implications of the results are discussed and overviewed at the end of the chapter.

10.2 WORSLEY SITE VISIT 1999

10.2.1 WORK PROGRAM

With Ross de Kretser from the University of Melbourne, a temperature controlled pressure filtration rig and Brookfield rheometer was transported to the Worsley Alumina Refinery on 27 July 1999. From 2-9 August 1999, the dewaterability of underflow samples from the second washer train were characterised using batch settling tests, stepped pressure filtration, the vane technique and rotational viscometry.

10.2.2 EXPERIMENTAL METHOD

Sampling

Underflow suspensions and overflow liquors were collected one at a time, from each stage of the Worsley red mud washing and dewatering process, with the exception of the drum filter from which only output filter cake was collected. An important consideration in sampling was to ensure that suspensions were representative of what was in the process vessel. To ensure this, the underflows were sampled before the pump and for overflows, it was ensured that no dilution occurred.

Characterisation

For each piece of process equipment (where practical), the following tests were performed and data recorded:

1) Measurement of the underflow temperature.
2) Recording of process equipment temperature setpoint, mud bed height and flocculant dosage.
3) Measurement of underflow solids volume fraction by weight loss on drying of suspension and liquor.
4) Measurement of liquor viscosity with a Cannon-Fenske viscometer and liquor density using a densitometer.
5) Measurement of gel point using an equilibrium batch settling test.
6) Measurement of dewaterability (compressive yield stress and hindered settling function) using stepped pressure filtration. Characterisation temperatures were as close as was practical to process setpoint temperatures, subject to the constraint that the filtration test rig could not operate above 90°C.
7) Measurement of the shear yield stress using the vane technique.
8) Measurement of suspension viscosity using rotational viscometry of a bob in a very large gap.

10.2.3 DEWATERABILITY RESULTS

Properties of the vessels characterised are listed below in Table 10.1 including temperature setpoints, measured temperatures and filtration characterisation temperatures and liquor viscosities at these temperatures.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Underflow Temperature Setpoint (°C)</th>
<th>Measured Temperature (°C)</th>
<th>Filtration Characterisation Temperature (°C)</th>
<th>Liquor Viscosity (mPa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Settler</td>
<td>105</td>
<td>96</td>
<td>90</td>
<td>1.990</td>
</tr>
<tr>
<td>1st Washer</td>
<td>80-85</td>
<td>78</td>
<td>80, (70)</td>
<td>1.092, (1.292)</td>
</tr>
<tr>
<td>2nd Washer</td>
<td>75-80</td>
<td>62</td>
<td>70</td>
<td>0.930</td>
</tr>
<tr>
<td>3rd Washer</td>
<td>65-70</td>
<td>62</td>
<td>70</td>
<td>0.768</td>
</tr>
<tr>
<td>4th Washer</td>
<td>60-65</td>
<td>43</td>
<td>50, (70)</td>
<td>0.877, (0.676)</td>
</tr>
<tr>
<td>Drum Filter</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 10.1: Worsley alumina refinery washer train process temperatures.

The underflow solids concentrations down the washer train are compared with the gel points determined from batch settling experiments at ambient conditions in Figure 10.1. All of the solids volume fractions quoted assume a solids density of 3720 kg m\(^{-3}\), so multiplication by 3720 gives the concentration in g/L. The underflow concentrations are similar to the gel points determined, which suggests that compressional dewatering is not employed in the settlers and washers. Compressional dewatering occurs at solids concentrations above the gel point because it is then that the suspension flocs form a continuously networked structure that transmits weight to compress the suspension below. It is understood that the flat bottomed washers are operated with low bed heights to ensure that rake torques are maintained at the reasonably low levels for which they were designed. The drum filter utilises compressional dewatering to compress the suspension to a solids volume fraction of 0.35, which is far in excess of the compression achieved in the settlers and washers.
Using the pressure filtration test rig, the dewaterability of the underflow suspensions was determined from stepped pressure filtration tests. The filtration test rig was operated at the filtration characterisation temperatures listed in Table 10.1. Process temperature variations during the sampling period were significant, but generally within 15°C of the characterisation temperatures which attempt to replicate typical process temperatures, subject to the constraint that the filtration test rig is limited to a maximum temperature of 90°C.

Compressive yield stress data are plotted in Figure 10.2. The compressibility values vary by up to 2% v/v with no reproducible trends within this range due to experimental uncertainty and process variability. Consequently, it is suggested that within experimental error, compressibility does not change down the washer train. The compressibility of these suspensions was found to be intermediate between the other sites investigated.

Hindered settling function data measured at process temperatures for each piece of process equipment is plotted in Figure 10.3. The settler sample is considerably less permeable than all of the other samples, due to its very higher liquor viscosity. With the exception of the settler sample, the permeability of the other units in the washer train are relatively close together. Nothing can be inferred from Figure 10.3 about the influence of flocculation conditions on permeability because all samples have different liquor viscosities. However, the effect of liquor viscosity can be easily corrected because permeability is proportional to liquor viscosity. The hindered settling functions corrected to a standard viscosity of 1 mPa s are shown in Figure 10.4. The trend in the results suggests that the flocculated network structure changes after the settlers to become more permeable in the washer train. Within the limits of reproducibility, the corrected permeabilities do not vary through the washer train, which suggests that the network structure is unaffected also.
Figure 10.1: Comparison of underflow solids volume fractions with gel point at Worsley refinery.

Figure 10.2: Compressive yield stress of Worsley refinery underflow suspensions at characterisation temperatures given.
Figure 10.3: Hindered settling function of Worsley refinery underflow suspensions at characterisation temperatures given.

Figure 10.4: Hindered settling function of Worsley refinery underflow suspensions corrected to a standard viscosity of 1 mPa s.

10.2.4 SHEAR RHEOLOGY RESULTS

Results for the viscosity shear rate behaviour of underflows from the washer train at the Worsley Refinery are plotted in Figure 10.5. As with the Pinjarra results, the data are spread
over about 1.5 orders of magnitude, with the lowest viscosity being attributed to the settler underflow and the highest viscosities being from the 3rd and 4th washer underflows, attributed to the higher solids and lower temperatures.

Results are presented in Figure 10.6 for the shear yield stress of the underflows from each washer. Additionally, selected samples were tested at different temperatures and over a range of solids concentrations. For the samples tested at varied temperatures (1st and 4th washer underflows) the yield stresses were observed to increase with increasing temperature.

Data were obtained probing the variation of the shear rheology of an underflow suspension with changing temperature. Results are presented in Figure 10.7 for the 4th washer underflow at 50 and 70°C. It is clear that there is an effect of temperature on the rheology with an increase in shear yield stress being observed at the higher temperature, but the reverse at higher shear rates.

Figure 10.5: Viscosity versus shear rate behaviour of Worsley underflow suspensions with temperatures and solids concentrations (volume %) indicated.
Figure 10.6: Shear yield stress data as a function of solids volume fraction for Worsley underflow suspensions with temperatures indicated.

Figure 10.7: Shear stress versus shear rate behaviour of Worsley 4th washer underflow at two temperatures with solids concentration (volume %) indicated. Note that the shear yield stress value at 70°C was determined by interpolation of results at other solids concentrations.
10.3 WORSLEY SITE VISIT 2000

10.3.1 WORK PROGRAM

The year 2000 Worsley site visit took place over two weeks from 24th July to 4th August 2000. The major dewaterability characterisation equipment requirements included numerous measuring cylinders, sample containers and temperature controlled water baths. For shear yield stress measurements, two techniques were used including the slump test and a Haake rheometer for the vane test. The work program, performed by me, involved dewaterability and shear yield stress characterisation of red mud underflow suspensions from two of the new deep cone thickeners. The units studied were operating as a settler (high rate) and a 5th washer (high rate). The characterisation for each of the vessel underflows included hindered settling function determinations over a wide range of solids concentrations using transient batch settling tests. Characterisation was completed in order to provide inputs to the steady state thickener model and the pressure filtration model, both of which are described in Chapter 7.

10.3.2 WASHER UNDERFLOW DEWATERABILITY AND SHEAR RHEOLOGY CHARACTERISATION

10.3.2.1 Experimental Method

Sampling

Underflow suspension samples were collected from the high rate settler and the high rate 5th washer at the Worsley Alumina Refinery. Overflow liquor was manually collected directly from the top of the vessels using a metal cup on the end of a metal rod.

Dewaterability Characterisation

Transient batch settling tests were conducted at 90°C for settler (high rate) underflow suspensions and at 50°C for 5th washer (high rate) underflow suspensions to obtain permeabilities at solids concentrations relevant to thickener operation. Equilibrium batch settling tests were conducted on each sample at ambient temperatures. The settling test procedures are detailed in Chapter 6.

Shear Rheology Characterisation

Numerous shear yield stress tests were conducted for both vessels over a range of solids concentrations at 80-90°C for settler suspensions and 40-50°C for 5th washer (high rate) suspensions. This involved taking the as-received underflow and either diluting or centrifuging it to change the solids concentration, sonicating the sample for 30 minutes at elevated
temperature, mixing slowly by hand for 50 revolutions, and then measuring the shear yield stress using either the vane test or the slump test (see Chapter 5 for details).

Temperature dependence was determined using the vane test on a settler (high rate) suspension. The method involved equilibrating the suspension temperature in a thermostated water bath, followed by mixing slowly by hand for 50 revolutions, measuring the temperature, measuring the shear yield stress and then remeasuring the temperature. This test was repeated over a range of temperatures in two cycles to confirm that precipitation was not compromising the results.

**10.3.2.2 Dewaterability Results**

Dewaterability characterisation results from 1999 contained an inadequate amount of permeability data at solids concentrations of relevance to thickener operations. The pressure filtration data, though useful, is at concentrations well above that which is achieved in a thickener, so transient batch settling tests were planned to characterise permeabilities near the gel point.

The new high rate settler and high rate 5th washer underflow suspension permeabilities were characterised using the initial settling rates from transient batch settling tests at a range of solids concentrations at 90°C and 50°C respectively. The as-sampled material properties of each of these suspensions is listed in Table 10.2.

The viscosities of the liquors as a function of temperature for both the high rate settler and the high rate 5th washer decrease by up to an order of magnitude over the range of temperatures from 20 to 100°C. This information is useful in predicting effects of temperature fluctuations, whether they be environmental or process based, on the permeabilities of Bayer process muds and hence thickener performance, but cannot be presented here for confidentiality reasons.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Settler Underflow (high rate)</th>
<th>5th Washer Underflow (high rate)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Underflow Solids Range, $\phi_u (v/v)$</td>
<td>CONFIDENTIAL</td>
<td>CONFIDENTIAL</td>
</tr>
<tr>
<td>Yield Stress Range, $\tau_y$ (Pa)</td>
<td>10</td>
<td>Above 100</td>
</tr>
<tr>
<td>Solids Density, $\rho_s \text{ (kgm}^{-3}\text{)}$</td>
<td>3720</td>
<td>3720</td>
</tr>
<tr>
<td>Liquor Density, $\rho_l \text{ (kgm}^{-3}\text{)}$</td>
<td>CONFIDENTIAL</td>
<td>CONFIDENTIAL</td>
</tr>
</tbody>
</table>

**Table 10.2:** Summary of as-sampled material properties of underflows of Worsley thickeners modelled.

Figure 10.8 presents the full characterisation of $P_y(\phi)$ over the range of solids from close to the gel point to high solids data. Note that the data used for the high end were obtained during the previous visit in 1999 from pressure filtration. The low solids data were obtained from equilibrium batch settling tests. Combination of the data from two separate visits is not considered problematic due to the relatively small differences observed between compressive
yield stress data from filtration compared to the large change in magnitude over the full solids concentration range (3.5 orders of magnitude).

**Figure 10.8:** Compressive yield stress as function of solids volume fraction for Worsley washers investigated. Data determined using transient batch settling and pressure filtration in 1999 and 2000.

Figure 10.9 presents the hindered settling function for the settler (high rate) and 5th washer (high rate) corrected to the modelling process temperatures of 105°C and 50°C respectively. The data spans the complete range of solids concentrations from well below the gel point to high solids concentrations where, as with the compressive yield stress data, filtration data from the 1999 visit has been combined with $R(\phi)$ data from settling experiments from the August 2000 visit. Justification for this integration of data from different site visits is as before. To account for the influence of temperature on liquor viscosity, the permeabilities have been corrected by the ratio of the test temperature liquor viscosities to the process temperature liquor viscosities.
Figure 10.9: Hindered settling function as a function of solids volume fraction for Worsley washers investigated at process temperatures (high rate settler at 105°C and high rate 5th washer at 50°C). Data determined using transient batch settling and pressure filtration in 1999 and 2000.

10.3.2.3 Shear Rheology Results

Figure 10.10 shows the shear yield stress as a function of solids concentration for the high rate settler and the high rate 5th washer suspensions as near to their process temperatures as was practical. Clearly, the high rate settler suspension exhibits a very sharp increase in structure at a solids volume fraction of 0.28-0.29 due to the significant presence of sand in the suspension. Conversely, the shear yield stress of the high rate 5th Washer suspension increases gradually with solids concentration due to the relatively low sand fraction. Empirical site knowledge suggests that suspensions with shear yield stresses greater than about 150 Pa are not readily pumpable and cause excessive rake torques. This upper limit enables the maximum practical underflow solids concentration to be determined from Figure 10.10 as 0.26 and 0.28 for the high rate settler and the high rate 5th washer respectively, not accounting for any margin of safety.

Figure 10.11 shows the shear yield stress as a function of temperature for a high rate settler suspension with a solids volume fraction of 0.248. The data shows an approximately linear increase in shear yield stress with temperature at a rate of 0.3148 Pa/°C. As a rule of thumb, the shear yield stress temperature variation could be approximated as 10% for every 10°C.
Figure 10.10: Shear yield stress as a function of solids volume fraction for Worsley suspensions investigated at test temperatures of 80-90°C for the high rate settler and 40-50°C for the high rate 5th washer.

\[
\tau_y = 0.3148 T + 17.313
\]

Figure 10.11: Shear yield stress as a function of temperature for a Worsley high rate settler suspension with a solids volume fraction of 0.248.
10.4 STEADY STATE THICKENER AND ROTARY DRUM FILTER MODELLING

10.4.1 WORK PROGRAM

The steady state thickening performance of numerous thickeners at the Worsley Alumina Refinery has been modelled using material properties and relevant geometric information. The thickeners include a high rate settler, a high rate 5\textsuperscript{th} washer and a flat bottomed 4\textsuperscript{th} washer. The material properties used include the dewaterability curve fits of underflow suspensions at process temperatures. Two types of modelling output have been presented:

i) Thickener underflow solids concentration as a function of feed solids flux calculated for a range of suspension bed heights.

ii) Solids residence time in the bed as a function of underflow solids concentration (linked to solids flux) for a range of different bed heights.

Comparison of the predicted output with the actual process output enables estimation of the permeability enhancement factor.

The performance of a rotary drum filter has also been modelled using the dewaterability of the 4\textsuperscript{th} Washer underflow as input data.

10.4.2 CURVE FITTING & PROCESS DATA

Steady state thickener modelling requires thickener dimensions, the feed solids concentration and suspension dewaterability in terms of the compressive yield stress and the hindered settling function. The experimental compressive yield stress and hindered settling function data from the high rate settler and high rate 5\textsuperscript{th} washer are shown in Figures 10.8 and 10.9, along with associated curve fits. The compressive yield stress data were fitted to Equation 7.3, while the hindered settling function data were fitted to Equation 7.6. Fitting parameter data for the two washers corrected to process temperatures (high rate settler 105°C, high rate 5\textsuperscript{th} washer 50°C), by a liquor viscosity correction of parameter $r_a$, are presented in Table 10.3 below. In addition, the parameters for the high rate 5\textsuperscript{th} washer have been liquor viscosity corrected to estimate the 4\textsuperscript{th} washer (flat-bottomed) underflow dewaterability and the rotary drum filter feed dewaterability at 50°C. The feed solids volume fraction used in modelling work was 0.04 for each thickener and 0.17 for the rotary drum filter.
<table>
<thead>
<tr>
<th>Vessel</th>
<th>$T$ (°C)</th>
<th>$\eta_{\text{liq}}$ (mPa s)</th>
<th>$\rho_{\text{liq}}$ (kg m$^{-3}$)</th>
<th>$\phi_{g}$</th>
<th>$p_{a}$</th>
<th>$p_{b}$</th>
<th>$p_{m}$</th>
<th>$p_{n}$</th>
<th>$r_{a}$</th>
<th>$r_{b}$</th>
<th>$r_{n}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Settler (high rate)</td>
<td>105</td>
<td>1.639</td>
<td>1346</td>
<td>0.191</td>
<td>82.3</td>
<td>-57.6</td>
<td>387</td>
<td>0.181</td>
<td>6.79E14</td>
<td>0</td>
<td>6.01</td>
</tr>
<tr>
<td>5$^{\text{th}}$ Washer (high rate)</td>
<td>50</td>
<td>0.647</td>
<td>1033</td>
<td>0.187</td>
<td>31.4</td>
<td>-2.24</td>
<td>13.7</td>
<td>0.859</td>
<td>1.64E13</td>
<td>0</td>
<td>3.71</td>
</tr>
<tr>
<td>4$^{\text{th}}$ Washer (flat bottomed)</td>
<td>50</td>
<td>0.877</td>
<td>1077</td>
<td>0.187</td>
<td>31.4</td>
<td>-2.24</td>
<td>13.7</td>
<td>0.859</td>
<td>2.23E+13</td>
<td>0</td>
<td>3.71</td>
</tr>
<tr>
<td>Rotary Drum Filter</td>
<td>50</td>
<td>0.877</td>
<td>1077</td>
<td>0.187</td>
<td>31.4</td>
<td>-2.24</td>
<td>13.7</td>
<td>0.859</td>
<td>2.23E+13</td>
<td>0</td>
<td>3.71</td>
</tr>
</tbody>
</table>

Table 10.3: Summary of $P_{s}(\phi)$ and $R(\phi)$ fitting parameter data for Worsley thickener underflows and rotary drum filter feed with $r_{n}$ corrected to account for liquor viscosities at process temperatures.

10.4.3 SETTLER (HIGH RATE) MODELLING RESULTS

Figure 10.12 shows the results of modelling the steady state thickening behaviour of the high rate settler. The model inputs included the high rate settler dimensions, the underflow dewaterability parameters listed in Table 10.3 and a feed solids volume fraction of 0.04. The results show the predicted solids flux fed to the thickener as a function of the underflow solids concentration for a range of suspension bed heights. The flux is in units of tonnes hour$^{-1}$ m$^{-2}$ of thickener surface area where the area used is the cross sectional area of thickener at the top of the bed. Additionally, lines corresponding to different yield stresses are indicated as a guide to the handleability of the underflow at different solids concentrations. The feed solids concentration and gel point are also indicated.

The output solids from the high rate settler suggested by the model predictions in Figure 10.12 are substantially different to the typical underflow solids observed in the process. Thus, assuming that the calculations are correct, there is something that effectively increases the permeability of the suspension, by a factor of 104.

There are two factors that may have contributed to this discrepancy. The first lies in the assumption that the material that is taken from the underflow of the thickener is representative of the flocculated material that feeds into the top of the bed. Observations of the effect of shear on freshly flocculated suspensions suggest that, once sheared, flocculated networks exhibit lower permeabilities. The second factor contributing to the discrepancy is the effect of shear on the flocculated solids due to raking. The simple 1-dimensional model for thickening employed here has no facility for the incorporation of shear and it is known that the high rate ALCAN type of thickener is highly sheared. Previous research has suggested that the effect of shear on a
flocculated network is to liberate floc-bound water, effectively increasing the permeability by a massive extent.

The shear yield stresses at various solids concentrations are also shown in Figure 10.12, showing the relationship between the flux, the underflow solids and the expected handleability of the material at those solids. The results in Figure 10.12 indicate that the settler underflow material would have had a very low shear yield stress. However, the indication is that if, for some reason, a region of the settler bed became stagnant and material resided in this region whilst still consolidating, then if it spent twice as long in the bed, the yield stress of the material in the stagnant region may increase to 100 Pa. Longer stagnation would result in even higher yield stress material being locally produced, leading to potential problems with rake torques and bogging. Thus the shear yield stress data in Figure 10.12 may be used to estimate the susceptibility of a settler to residence time distribution problems. The other factor not considered here is auto precipitation, where the added problems associated with auto precipitation in a stagnant region would greatly exacerbate the increases in yield stress.

The thickener model outputs may be converted to a bed solids residence time, indicating the average time a solid particle would spend in the bed. Figure 10.12 shows the predicted bed solids residence time versus underflow solids for the high rate settler. The predicted underflow solids corresponding to the true solids flux is outside the range of the data in Figure 10.12; however if the solids corresponding to the effective flux (including the effect of raking) is used, a bed solids residence time of around 100-200 hours is indicated for the underflow solids concentration with a bed height between 5-10 m. Scaling this residence time by the effective increase in permeability obtained through raking, a factor of roughly 100, indicates that the real residence times of solids in the bed are of the order of 1 – 2 hours.
Figure 10.12: Steady state thickener modelling predictions based on Worsley high rate settler underflow sample dewaterability, showing solids flux and solids residence time in suspension bed as functions of solids volume fraction for various bed heights.
10.4.4 5TH WASHER (HIGH RATE) MODELLING RESULTS

Figure 10.13 shows the solids flux – underflow solids relationship for the high rate 5th washer. The typical process flux in this washer is similar to that for the high rate settler. Again, this flux corresponds to the permeability-limited regime of operation of the washer.

Figure 10.9 showed earlier that at low solids the $R(\phi)$ data (which dictates washer output in this regime of operation) for the high rate 5th washer underflow were higher than for the high rate settler. This is reflected in the lower underflow solids for a given solids flux.

Just like for the high rate settler, a significant discrepancy is observed between the model output predictions and the observed underflow solids from underflow sampling. Reasons for the discrepancy have already been discussed. Note that the observed underflow solids for the 5th washer are higher than for the settler, indicating more efficient raking in the final washer. The “permeability enhancement factor” for the high rate 5th washer is of the order of 200. The results show that the actually observed underflow solids outputs are moving into the regime of compressibility limited operation for small bed heights.

As with the settler predictions, shear yield stress data are also presented in Figure 10.13. The results indicate that higher yield stress materials will be encountered for lower underflow solids concentrations than in the settler. This suggests that particle size segregation should be easier to control in the 5th washer.

Figures 10.13 also shows the bed solids residence time for the high rate 5th washer as a function of the underflow solids concentration. Using the same method as was used for the settler, the solids residence times estimated based on the measured underflow solids concentrations and the permeability enhancement factor are from around 0.4 to 1.5 hours for bed heights of between 0.5 and 5 m.
Figure 10.13: Steady state modelling predictions of high rate thickening performance based on Worsley 5th Washer underflow sample dewaterability, showing solids flux and solids residence time in suspension bed as functions of solids volume fraction for various bed heights.
10.4.5 4TH WASHER (FLAT BOTTOMED) MODELLING RESULTS

An estimate of the flat bottomed 4th washer underflow dewaterability at 50°C is provided in Table 10.3. The properties are basically the same as for the high rate 5th washer underflow with the exception of a scale factor difference for the $R(\phi)$ magnitude. Modelling of this flat bottomed thickener will give a similar solids flux versus underflow solids concentration curve that only needs to be corrected for the $R(\phi)$ scale factor. If the vessel is operating in a permeability limited regime, then this approximation will be accurate. 4th washer underflow sampling in 1999 produced an underflow solids volume fraction of 0.193 for a solids flux of 0.137 tonnes hr$^{-1}$ m$^{-2}$ and a suspension bed height of 1-2 metres. For this underflow solids concentration with a suspension bed height of 1 metre or more, the predictions in Figure 10.13 suggest that the process is permeability limited. At the underflow solids concentration of 0.193, the predicted solids flux is $0.01 \times (0.647/0.877) = 0.00738$ tonnes hr$^{-1}$ m$^{-2}$ (liquor viscosity corrected). Therefore, the permeability enhancement factor is calculated to be 18.6. The difference relative to the high rate 5th washer is clear.

10.4.6 ROTARY DRUM FILTRATION MODELLING

Some of the underflow from the 4th washer is fed to a bank of rotary drum filters for the final stage of dewatering before pumping to the bauxite residue disposal area. The reader is referred to Chapter 7, section 7.3 for a description of rotary drum filtration and associated process modelling calculations.

The rotary drum filter feed suspension dewaterability curve fit parameters shown in Table 10.3 enable prediction of filtration behaviour, where a filter cake is assumed to form from the membrane or filter cloth. For a specified pressure gradient, $\Delta P$, the solids concentration at the membrane, $\phi_{base}$, is directly determined from the compressive yield stress. The average solids concentration of the filter cake, $\phi_{cake}$, is assumed constant during filtration for a constant specified pressure gradient. The results of this analysis are presented and compared with the compressive yield stress in terms of solids volume fraction below in Figure 10.14. This data is also presented in terms of solids weight fraction in Figure 10.15, but it should be noted that the washing does increase the solids weight fraction due to liquor density changes. The filtration parameter $\beta^2$ is predicted in Figure 10.16 as a function of applied pressure gradient, $\Delta P$, for rotary drum filter feed at 50°C with feed solids concentrations of 17 % v/v (41.4 % w/w) using dewaterability curve fits given in Table 10.3. For an applied vacuum pressure of 50 kPa, the predictions in Figures 10.14-10.16 are summarised in Table 10.4.
Table 10.4: Summary of predicted solids concentrations for filtration of Worsley rotary drum filter feed at 50°C with applied vacuum pressure of 50 kPa and feed solids concentration of 17% v/v (41.4% w/w) using dewaterability curve fits given in Table 10.3.

Filter operation has been modelled assuming a feed solids volume fraction of 0.17 (41.4% w/w), an applied vacuum pressure of 50 kPa and a filter cloth area of 100 m². Throughputs for filters with cloth areas that differ from 100 m² can be scaled accordingly. It has been assumed that approximately 25% of the drum is submerged under normal operation. The height of the filter cake, $h_{cake}$, has been plotted as a function of submerged filtration time in Figure 10.17, where $h_{cake}$ is a function of cycle time and applied pressure, but not affected by throughput.

According to Table 10.4, 50 kPa of vacuum pressure gives average filter cake solids volume fraction, $\phi_{cake}$, of 0.334 (63.3% w/w) assuming insignificant flow resistance in the cloth. This prediction compares well with the measured value of 0.351. The predicted rotary drum filter solids throughput is calculated using

$$SolidsThroughput = \frac{\rho_{sol} \phi_{cake} h_{cake} A_{cloth}}{t_{cycle}}, \quad (10.1)$$

where cycle time is given by

$$t_{cycle} = \frac{t_{submerged}}{0.25}, \quad (10.2)$$

Solids throughput predictions are shown in Figure 10.18 as a function of cycle rate.

Further modelling of the cake washing would require an estimate of the cloth area subjected to washing and the temperature of the wash water. Reliable prediction of the extent of washing is beyond the scope of this analysis, so instead, an approximate tool is proposed. The following equation provides an initial guess of the potential wash liquor throughput from the rate of filtration at the end of the cake formation stage.
\[ WashWaterThroughput = \frac{\rho_{\text{lig}} A_{\text{clothwashed}}}{2} \left( \frac{\beta^2}{t_{\text{submerged}}} \right)^{1/2}. \] \hspace{1cm} (10.3)

**Figure 10.14:** Comparison of filter cake solids volume fraction in filtration for a specified pressure gradient with compressive yield stress for rotary drum filter feed at 50°C with feed solids concentrations of 17% v/v (41.4% w/w) using dewaterability curve fits given in Table 10.3.
Figure 10.15: Comparison of filter cake weight fraction in filtration for a specified pressure gradient with compressive yield stress for rotary drum filter feed at 50°C with feed solids concentrations of 17 % v/v (41.4 % w/w) using dewaterability curve fits given in Table 10.3. Note that a washed suspension has a different solids weight fraction to an unwashed suspension due to liquor density differences. There may also be other differences in material properties due to washing not accounted for here.

Figure 10.16: Prediction of $\beta^2$ as a function of applied pressure gradient for filtration of rotary drum filter feed at 50°C with feed solids concentrations of 17 % v/v (41.4 % w/w) using dewaterability curve fits given in Table 10.3.
Figure 10.17: Prediction of filter cake height as a function of submerged filtration time for filtration of rotary drum filter feed at 50°C with feed solids concentrations of 17 % v/v (41.4 % w/w) using dewaterability curve fits given in Table 10.3.

Figure 10.18: Prediction of solids throughput as a function of cycle rate for rotary drum filtration of rotary drum filter feed at 50°C with feed solids concentrations of 17 % v/v (41.4 % w/w) using dewaterability curve fits given in Table 10.3.
10.5 GENERAL DISCUSSION AND OBSERVATIONS

10.5.1 WASHER TRAIN UNDERFLOW DEWATERABILITY

Overall, Worsley suspensions appear to be intermediate in compressibility and permeability relative to other sites which intuitively seems inconsistent with the low flocculation doses applied. Higher compressibilities and lower permeabilities would have been anticipated, but this may be due to feed well design and the methodology employed in floc formation, or variations in particle size distribution.

Because of the reasonably high suspension compressibility, Worsley has the potential to achieve greater dewatering, possibly to over 20% v/v, but this was yet to be realised with flat bottomed thickeners in 1999. The significant issue arising from this analysis is the low extent of washing and low underflow solids concentrations achieved in the washer train. An explanation for this lack of performance is possibly the low process temperatures, suspected low bed heights and low rates of washing water addition.

In 1999, process temperatures were up to 20°C below process set-points due to various process instabilities during the sampling period. Maximising process temperatures will lower liquor viscosities and significantly improve settling and compression rates and provide higher underflow concentrations, if desired.

Secondly, the flat bottomed vessels do not appear to utilise compressional dewatering to achieve high underflow solids concentrations, because the vessels are operated with high solids fluxes and the underflow solids concentration is permeability limited. For high solids to be achieved, the solids flux would need to be reduced. If the solids flux were reduced enough, high underflow concentrations would be controlled by suspension bed height. Process data on bed heights suggest that bed heights were low, and are kept low to control rake torques. The shear yield stress of a suspension increases with increasing concentration and determines the rake torque and pumpability. The maximum torque that the rakes are allowed to withstand or the maximum pumpable solids concentration determines the maximum underflow solids concentration and bed height. The limitation that prevents higher underflow concentrations from being pursued currently is that the flat based thickeners already operate at close to rake torque limits.

The characterisation of suspension dewaterability and process modelling for two high rate thickeners, one operating as a settler and one operating as a 5th washer was performed. Modelling results show the region of operation of both the settler and the 5th washer to be predominantly in the “permeability limited region” of thickener operation, meaning that the
output solids concentration will be dominated by the low solids permeability of the material (i.e. settling rates at low solids concentrations). The effect of bed height is predicted to be minimal. This knowledge implies that optimisation of high rate thickener operation requires improvement in the permeability and settling rate of the material from the feed solids up to just above the gel point and optimisation of the gel point itself.

The predicted underflow solids at typical process throughputs in the high rate thickeners investigated was lower in each case than the measured values. The differences between the predicted and measured underflow solids is partly attributed to the effects of shearing on the samples tested during the thickening and sampling processes (which will tend to reduce the permeability). In addition, the effect of raking is not accounted for in the 1-D thickener model employed. These combined effects, suggest that the “effective permeability” in the high rate thickeners is of order of 100-200 times higher than that measured directly. The permeability enhancement defined in this manner, shown in Table 10.5, may be used as an empirical measure of raking effectiveness for different thickening operations.

Shear yield stress results were also determined for the underflows of both the high rate settler and the high rate 5th washer. The results allow combination of knowledge of the throughput of a thickener and its expected output with the handleability of the underflow. This comparison gives indications of the consequences of process fluctuations and thickener dead zones. The solids residence time in the bed can influence what yield stress a material may attain in the event of inhomogeneities or “dead zones” causing material to reside for longer in the bed, thus dewatering to a higher solids. Such high solids zones could be a cause of rake torque and bogging problems.

<table>
<thead>
<tr>
<th>Vessel</th>
<th>Type</th>
<th>$T$ (°C)</th>
<th>$PE_{\text{underflow}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Settler</td>
<td>(high rate)</td>
<td>105</td>
<td>104</td>
</tr>
<tr>
<td>5th Washer</td>
<td>(high rate)</td>
<td>60</td>
<td>200</td>
</tr>
<tr>
<td>4th Washer</td>
<td>(flat bottomed)</td>
<td>60</td>
<td>18.6</td>
</tr>
</tbody>
</table>

Table 10.5: Summary of permeability enhancement in thickeners at Worsley alumina refinery.

10.5.2 FILTRATION

Washing and dewatering at Worsley relies heavily on the rotary drum filters. The drum filters wash the cake with hot water and achieve excellent compression. Suspension compressibility in the drum filters has been modelled using the compressibility of the final washer, which is similar to the filter feed. The permeability will be better than that of the feed because of the washing that occurs and subsequent dilution of the liquor.
The drum filters currently operate at about 50 kPa and produce a filter cake with a solids volume fraction of approximately 0.35. The modelling predictions give an average filter cake solids volume fraction of 0.334 at 50 kPa increasing to 0.353 if the vacuum pressure was increased to its maximum of 100 kPa. The predicted increase of only 0.019 suggests that there isn’t much scope for increasing the already high solids concentration output. None-the-less, there is still potential to optimise filter throughput and extent of washing through cycle rates. In addition, more effective washing in the washer train would allow a higher filter throughput.

Use of flocculation and the addition of filter aids in filtration is commonly used to improve permeability and also reduce fouling by ensuring that the filter cake builds homogeneously on the membrane and does not separate on the basis of particle size.

Modelling predicts that drum rotation speed can be varied to operate at any solids throughput, but that it does not influence average cake solids concentrations or extent of washing. The modelling predicts that the only limits on throughput are the mechanically viable range of drum rotation.

The predicted filter cake solids concentration is lower than the estimates of actual values provided and is likely to be due to drying of the filter cake through suction of air into the cake at some stage in the washing and filtration cycle.

### 10.6 CONCLUSIONS

Experimental dewaterability characterisation and solid-liquid separation equipment process modelling for the Worsley Alumina Refinery included the following:

- Auditing of the process employed to wash and dewater red mud suspensions.
- Shear rheology and dewaterability characterisation of underflows from each solid-liquid separation stage.
- More complete dewaterability characterisation of underflows from two new high rate thickeners operating as a settler and a 5th washer enabling more appropriate hindered settling function curve fitting.
- Steady state thickener modelling of the high rate settler, high rate 5th washer and flat bottomed 4th washer, with evaluation of the permeability enhancement of raking and evaluation of solids residence time in the suspension bed.
- Rotary Drum Filtration modelling with prediction of filter cake solids concentration, filter cake thickness, solids throughput and the influence of drum speed.
In 1999, characterisation and modelling at Worsley revealed how red mud suspensions vary from having thick liquors at over 100°C in the settlers to having dilute liquors in the washed rotary drum filter cakes at reduced temperatures. The suspension shear rheology and dewaterability was shown to be very consistent throughout the washer train. Effects due to temperature and liquor viscosity were largely scalable and there was only a minor permeability improvement down the washer train attributable to thinner liquors and increased cumulative flocculant dose. More complete permeability characterisation performed on red mud produced as underflows from a new high rate settler and a new high rate 5th washer demonstrated how permeability varies by more than five orders of magnitude from low to high solids concentrations. Modelling results demonstrate that both vessels operate in a permeability-limited region of operation. The options open for optimisation of dewatering involve increasing the permeability of the muds at low solids concentrations through increasing the free settling rate, the gel point and also the bed height (if in the converging section where the effective bed solids flux would be improved). The modelling results also show the improvements in dewatering that are observed through internal raking and shear processes in the high rate thickeners relative to the flat based thickeners. An effective permeability enhancement factor of 100-200 was attributed to raking and shear processes in the high rate thickeners compared with only 18.6 for a flat based washer. Modelling results show that improvements in extent of dewatering can potentially be achieved with higher bed heights and lower solids fluxes, subject to rake torque limits not being exceeded. The relationship between shear yield stress and the flux - underflow solids relationship is also discussed with respect to stagnation and rake torque problems. Rotary drum filtration modelling shows that solids throughput can be controlled by cycle rate alone.
Chapter 11

QUEENSLAND ALUMINA REFINERY

11.1 INTRODUCTION

The Queensland Alumina Limited (Gladstone) Alumina Refinery located on in the port city of Gladstone 500 km north of Brisbane in Queensland processes bauxite from Weipa in Northern Queensland. The refinery is the world’s largest with an annual capacity of 3.65 million tonnes of alumina (QAL 2001).

The QAL Refinery operates an extensive system of washing, dewatering and neutralisation of red mud suspensions. The QAL red mud washing and dewatering process has 7-8 key solid-liquid separation stages. The first stage includes a number of settlers running in parallel at over 100°C. The next stages include a number of counter-current washer trains also running in parallel and consisting of 4-5 washers in series in each train. In the washer trains, the temperature reduces at every process stage to about 50°C in the final washer. The settlers and washers all have a converging base geometry. The second last stage involves neutralisation by diluting and mixing with seawater in a waste tank at about 35°C. The suspension is then pumped to the residue disposal area where the suspension is further diluted with seawater before the final dewatering stage of consolidation in the residue disposal area. Neutralised liquor run-off from the residue disposal area is filtered before being discharged into the environment, often with a lower dissolved solids concentration than seawater.

In 1999, on site dewaterability and shear rheology characterisations were performed on underflow suspensions from a settler, an entire washer train and a waste tank underflow at as close to process temperatures as was practical. In 2000, more focussed underflow dewaterability characterisations were performed on 3rd washer, 5th washer and waste tank underflow suspensions.

Also in 2000, on site laboratory flocculation tests and dewaterability characterisations were performed on simulated 5th washer feed suspensions and a waste tank suspension. Work included flocculation trials of a copolymer (sodium polyacrylate / polyacrylamide) flocculant, a hydroxamate flocculant and a modified polyacrylamide flocculant on a simulated 5th washer feed. Also, the same copolymer flocculant and a non-ionic flocculant were both tested on a
waste tank sample further diluted with seawater. The waste tank testing also investigated the
effect of making up flocculant in a caustic or non-caustic liquor.

11.2 QAL SITE VISIT 1999

11.2.1 WORK PROGRAM
With Ross de Kretser from the University of Melbourne, a temperature controlled pressure
filtration rig was transported to the Gladstone Alumina Refinery in September 1999. From 6-11
September 1999, the dewaterability and shear rheology of underflow samples from each stage of
solid-liquid separation were characterised using batch settling tests, stepped pressure filtration,
the vane technique and rotational viscometry.

11.2.2 EXPERIMENTAL METHOD

Sampling
Underflow suspensions and overflow liquors were collected one at a time, from each stage of
the QAL red mud washing and dewatering process. An important consideration in sampling
was to ensure that suspensions were representative of what was in the process vessel. To ensure
this, the underflows were sampled before the pump and for overflows, it was ensured that no
dilution occurred.

Characterisation
For each piece of process equipment, the following tests were performed and data recorded:

1) Measurement of the underflow temperature.
2) Recording of process equipment temperature setpoint.
3) Measurement of underflow solids volume fraction by weight loss on drying of suspension
   and liquor.
4) Measurement of liquor viscosity with a Cannon-Fenske viscometer and liquor density using
   a pycnometer.
5) Measurement of gel point using an equilibrium batch settling test.
6) Measurement of dewaterability (compressive yield stress and hindered settling function)
   using stepped pressure filtration. Characterisation temperatures were as close as was
   practical to process setpoint temperatures, subject to the constraint that the filtration test rig
   could not operate above 90°C.
7) Measurement of the shear yield stress using the vane technique.
8) Measurement of suspension viscosity using rotational viscometry of a bob in a large gap.
11.2.3 DEWATERABILITY RESULTS

Properties of the vessels characterised at QAL in 1999 are listed below in Table 11.1 including logged process temperatures, measured underflow temperatures, temperatures at which samples were characterised. It is noted that one washer in the train was offline for de-scaling and as a result, there were only 4 washers in the train.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Underflow Temperature Logged (°C)</th>
<th>Underflow Temperature Measured (°C)</th>
<th>Filtration Characterisation Temperature (°C)</th>
<th>Liquor Viscosity (mPa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Settler</td>
<td>95</td>
<td>90</td>
<td></td>
<td>1.874</td>
</tr>
<tr>
<td>1st Washer</td>
<td>70, 79</td>
<td>90</td>
<td></td>
<td>0.664</td>
</tr>
<tr>
<td>2nd Washer</td>
<td>80</td>
<td>58, 69</td>
<td>70</td>
<td>0.605</td>
</tr>
<tr>
<td>3rd Washer</td>
<td>65</td>
<td>54, 55</td>
<td>70</td>
<td>0.539</td>
</tr>
<tr>
<td>4th Washer</td>
<td>50</td>
<td>45, 50, 50</td>
<td>50</td>
<td>0.654</td>
</tr>
<tr>
<td>Waste Tank</td>
<td>32</td>
<td>50</td>
<td>50</td>
<td>0.642</td>
</tr>
</tbody>
</table>

Table 11.1: QAL washer train process temperatures.

The underflow solids concentrations at all stages of the washing and dewatering process are compared with the gel points determined from batch settling experiments at ambient conditions in Figure 11.1. All of the solids volume fractions quoted assume a solids density of 2900 kg m\(^{-3}\), so multiplication by 2900 gives the concentration in grams per litre (g/L). The underflow concentrations are all above the gel points determined, which suggests that compressional dewatering is employed in all settlers and washers. The exception is the waste tank underflow, but this is a holding tank, not a thickener. Compressional dewatering occurs at solids concentrations above the gel point because the suspension flocs can then form a continuously networked structure that transmits weight to compress the suspension below.

Using the pressure filtration rig, the dewaterability of the underflow suspensions was determined from stepped pressure filtration tests. The filtration rig was operated at the filtration characterisation temperatures listed in Table 11.1. Process temperature variations during the sampling period were significant, but generally within 15°C of the characterisation temperature.

The compressive yield stress data for each piece of process equipment is plotted in Figure 11.2. The compressibility values suggest that the settler sample is slightly less compressible than the washer train samples, but in general, compressibility does not change down the washer train. The waste tank sample is significantly less compressible than the other samples. Influences that cause this change include mixing with seawater to change the suspension pH, surface chemistry and precipitation induced through this dilution process.

The hindered settling function data measured at process temperatures for each piece of process equipment is plotted in Figure 11.3. The settler sample is considerably less permeable than all
of the other samples, due to its significantly higher liquor viscosity. With the exception of the settler sample, the other washer train permeabilities are relatively close together. Nothing can be inferred from Figure 11.3 about the influence of flocculation conditions on permeability because all samples have different liquor viscosities. However, the effect of liquor viscosity can be easily corrected for because permeability is proportional to liquor viscosity. The hindered settling function corrected to a standard liquor viscosity of 1 mPa s is shown in Figure 11.4. The trend in the results suggests that the flocculated network structure becomes more permeable down the washer train from the settler to the 3rd washer. The other notable feature is the low permeability of the waste tank sample. This comparatively low permeability is influenced by being mixed with sea water to give a pH of around 9 and is also influenced by not being flocculated after the mixing.

Overall, the dewaterability of QAL suspensions appears to be high compared with other sites, but there is still scope for maximising the benefit from this advantage.

**Figure 11.1:** Comparison of underflow solids volume fractions with gel point at QAL refinery.
Figure 11.2: Compressive yield stress of QAL refinery underflow suspensions at characterisation temperatures given in Table 11.1.

Figure 11.3: Hindered settling function of QAL refinery underflow suspensions at characterisation temperatures given in Table 11.1.
Figure 11.4: Hindered settling function of QAL refinery underflow suspensions corrected to a standard viscosity of 1 mPa s.

11.2.4 SHEAR RHEOLOGY RESULTS

Results for the rheological characterisation of samples from the QAL refinery are presented in Figures 11.5 to 11.7 including suspension viscosity and shear yield stress versus solids concentration for a range of temperatures.

Results for the suspension viscosity versus shear rate behaviour of the QAL Refinery washer underflow suspensions are presented in Figure 11.5. All of the data exhibit power law behaviour with a similar slope. However, for these results, the spread of data is only just over 1 order of magnitude with the highest viscosity being for the settler underflow and the lowest being for the waste tank underflow. Notably, the solids concentration of the settler underflow was very high, almost twice that of the other refineries, contributing to the higher viscosity. The solids concentration from the waste tank underflow was very low. Given that the underflow solids concentrations of the 1st, 2nd, 3rd and 4th washers are all similar, Figure 11.5 shows that there is very little difference between them in terms of suspension viscosity.

The shear yield stress of underflow suspensions, at as-received underflow concentrations and concentrated/diluted to a range of solids concentrations, are presented in Figure 11.6. Comparison of the results for washers where a range of solids were tested reveals small differences, however most results from each washer appear to fall, in a broad sense, on a single curve. An exception was the waste tank output sample which had a much higher shear yield
stress than those of any of the washers. The “gel like” precipitate formed in the neutralisation of the Bayer process liquors with sea water most likely is the cause of the higher yield stresses. Within Figure 11.6, data for the 4th washer underflow are also presented for tests at 50 and 70°C. As at other sites, a clear increase in the yield stress with increasing temperature is apparent over the range of solids concentrations tested. Figure 11.7 highlights the temperature differences in the 4th washer results, showing a 15-30% increase in shear yield stress for the 20°C temperature increase.

Figure 11.5: Viscosity versus shear rate behaviour of QAL underflow suspensions with test temperatures and solids concentrations (volume %) indicated.
Figure 11.6: Shear yield stress data as a function of solids volume fraction for QAL underflow suspensions with test temperatures indicated.

Figure 11.7: Shear yield stress data as a function of solids volume fraction for QAL 4\textsuperscript{th} washer underflow suspensions at test temperatures of 50\textdegree{}C and 70\textdegree{}C.
11.3 QAL SITE VISIT 2000

11.3.1 WORK PROGRAM

The year 2000 site visit conducted by me with the assistance of Ross de Kretser in the first week took place over two weeks from 4th to 15th December 2000 in two parts.

The first part of the work program involved dewaterability characterisation of the red mud suspensions from the underflows of the 3rd washer, 5th washer and from the seawater diluted underflow of the waste tank. Characterisation was completed in order to provide inputs to the steady state thickening model that has been developed. The characterisation for each washer underflow included compressive yield stress and hindered settling function characterisation over a wide range of solids concentrations.

The second part of the work program involved plunger flocculation tests. The tests involved characterisation of initial settling rate, gel point, permeability near the gel point and pressure filtration. A copolymer (sodium polyacrylate / polyacrylamide) flocculant, a hydroxamate flocculant and a modified polyacrylamide were tested on a simulated 5th washer feed over a range of doses. The same copolymer flocculant and a non-ionic flocculant were tested on a waste tank sample further diluted with seawater with reduced performance observed. The bad results were suspected to be due to precipitation products from the reaction of sodium hydroxide with sea water, so the tests were repeated with no caustic present in the made up flocculant with dramatically improved results.

11.3.2 WASHER AND WASTE TANK UNDERFLOW DEWATERABILITY CHARACTERISATION

11.3.2.1 Experimental Method

Sampling

Underflow suspension samples were collected from a 3rd washer, a 5th washer and a sea water diluted waste tank in a washer train at the QAL Alumina Refinery. Overflow liquor was collected from overflow pump outlets.

Characterisation

Dewaterability characterisation involving gel point tests, transient batch settling tests and pressure filtration tests were conducted on each sample at 60°C for the washer samples and 30°C for the seawater diluted waste tank sample. The transient batch settling tests were conducted on
each sample to obtain permeabilities at solids concentrations relevant to thickener operation. The test procedures are detailed in Chapter 6.

11.3.2.2 Dewaterability Results

Dewaterability characterisation results from 1999 contained an inadequate amount of permeability data at solids concentrations of relevance to thickener operations. The pressure filtration data, though useful, is at concentrations well above that which is achieved in a thickener, so transient batch settling tests were planned to characterise permeabilities near the gel point. 3rd washer, 5th washer and seawater diluted waste tank underflow suspension permeabilities were characterised using the initial settling rates from transient batch settling tests at a range of solids concentrations at 60°C for the washers and 30°C for the waste tank sample. The as-sampled material properties of each of these suspensions is listed in Table 11.2. The 3rd washer underflow solids concentration of 0.098 is unusually low and may be due to a temporary process instability.

For each sample, the hindered settling function corrected to the modelling process temperature is plotted in Figure 11.8, where the low volume fraction data (φ < 0.2) comes from transient batch settling tests and the high volume fraction data comes from pressure filtration tests. The compressive yield stresses are plotted in linear and log-linear co-ordinates in Figures 11.9 and 11.10 where the low pressure data (<1 kPa) comes from equilibrium batch settling tests and the high pressure data comes from pressure filtration. Changes in suspension dewaterability between 1999 and 2000 are almost undetectable, which is quite a surprise for an industrial process with such variable process conditions. The lower compressibility and permeability observed for the waste tank sample in 1999 is again observed in 2000. The extra data, at low volume fraction, produced in 2000 provides a more complete picture of how the hindered settling function varies at solids concentrations of interest to thickener operations. This new data enables more representative curve fitting and thus allows more appropriate modelling predictions. All suspensions sampled had a low shear yield stress, below 20 Pa.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>3rd Washer U/F</th>
<th>5th Washer U/F</th>
<th>Waste Tank U/F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solids Volume Fraction</td>
<td>φ_u (v/v)</td>
<td>0.098</td>
<td>0.169</td>
<td>0.065</td>
</tr>
<tr>
<td>Yield Stress</td>
<td>τ_y (Pa)</td>
<td>&lt;20</td>
<td>&lt;20</td>
<td>&lt;20</td>
</tr>
<tr>
<td>Solids Density</td>
<td>ρ_sol (kg m⁻³)</td>
<td>2900</td>
<td>2900</td>
<td>2900</td>
</tr>
<tr>
<td>Test Temperature</td>
<td>T_t (°C)</td>
<td>60</td>
<td>60</td>
<td>30</td>
</tr>
<tr>
<td>Modelling Temperature</td>
<td>T_m (°C)</td>
<td>70</td>
<td>50</td>
<td>30</td>
</tr>
</tbody>
</table>

Table 11.2: Summary of as-sampled material properties of QAL washer suspensions characterised.
Figure 11.8: Hindered settling function, $R(\phi)$, as a function of solids volume fraction, $\phi$, for QAL washers investigated at modelling temperatures given in Table 11.2. Data determined using transient batch settling and pressure filtration in 1999 and 2000.

Figure 11.9: Compressive yield stress, $P_y(\phi)$, as a function of solids volume fraction, $\phi$, for QAL washers investigated using transient batch settling and pressure filtration in 1999 and 2000.
Figure 11.10: Compressive yield stress, $P_y(\phi)$, in logarithmic co-ordinates as a function of solids volume fraction, $\phi$, for QAL washers investigated using transient batch settling and pressure filtration in 1999 and 2000.

11.3.3 FLOCCULATION TESTING

11.3.3.1 Experimental Method

*Materials*

**Suspension:** 5th washer underflow suspension

**Process Liquor:** 5th washer overflow liquor

Seawater

**Flocculants:**

- Alclar W50, an anionic copolymer (sodium polyacrylate / polyacrylamide) flocculant supplied by CIBA Specialty Chemicals in powder form.
- Superfloc 1240, a modified polyacrylamide flocculant supplied by Cytec as a water in oil emulsion.
- HX400, a hydroxamate flocculant specially supplied by Cytec in powder form.
- Superfloc N100, a non-ionic flocculant supplied by Cytec in solid form.
Flocculant Makeup

Dilution of the powder and emulsion flocculants required different methods, which are detailed below:

- **Powder Flocculant Makeup:**
  The powder flocculants were prepared at 0.025% w/v by the following method:
  1. weigh out 0.25 g of polymer
  2. add 2 mL of ethanol or methanol
  3. add 98 mL of 17 g/L NaOH
  4. mix by hand (about 2 hours) until dissolved
  5. mix 10 mL of dissolved flocculant with 90 mL of 17 g/L NaOH

When the powder flocculant was later made up in the absence of caustic, de-ionised water was substituted for 17 g/L NaOH.

- **Emulsion Flocculant Makeup:**
  The water in oil emulsion flocculants were inverted and diluted to 0.1% v/v by the following method:
  1. mix by hand (about 2 hours) until dissolved
  2. create a vortex by stirring 198 mL of 17 g/L NaOH
  3. add 2 mL of flocculant emulsion slowly to the edge of the vortex with a syringe
  4. create a vortex by stirring 180 mL of 17 g/L NaOH
  5. add 20 mL of diluted flocculant slowly to the edge of the vortex with a syringe

Suspension Makeup

For the flocculation trials, a simulated 5th washer feed suspension and a simulated waste tank thickener feed were prepared by the methods given below:

- **Simulated 5th Washer Feed**
  Underflow suspension (5th washer) was diluted with process liquor (5th washer overflow) to 50.4 g/L in 4 by 20 litre drums to simulate a 5th washer suspension feed.

- **Simulated Waste Tank Thickener Feed**
  Waste tank underflow suspension is a final washer underflow diluted predominantly with seawater. Operation of a thickener with this suspension would involve further dilution with seawater before flocculation. 1 part waste tank underflow suspension was mixed with 2.8 parts seawater on a volume basis to simulate a waste tank thickener feed at 51.2 g/L.
Flocculation Test Method

The plunger flocculation method was used in all flocculant trials at QAL. The method is briefly detailed below. In a thermally controlled water bath at 30 and 60°C, 1 L measuring cylinders were filled with suspension. A suspension was plunged strongly a few times to homogenise the sample, made up flocculant was added and the suspension was plunged 3 times over 12 seconds using a timer. The settling time for the bed height to fall from 900 mL to 700 mL was recorded. This method can be used to measure relatively high settling rates because of the 10 cm distance from the 900 to 700 mL marks, but the method can lead to errors when settling slows before reaching the 700 mL mark.

Dewaterability Characterisation

For selected samples, dewaterability characterisation was conducted. This included batch settling tests for compressibilities and permeabilities at solids concentrations near the gel point and pressure filtration tests for compressibilities and permeabilities high solids concentrations.

11.3.3.2 5th Washer Flocculant Test Results

Flocculant trials were conducted using the plunger method with a simulated 5th washer feed suspension at 60°C. The suspension was prepared at 50.4 g/L by dilution of 5th washer underflow suspension with 5th washer overflow suspension. The flocculants tested were

- Alclar W50, an anionic copolymer (sodium polyacrylate / polyacrylamide) flocculant supplied by CIBA Specialty Chemicals.
- HX400, a hydroxamate flocculant specially supplied by Cytec.
- Superfloc 1240, a modified polyacrylamide flocculant supplied by Cytec.

Free Settling Rate & Permeability

As shown in Figure 11.11, settling rates generally increase with flocculant dose for all flocculants tested. The settling rates of the AW50 and S1240 flocculants are significantly higher than for the HX400 flocculant at similar dosages. Note that the observation of the lower settling rate for the HX400 flocculant is not significant given that it is normally not used for flocculation in a low caustic environment at 60°C and is often also added in combination with another flocculant.
Figure 11.11: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated QAL 5th washer feed at 50.4 g/L, flocculated by the plunger method with a range of flocculants.

Selected flocculated samples were concentrated to 5 times their original concentration and the initial settling rate was determined from transient batch settling tests to determine relative permeabilities near the gel point. The free settling rate results near the gel point, shown in Figure 11.12, give trends in accordance with those of the free settling rates at the flocculation solids concentration. After 24 hours of settling, the suspensions were sheared by plunging 40 times at 2 seconds per plunge and the initial settling rates were recorded again. The shear significantly reduced the settling rates of the AW50 and S1240 samples. The reduction was so extensive that the S1240 suspensions then settled as slowly as the unflocculated sample. In contrast, there was actually a slight increase in the settling rate of the HX400 suspensions after shearing.

Selected samples were also characterised by pressure filtration tests. The hindered settling function results at high solids concentration from pressure filtration, shown in Figure 11.13, are relatively close together, with only small differences between the flocculants. AW50 and HX400 flocculants both showed a reduction in the hindered settling function with increasing flocculant dose.
Figure 11.12: Concentrated free settling rate, $u_{fs}(\phi)$, ($\phi = 0.087$, 252 g/L) as a function of flocculant dose for flocculated simulated QAL 5th washer samples.

Figure 11.13: Hindered settling function, $R(\phi)$, obtained by pressure filtration of QAL 5th washer underflow suspension and a selection of flocculated simulated QAL 5th washer samples.

Compressibility

The freshly flocculated simulated 5th washer feed suspensions were very non-homogeneous and this made traditional gel point analysis impractical. Therefore, to obtain a relative measure of
compressibility near the gel point, each sample was transferred to an individual container with a 58 mm internal diameter and the equilibrium suspension height was recorded after 2 days. From these heights, the average equilibrium solids volume fractions, $\phi_{av}$, were calculated, as shown in Figure 11.14. The AW50 $\phi_{av}$ values were actually higher than for the unflocculated sample when the dose was less than 70 g/t. This phenomenon has been observed at other sites for this copolymer type of flocculant. The dropping off in the value of $\phi_{av}$ for doses of all flocculants greater than 100 g/t could be due to excessive flocculant.

The compressibilities of selected samples at high solids were determined from pressure filtration tests, as shown in Figure 11.15. All flocculated samples were less compressible than the unflocculated sample. For the HX400 and the S1240 flocculants, the compressibility was reduced slightly further with increasing dose, but the trend was reversed for AW50. Regardless of the trends noted, all variations were very small and the observed trends could be invalidated by the magnitude of experimental error with a solids volume fraction magnitude of about 0.01.

![Figure 11.14](image)

**Figure 11.14:** Average equilibrium solids volume fractions, $\phi_{av}$, as a function of flocculant dose for simulated QAL 5th washer samples flocculated by the plunger method.
Figure 11.15: Compressive yield stress, $P_y(\phi)$, as a function of solids volume fraction, $\phi$, obtained by pressure filtration of QAL 5th washer underflow suspension and a selection of flocculated simulated QAL 5th washer samples.

11.3.3.3 Sea Water Diluted Waste Tank Underflow Flocculant Test Results

Flocculant trials were conducted using the plunger method with a simulated seawater mixed waste tank thickener feed suspension at 30°C. The suspension was prepared at 51.2 g/L by dilution of 1 part waste tank underflow suspension with 2.8 parts seawater. The flocculants tested were

- Alclear W50, an anionic copolymer (sodium polyacrylate / polyacrylamide) flocculant supplied by CIBA Specialty Chemicals.
- Superfloc N100, a non-ionic flocculant supplied by Cytec.

The flocculants were made up in 17 g/L NaOH and also in de-ionised water to test the effect of caustic addition on flocculation performance.

Free Settling Rate & Permeability

As shown in Figure 11.16, settling rates generally increase with flocculant dose for all flocculants tested. The settling rates of the AW50 and SN100 flocculants were almost indistinguishable. The key result here is that settling rate is very low when the flocculant is made up in 17 g/L NaOH, even at very high dosages. The explanation for this could be that when the caustic mixes with the seawater, the precipitation of fine particles inhibits the
floculation mechanism. However, when the flocculant is made up in de-ionised water, very high settling rates are observed because precipitation is prevented.

Waste tank samples flocculated with flocculant made up in 17 g/L NaOH were concentrated to over three times their original concentration and the initial settling rate was determined from transient batch settling tests to determine relative permeabilities near the gel point. The free settling rate results near the gel point are shown in Figure 11.17. Low settling rates are observed with only small increases in settling rate with flocculant dose. After one day of settling, the samples were then sheared by plunging 40 times at a rate of 2 seconds per plunge. The settling rates were then measured again and interestingly, they had increased significantly. The explanation for the increase could be that the effect of the fine precipitate had diminished over time or that floc densification occurred with shear.

Selected samples were also characterised by pressure filtration tests. The hindered settling function results at high solids concentration from pressure filtration are shown in Figure 11.18. The key observation is that there is only a small variation in the hindered settling function at high solids concentrations relative to the massive variations in settling rates immediately following flocculation.

![Figure 11.16: Free settling rate, $u_{fs}$, as a function of flocculant dose for simulated QAL waste tank thickener feed at 51.2 g/L, flocculated with a range of flocculants by the plunger method.](image-url)
Figure 11.17: Concentrated free settling rate, $u_{fs} (\phi)$, ($\phi = 0.060$, 174 g/L) as a function of flocculant dose for flocculated simulated QAL waste tank thickener samples.

![Free Settling Rate vs Flocculant Dose](image1)

Figure 11.18: Hindered settling function, $R(\phi)$, obtained by pressure filtration of QAL waste tank underflow suspension and a selection of flocculated simulated waste tank thickener samples.

![Hindered Settling Function](image2)

Compressibility

The compressibilities of selected flocculated simulated waste tank thickener samples at high solids concentrations were determined from pressure filtration tests, as shown in Figure 11.19.
The unflocculated waste tank sample is much less compressible than both the flocculated and unflocculated 5th washer samples shown in Figure 11.15. Yet, flocculation of the waste tank sample still caused a further reduction in compressibility, for all flocculants tested.

![Waste Tank Flocculation Trial](image)

**Figure 11.19:** Compressive yield stress, $P_y(\phi)$, as a function of solids volume fraction, $\phi$, obtained by pressure filtration of QAL waste tank underflow suspension and a selection of flocculated simulated QAL waste tank thickener samples.

11.4 STEADY STATE THICKENER MODELLING

11.4.1 WORK PROGRAM

The steady state thickening performance of a 5th washer at the QAL Alumina Refinery has been modelled using material properties and relevant geometric information. The material properties include the dewaterability curve fits of a typical flocculated simulated feed suspension and an underflow suspension at process temperatures. Two types of modelling output have been presented:

i) Thickener underflow solids concentration as a function of feed solids flux calculated for a range of suspension bed heights.

ii) Solids residence time in the bed as a function of underflow solids concentration (linked to solids flux) for a range of different bed heights.

Comparison of the predicted output with the actual process output enables estimation of the permeability enhancement factor.
Consolidation in residue disposal areas has been modelled using the dewaterability curve fits of the final washer underflow suspension, corrected to the liquor viscosity at 20°C.

### 11.4.2 CURVE FITTING & PROCESS DATA

Steady state thickener modelling requires thickener dimensions, the feed solids concentration and suspension dewaterability in terms of the compressive yield stress and the hindered settling function. The thickener modelled in this study is a 5th washer with a truncated converging base as shown in the schematic in Figure 7.4. The feed solids concentration used in modelling work was a solids volume fraction of 0.055. The operating solids flux of the 5th washer had an average value of about 0.088 t hr⁻¹ m⁻².

Figure 11.20 presents the full characterisation of \( P_y(\phi) \) over the range of solids concentrations from close to the gel point to high solids data for a 5th washer underflow sample and for a flocculated simulated 5th washer sample. The low solids data were obtained from sedimentation tests, while the high solids data were obtained from pressure filtration. The flocculated sample is a simulated 5th washer feed flocculated by the plunger method with 30 g/t Alclar W50 copolymer flocculant. Clearly, the freshly flocculated simulated feed sample is less compressible than the underflow sample. The difference is believed to be due to the underflow having experienced moderate shear in the washer bed. The compressive data were fitted to an exponential-power law functional form given by Equation 7.3, with the \( P_y(\phi) \) fitting parameter data listed for each sample in Table 11.3 below.

Figure 11.21 presents the hindered settling function over the complete range of solids concentrations from well below the gel point to high solids concentrations for the 5th washer underflow sample and for the flocculated simulated 5th washer sample. The hindered settling function data were fitted to an exponential-power law functional form given by Equation 7.7, with the \( R(\phi) \) fitting parameter data for each sample presented in Table 11.3 below.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( T ) (°C)</th>
<th>( \phi_g )</th>
<th>( p_a )</th>
<th>( p_b )</th>
<th>( p_m )</th>
<th>( p_n )</th>
<th>( r_a )</th>
<th>( r_b )</th>
<th>( r_c )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Underflow</td>
<td>50</td>
<td>0.126</td>
<td>31.502</td>
<td>-3.630</td>
<td>34.454</td>
<td>0.772</td>
<td>23.458</td>
<td>17.092</td>
<td>0.833</td>
</tr>
<tr>
<td>Flocculated</td>
<td>50</td>
<td>0.134</td>
<td>35.504</td>
<td>-9.730</td>
<td>33.569</td>
<td>0.507</td>
<td>28.593</td>
<td>12.092</td>
<td>0.587</td>
</tr>
</tbody>
</table>

**Table 11.3:** Summary of \( P_y(\phi) \) and \( R(\phi) \) fitting parameter data for QAL 5th washer underflow and flocculated simulated QAL 5th washer sample both at 50°C.
Figure 11.20: Compressive yield stress in normal and logarithmic co-ordinates as a function of solids volume fraction for QAL 5\textsuperscript{th} washer underflow and flocculated simulated QAL 5\textsuperscript{th} washer sample.
For transient consolidation modelling, the compressive yield stress, $P_y(\phi)$, data for the 5th washer underflow was fitted to Equation 7.3, while the hindered settling function, $R(\phi)$, data corrected to 20°C, was fitted to Equation 7.4. Fitting parameter data for the sample are presented in Table 11.4.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$T$ (°C)</th>
<th>$\phi_a$</th>
<th>$p_a$</th>
<th>$p_b$</th>
<th>$p_m$</th>
<th>$p_n$</th>
<th>$w$</th>
<th>$m$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Underflow</td>
<td>20</td>
<td>0.126</td>
<td>31.502</td>
<td>-3.630</td>
<td>34.454</td>
<td>0.772</td>
<td>1.979E8</td>
<td>-18.583</td>
</tr>
</tbody>
</table>

Table 11.4: Summary of $P_y(\phi)$ and $R(\phi)$ fitting parameter data for QAL 5th washer underflow sample at 20°C.

A liquor material balance can be used to indicate the fractional liquid recovery in the washer as a function of underflow solids concentration, showing the overflow flowrate as a fraction of the total liquid fed to the washer for a given feed solids concentration. Figure 11.22 shows the results for the 5th washer using a feed solids volume fraction of 0.055. At the measured underflow solids volume fraction of 0.169, 71% of the liquor fed to the 5th washer is recovered. Recovery will decrease rapidly as the underflow solids concentration decreases, but will only increase to 80% when the solids concentration rises over 0.22. Because no liquor is recovered from the residue disposal area, this implies that 29% of the liquor fed to the final washer is never recovered.

Figure 11.21: Hindered settling function in logarithmic co-ordinates as function of solids volume fraction for QAL 5th washer underflow and a flocculated simulated QAL 5th washer sample both at 50°C.
**Figure 11.22:** Fractional liquid recovery in 5th washer as a function of underflow solids volume fraction using a feed solids volume fraction of 0.055 (160 g/L).

### 11.4.3 5TH WASHER MODELLING RESULTS

*Predictions based on washer underflow sample dewaterability*

The steady state operation of the 5th washer was modelled using the washer dimensions, a feed solids volume fraction of 0.055 and the dewaterability curve fits of the underflow sample as inputs. The results in Figure 11.23 show the predicted steady state solids flux fed to the thickener and solids residence time in the bed as functions of underflow solids volume fraction for a range of bed heights. The solids flux is in units of tonnes per hour per m$^2$ of thickener surface area where the area used is the cross sectional area of thickener at the top of the bed. The feed solids concentration and gel point are also indicated.

For a typical bed height of 2-5 metres (below the top of the converging section), the true solids flux ranges from 0.615 t hr$^{-1}$ m$^{-2}$ for 2 m of bed height to 0.165 t hr$^{-1}$ m$^{-2}$ for 5 m of bed height. The measured underflow solids concentration of 0.169 listed in Table 11.2 earlier. According to the predictions in Figure 11.23, an underflow solids of around 0.17 would be attained at a solids flux of 0.00384 t hr$^{-1}$ m$^{-2}$ for 2 m of bed height and 0.00396 t hr$^{-1}$ m$^{-2}$ for 5 m. Thus, assuming that the predictions are correct, there is some additional factor that effectively increases the permeability of the suspension, by a factor of 16.0 for 2 m and 4.1 for 5 m of bed height.
Reasons for permeability enhancement have already been suggested in previous chapters and are overviewed in Chapter 13. From the bed solids residence time plot in Figure 11.23, at the actual underflow solids concentration of 0.169, the model predicts a bed solids residence time of 11.57 to 22.96 hours for a bed height between 2 and 5 m. Scaling this residence time by the effective increase in permeability obtained through raking (a factor of 16.0 and 4.2), indicates that the corrected residence times of solids in the bed are of the order of 0.7 to 5.5 hours (2 and 5 m respectively).
Figure 11.23: Steady state thickener modelling predictions based on QAL 5th washer underflow sample dewaterability showing solids flux and solids residence time in bed as functions of underflow solids volume fraction for various bed heights.
Predictions based on flocculated sample dewaterability

Figure 11.21 showed that at low solids, the \( R(\theta) \) data (which dictate washer output in this regime) for the underflow sample was higher than for the freshly flocculated sample. This implies that at low solids concentrations, the freshly flocculated sample is more permeable than the underflow sample and this is reflected in the flocculated sample’s higher predicted underflow solids concentration for a given solids flux.

Figure 11.24 shows the solids flux and residence time versus underflow solids concentration relationship for the same 5\(^{th}\) washer, with the same initial solids volume fraction of 0.055, but with the flocculated sample dewaterability curve fits. Again, the actual solids flux in the process of 0.165 t hr\(^{-1}\) m\(^{-2}\) for 5 m of bed height and 0.615 t hr\(^{-1}\) m\(^{-2}\) for 2 m of bed height, correspond to a predicted underflow solids concentration of 0.09-0.14. A discrepancy is still observed between the model output predictions and the observed underflow solids concentration. Reasons for the discrepancy have been discussed. Note that the flocculated sample is probably more representative of the suspension in the washer than the underflow sample, so the same permeability enhancement calculations are repeated here.

According to the prediction is Figure 11.24 an underflow solids of around 0.17 would be attained with a solids flux of 0.0709 t hr\(^{-1}\) m\(^{-2}\) for a bed height of 2-5 m. The permeability enhancement factor is calculated to be 8.7 for 2 m of bed height and 2.3 for 5 m of bed height. From the bed solids residence time plot in Figure 11.24, at the actual underflow solids concentration of 0.17, the model predicts a bed solids residence time of 6.75 to 13.25 hours for a bed height between 2 and 5 m. Scaling this residence time by the effective increase in permeability obtained through raking, a factor of 2.3-8.7, indicates that the corrected residence times of solids in the bed are of the order of 0.8 to 5.7 hours, (2 and 5 m respectively) which compares well with the 0.7-5.5 hours calculated from the underflow sample analysis.

Comparison of the permeability enhancement factors for the flocculated versus the underflow sample indicate that the effect of flocculation is to enhance permeability by a factor of 1.8 while raking enhances permeability by a factor of 2.3-8.7. Whilst these figures are qualitative, in that the permeability of the solids in the feed well may not be represented exactly, the numbers do correlate with samples tested from other thickening devices.
Figure 11.24: Steady state thickener modelling predictions based on laboratory flocculated QAL 5th washer sample dewaterability showing solids flux and solids residence time in bed as functions of underflow solids volume fraction for various bed heights.
11.5 RESIDUE DISPOSAL AREA CONSOLIDATION MODELLING

Based on the data that has been collected, it appears that suspension that has recently been mixed with seawater has reduced dewaterability in terms of both compressibility and permeability. However, there is evidence in Figure 11.17 to suggest that at least some of this loss of dewaterability may be temporary, but a dedicated program of work would need to be conducted to confirm this postulate. Assuming that dewaterability can be recovered in a reasonably short period of time after mixing with seawater, the dewaterability curve fits of the final washer underflow at 20°C given in Table 11.4 has been used in disposal area consolidation modelling.

The transient consolidation of 20 m of suspension in a residue disposal area with an initial solids volume fraction of 0.2 has been modelled. The results are shown in Figures 11.25 and 11.26. The predicted suspension height falls relatively linearly for about the first 80 days and then slows until consolidation is virtually complete after 500 days. Unfortunately, the effects of thixotropic build up of structure are not accounted for in this modelling. The likely effect of thixotropy is that consolidation will actually terminate earlier than predicted here.

**Figure 11.25:** Transient consolidation modelling predictions based on QAL 5th washer underflow sample dewaterability at 20°C, showing suspension height as a function of time for an initial solids volume fraction of 0.20 and an initial suspension height of 20 m.
Figure 11.26: Initial and steady state consolidation modelling predictions based on QAL 5th washer underflow sample dewaterability for an initial solids volume fraction of 0.20 and an initial suspension height of 20 m.

11.6 GENERAL DISCUSSION AND OBSERVATIONS

11.6.1 WASHER TRAIN UNDERFLOW DEWATERABILITY

1999 characterisation results suggest that compressibility in QAL washer trains is lower than at other sites, while the flocculant audit indicates that the flocculant dosing level is high relative to other sites. The fact that the low compressibility does not vary down the washer train suggests over-flocculation. Permeability values corrected for a standard viscosity of 1 mPa s, clearly show improving permeability down the washer train, but interestingly, this improvement is not reflected in improved underflow solids concentrations for the 2nd, 3rd and 4th washers as shown in Figure 11.1. The underflow solids volume fraction of 0.207 achieved in the 1st washer is much higher than the 0.16 to 0.17 achieved in the 2nd to 4th washers. The lower temperatures at the back end of the washer train increases liquor viscosity and decreases permeability, but not enough to account for the loss of performance.

For converging base thickeners, high underflow solids concentrations are controlled by suspension bed height. Process data on bed heights has not been made available, but the results here suggest that bed heights in the 2nd, 3nd and 4th washers were lower than for the 1st washer. Achievable underflow concentrations are limited by the maximum bed height, which is linked to the suspension shear yield stress. The shear yield stress of a suspension increases with
increasing concentration and determines the rake torque and pumpability. The maximum torque that the rakes are allowed to withstand or the maximum pumpable solids concentration determines the maximum underflow solids concentration. If the current output solids concentration is not causing handling issues relating to rake torque and pumpability, it is suggested that operating with higher bed heights be considered to improve underflow solids concentrations.

Consequently higher underflow concentrations are achievable in the 2nd, 3rd and 4th washers, possibly to over 20% v/v simply with higher bed heights. This improvement would not improve caustic recovery significantly as the overall extent of washing is already substantial, so there is scope for reducing flocculant dose without decreasing process effectiveness. Alternatively, there is potential for higher throughputs with current equipment.

11.6.2 UNDERFLOW SHEAR RHEOLOGY

According to characterisations in 1999, the shear rheology of the settler and washer suspensions are all very similar and show similar shear yield stress behaviour. The key exception being the waste tank suspension which has a much higher shear yield stress. It was demonstrated in Figure 11.7 that temperature increases the shear yield stress.

11.6.3 FLOCCULATION TESTING

Flocculation testing of final washer suspensions has shown that flocculant dose increases free settling rate. However, the hydroxamate flocculant produced significantly lower settling rates than the anionic copolymer (sodium polyacrylate / polyacrylamide) flocculant and the modified polyacrylamide flocculant. Shearing the suspension after flocs had been produced was found to reduce the permeability in transient batch settling tests. At high solids concentrations, there was no discernible permeability difference between different flocculant type and dose. Flocculation was found to reduce compressibility at high solids concentrations, but there were no discernible dose trends.

Flocculation testing of sea water diluted waste tank suspensions with an anionic copolymer (sodium polyacrylate / polyacrylamide) flocculant and a non-ionic flocculant produced very low settling rates when the flocculant was made up in 17 g/L NaOH. These settling rates were insensitive to flocculant type and dose. However, when flocculant was made up in de-ionised water, the settling rates improved by a factor of 4 to 30 with settling rate increasing with dose, but insensitive to flocculant type. In transient settling tests, it was found that for suspensions flocculated in 17 g/L NaOH, rehomogenisation through shearing caused an increase in settling
rate, possibly because the precipitate had been given enough time to agglomerate onto the larger solids particles. In addition, compressive yield stress was found to increase further when suspensions were flocculated with a flocculant made up in sodium hydroxide solution.

11.6.4 PROCESS EQUIPMENT MODELLING

Steady state thickener modelling of the final washer using the dewaterability from both a flocculated feed and an underflow suspension, both predict different dewatering performance. However, neither predict actual performance accurately and a permeability enhancement factor of 2-8 is required to predict the effects of raking and shear processes within the thickener bed. Consolidation modelling of a residue disposal area has predicted that consolidation should be relatively complete within the space of one year, which can be useful information for rehabilitation considerations.

11.7 CONCLUSIONS

Experimental dewaterability characterisation and solid-liquid separation equipment process modelling for the QAL, Gladstone Alumina Refinery included the following:

- Auditing of the process employed to wash and dewater red mud suspensions.
- Shear rheology and dewaterability characterisation of underflows from each solid-liquid separation stage.
- More complete permeability characterisation of 3rd-washer, 5th washer and waste tank underflows near the gel point enabling more appropriate hindered settling function curve fitting.
- Flocculant trials with a simulated 5th washer feed suspension comparing settling rate and other dewaterability parameters for a copolymer (sodium polyacrylate / polyacrylamide) flocculant, a modified polyacrylamide flocculant and a hydroxamate flocculant over a range of doses.
- Flocculant trials doses with a simulated seawater diluted waste tank thickener feed suspension comparing settling rate and other dewaterability parameters for a copolymer (sodium polyacrylate / polyacrylamide) flocculant and a non-ionic flocculant over a range of flocculant doses. The influence of NaOH in the made up flocculant was shown to cause significant reduction in the flocculated suspension permeability.
- Steady state thickener modelling of the 5th washer, with evaluation of the permeability enhancement of raking and flocculation, solids residence time in the suspension bed and liquor recovery.
- Transient residue disposal area consolidation modelling.
QAL site visit testing allowed characterisation of washer dewaterability with permeability near the gel point and plunger method flocculation trials on a 5th washer simulated feed and a seawater diluted waste tank sample. The flocculants tested included a copolymer (sodium polyacrylate / polyacrylamide) flocculant, a hydroxamate flocculant, a modified polyacrylamide flocculant and a non-ionic flocculant. A key finding for the seawater diluted sample was that the presence of caustic in the made up flocculant reduced flocculation performance due to precipitation. Steady state washer modelling was performed to evaluate the influence of rakes on solids output. It is concluded that rakes increase the effective suspension permeability by a factor of 2-8. Transient consolidation modelling shows that consolidation in residue disposal areas is quick and should be complete within two years.
SECTION D

SUMMARY
OF
RESULTS, CONCLUSIONS AND FUTURE DIRECTIONS
Chapter 12

GENERIC PROPERTIES OF RED MUD SUSPENSIONS

12.1 INTRODUCTION
Red mud washer train suspensions from the ALCOA, Pinjarra Alumina refinery, the Worsley Alumina Refinery and the Queensland Alumina (QAL) Refinery have been characterised as described in the previous three chapters. Although each of the three refineries studied use the Bayer process, they use different bauxite ores which are processed differently at each plant. In order to understand the performance of each refinery, these similarities and differences must be recognised and are overviewed in this chapter. Initially, the properties of the red mud solids and process liquors are overviewed in sections 12.2 and 12.3. Knowledge gained from suspension shear rheology and dewaterability at each sponsor site is overviewed in sections 12.4 and 12.5. Section 12.6 summarises the key conclusions from the investigation about red mud properties.

12.2 SOLIDS

12.2.1 MINERALOGY

Bauxite

The term bauxite is used to refer to “sedimentary rocks that contain economically recoverable quantities of the aluminium minerals gibbsite, boehmite and diaspora” (Hudson et al. 1985). The mineralogy of bauxite ore used as a feed source for the production of alumina via the Bayer process contains significant variations in composition. Every orebody is different and requires uniquely different processing operations. The principal components of bauxites include alumina, silica, iron oxides and titania, but there are many other impurities. The alumina is present in recoverable and non-recoverable forms. The recoverable forms include a trihydrate form gibbsite (Al$_2$O$_3$.3H$_2$O) and monohydrate forms including boehmite (Al$_2$O$_3$.H$_2$O) and diaspore (Al$_2$O$_3$.H$_2$O). Gibbsite dissolves readily in sodium hydroxide solution at temperatures below 150°C and thus digestion is generally achieved at 150-200°C wheras boehmite requires elevated temperatures (above 200°C) to dissolve readily in sodium hydroxide solution and thus digestion requires much higher temperatures and pressures (QAL 2001; Worsley 2001).

The composition of bauxites used in the Alcoa (Pinjarra), Worsley and QAL refineries are provided in Figure 12.1. Both Alcoa and Worsley obtain their bauxite from the Darling range...
where the recoverable alumina is mainly gibbsite. Worsley bauxite is mined near Boddington, separate from where Alcoa obtains its ore (Worsley 2001). Both ores contain an average of about 30-32% recoverable alumina in the form of gibbsite, enabling low temperature operations (Worsley 2001). QAL bauxite is mined at Weipa in North Queensland (QAL 2001). QAL refines two grades of bauxite, the bulk of which is the monohydrate grade which contains gibbsite and boehmite and the trihydrate grade which contains mainly gibbsite (QAL 2001). Consequently, monohydrate grade bauxite undergoes high temperature extraction under pressure in digesters, while trihydrate grade material is added as a "sweetening bauxite" to the flash tanks where temperatures are less than 200°C (QAL 2001). The non-recoverable alumina component of bauxite is generally insoluble aluminosilicates.

The silica content includes silica (quartz) and aluminosilicates or clay materials such as kaolin, halloysite and montmorillonite. Silica is relatively unreactive at temperatures up to 200°C, but the high temperatures required for boehmite dissolution do dissolve a portion of the quartz present. The clay minerals are generally very reactive and dissolve under Bayer conditions. However, silicon present in the liquor precipitates as a sodium-alumino-silicate desilication product (DSP) which is discarded with the red mud and results in a loss of hydroxide and extractable alumina (Hudson et al. 1985). As shown in Table 12.1, Darling range bauxite has a reasonably high silica content, but only a small fraction of it is dissolved in the low temperature digestion processes at Pinjarra and Worsley. The significant silica concentration at QAL leads to significant loss of sodium hydroxide and alumina. The most common iron minerals present in the ore are goethite and hematite which are practically inert under the conditions of the Bayer process (Hudson et al. 1985).

<table>
<thead>
<tr>
<th>Composition (%)</th>
<th>Alcoa Darling Range</th>
<th>Worsley Darling Range</th>
<th>QAL Weipa Monohydrate</th>
<th>QAL Weipa Trihydrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>Total chemical</td>
<td>35-37</td>
<td>37</td>
<td>54.6</td>
</tr>
<tr>
<td></td>
<td>Total available</td>
<td>32</td>
<td>31</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Trihydrate</td>
<td>31</td>
<td>40</td>
<td>44</td>
</tr>
<tr>
<td></td>
<td>Monohydrate</td>
<td>1</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>Total</td>
<td>24</td>
<td>2-8</td>
<td>5.6</td>
</tr>
<tr>
<td></td>
<td>Reactive</td>
<td>1.2, 1-1.5</td>
<td>1.5</td>
<td>4.6</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td></td>
<td>18</td>
<td>33</td>
<td>12</td>
</tr>
<tr>
<td>TiO₂</td>
<td></td>
<td>1</td>
<td>3</td>
<td>2.6</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td></td>
<td>20</td>
<td>22</td>
<td>25</td>
</tr>
</tbody>
</table>

Table 12.1: Composition of alumina refinery bauxite composition (Debney 1993; Milne and Rickleman 1993; Newchurch et al. 1993; Alcoa 2001a; QAL 2001).

The important components of the ore from a production perspective, include the amount of recoverable alumina and its chemical form along with other soluble minerals that cause sodium
hydroxide loss and organics, from tree roots for example, that alter the dissolution chemistry (Hudson et al. 1985).

**Red Mud**

Alumina refining produces a fine bauxite residue, which is deposited in specially constructed dams (Worsley 2001). The residue is red in colour due to the high iron oxide content and is commonly termed red mud. For the majority of alumina refineries around the world, 1-1.5 tonnes of waste residue is typically produced for each tonne of alumina (Alcoa 2001b). Australian bauxites from the Darling range and Weipa are low grade and produce low alumina yields by world standards with about 2-2.5 tonnes of waste residue produced for each tonne of alumina produced (Cooling and Glenister 1992; Alcoa 2001b; QAL 2001; Worsley 2001). The red mud solids contain the barren insoluble bauxite residue and also the desilication product (DSP) formed in the alumina process and their mineralogical composition is summarised in Table 12.2. From Pinjarra, the key components have been identified as goethite, quartz and muscovite (Green 1997).

<table>
<thead>
<tr>
<th>Composition (%)</th>
<th>Alcoa (Green 1997)</th>
<th>QAL (Kane 1999)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>18.7</td>
<td>25</td>
</tr>
<tr>
<td>SiO₂</td>
<td>36.4</td>
<td>19</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>26.3</td>
<td>29</td>
</tr>
<tr>
<td>TiO₂</td>
<td>2.62</td>
<td>6</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>14.1</td>
<td>8</td>
</tr>
</tbody>
</table>

**Table 12.2:** Composition of alumina refinery red mud (Green 1997; Kane 1999).

**12.2.2 PARTICLE SIZE DISTRIBUTION**

The particle size distribution of red mud suspensions varies from site to site and depends on the sand fraction, the extent of crushing and grinding of the ore, numerous mineralogical factors and the way in which precipitates such as DSP are formed. If comminution creates particles that are too fine, then washing and dewatering becomes very difficult because permeability becomes very low. The main way in which refineries manipulate red mud particle size distribution is by removing and recombining the sand fraction with the suspension at various stages of the washing and dewatering process. Many refineries remove the sand fraction by means of hydrocycloning either before the settler/thickener or before the first washer to reduce some of the wear and tear of washing and dewatering suspensions with coarse particles. The sand fraction is used for dam wall construction, for permeable drainage layers in residue disposal areas and some is often recombined with the suspension prior to discharge into residue disposal areas.
The particle size distribution of an Alcoa (Pinjarra) superthickener underflow suspension, containing the sand fraction, measured by Green indicate that on a volume basis 10% is less than 1.2 µm, 50% is less than 15 µm and 90% is less than 267 µm (Green 1997).

### 12.2.3 DENSITY

The density of the red mud solids vary across all sites due to differences in ore composition and process operations. Red mud solids density estimates are 3100 kg m\(^{-3}\) at Pinjarra and 3680-3720 kg m\(^{-3}\) at Worsley with the high density at Worsley due to a high Fe\(_2\)O\(_3\) content and low SiO\(_2\) content. QAL processes several different grades of bauxite where each bauxite produces a mud with different chemical and mineralogical make-up. The red mud solids density ranges from ~2900 kg m\(^{-3}\) to ~3400 kg m\(^{-3}\) depending on the grade of bauxite used. The main constituent affecting the density of the red mud is the amount of Fe\(_2\)O\(_3\), which can vary from 25-51% at QAL (Kane 1999).

### 12.2.4 POROSITY

The remaining particles containing the barren insoluble residue of the ore and the precipitated DSP with zeolite structure (Hudson et al. 1985) is extremely porous. Red mud solids surface areas are reasonably high with measured values of the order of 10-20 m\(^2\) per gram of solid (Geppert 1981) and 25 m\(^2\)/g (Sarmiento 1978). The high porosity leads to significant difficulty in washing all caustic from the mud before disposal and rehabilitation. No matter how much the suspension is washed, there always seems to be more caustic leaching out, potentially for years, and this is a key reason why red mud residue disposal areas require long term management to minimise the risk of contamination of the surrounding environment. From a more positive perspective, the high porosity of red mud is useful for a number of applications including neutralisation of acidic soils and many contaminant absorption applications. In fact, the good adsorption properties of alumina refinery precipitates are exploited in “liquor polishing” where contaminants are selectively removed from the liquor by filtration with tricalcium aluminate hexahydrate (TCA) (Whittington 1996).

### 12.3 PROCESS LIQUOR

#### 12.3.1 CHEMICAL COMPOSITION

The principal components of Bayer process liquors dissolved in aqueous solution are sodium aluminate, sodium hydroxide and a cocktail of organics species. The liquor is very concentrated with a total dissolved solids concentration well in excess of 200 g/L. Bayer liquors are
produced from the dissolution of components of the bauxite ore that are soluble in concentrated sodium hydroxide solution at high temperatures. The soluble components of the ore include alumina in the form of gibbsite at temperatures up to 200°C and sometimes boehmite and diaspore at higher temperatures. Also included is silica in the form of the aluminosilicate kaolin for example. This silica concentration is reduced to acceptable levels by the addition of lime, CaO, which causes the precipitation of the silicon containing desilication product cancrinite (Hudson et al. 1985). Organics are extracted from tree roots and decayed organic matter present in the ore (Hudson et al. 1985). Much of the entrained organic matter is degraded to the relatively stable oxalate anion (Mckinnon et al. 1999). If the sodium oxalate concentration is allowed to continually increase in the process then it can co-precipitate with gibbsite leading to product quality issues (Mckinnon et al. 1999), so in many processes liquor oxalate levels are controlled by oxalate burning for example. In addition there are countless other non organic impurities in the ore that dissolve into the Bayer liquor including cations such as gallium, potassium, iron (III), calcium and anions such as carbonate, sulphate, fluoride and phosphates, all of which can significantly alter the liquor chemistry (Hudson et al. 1985).

In the washing and dewatering of red mud suspensions in settlers/thickeners, washers and other process dewatering equipment, the residue is washed and filtered to remove as much caustic soda as possible prior to pumping to the residue disposal areas. The red mud washer train liquors cover a wide range of consistency from a very thick black liquid with high dissolved solids concentrations in the settlers or thickeners to low dissolved solids in the final washer before pumping to the residue disposal areas. However, a small amount of caustic soda always remains in the residue resulting in an alkaline pH (Worsley 2001).

### 12.3.2 PHYSICAL AND CHEMICAL PROPERTIES

On site, standard liquor analysis involves determination of total alumina, total caustic as sodium carbonate and total soda, also as sodium carbonate, all in g/L. In addition, liquor density, dry solids by oven drying (w/w) and liquor viscosity were measured for most suspensions characterised. The range of values for these properties is summarised in Table 12.3. Liquor samples collected from the Alcoa (Pinjarra), Worsley and QAL refineries in 1998 were transported to Melbourne for analysis. The first observation when the samples arrived was that most samples had been subject to precipitation, even the settler/thickener and first washer samples that were allegedly stabilised with sodium gluconate. The liquors from the washer trains on each site were analysed in terms of pH, electrical conductivity and density. The range in determined values are also presented in Table 12.3.
Table 12.3 shows that the operation of washing and dewatering red mud suspensions is very similar at each site in terms of the variation in process conditions and liquor properties from the start to the end of the washer trains. The liquors start in the settlers at about 400 g/L solids and are diluted to about 70 g/L in the final washer with the pH being reduced from over 14 to around 13. The suspension pH has been demonstrated to have a significant effect on the surface properties in many particulate suspensions. It is the pH that determines the surface charges which contribute to the magnitude of inter-particle forces and determine the strength of the network structures. The effects are evident in shear and compression. However, because of the constantly high pH throughout the process, it is expected that there is no significant change in surface chemistry due to pH effects for red mud suspensions. Liquor densities reduce from about 1300 kg m$^{-3}$ for the high solids settler liquors to close to that of water for the dilute final washer liquors, depending on the extent of washing by the final washer. Liquor viscosities in the settlers/thickeners are relatively high above 1.5 mPa s, however because of the reducing temperature down the washer train, the liquor viscosities only reduce gradually down the trains to approach that of water at the end of the train at around 0.6-0.9 mPa s.

It is noted that the compositions of the final washer liquors do not represent the end of the red mud washing and dewatering process at any of these sites. At Alcoa (Pinjarra) there is a further superthickener stage in addition to consolidation and liquor recovery from sand layers in residue disposal. At Worsley there is a further filtration or thickening stage followed by liquor recovery from residue disposal. At QAL the final washer underflow mud is mixed with seawater that helps to neutralise and buffer the pH of the liquor and causes precipitation of liquor components.

<table>
<thead>
<tr>
<th>Operating Temperature ($^\circ$C)</th>
<th>Thickener/Settler</th>
<th>Final Washer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total alumina, TA (g/L)</td>
<td>90-150</td>
<td>15-30</td>
</tr>
<tr>
<td>Total caustic as sodium carbonate, TC (g/L)</td>
<td>200-210</td>
<td>25-45</td>
</tr>
<tr>
<td>Total soda as sodium carbonate, TS (g/L)</td>
<td>240-260</td>
<td>30-60</td>
</tr>
<tr>
<td>Density @ 25$^\circ$C (kg m$^{-3}$)</td>
<td>1300-1360</td>
<td>1010-1090</td>
</tr>
<tr>
<td>Dry solids by oven drying (w/w)</td>
<td>0.32-0.38</td>
<td>0.014-0.10</td>
</tr>
<tr>
<td>Viscosity @ Operating Temperature (mPa s)</td>
<td>1.5-2.0(@ 105$^\circ$C)</td>
<td>0.6-0.9(@ 50$^\circ$C)</td>
</tr>
<tr>
<td>pH</td>
<td>14.0 - 14.5$^*$</td>
<td>12.8 – 13.4</td>
</tr>
</tbody>
</table>

*Note that there was significant precipitation of gibbsite and oxalates in all settler samples before analysis.

### 12.3.3 SUPERSATURATION, INSTABILITY & PRECIPITATION

All Bayer liquors transported to Melbourne were subject to precipitation. This precipitation was even observed in liquors from the end of the washer train, suggesting that the sodium aluminate
component is always supersaturated. This has significant implications for processing of red mud suspensions and disposal in residue disposal areas where precipitation will influence suspension properties. Precipitation is proposed as the principal cause of thixotropic phenomena observed for red mud suspensions.

12.4 SUSPENSION SHEAR RHEOLOGY

12.4.1 INTRODUCTION

Shear rheology determines the flow and pumpability of rheological suspensions. The key measures of shear rheology investigated in this work include shear stress versus shear rate, viscosity versus shear rate and shear yield stress versus solids concentration behaviour. The key factors that influence shear rheology include solids concentration, temperature, particle size distribution, liquor properties and numerous chemical factors such as flocculation, pH etc..

Numerous studies have been completed over the past twenty years on the shear rheology characteristics of red mud (Sarmiento 1978; Nguyen 1983a; Nguyen and Uhlherr 1983; Nguyen and Boger 1985b; Pashias 1997). These tests have included both shear yield stress and shear stress versus shear rate characterisations using a range of devices including the vane (Nguyen 1983b; Nguyen and Boger 1985a), the slump test (Pashias et al. 1996), rotational viscometers with cup and bob or cone and plate configurations (Nguyen and Boger 1992) and capillary devices (Nguyen and Boger 1992). The aim has been to characterise the yielding and flow of red mud as a function of solids concentration and to help in the prediction of the pipelining of red mud slurries. The work has mainly been performed at room temperature on end of process red mud samples that have been collected on site and transported to the laboratory. Observations of aged red mud slurries at room temperature indicate that they are thixotropic yield stress materials and highly shear thinning, but do not fully recover the initial structure after shear (Nguyen 1983a; Nguyen and Uhlherr 1983; Nguyen and Boger 1985b; Pashias 1997). Pumping and processing of thixotropic materials has a number of design implications for process equipment, especially if operations such as thickening or pipelining are interrupted.

The aims of the shear rheology work, presented in this thesis in Chapters 9-11, were to examine trends in the shear rheology of red mud at process temperatures throughout the washer trains at each sponsor site and to establish the basis for red mud thixotropy.
12.4.2 OVERVIEW OF RED MUD SHEAR RHEOLOGY

Shear Yield Stress

For red mud suspensions, the exponential rise in shear yield stress with solids concentration is clearly shown in Figures 9.7, 10.6, 10.10 and 11.6. The effect of temperature is to modify interparticle forces, which is observed as a change in the shear yield stress. The effect of temperature on the shear yield stress of red mud suspensions has been demonstrated at all sites and at both the front and back end of washer trains in Figures 9.7, 10.6, 10.11, 11.6 and 11.7. As a rule of thumb, for every 10°C increase in temperature, there is a 10% increase in the shear yield stress.

References to the measured influence of temperature on the shear yield stress of particulate suspensions are rare. Results from two pieces of work from the literature contradict the observations presented here and suggest that the shear yield stress decreases with increasing temperature (Foong et al. 1995; Liu and Tseng 1998). Both Foong et al. and Liu et al. determined the shear yield stress by extrapolation of shear stress versus shear rate data to zero shear rate. The work by Foong et al. (Foong et al. 1995) involved rheological characterisation of a 58% v/v carbonyl iron suspension in ethylene vinyl acetate/beeswax binder, at 130-180°C, using a cone and plate rheometer. There are slip issues associated with the very low shear rates required for accurate yield stress extrapolation that were not adequately addressed. The work by Liu et al. (Liu and Tseng 1998) involved rheological characterisation of highly concentrated zirconia-wax suspensions at temperatures of 58.5-85°C using a capillary rheometer. The significant limitation of this work was that the lowest measured shear rates were 100 s⁻¹, which is excessively high for accurate shear yield stress extrapolation. In addition, the authors (Foong et al. 1995) suggest that the viscosity of the wax binder is similar to that of water at the characterisation temperatures, however, no evidence is provided to verify this postulate. It is conceivable that the binder has non-Newtonian behaviour that varies with temperature and contributes to the increase shear yield stress with temperature. As a result, the findings of the referenced articles are considered slightly suspect and also not directly related to red mud suspensions.

To verify that the shear yield stress is indeed a function of temperature, a program of work was conducted on a model suspension system by Haryanto Senlitonga (Senlitonga 2000). The model system was a coagulated zirconia suspension in aqueous 0.01 M KNO₃. Testing was conducted at a solids volume fraction of 0.15, over a wide pH range and at temperatures of 15°C, 25°C and 45°C using the vane technique in a thermally controlled sample cup. The starting pH was around 7.0 with pH adjustments achieved using aqueous solutions of 3 M HNO₃ and 10 M of KOH. The shear yield stress results for each temperature are shown in Figure 12.1 as...
a function of pH. The results in Figure 12.1 clearly show that the shear yield stress of the zirconia suspension increases with increasing temperature and is consistent with red mud observations.

The standard assumption that the shear yield stress of a flocculated suspension is not influenced by temperature is not supported by this work. This industrially important knowledge has been overlooked in the past because traditional methods of determining the shear yield stress involve extrapolation of shear stress versus shear rate data to a shear rate of zero. The vane technique has allowed fast and accurate determination that clearly shows the extrapolations can lead to incorrect conclusions. The practical implication of this finding is that the effect of temperature on shear yield stress needs to be considered in many processing applications.

![Graph showing shear yield stress as a function of pH at different temperatures](image)

**Figure 12.1:** Shear yield stress as a function of pH at a range of temperatures for a 15% v/v zirconia suspension measured using the vane technique (Senlitonga 2000).

**Suspension Viscosity**

The effect of solids concentration dramatically increasing suspension viscosity has been clearly demonstrated by previous workers (Nguyen 1983a; Pashias 1997) and is the expected effect based on rheological principles. The effect of increased dissolved solids concentration in the liquor is to increase the liquor viscosity and thus also increase suspension viscosity. The effect of increased temperature is to lower the liquor viscosity and thus also lower suspension viscosity. The significant reduction in dissolved solids concentration in the liquor down a washer train has only a moderate effect on suspension viscosity because of the reduction in temperature down the train, as demonstrated in Figure 12.2. Figure 12.2 compares the viscosity versus shear rate behaviour of each of the settler/thickener and final washer underflows at each
of the sponsor sites, which represents the upper and lower bounds of all of the data obtained. Comparison of the settler underflow rheology shows that for Worsley and Pinjarra, where the solids were effectively the same, the rheology was almost identical. The viscosity of the QAL settler underflow was much higher due to its higher solids concentration. Comparison of the final washing stage underflow rheology reveals that again, allowing for the variations in solids, the muds show very similar rheological behaviour. The main feature of Figure 12.2 is that the shear rheology of the washer underflows from each site are largely a function of solids concentration and liquor viscosity, with mud specific differences being much less significant. This result supports the findings of the permeability/compressibility study conducted at the same sites which showed that in dewaterability terms, the various red mud samples are very similar (See section 12.5).

![Figure 12.2: Comparison of viscosity versus shear rate behaviour of underflows from selected washer stages for each sponsor site in 1999. Temperature and solids concentration (volume %) are indicated.](image)

**Figure 12.2** Competing Temperature Effects

The competing effects of increased temperature causing an increase in shear yield stress while lowering liquor viscosity and causing a lowering of shear stress at higher shear rates is well displayed in Figure 10.7. The competing temperature effects can complicate predictions of flow behaviour, where temperature changes are involved, on top of the already complex thixotropic behaviour.
**Thixotropy**

For all of the shear rheology testing on site, reported in Chapters 9-11 in terms of shear yield stress and suspension viscosity, no thixotropic breakdown or recovery data has been presented. This is because no significant breakdown of shear stress was observed during rheological characterisation of the fresh suspensions at process temperatures. Some minor increases in shear yield stress were observed with 1-2 hours at rest with no shear, but these increases were consistently removed with only light mixing and it was not clear whether these increases were due to a settling related increase in solids concentration or a thixotropic build up of structure. As a result, all samples were lightly mixed immediately prior to characterisation. The samples tested included fresh thickener/settler and washer underflow suspensions, sampled before the centrifugal underflow pumps to best represent the rheology in the thickeners/settlers and washers. Nonetheless, investigation of thixotropy in cooled settler underflow samples by shearing with an impeller at 500 rpm showed a 30 % decrease in shear yield stress after 4 hours of shearing. This reduction in suspension strength can be produced in the plant through the shear processes involved with rakes, centrifugal underflow pumps and pipeline transport, as observed by previous workers (Pashias 1997). In general, different shear rates produce different extents of thixotropic breakdown over different time scales and aging of red mud suspensions in the absence of shear allows increases in the strength of network structures.

There are two observed levels of structure in “fresh” red mud suspensions. First, there is a relatively weak structure with a high rate constant for the breakdown and recovery process under relatively low shear rates observed as shear thinning. Second, there is a stronger network structure with longer time scales of breakdown and recovery under higher shear rates that is observed as thixotropy.

The range of potential observations indicate that the shear rheology of red mud should be determined for the specific shear rate range, temperature range and time scale in the application of interest. The differences in rheology observed between fresh and aged samples makes it unlikely that on-site rheology could be duplicated in off-site studies. However, for situations where on site testing is not possible previous authors have suggested that simply shifting the shear stress data to match the correct shear yield stress should be reasonably accurate (Pashias 1997).

### 12.4.3 ORIGINS OF THIXOTROPY

A fundamental knowledge of thixotropy is important in the design and operation of mixing processes, rakes, pumps and pipelines. The thixotropy of red mud suspensions has been characterised by many workers (Sarmiento 1978; Nguyen 1983a; Nguyen and Uhlherr 1983;
Thixotropy is postulated to be due to a range of properties including the strength of particle-particle interactions, particle size and shape and the chemical molecular environment at particle surfaces. All of these factors can result in the rate of suspension breakdown in shear being substantially different from the rate of recovery. Particulate suspensions with high solids concentrations often exhibit thixotropic behaviour. The phenomenon is usually associated with structural changes in a suspension during shearing. The potential influences of particle shape and liquor precipitation are discussed here.

12.4.3.1 The Influence of Particle Shape

Introduction

There are numerous examples of both model and industrial particulate suspensions that exhibit thixotropic behaviour due to particle shape effects. A typical example is a montmorillonite clay suspension at low background salt concentration. Under these conditions, the individual clay platelets dissociate completely into units with a thickness of order 10Å. The lateral dimension is usually between 0.2 and 2 µm, giving the particles an aspect ratio of between 200 and 2000. The effective volume fraction of a fully dispersed particle suspension is thus substantially greater than the true volume fraction. Alignment in shear reduces the effective volume fraction dramatically and the suspensions are characteristically highly shear thinning. The recovery of random particle alignment on the cessation of shear is slow compared to the shear alignment process and consequently, the suspensions are observed to be thixotropic. The time scale of the recovery or randomisation of particle orientations in the suspension is usually dependent on both the volume fraction of particles in suspension and the strength of particle-particle interactions.

In a study of the thixotropic behaviour of a coal tailings suspension (de Kretser and Boger 1992; de Kretser and Boger 1994; de Kretser et al. 1997) containing significant quantities of a montmorillonite clay, the thixotropy of the suspension was observed to decrease dramatically as the solids volume fraction increased. At high concentrations, thixotropy was not observed. It was postulated that the ability to randomise under such circumstances was severely restricted by the presence of nearest neighbour particles. To examine the effect of inter-particle forces on clay particle alignment and hence the effective volume fraction, de Kretser et al. (de Kretser et
al. 1997) used the same clay filled coal tailings suspension. They added salt to the suspension to reduce both the length scale and the magnitude of the repulsive forces between the clay plates. The yield stress of the suspension was observed to increase as the particles coagulate and form a network structure (Scales et al. 1998b). The yield stress then decreased as further coagulation led to clay particle alignment into tactoids. The decrease in yield stress with an increase in inter-particle force was postulated to be due to the decrease in the effective volume fraction of the particles being greater than the increase in inter-particle strength. Suspension thixotropy, measured as the ratio of the suspension yield stress after breakdown to an equilibrium state versus the yield stress after twenty four hours, decreased monotonically as a function of added salt. One may conclude on this basis that coagulation processes always reduce the ability to randomise. Analogous to the high volume fraction case, the suspensions showed very little thixotropy under strong particle-particle interaction conditions where the ability to randomise is grossly restricted. Recent work on a synthetic clay suspension shows similar trends (Tateyama et al. 1997a; Tateyama et al. 1997b).

Red Mud

Transmission Electron Microscope (TEM) analysis of a range of red mud samples from Kwinana and other sites around the world has been completed. The particulate structure of undialysed red mud samples was difficult to elucidate although the structure of dialysed samples revealed the presence of needle shaped particles with major dimensions from 10 to 100 nm and an aspect ratio of about 5, as shown in the TEM micrograph of Figure 12.3. These needles are likely to be hematite and/or precipitated oxalates, but have not been investigated further. The question of interest is whether the presence of these asymmetric particles has the same influence on suspension thixotropy as the higher aspect ratio clay platelets.

Varying the electrolyte concentration, which in turn influences the strength of particle interactions in a controlled manner is an effective method of exploring the influence of particle shape on red mud thixotropy. Thus, an aged sample of red mud from the Alcoa Kwinana refinery, Kwinana, Western Australia, was dialysed to reduce the sodium hydroxide electrolyte concentration from 0.13 M to $10^{-4}$ M with a pH of 10. The sample was then dialysed to sodium chloride concentrations of $10^{-3}$ M and 1 M. The suspensions were sheared to an equilibrium shear yield stress of 35 Pa, over six days as shown in Figure 12.4. The shear yield stress recovery of the dialysed suspensions was measured using the vane technique over seven days, as shown in Figure 12.5. The red mud suspension dialysed to $10^{-3}$ M NaCl recovered more quickly to a higher shear yield stress even though the same suspension was the easiest to break down. Clearly, there are some chemical phenomena and possibly some particle shape effects in the observed differences in the rate and extent of structural breakdown and recovery. However,
more work would be required to validate the theory that particle shape was responsible for some of the differences and thus a contributing factor towards red mud thixotropy.

Figure 12.3: TEM photograph of dialysed red mud from Alcoa’s Kwinana refinery.

Figure 12.4: Thixotropic breakdown of dialysed red mud from Alcoa’s Kwinana refinery
12.4.3.2 The Influence of Liquor Precipitation

All liquors in red mud washer trains are supersaturated with sodium aluminate and sodium oxalates (Mckinnon et al. 1999). This supersaturation is exacerbated by the reduction in temperature down the train, right out to the residue disposal areas. The presence of red mud solids acts as a catalyst for co-precipitation of gibbsite, sodium oxalates and other species from the supersaturated liquor (Mckinnon et al. 1999). The continued precipitation onto the solid surfaces binds them together and continuously builds up structure in the network suspension.

The observation of increased rheological strength in red mud with sample ageing time suggests that the strong network structure of aged samples is almost certainly due to liquor instability whereby precipitation causes irreversible welding of particles. Breaking of these bonds produces a breakdown in the viscosity with time that is shear rate and time dependent. The observation of Nguyen (Nguyen 1983a) that full recovery of aged structure is never achieved after shear lends weight to this theory because precipitated liquor will not precipitate again. The initially rapid and then slow recovery of structure after shearing is proposed to be due to the freshly formed new crystalline surfaces catalysing precipitation, with the active surfaces gradually poisoned by various liquor components. These ideas lead logically to a wide range of potential experiments that would quantify the rate and extent of thixotropic processes as a function of temperature, liquor supersaturation, liquor components and shear conditions.
12.5 SUSPENSION DEWATERABILITY

12.5.1 INTRODUCTION

Relatively comprehensive dewaterability characterisation of red mud washer train suspension dewaterability has been presented in Chapters 9-11. During this characterisation, a number of generic trends have been observed. These trends have been summarised in this section. Topics considered include important issues during characterisation such as temperature, sample age, chemical hazards, evaporation and sand fraction. The effect of characterising aged rather than fresh suspensions is overviewed. The compressibility and permeability trends from the front to the back end of the washer trains are overviewed with emphasis on similarities and differences between sites. To finish off, comprehensive sample dewaterability characterisation over the full range of solids concentrations is evaluated in terms of its accuracy and applicability to modelling the operation of process equipment.

12.5.2 CONSIDERATIONS WHEN CHARACTERISING RED MUD SUSPENSIONS

When sampling red mud suspensions for dewaterability characterisation, there are numerous issues that need to be considered. The first consideration is to collect suspension samples from locations where suspension properties are representative of those in the process that one wishes to model or understand. In addition, it is also important that the process of sampling and characterisation does not excessively degrade the sample and lead to non-representative characterisation results. In reality, most characterisations are performed on samples that provide the best practical balance between experimental time used and knowledge gained.

When trying to determine flocculated red mud washer dewaterability, there are a number of problems. Firstly, freshly flocculated suspensions can not be collected from the feedwell, and if they could be, the process of sample collection would probably destroy the sensitive floc structure. Even the difficult task of sampling from the washer mud bed has been shown to destroy the floc structure. The other sampling location is the washer underflow that has been raked, but not strongly sheared as it would be in the centrifugal pump immediately downstream of the underflow sample point. Underflow suspensions provide a consistent rheology/dewaterability for characterisation purposes. Unfortunately, dilution of underflows to lower solids concentrations produces floc structures that are not representative of unsheared samples in the feedwell. To obtain a suspension that is representative of a feedwell suspension requires flocculation of a simulated feed suspension. Common flocculation methods employed include the plunger method, a stirred baffled reactor and a shear vessel. No lab based
flocculation test can reproduce feedwell conditions exactly and each method has its own inherent advantages and disadvantages in terms of shear rate range, being batch or continuous and the ability to suspend coarse sand particles.

Another important consideration is temperature. Often high temperature samples cannot be characterised at the high process temperatures because of equipment limitations, problems with excessive evaporation or unacceptable sampling hazards. Therefore an optimum lower temperature must be chosen to eliminate these problems while maintaining a reasonably representative sample. Some of the issues involved with temperature changes include changes in liquor density and viscosity, but these can be measured. Changes in compressive yield stress are usually minor. However, changes in the hindered settling function which are almost exclusively due to liquor viscosity can be corrected, but changes in the hydrodynamic and chemical environment due to temperature changes can sometimes affect flocculant activity and cannot be corrected.

Due to the fact that red mud liquors are inherently unstable, long test times will lead to uncertainty in the results due to liquor precipitation. Unfortunately, the effects of precipitation due to liquor ageing are not understood and cannot be corrected.

The final important issue is sample degradation in the characterisation process. It is very difficult to perform accurate dewaterability characterisations over a wide range of solids concentrations due to the extreme shear sensitivity of fresh floc structures. This issue is addressed by shearing freshly flocculated test suspensions as little as possible during handling. Unfortunately, even small amounts of shear can reduce permeabilities by a factor of ten, so the key to gaining knowledge is to be as consistent and controlled in sample handling as possible. The implications of shear degradation are addressed later in this chapter.

12.5.3 FRESH VERSUS AGED SAMPLE CHARACTERISATION

Red mud samples were collected from each site and brought back to Melbourne for testing. As already noted earlier in this chapter in section 12.3.2, all samples were subject to some precipitation, even those stabilised with sodium gluconate. As a result, the samples were very non-homogeneous with coarse sand particles at the base with red mud immediately above. The feeds and underflows from the thickener and the first few washers had a brown and then white precipitate above the red mud, with scaling above that. The end of washer train suspensions generally had only a layer of scale above the red mud. The white precipitate and the scale was physically removed for testing, but the results are not presented here because they are not considered representative of the true material properties. Those considering dewaterability
characterisation of red mud suspensions offsite should endeavour to perform testing only on end of washer train suspensions or to transport the liquor and solids separately.

12.5.4 THICKENER UNDERFLOW SUSPENSIONS

Dewaterability characterisation on underflows from the front to the back end of the red mud washing and dewatering processes at each sponsor site in 1999 have been presented in Chapters 9-11 in sections 9.2.3, 10.2.3 and 11.2.3. Across all sites there are some differences and generic trends that can be observed.

Figure 12.6 presents an overview of compressive yield stress, $P_y(\phi)$, thickener/settler and final washer data from each site. Clearly, the compressive yield stress was very variable at Pinjarra due to processing differences along the process, while Worsley and QAL red muds were relatively unchanged throughout their respective processes. An important point of note is that these similar results are presented in terms of solids volume fraction. However if the data were presented in terms of the more commonly used solids weight fraction, then reductions in liquor density down the washer trains would cause the thickener/settler results to vary from the final washer results. The inference here is that liquor properties generally have little effect on the compressive yield stress on a volume basis, but not necessarily on a weight basis. The other important influence on compressive yield stress that has been observed, but not highlighted in this report to date, is coarse sand fraction. In general, addition or removal of some sand does not affect the compressive yield stress of the rest of the suspension, and can thus be corrected for without any further experiments if necessary.

Figure 12.7 presents an overview of hindered settling function, $R(\phi)$, data for a thickener/settler and final washer at each site. Clearly, the suspensions are 4-6 times as dewaterable at the back end compared with the front end of the train, due mainly to differences in liquor viscosity. In Figure 12.8, the influence of liquor viscosity is corrected for and the suspensions at the back end are only twice as permeable as those at the front end. This trend of improved permeable network structure down the washer train is proposed to be primarily due to increased cumulative flocculant dose and gradual reductions in liquor viscoelasticity.
Figure 12.6: Comparison of compressive yield stress, $P_y(\phi)$, behaviour of underflows from selected washer stages for each sponsor site in 1999.

Figure 12.7: Comparison of hindered settling function, $R(\phi)$, behaviour of underflows from selected washer stages for each sponsor site in 1999.
Figure 12.8: Comparison of hindered settling function, $R(\phi)$, behaviour corrected to standard liquor viscosity of 1 mPa s for underflows from selected washer stages for each sponsor site in 1999.

### 12.5.5 FLOCCULATED RED MUD SUSPENSIONS

In 2000, more complete permeability characterisations of selected washer underflows were performed at each site over the full range of solids concentrations of relevance to most processing operations and the results are presented in Figures 9.8, 10.9 and 11.8. At Pinjarra and QAL, flocculation tests were conducted to determine the differences in dewaterability caused by flocculation. Flocculation was observed to reduce the compressibility of a suspension for all solids concentrations at Pinjarra (See Figures 9.19), however increased dose was observed to increase the gel point slightly (See Figure 9.18). At QAL, the similar trends were observed (See Figures 11.14 and 11.15), but the gel point went down for doses above 100 g/t. At both Pinjarra and QAL, flocculation was observed to improve suspension permeability significantly at low to intermediate solids concentrations while not causing significant improvements at high solids concentrations (See Figures 9.22 and 11.21). Increasing flocculant dose was observed to improve suspension permeability significantly at low to intermediate solids concentrations (See Figures 9.14, 9.15, 9.16, 11.11 and 11.12), while not causing significant improvements at high solids concentrations (See Figures 9.17 and 11.13).

Figure 12.9 demonstrates the effect of flocculation in reducing compressibility and improving permeability. In addition, it must be recognised that the observed flocculated suspension dewaterabilities would have been altered by floc degradation in all cases that involved sample
handling after flocculation and before characterisation. For compressibilities, the true gel point is likely to be lower than the observed value due to the destruction of some floc structure in the gel point test method. In addition, the true permeabilities would have been significantly better than the observed permeabilities because of the floc degradation involved in sample transfers for transient batch settling tests and pressure filtration tests.

The proposed effect of sample degradation is also shown in Figure 12.9 as the difference between the true and observed permeability. This sample degradation argument does highlight the inaccuracies of the freshly flocculated suspension characterisation results presented in sections 9.3.3 and 11.3.3, but they should not be dismissed altogether, because they do provide useful information about the sensitivity of flocs to shear. In addition, these will remain the best sets of results available until better characterisation methods are developed.
Figure 12.9: Comparison of typical red mud hindered settling function, $R(\phi)$, and compressive yield stress, $P_y(\phi)$, curves for an observed underflow suspension characterisation, an observed flocculated suspension characterisation and a true flocculated suspension before any material handling degradation.
12.6 OVERVIEW OF RED MUD PROPERTIES

Solids

The principal differences in red mud solids are the iron concentration which is the main factor that influences solids density and the sand fraction which affects the particle size distribution and can influence suspension dewatering behaviour.

Process Liquor

Process liquors vary significantly from the front to the back end of a washer train. At the front end, the liquor is very caustic, has over 400 g/L of dissolved components at 100-110°C, is reasonably thick with a viscosity of over 1.5 mPa s and is probably slightly viscoelastic. At the back end of the washer train, the liquor is relatively dilute, often at less than 50°C, with a density and viscosity similar to that of water, but with a high pH and a significant level of sodium aluminate supersaturation that can precipitate as gibbsite.

Suspension Shear Rheology

On a solids volume fraction basis, suspension shear rheology tends to be relatively consistent throughout a washer train. Above the gel point, red mud suspensions exhibit a shear yield stress that increases exponentially with solids concentration, while suspension viscosity decreases with increasing shear rate (shear thinning). Significant viscosity variations are caused by liquor viscosity differences, chemical changes and removal or addition of sand.

Origins of Thixotropy

The origins of thixotropy in red mud suspensions are still uncertain. However, it seems that the influence of particle shape and precipitation of gibbsite from supersaturated liquor may hold the key to improved understanding the rates and extent of thixotropic breakdown and recovery in the future.

Dewaterability

Compressive yield stress behaviour at the front of the washer train is very similar to that at the end of the washer train. Key reasons for variations are flocculant type and dose, extent of shearing, chemical changes and sand removal or addition. Hindered settling function behaviour varies by many orders of magnitude as a function of solids concentration and is strongly related to liquor viscosity. With the influence of liquor viscosity removed, it is revealed that the permeability of the network structure gradually improves down the washer train due to the cumulatively increasing flocculant dose and possibly reduced liquor viscoelasticity.
**Flocculation**

The effect of flocculation is to decrease compressibility slightly, but to improve permeability at low to intermediate solids concentrations. However, this highly permeable floc structure is extremely sensitive and easily destroyed by shear processes in thickening operations, so that the underflows are significantly less permeable at low to intermediate solids concentrations.

**Temperature Dependence**

There are two significant ways in which temperature influences the properties of red mud suspensions. The first is to affect the strength of the suspension network structure which manifests itself most obviously as an increase in the shear yield stress and the compressive yield stress with increasing temperature. The phenomenon of increasing shear yield stress and compressive yield stress with increasing temperature has since been observed in numerous other metal oxide systems and it is proposed that the phenomenon is common to many metal oxide suspensions. Use of the vane technique has shown that extrapolation of shear stress versus shear rate data to estimate the shear yield stress almost invariably leads to incorrect conclusions. The other way in which temperature influences red mud properties is in rate behaviour where a decrease in the suspension viscosity and hindered settling function with increasing temperature is due mainly to a reduction in the liquor viscosity.
Chapter 13

COMPARISON OF THICKENER DEWATERING
PREDICTIONS WITH OPERATIONAL REALITY

13.1 INTRODUCTION
This chapter explains how red mud dewaterability characterisation test results generated on site have been used in steady state thickener model predictions to improve understanding of the influences on process performance. Comparison of performance predictions with actual performance has led to some key observations, which are listed and discussed. The variables that contribute to and influence dewatering performance predictions are also discussed. The discussion will address issues of material property changes due to sampling from a thickener underflow, directly from the bed within a thickener and using freshly flocculated material in the laboratory. Then, most importantly, the key observations of permeability enhancement are discussed in terms of the effect of raking and its quantification. Finally, an attempt has been made to explain the origins of permeability enhancement.

13.2 KEY OBSERVATIONS
Comparison of steady state thickener model predictions with operational outputs was a key objective of the final round of site work in 2000. Details of the individual modelling work completed and comparisons against operational outputs are presented for each site in Chapters 9, 10 and 11. Comparison of model predictions with reality was conducted by observing the typical solids throughput experienced in a given thickener, reading off the predicted underflow solids concentration corresponding to this solids flux from the flux-concentration curve developed using the model and dewaterability data gathered on site and comparing the predicted and observed underflow solids concentrations. The key observations were that most thickeners operate such that the underflow solids concentration is limited by the permeability of the suspension, that the steady state thickener model under-predicts performance and that it over-predicts average solids residence times.

13.2.1 PERMEABILITY LIMITED OPERATION
For almost every thickener modelled, given the operational underflow solids concentration and suspension height, thickener operation is predicted to be permeability limited. This means that the effect of differences in the compressive yield stress will be of limited importance to
thickener output solids concentrations. This implies that for a given solids flux, the underflow solids concentration will not increase for increased suspension bed height. For permeability limited operation, the thickener underflow solids concentration will be affected primarily by the permeability of the suspension at low solids concentrations from the feed solids concentration to the underflow solids concentration. Whilst flocculant selection is currently based on optimising the settling rate at a single solids concentration, the previous point implies that flocculant selection should be based on settling behaviour characterised over the relevant range of solids concentrations. The implication is that optimisation of permeability limited dewatering requires improved suspension permeabilities. This improved permeability, however, is not just required as an improved settling rate at the feed solids concentration, but at all solids concentrations from the feed solids concentration to the underflow solids concentration.

13.2.2 THICKENER MODEL UNDER-PREDICTS PERFORMANCE

A common outcome of all of the comparisons was that the thickener model consistently under-predicts the underflow solids concentration achievable in a thickener when compared to operational reality. The magnitude of this under-prediction is quantified in terms of a permeability enhancement factor. The correction factor and its origins form the subject of discussion later in this chapter. Understanding the relative contribution of shear and flocculation to the amount of under-prediction provides a basis for improved understanding of dewatering processes and provides a clear direction for the optimisation of dewatering in a thickener.

13.2.3 THICKENER MODEL OVER-PREDICTS SOLIDS RESIDENCE TIME

Conversely, the under-prediction of performance also over-predicts the true solids residence time. However, the trends predicted are still expected to be valid. In addition, reasonably indicative solids residence times can be determined from the model predictions divided by the permeability enhancement factor. Operational solids residence times are expected to be important for red mud settlers and first washers because of alumina product loss due to precipitation. The longer the solids residence time, the more precipitation is likely to occur. As a result, the solids residence time predictions can provide a guide to help minimise product loss. For later stage washers, precipitation rates are much slower and the solids residence time is not as critical to process optimisation.
13.3 THICKENING PERFORMANCE PREDICTION INFLUENCES

As has already been noted throughout this work, there are numerous variables that influence thickening performance. Model predictions suggest that thickener dimensions, solids flux, suspension bed height and suspension dewaterability can completely determine thickening performance. Unfortunately, as has already been highlighted in Chapter 12, suspension dewaterability varies from the feedwell to the underflow due to shear processes and it is difficult to reliably characterise such variations. Thickener performance predictions have been based on suspension dewaterability characterisations of both thickener underflow suspensions and laboratory flocculated suspensions, each having properties that reflect their processing and characterisation history. The way in which the characterisation protocol employed causes variations in the measured dewaterability and therefore influences thickening performance predictions is discussed in this section. Observed dewaterability variations when comparing the baffle reactor and the plunger test are discussed, in addition to the effect of shearing during sampling and characterisation.

13.3.1 BAFFLE REACTOR VERSUS PLUNGER TEST

A key development in laboratory testing of flocculation is the implementation of the baffle reactor for reproducible flocculation. An advantage of the baffle reactor is the integration of the well quantified shear conditions in the vessel with large enough volumes of material to conduct dewaterability characterisations using batch settling, gravity permeation and pressure filtration tests. Alternatively, the plunger test requires replicate tests to produce enough sample for comprehensive dewaterability characterisation. Whilst some variability existed during initial utilisation of the baffle reactor during site work (See Chapter 9), refinement of the technique gave comparable reproducibility to the plunger test and demonstrated similar trends in observed settling rate and gel point with increasing floc dose. The work has shown that the results of the plunger test can be mimicked in a constant shear reactor. The ability to control shear during flocculation is an important component of floc formation tests that seek to either mimic shear regimes in operational feedwells or to determine optimum shear regimes, as is being attempted by Hulston (Hulston and Scales 2001) with a baffle reactor. Aside from the determination of the initial settling rate, both methods suffer from the limitation that sample handling requirements for dewaterability characterisation at higher solids concentrations tends to degrade freshly flocculated suspensions and reduce the measured permeability (See Figure 12.9). This is the case for transient batch settling tests, gravity permeation tests and pressure filtration tests.
13.3.2 SHEAR DURING SAMPLING AND CHARACTERISATION

The most obvious initial focus in understanding the origins of the difference between observed and predicted underflow solids concentrations is in the quality of the dewaterability data fed to the model. As has been observed in the characterisation results in Chapters 9, 10 and 11, suspension permeability is a strong function of shear history. When freshly flocculated, permeability is very high across all solids concentrations. However, even the smallest amounts of shear can destroy the permeable floc structure to the extent that permeabilities are reduced by up to a factor of 100 at feed solids concentrations. The only simple method of collecting relatively representative samples of thickener suspension is through collecting thickener underflow suspensions. To ensure that the suspensions are reasonably representative, the samples are collected upstream of the centrifugal pumps that impart significant shear. Indeed, the shear processes inside a thickener lead to an underflow sample permeability that is significantly worse than in the feed well. This raises the question of where to sample thickener suspensions. Work conducted during the 2000 ALCOA site visit (Chapter 9) compared the permeability of suspension taken from the underflow of a thickener with the permeability of the suspension taken directly from the suspension bed inside the same thickener. It was shown that there was little or no difference between the measured permeabilities of the bed-sampled and underflow-sampled suspensions. It is expected that any potential differences were not observed because of degradation to the shear sensitive bed sample permeability during sampling and characterisation, rendering it basically the same as the underflow sample.

13.3.3 FLOCCULATED SAMPLES VERSUS UNDERFLOW SAMPLES

During the year 2000 site visits, to both ALCOA and QAL (Chapters 9 and 11), a number of dewaterability characterisations were conducted using freshly flocculated material, flocculated using either the baffle reactor or the standard plunger test. The results from these tests demonstrated that a freshly flocculated sample has a higher permeability than an underflow sampled material. The magnitude of the improvement in permeability varies as the solids concentration changes; however, at feed solids concentrations the improvement can be a factor of 10 to 100, dropping to a factor of around 1 to 2 at high solids concentrations. Once the solids concentrations have reached the typical concentrations of the underflow of a thickener, the differences in permeability between a freshly flocculated and an underflow-sampled material are minimal relative to experimental error. However there are also some characterisation method degradation issues to consider. Consequently, at intermediate solids concentrations, the differences between freshly flocculated and underflow suspension permeabilities is even greater than measurements indicate. Freshly flocculated suspensions are generally slightly less compressible than sheared underflow samples. However, exceptions are common at solids
concentrations near the gel point due to the type of flocculant or the shear regime during flocculation.

The optimum condition for thickening, flocculated or sheared (underflow), will generally depend on how the thickener is being operated. According to the modelling predictions, at high solids fluxes, the flocculated suspension produces the highest underflow solids concentration, while at low solids fluxes, the underflow suspension produces the highest underflow solids concentration. For the observed underflow solids concentration in the final washer at Pinjarra, the predicted solids fluxes determined from the flocculated suspension characterisations was a factor of 2.5 higher than that determined from the underflow suspension characterisation. For the final washer at QAL, the factor was 1.8.

13.4 THICKENING PERFORMANCE INFLUENCES

In addition to the thickening performance prediction influences already discussed, there are some additional non-ideal influences not accounted for in the model. This non-ideal behaviour involving the thickener model under-predicting performance is interpreted as an effective permeability enhancement and has been attributed to shear processes such as raking. The relative effects of flocculation and raking in thickening operations are quantified in the following section. The interaction between raking, the suspension shear yield stress and thickener operation is then discussed. In addition, non-ideal raking effects that might vary with flocculation conditions are also discussed.

13.4.1 EFFECTIVE PERMEABILITY ENHANCEMENT OF RAKING

Characterisation issues aside, modelling of the output of a thickener for various material property and process condition inputs has a number of limitations. Even if the effects of treatment of the sample during dewaterability characterisation have been taken into account, effects relating to non-ideal operation of the thickener and, in particular, raking in the bed of a thickener are not considered in the 1-D steady state thickener model. However, a quantification of the deviation from model predictions may be obtained from the ratio of the actual operational solids flux relative to the predicted solids flux for the actual suspension bed height and underflow solids concentration. This ratio is termed the permeability enhancement or PE factor. Table 13.1 below summarises the findings from modelling of thickeners at sponsor sites, based on both freshly flocculated and underflow suspension dewaterabilities. In Table 13.1, permeability enhancement has been attributed partially to flocculation and partially to shear processes such as raking. All of the PE of flocculation values presented are given as minimum values because the true suspension permeability is higher than that measured in the
characterisations due to sample degradation issues when characterising freshly flocculated suspensions. Conversely, the PE of raking values presented are maximum potential values for the same reason.

Over the three sponsor sites investigated, PE factors ranging from 4 to 200 have been observed. As noted in Chapter13 (section 13.3.3), the PE of flocculation, of the order of 2, gives a clear indication of the magnitude of improvement available to a process via optimising the flocculation process itself. After removing flocculation effects, the resultant PE of raking ranges from 2 to 100. Therefore, the single most significant factor in improving or optimising dewatering in a thickener is the effect of raking. Whilst this type of analysis is a new and empirical approach, there already appears to be a correlation between the type of thickener and the magnitude of the PE Factor for raking. The highest levels of PE Factor observed have been for deep cone thickeners that have extensive raking in terms of raking frequency and the vertical height of the raked zone. The converging base and flat bottomed thickeners, having PE of raking factors less than 16, are generally only raked moderately because of operational torque limits. The generally observed trend is that the more extensive the raking, the higher the PE factor.

<table>
<thead>
<tr>
<th>Refinery</th>
<th>Solids Flux</th>
<th>Thickener Design</th>
<th>Measured PE Factor (underflow) (based on underflow sample)</th>
<th>Measured PE Factor (flocculated) (based on flocculated sample)</th>
<th>PE of Flocculation (PEF)</th>
<th>PE of Raking (PR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Worsley</td>
<td>High</td>
<td>Deep Cone</td>
<td>104</td>
<td>&gt;2*</td>
<td>&lt; 52</td>
<td></td>
</tr>
<tr>
<td>High</td>
<td>Deep Cone</td>
<td>200</td>
<td>&gt;2*</td>
<td>&lt; 100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Medium</td>
<td>Flat Bottomed</td>
<td>18.6</td>
<td>&gt;2*</td>
<td>&lt; 9.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pinjarra</td>
<td>Low</td>
<td>Converging Base</td>
<td>39</td>
<td>16.3</td>
<td>&gt; 2.5</td>
<td>&lt; 16</td>
</tr>
<tr>
<td>Gladstone</td>
<td>Medium</td>
<td>Converging Base</td>
<td>4.1-16.0</td>
<td>2.3-8.7</td>
<td>&gt; 1.8</td>
<td>&lt; 2.3</td>
</tr>
</tbody>
</table>

*Table 13.1: Summary of steady state thickener modelling permeability enhancement at sponsor refineries.*

13.4.2 SHEAR YIELD STRESS, BED HEIGHT AND RAKE TORQUE

Thickener rake torques depend on the suspension bed height and the shear yield stress of the suspension. Mechanical constraints often provide an upper limit for operational rake torques. This is usually due to limitations in the strength of the rake arms or the power of the rake motor. The process variables that contribute to rake torques include rake speed, suspension bed height and suspension shear yield stress (function of solids concentration). There is usually a range of rake speeds over which the torque is kept relatively low. However, at higher rake speeds, the torque will be too high, while at very slow rake speeds, the rake may get bogged. Varying the
Suspension bed height is a simple method of controlling the rake area exposed to suspension with a shear yield stress, thus controlling rake torque. Thickener modelling has predicted that suspension bed height has little effect on thickener output as a result of compressive dewatering. However, given the observations of high PE Factors for raking, the location of the rake relative to the bed height may be a significant variable in thickener output. As a result, if the bed height is such that only a small fraction of the rake is immersed in the bed, then maximum benefits from the raking may not be achieved. Alternatively, if the rake is completely submerged below the bed, this doesn’t provide an adequate pathway for significant channelling. Additionally, variations in the flocculation regime can affect the shear yield stress at a given solids concentration and as a result, also influence the rake torque.

13.4.3 FLOCCULANT TYPE AND DOSE
Flocculant type and dose can influence suspension dewaterability and in turn, influence thickener modelling performance predictions. However, in the presence of shear forces due to rakes and the like, different flocculant types and doses may respond differently. Future research will need to focus on the link between floc type and structure and obtaining maximum benefit from raking. Some floc structures may be weak and degrade easily, while modified flocculation conditions may produce dense compact flocs that maintain their structure with less permeability degradation in the presence of shear.

13.4.4 SAND SEGREGATION
The presence of large amounts of sand in a process may place further constraints over the practical operational range of a thickener. Maintenance of a homogenous distribution of sand in a bed will require a minimum level of bed stiffness to prevent sand from settling relative to the rest of the suspension. Without this stiffness, segregation may occur which can produce dead zones in a thickener, leading to rake bogging. In principle, prevention of sand segregation can be accomplished by changing the flocculant type or dose to give a stiffer bed at a given solids concentration. This stiffer bed can be characterised through shear yield stress or gel point testing.

13.4.5 ORIGINS OF PERMEABILITY ENHANCEMENT
Understanding the fundamental aspects of shear-induced dewatering is critical to improving design of thickeners to optimise dewatering. The observed permeability enhancement in thickener operation is believed to be due to a number of phenomena involving shear processes in the presence of compressive forces. Angled bottoms on thickeners give a non-vertical normal
force that manifests itself as a shear force under compression in a mud bed. Similarly, the actions of rakes and shear rods create a shear force perpendicular to the compression in a mud bed. As a result, the shearing creates locally enhanced compression in front of the rakes and rods, while solids free liquor builds up behind the rake. However, improvement in the overall effectiveness of dewatering is only achieved if the liquor has greater opportunity to escape to the surface of the mud bed. It is suggested that liquor bubble rise and liquor channelling could be the mechanisms by which the liquor escapes. However, experiments have not been conducted to assess the importance of these phenomena.
Chapter 14

CONCLUSIONS AND FURTHER WORK

14.1 CONCLUSIONS AND MAJOR OUTCOMES

The developments presented in this thesis establish a foundation for the assessment of the dewaterability of suspensions in terms of the compressive yield stress, \( P_y(\phi) \), and the hindered settling function, \( R(\phi) \). This foundation is enabling the creation of new knowledge in separation processes, including for example, quantification of the role of flocculants in filtration, thickening and consolidation of particulate suspensions.

Suspension dewaterability characterisation techniques and process equipment modelling tools have been developed based on a fundamental theory of dewatering. The characterisation techniques involved batch settling, gravity permeation and pressure filtration tests and were used in on site red mud dewaterability characterisation at the Pinjarra, Worsley and QAL alumina refineries. The characterisation results were brought together to illustrate the generic properties of red mud suspensions. Process equipment modelling tools were developed to predict pressure filtration, steady state thickening and transient consolidation of suspensions based on dewaterability characterisation results. The red mud characterisation results were applied to the process modelling tools to predict the performance of thickeners, washers, rotary drum filters and tailings consolidation in residue disposal areas. Thickener performance predictions were compared with actual performance and deviations attributed to shear processes such as raking. Directions for processing optimisation were also identified.

Dewaterability Characterisation Technique Development

Experimental techniques have been developed for comprehensive dewaterability characterisation of particulate suspensions over the full range of solids concentrations from separation equipment feeds to outputs. These techniques include equilibrium batch settling, transient batch settling, gravity permeation, stepped pressure filtration and \( \phi_\infty \) estimation. Equilibrium batch settling is for determination of the gel point and low \( P_y(\phi) \) values. Transient batch settling is for the determination of \( R(\phi) \) at low to intermediate solids concentrations. Gravity permeation is for the determination of \( R(\phi) \) at an intermediate solids concentration, especially when suspension sample availability is limited. Stepped pressure filtration is for the timely determination of both \( P_y(\phi) \) and \( R(\phi) \) values for applied pressures above 5 kPa. The \( \phi_\infty \)
estimation technique provides more accurate determination of \( P_y(\phi_\infty) \) values when a compressibility filtration test is incomplete and also provides a value of \( D(\phi_\infty) \) from the same data. The development of these methods has involved laboratory work on model systems and testing of the methods on-site using red mud slurries at process temperatures. These new dewaterability characterisation techniques are generically applicable to particulate suspensions in a wide range of industries.

**On Site Red Mud Dewaterability and Shear Rheology Characterisation**

Red mud dewaterability and shear rheology characterisation was performed at process temperatures on underflow suspensions for an entire washer train at each refinery. Results from pressure filtration tests illustrated that, in spite of the great variation in the mineral make up of red mud suspensions across all sponsor sites, the dewaterability properties of all of these materials demonstrated little variability. It was observed that the variability in properties across a single sponsor site was as great as the variability observed across all sites. A similar observation came out of the shear rheology testing in that the variability in shear rheology within a single sponsor site was of the same order as the variability across all sites. Liquor viscosity was observed to be the largest factor contributing to variation in the permeability of the red mud suspensions tested. Normalisation of the dewaterability behaviour allowed removal of the effects of the variation in liquor viscosity and facilitated comparison of the effect of washer stage and flocculation on the underflow dewaterability to be examined. Variations in the dewaterability down the washer train, after the thickener/settler, were observed to be minimal after removal of the variation in liquor viscosity. Again, across all of the sponsor sites, the same trends were observed.

The shear yield stress of red mud suspensions was observed to increase with increasing temperature. This phenomenon has not been widely reported until recently because yield stresses have historically been determined by extrapolation rather than through the vane test or the slump test. It is proposed that this phenomenon is generic to a wide range of particulate suspensions and also manifests itself as an increase in \( P_y(\phi) \) with an increase in temperature. Thixotropy in red mud suspensions was observed to be relatively insignificant over the short time scale of dewatering processes. It is concluded that thixotropic build up of structure is most significant in thickener stagnant zones and in residue disposal areas. It is proposed that the origins of thixotropic build up are partially related to the influence of particle shape, but predominantly due to precipitation products from supersaturated liquor.

The role of a range of flocculants in changing the permeability and compressibility of red mud suspensions has been examined. The work involved dewaterability characterisation after
flocculation to establish correlations between process variables such as flocculant type, dose and shear history. Results demonstrated that flocculation can improve permeabilities by a factor of up to 100 at very low solids concentrations, but only by a factor of up to 2 at high solids concentrations. It was also demonstrated that just like flocculant type and dose, the shear regime during flocculation can also have a significant influence on the resultant suspension dewaterability.

Additional characterisation work was performed on seawater neutralised suspensions. The addition of seawater was observed to have a negative effect on both the compressibility and permeability of the suspensions. When these neutralised suspensions were flocculated, it was observed that flocculation was most effective in improving dewaterability when the flocculant make-up solution did not contain any caustic. The main reason for the loss of dewaterability has been demonstrated to be due to precipitation products from the mixing of seawater with caustic. Interestingly, the effect of this precipitation reduced with time and a partial recovery of permeability was observed.

*Development of New P*(f) and R*(f) Functional Forms*

For the first time, the true shape of both *(f)* and *(f)* curves have been determined experimentally from separation equipment feed solids concentrations to output solids concentrations. Fitting traditional functional forms to these curves has proven inadequate and thus new functional forms have been developed to provide a better representation of the shapes of these curves.

*Development of Process Equipment Modelling Tools*

A filtration model (Landman and White 1997), a steady state thickening model and a transient consolidation model have been developed. These models have been converted into mathematical code, enabling prediction of dewatering performance from *(f)* and *(f)* curve fits that range from feed solids concentrations to well beyond output solids concentrations. These new modelling tools are generic to the assessment of dewatering performance and are applicable to a wide range of industries.

*Process Equipment Modelling*

Modelling of rotary drum filtration of red mud suspensions predicted how process variables contribute to overall performance. From these predictions, directions for filtration optimisation were identified.
Steady state thickener modelling predictions based on the dewaterability of thickener underflow suspensions and flocculated simulated thickener feed suspensions have established that these devices operate in a permeability-limited regime. This implies that attainment of higher solids concentration is dependent only on suspension permeability and is not influenced by suspension compressibility. The predictions demonstrate that whilst the compressional properties of the floc network are important, they are not being exploited in most Bayer process thickeners and washers. As such, there is potential for disposal at higher solids concentrations.

Modelling of the effect of flocculation on thickener dewatering suggests that flocculation can improve throughput by a factor of at least 2, for the same underflow solids concentration. The influence of the settling rate immediately after flocculation (a commonly used gauge of floc performance) was found to be less important than the permeability of the floc network at solids concentrations near the gel point. This is a major outcome for the quantitative assessment of the role of flocculants in dewatering. The expected change in output solids concentration from a thickener due to changes in flocculant type or dose can now be quantitatively assessed.

Thickener modelling predictions suggest that the role of raking in dewatering is significant. Results for the thickeners that were modelled demonstrate that effective permeability enhancement factors due to raking of 2 to 100 can be achieved. This wide variation is attributed to variations in the intensity with which the suspension bed is raked in different thickeners. In the absence of rake torque limitations, raking represents the single most important parameter in achieving enhanced dewaterability or throughput in current facilities. For torque limited devices, benefit can be gained by strengthening rakes to ensure that equipment has sufficient raking capacity to allow more intense raking. Otherwise, only enhancement of floc network permeability through new flocculant choices and controlled shear manufacture of flocs is likely to achieve improved dewatering performance.

Modelling predictions of transient consolidation of red mud suspensions in residue disposal areas, gave an indication of the time-scale of settlement.

### 14.2 FURTHER WORK & FUTURE DIRECTIONS

The conclusions and major outcomes of this work lead to logical directions for future work. The knowledge gained can be utilised to refine and further improve the methods that have been developed. The characterisation and modelling methods can be put to practical use in solving industrial problems, where the scope to further the current research and exploit the outcomes is significant. Modelling the performance of entire washer trains, rather than single pieces of process equipment at a time has the potential for significant performance improvements. In
addition, future research can seek to understand the unexplained phenomena observed in this work and feed the results back to further develop and refine the characterisation and modelling tools.

**Dewaterability Characterisation Method Improvements**

In the long term, the key to demystifying the uncertainties in suspension dewaterability will probably lie in new characterisation methods that are yet to be fully developed. Current examples of significant potential include obtaining more dewaterability characterisation data from a single settling test and automated floc density analysis. Improved dewaterability characterisation from a single batch settling test would have the advantage that the shear degradation of floc structure does not compromise the accuracy of dewaterability characterisations. Automated floc size and density analysis will provide more detailed information about floc structure and its interaction with dewaterability, especially when subjected to shear. With these characterisation tools developed, understanding and prediction of thickener operation will be significantly improved.

**Flocculation**

The utilisation of new flocculation strategies considering flocculant type, dose and shear regime can now be quantitatively investigated for improved operation of filtration and thickening equipment. This involves using dewaterability characterisation results to predict dewatering performance. Generally, improvements in both compressibility and permeability are desirable, but when there is a trade-off, only process modelling can predict the optimum conditions. A step change in the improvement in dewaterability might be achieved if flocculation could produce rapidly settling, dense and compact flocs that were held together in macro-flocs to improve clarity. Developments in research of this nature will rely heavily on the development of new characterisation techniques including dewaterability characterisation from a single settling test and automated floc density analysis.

**The Interaction Between Flocculation and Raking**

The role of rakes in improving dewatering has been highlighted. Work presented in this thesis has shown that exploitation of raking has large potential benefits in terms of dramatically improving thickener performance, regardless of whether the aim is to increase solids throughput or underflow solids concentration. Though thickener modelling can predict the optimum flocculation conditions when shear processes are absent, model predictions must be treated with caution when there is significant raking involved. As a result, there is no substitute for experimental observations. Experimental work could investigate the effect of raking in a pilot scale thickener for a range of operational throughput scenarios, from low to high solids
throughputs. This work could also examine the influence of the flocculation regime on thickening performance in the presence of shear, since current knowledge cannot predict what flocculation conditions produce the most significant dewaterability enhancement due to raking. The ultimate aim of this experimental research would be to identify flocculation regimes that produce high density, high permeability flocs which do not degrade significantly under shear. Achieving this aim would both improve current operations and potentially change the way that flocculation is performed.

In situations where exploitation of further raking is not possible due to torque limitations, research should instead be directed towards flocculation conditions that alter the suspension shear rheology.

*Investigating Unexplained Phenomena*

There has been a wide range of interesting phenomena observed in the work presented in this thesis and future work could investigate the origins of these phenomena. One notable observation is the increase in shear yield stress with temperature and the link to similar behaviour in the compressive yield stress. Another significant observation requiring investigation is the effective permeability enhancement of raking, possibility due to liquor channelling and bubble rise.

*Multi-Dimensional Modelling*

Continuing developments in multi-dimensional modelling of dewatering operations will enable verification of the applicability and weaknesses of the one-dimensional models that have been applied in this thesis. In addition, the multi-dimensional models have the potential to probe the effect on shear in dewatering. The complication in applying these complex multi-dimensional methods is that they require extensive computer time using approaches such as computational fluid dynamics, CFD.

*Process Optimisation Modelling*

It is very important that process configurations optimise the caustic and alumina recoveries in the red mud washer trains. This involves maximising the solids concentration and minimising the caustic and sodium aluminate concentrations in the liquor sent to the mud lakes. In addition, it also involves minimising the solids residence time in the washer train where the supersaturated sodium aluminate precipitates as gibbsite. Modelling the steady state material balance of an entire washer circuit is a feasible option if the outputs of each piece of process equipment can be reliably predicted. The only requirement is that material properties can be adequately predicted as a function of chemical composition and temperature. Based on the
developments in dewaterability characterisation and solid-liquid separation equipment performance prediction substantiated in this thesis, an algorithm could be developed for modelling the steady state operation of a red mud washer circuit and optimising for a wide range of process variables.

14.3 OVERVIEW

The work presented in this thesis has set a new benchmark for the determination of dewatering performance. It has quantified the operational parameters that govern dewatering performance in the washing of red mud and provides new directions for the choice and assessment of dewatering performance for flocculants. It has given a firm direction for future research efforts and provides practical tools for the design and optimisation of solid-liquid separation operations in the minerals and other particulate fluids processing industries.
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